Book of Abstracts

E-MRS Fall Meeting 2006

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Notice

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Table of Contents

Welcome

The European Materials Research Society

2006 Fall Meeting

Warsaw University of Technology
Warsaw (Poland)
4th - 8th September, 2006

We are pleased to welcome delegates to the 2006 Fall Meeting of the European Materials Research Society (E-MRS).This is the 5th E-MRS Fall Meeting. The first E-MRS Fall Meeting hosted 2 symposia and about 200 participants. In 2006 there are 685 submitted contributions, 10 symposia, plenary session, 5 associated events, and about 800 participants from 60 countries . The success of the initiative has been possible due to good cooperation of the Organizers in Warsaw with the E-MRS Headquarters in Strasburg, financial support from many sources (in particular from WUT and Polish Ministry of Science and Higher Education), very good work and engagement of over 100 symposia chairpersons, as well as hard work of all participants that presented oral and poster presentations. The E-MRS Fall Meeting in Warsaw is now a strong point in the European Research Area. It contributes to strong integration of researchers from the whole Europe, and permits to follow the most important trends in the development of materials science and engineering. Each year new initiatives are undertaken to improve the meeting.This year the meeting will be connected with Czochralski Award Ceremony. The Conference Organizers and Committee hope that the E-MRS Fall Meeting meets your expectations, provides enlightenment in your subject area, enables you to make new contacts and, very importantly, proves to be a worthwhile and enjoyable experience from the scientific and social viewpoint.

Organisers

Conference Organisers:

European Materials Research Society (****)
Polish Materials Science Society
Warsaw University of Technology (*)
Institute of Physics, Polish Academy of Sciences (**)
Institute of High Pressure Physics, Polish Academy of Sciences (***)

The conference chairpersons:

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M. Lewandowska (*)
W. Lojkowski (***)
A. Mycielski (**)
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(1) Warsaw University of Technology
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(3) Institute of High Pressure Physics, Polish Academy of Sciences
(4) European Materials Research Society, E-MRS

E-MRS European Co-ordination Group:

- J. P. Massué E-MRS President
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The European Materials Research Society (E-MRS) is a non profit scientific association founded in 1983. It focuses on creating the synergy between interdisciplinary, innovative technologies, spreading and exchanging information and promoting technology transfer from public institutions to industry. The primary objective of E-MRS is to promote and enhance the efficiency of research in Europe in the field of Advanced Materials. E-MRS seeks to quickly inform researchers of the scientific and technological developments in their field of interest from the rest of the world through the society's links with other MRS societies belonging to the International Union (IUMRS).

E-MRSAnnounces New Opportunities for its Members

The Executive Committee of the European Materials Research Society (E-MRS) has made the decision to diversify and extend the activities of the Society toward a variety of items beyond the organization of the E-MRS Spring and Fall Meetings. The Society's three new initiatives are books, short courses, and workshops.
E-MRS seeks authors and editors interested in preparing books on

topics such as materials science, materials engineering, and materials processing. The books would be published in the frame of the Elsevier/E-MRS Publishing Agreement related to a monograph series of books titled the "E-MRS Monograph Work." Based on the contents of books in the monograph series, E-MRS plans to develop short courses to be held in conjunction with the Spring and Fall Meetings.

For its third initiative, E-MRS plans to establish Advanced Research Workshops on new topics in order to strengthen and develop the relationships between scientists from various European countries. The Society would seek sponsorship from research agencies such as the European Science Foundation and NATO. The output of these workshops is to initiate research activities in Europe, and prepare the ground to develop future collaborative research programs or scientific networks.

For more information, contact E-MRS at 23 Rue du Loess, BP 20, 67037 Strasbourg Cedex 02, France; tel. +33-(0)3-88-10-63-72; fax +33-(0)3-88-10-62-93; e-mail emrs@emrs.c-strasbourg.fr; or access Web site www.emrs-strasbourg.com.

The Polish Materials Science Society

The Polish Materials Science Society (PMSS) was founded in December 2004 and promotes interdisciplinary cooperation between scientists working in different scientific areas - materials science and engineering, chemistry, physics, biology, medicine etc. The PMSS closely collaborates with E-MRS in the organisation of the E-MRS Fall Meeting.

Programme

Introduction

The E-MRS 2006 Fall Meeting will consist of two Plenary Sessions, and 10 scientific symposia which will run concurrently. The Opening Ceremony together with the Czochralski Award Ceremony will take place on Monday 4th September at 11:00 am. Poster sessions, common for all the symposia, will be organized on Monday, September 4, 17:20-19:00 and Wednesday, September 6, 15:50-17:20.

The detailed timetable and abstracts for papers to be presented in each symposium will be found in the appropriate section.

The Czochralski Award Ceremony

Willing to commemorate the contribution of Jan Czochralski to the materials science, technology transfer to industry, and international collaboration, E-MRS instituted an award in the name of Jan Czochralski. The Society formally legalised the Czochralski Award for Achievements in Materials Science and to date two recipients have been honoured: Professor Dr Walter Heywang, formerly Director of Research of Siemens, and Professor Dr Boris Paton, the long term President of the Ukraine National Academy of Science.

In the year 2006 – European Materials Research Society, Polish Academy of Sciences, Polish Materials Science Society, and Polish Society for Crystal Growth established the International Chapter of the Jan Czochralski Award. The first recipient of Czochralski Award selected by the Chapter will be Prof. Thaddeus B. Massalski.

Plenary Sessions:

1. Monday, September 4, 11:00 - 12:30
2. Wednesday, September 6, 09:30 - 10:30, 11:00 - 12:30

Poster Sessions:

1. Monday, September 4, 17:20 - 19:00
2. Wednesday, September 6, 15:50 - 17:20

The same posters are presented at both Sessions

Symposia:

SYMPOSIUM A, Room 134:
Nanostructured composite films: synthesis, characterization, properties, and applications

SYMPOSIUM B, Room 144:
Nanomaterials in catalysis

SYMPOSIUM C, Room 315:
Doped nanopowders: synthesis, characterisation, applications

SYMPOSIUM D, Room 309:
Polymer materials modified by nanoparticles

SYMPOSIUM E, Room 213:
Dilute magnetic materials for spintronic applications

SYMPOSIUM F, Room 219:
Wide Band Gap II-VI Semiconductors: Growth, Characterization and Applications

SYMPOSIUM H, Room 206:
Multiscale kinetic modelling of materials

SYMPOSIUM I, Room 226:
Phase Diagrams, Phase Stability: Theory and Applications

SYMPOSIUM J, Room 231:
Surface functionalization and activation of biomaterials

SYMPOSIUM K, Room 208:
Complex oxide materials for new technologies

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Satellite events coordinated with the E-MRS Fall Meeting:

- Fall school on thermal analysis (3rd and 4th September)
- Thin-layered materials - investigation, technology and application (3rd September)
- COST action D30 Meeting (3rd and 4th September). 4th Sept. Room 315, the same as Symposium C.
- Technology Commercialisation Market Place (7th September. 17.30)
- Modeling real materials from first principles (5th and 6th September)

Sponsors

THE E-MRS FALL MEETING 2006 is supported by:

	European Materials Research Society
	Polish Ministry of Science and Higher Education
	The Polish Materials Science Society
	Faculty of Materials Science and Engineering, Warsaw University of Technology
	Institute of Physics, Polish Academy of Sciences
	Institute of High Pressure Physics, Polish Academy of Sciences

Symposia

Plenary Session

Programme

Monday, 4 September

Opening / Czochralski Award Ceremony
Monday morning, 4 September, 11:00

11:25 Invited oral

Alloy Phase Diagrams: Opportunities, Problems and Applications

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In the context of materials, there is probably no other broad subject that impinges more on the various aspects of materials science and technology than the description of phase stability in terms of temperature and composition, known as a Phase Diagram. Phase diagrams are the "road maps" that guide and direct us to our numerous goals in fabrications, developments, heat treatment, properties and basic science. In this presentation, I shall discuss briefly the three laws of thermodynamics, including considerations of how phase diagrams are determined and assessed, and how they can be judged in terms of thermodynamic parameters. I shall then turn attention to metastability and concepts that arise when, because of kinetics, ideal equilibrium conditions can be suppressed or altered by heat treatment, such as cooling, rapid quenching, annealing etc. The use of phase diagrams and opportunity which they present for development of unusual properties, or structures, or features will be considered next, and will be followed by expectation of phase behavior, and phase diagram features, at temperatures between 0C and 0K, where the third law becomes increasingly important and kinetics are reduced. Finally, the process of assessing phase diagrams through the examination of published work on experimental determinations, on modeling calculations, and on measured thermodynamic quantities will be considered. Here, I will stress the usefulness of international cooperation in this whole area.

12:10 Invited oral

Recent initiatives to create a materials community in Europe

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On the globalized world market, the competition has become extremely strong and new countries become increasingly active in RDT and even high tech activities. At the Lisbon European Summit it was decided that Europe should strongly increase its efforts in research and innovation. Until presently, not too much bas been achieved and even if it has become quite clear that without a very active RDT in Europe it will not be possible to maintain the level of the present GNP, and consequently certain jobs. In the field of materials, one considers that about 10 million jobs are related to "materials" ("metallurgy" in OECD terminology)

During this presentation, we shall review the different initiatives we have taken in the frame of the legalised "European Materials Forum"(EMF) over the last year. Presently, most of the European or national societies dealing with materials are directly or associated to EMF, in total over 130. During the last year, our efforts have been directly into the following directions:

- Establish stronger links among the researchers in the 25 European countries involved in the field of materials; try to involve also other European not in the Union
- Advocate for a more efficient and more transparent RDT policy in Europe
- Develop a new model for INNOVATION, bridging research to industry and investors
- Elaborate a model for a testing at small scale of the European Institute of Technology (EIT)
- Launch common actions through the existing European instruments

Wednesday, 6 September

Plenary Session
Wednesday morning, 6 September, 9:30

9:30 Invited oral

Space Programs: a challenge for material engineering

Zbigniew Klos, Jerzy Grygorczuk

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Space Programs are, by their nature, interdisciplinary and prefer holistic approach. Reliability and redundancy are governing principles in preparing space missions. The space sector is generally more oriented toward using and combining existing technologies than toward generating new ones. Although, this "spin-in" approach dominates in the policy of primary space contractors, it still leaves room for new innovative elements and solutions provided by subcontracting enterprises, usually belonging to the state-of-art unit in material engineering. The good example of that is the parallel activity of the European Space Technology Master Plan (ESTMP) - outlook for future and solutions for space instrumentation implemented now in the planetary probes. The technology of material coating, tribology and advanced miniaturization are key areas that respond to continuously increasing requirements for new sensors, power sources and propulsion systems with very low mass, volume and power consumption. The main challenge of space engineering follows from unusual environmental requirements and constraints (heavy loads and vibration during launch, microgravity, vacuum, thermal gradients, increased

doses of radiation) that forces space specific solutions. The Space Research Centre PAS has been involved in design, development, tests and analyses of the advanced instruments prepared for European landers on Titan (Cassini /Huygens) and cometary nucleus (Rosetta). In particular, an original cometary nucleus penetrator on-board Philae(Rosetta lander), working at unusual conditions in space, was a challenge for mechanical design and material engineering. The device contains novel solutions and was developed within close international cooperation.

Coffee break

Wednesday morning, 6 September, 10:30

Plenary Session

Wednesday morning, 6 September, 11:00

11:00 Invited oral

Peculiarities of mechanical behavior of nanocrystalline and noncrystalline materials

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The review of the mechanical behavior of nanocrystalline (NQ) and noncrystalline (amorphous (MG) and quasicrystalline (QC)) materials is given. Peculiarities of deformation mechanism for NQ materials are connected with the practically absence of dislocations in the body of grain (dislocations go out into the grain boundaries due to image forces) and realization of deformation in the grain boundaries. In MG the dislocation mechanism of deformation is discussed for the range of nonhomogenious deformation. Strain hardening and localization of deformation in MG is considered. Yield stress σ_s of MG consists of thermal and very big athermal components. Thermal component is close to the thermal component of crystalline state. σ_s and hardness H of MG are very high, but NQ can have higher values of σ_s and H if d < 100 nm.
Plastic deformation of quasicrystals is of dislocation type, but Burgers vector of dislocations contains phason component, and dislocation movement is accompanied by the gradual destroying of quasicrystalline structure and in some cases by phase transition to crystalline state. The hardness of quasicrystals increases after annealing and decreases during deformation. QC with the nanosize grains (NQC) is the especial class of materials, because energy of dislocation connected with phason defects is proportional to d (not lnd as elastic energy of dislocations in crystals).
For this reason plasticity of NQC is more than in QC. Composite materials on the base of NQ, MG and QC can have high level of mechanical properties.

11:45 Invited oral

Fabrication of ordered atomic-scale structures - a step towards future atomic-scale technology

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The quest of a reliable method for fabricating ordered atomic-scale structures is a prequisite for future atomic-scale technology. The interest in such nanostructured materials, consisting of building blocks of a small number of atoms or molecules, arises from their promising new optic, catalytic, magnetic and electronic poperties. Here we present three examples concerning atomic and supramolecular self-assembly investigated by low-temperature scanning tunneling microscopy (STM). (i) The self-assembly of a two-dimensional array of individual Ce adatoms on a metal surface based on long-range interactions between adatoms mediated by surface state electrons [1,2]. (ii) The conservation of chirality in a hierarchical supramolecular self-assembly of pentagonal symmetry of the organic molecule rubrene on a reconstructed Au(111) surface [3]. (iii) Using the highly localized current of electrons tunneling through a double barrier STM junction, we excite luminescence from a selected C60 molecule in the surface layer of fullerene nanocrystals self-assembled on an ultrathin NaCl film on Au(111). In the observed fluorescence and phosphorescence spectra, pure electronic as well as vibronically induced transitions of an individual C60 molecule are identified, leading to unambiguous chemical recognition on the single-molecular scale [4]. [1] F. Silly, M. Pivetta, M. Ternes, F. Patthey, J. P. Pelz, and W.-D. Schneider, Phys. Rev. Lett. 92, 016101 (2004). [2] M. Ternes, C. Weber, M. Pivetta, F. Patthey, J. P. Pelz, T. Giamarchi, F. Mila, and W.-D. Schneider, Phys. Rev. Lett. 93, 146805 (2004). [3] M.-C. Blüm, E. Cavar, M. Pivetta, F. Patthey, W.-D. Schneider, Angew. Chem. Int. Ed. 44 , 5334 (2005). [4] E. Cavar, M.-C. Blüm, M. Pivetta, F. Patthey, M. Chergui, and W.-D. Schneider, Phys. Rev. Lett. 95, 196102 (2005).

Symposium A

Welcome

The fundamental properties of materials, coatings and thin films are remarkably altered as the size of their constituent grains decreases to a nanometer scale. Novel nanostructured (made of nanosized grains or building blocks) and composite (formed by clusters embedded in matrices) coatings or thin films offer unique and entirely different mechanical, tribological, electrical, optical, catalytic, dielectric and magnetic properties compared with conventional micrometer or millimeter-size materials or films owing to their distinct size, shape, morphology, topology, surface and interface chemistry. For instance, the nanosized grains, which can be made of metal, carbide, nitride or oxide phase are embedded in an amorphous carbon or silicon dioxide matrix.

Several major research and development programs devoted to nanostructured composite films encompass diverse aspects including synthesis, processing and fabrication technologies, spectroscopic characterization, physico-chemical properties, structure-property relationships and future applications in various fields (aerospace, automobile, cutting tools, catalysts, batteries, sensors, micro- and optoelectronics, modulators, transducers solar cells, medical implants, etc.).

A strong specificity is associated with the mechanisms and methods of deposition as well as with the characterization techniques of functional properties of nanostructured composite films dedicated to various advanced technologies. The synthesis and characterization of this novel generation of films remain difficult in spite of recent advances. However, potential applications are now more or less precisely defined and start to arise in a variety of cutting edge technologies.

The major aim of this symposium is to offer an overview and a forum on the recent advances in the interdisciplinary field of nanostructured composite films. Researchers and engineers from different disciplines of science and engineering will be brought together to share their knowledge and expertise on these novel films involved in various emerging applications.

The symposium will address progress in the development and applications of coatings and thin films as active components where their nanostructure, morphology and composition enhance the physico-chemical properties or ensure specific properties. Papers are solicited on topics corresponding to one or more of the following areas : (1) the preparation of films by sputtering, activated reactive evaporation, ion plating, chemical vapor deposition, plasma-assisted deposition, pulsed laser ablation and hybrid techniques, (2) the advanced techniques for the characterization of the microstructure, morphology, size of particles embedded in the matrix, (3) the application of nanostructured composite films to enhance the mechanical, tribological, dielectric, optical, catalytic and magnetic properties of devices.
The specific topics covered by the symposium include diverse aspects related to nanostructured composite films :
1) synthesis, growth mechanisms and modeling
2) characterization of films
3) mechanical and tribological properties
4) electrical and dielectric properties
5) optical properties
6) catalytic properties
7) magnetic properties
8) applications for microelectromechanical systems (MEMS) and sensors
9) applications for optical devices
10) biological and biomedical applications

Scientific Committee:

G. Abadias (France), M. Benlahsen (France), I. Bertoti (Hungary), M.-P. Besland (France), H. Biederman (Czech Republic), N.J.M. Carvalho (Belgium), M. Cekada (Slovenia), K. Cooke (U.K.), I. Dahan (Israel), G. Dennler (Austria), X. De La Fuente (Spain), M. Farle (Germany), A. Galdikas (Lithuania), A. Gonzalez-Elipe (Spain), H. Hofsäss (Germany), U. Jansson (Sweden), W. Kautek (Austria), S. Kennou (Greece), H. Klostermann (Germany), Z. Kutnjak (Slovenia), N. Laidani (Italy), D. Leinen (Spain), M. Leonowicz (Poland), P. Lobotka (Slovakia), S. Logothetidis (Greece), J.C. Lopez (Spain), B. Major (Poland), A. Matthews (U.K.), F. Maury (France), C. Meunier (France), T.-P. Nguyen (France), S. Novak (Czech Republic), P. Oelhafen (Switzerland), K. Oskomov (Russia), E. Pascual (Spain), L. Poperenko (Ukraine), N. Radic (Croatia), J.-P. Rivière (France), R. Sanjines (Switzerland), A. Savan (Germany), A. Schüler (Switzerland), M. Stüber (Germany), S. Tamulevicius (Lithuania), V. Teixeira (Portugal), P.-Y. Tessier (France), S. Zhang (Singapore)

Organisers

- **Prof. Yves M. Pauleau**, National Polytechnic Institute of Grenoble, CNRS-LEMD, Grenoble, France - Symposium Chairman
- **W. Gulbinski**, The Technical University of Koszalin, Faculty of Mechanical Engineering, Dep. of Physics, Koszalin, Poland
- **P. Patsalas**, University of Ioannina, Dep. of Materials Science and Engineering, Ioannina, Greece

Proceedings

The manuscripts submitted to this symposium and accepted on the basis of the referee procedure adopted for regular papers would be published in the international scientific journal **"Reviews on Advanced Materials Science"**.

Programme

Monday, 4 September

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

the Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break

Monday afternoon, 4 September, 12:30

Tribological properties of composite films

Monday afternoon, 4 September, 14:00

Chair: Juan Carlos Sánchez-López, Thien Phap Nguyen

14:00 Invited oral

Nanocomposite TM-dichalcogenides sputtered self-lubricating coatings alloyed with C

Albano Cavaleiro, Tomas Polcar, Manuel Evaristo

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Transition metal dichalcogenides (TMD) are compounds with unique and unusual properties based on the extreme degree of anisotropy of the layered structure, which make them suitable as solid lubricants. TMD exists in the MX_2 form, where M=Mo,W,Nb and X=S,Se,Te. The deposition of TMD systems leads usually to films with very high porosity with the consequent low values in hardness and adhesion to the substrates. Thus, their tribological behavior was unsuitable whenever high loads were applied or tests were carried out in moisture containing atmospheres.There are a lot of different possibilities to improve the tribological behavior of these coatings. One of the most successful ways is to deposit a composite material associating high strength materials with self-lubricants. Recently, nanocomposite structured coatings were deposited associating a hard DLC phase with WS_2 permitting to reach very low friction coefficients in a large range of environments. On the other hand, there are studies showing that diselenides have improved resistance to water in relation to sulfides.It is the aim of this talk to compare the tribological performance of nanocomposite coatings based in two different TMD systems, a sulphide (W-S) and a selenide (Mo-Se). In both cases, the coatings were deposited with increasing contents of carbon (up to 70at.%) by decreasing the number of TMD pellets placed in the C target during the sputtering deposition. Significant improvements of the mechanical properties (hardness, adhesion) in more than one order of magnitude were reached. Pin-on-disc performed in different testing conditions showed that W-S-C system outstand Mo-Se-C coatings at higher temperatures whereas the inverse occurred concerning the global behaviour in environments containing different humidity ratios

14:30 Oral

Nanostructured Composite Films for high temperature tribological applications

Hanna Wiśniewska Weinert, Volf Leshchynsky, Monika Gierzyńska-Dolna

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Transition-metal dichalcogenides (MoS_2, WS_2, etc.) are the most common solid lubricants for high temperature applications. They are applied as powders and coatings burnished the surface from powders, or deposited by spray and vacuum deposition methods. The goal of the paper is to develop nanocomposite coatings made of Boron Nitride and transition-metal dichalcogenide phases by advanced pressure saturation of porous sintered matrix and following severe deformation technique. Modern PM technologies were used to produce low cost high quality bearings with long-term performance and reliability in critical applications (high loads and sliding velocities, high temperatures). To comply with endurance requirements of high temperature applications, the nanostructuring approach was explored. In this technology solid lubricant particles were pressed into a supporting porous matrix and modified into nanostructured composite films by surface deformation. The nanostructured composite films consist of 10-50nm BN crystals and WS2 sheets embedded in nitrided stainless steel powder matrix. These BN/WS_2 nanostructured coatings demonstrate low friction and wear in tests performed in humid air at the temperatures up to 5000C. Coatings are found to adapt to the test conditions, which results in BN and WS_2 nano-sheets generation and permanent supply of solid lubricant from the surface reservoirs. These adaptive mechanisms achieve low friction coefficients of 0.05-0.1 and high endurance in high temperature exploitation friction tests. Correlations among BN/WS2 chemistry, structure, hardness, friction and wear are discussed. The tremendous potential of such nanostructured solid lubricant composite films for aerospace tribology is demonstrated.

14:50 Oral

Nickel/hydrogenated amorphous carbon composite films deposited in acetylene/argon microwave plasma discharge

Sławomir Kukiełka[1,2], Witold Gulbiński[1], Yves Pauleau[2], Sergey N. Dub[3], Jean-Jaques Grob[4]

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Nickel/hydrogenated amorphous carbon composite films have been deposited on silicon and stainless steel substrates by combining sputter-deposition of metal and microwave plasma-assisted chemical vapor deposition of carbon from argon-acetylene mixtures containing 10% to 100% of C_2H_2. The composition of films was investigated as a function of the C_2H_2 concentration by Rutherford backscattering spectroscopy, nuclear reaction analysis and elastic recoil detection analysis. The crystallographic structure of the films was identified by X-ray diffraction techniques. The grain size of carbide, Ni_3C, detected in all samples, was found to be in the nanometer range. The nanohardness and Young modulus of films were deduced from the load-displacement nanoindentation curves. The maximum hardness of 13 GPa was found for films produced from gas mixtures containing 25 to 35% of C_2H_2. The magnitude of residual stresses was determined by measurements of the curvature radius of silicon sub-

strates. The maximum value of compressive residual stresses was equal to - 0.8 GPa for films deposited in gas mixtures containing 25 to 35% of C_2H_2. The ball-on-disk tribological tests were conducted in room air at 20°C. The friction coefficient was as low as 0.22 for films deposited in gas mixtures containing 70% of C_2H_2.

15:10 Oral

Surface chemistry and oxidation-driven Fe-segregation on the surface of Fe40Ni40B20 bulk metallic glasses

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Metallic glasses (MGs) are a category of materials that are characterized by their amorphous structure and metallic bonds. Owing to their disordered structure, MGs possess unique properties that make them attractive for mechanical and tribological applications. In this work, we study the surface chemistry of Fe40Ni40B20 MGs, which are promising materials fulfilling the requirements for various industrial applications. The surface and bulk structure are investigated by X-Ray Diffraction (XRD), Auger Electron Spectroscopy (AES), X-Ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS). Different XRD geometries, as well as sputter etching during XPS and SIMS experiments were used to study the structural and chemical profile of these glasses. The findings of this study include the segregation of Fe on the MG surface. We found a strong correlation between the concentration of trivalent Fe (oxide form) and the concentration of O, and comparing the [Ni]/[Fe] and [Ni]/[Fe3+] we concluded that the excess of [Fe] on the surface is attributed to preferential surface oxidation. This behavior is reported for the first time for a glass system, and it is similar to the corresponding oxidation behavior of Fe-Ni crystalline alloys. It is worth to emphasize that such oxidation occurs in a glass system, although O diffusion is expected to be limited compared to the Fe-Ni crystalline alloys. Depth profile experiments in combination to exposurre of clean glass surfaces to O-ambient have shown that the oxidation-driven Fe-segregation on the surface is a reversible and repeatable process. XPS has also detected that in the buried layers the concentration of trivalent Fe is higher than it would be expected for the corresponding O concentration. This observation might be attributed to the coordination of Fe in the glass state.

Coffee break
Monday afternoon, 4 September, 15:30

15:30 Oral

The influence of temperature, surface modification, deposition method and porosity on tribological properties of titanium dioxide thin films

Ireneusz Piwoński

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Titanium dioxide exhibits many interesting features, which make this material applicable in solar cells, as photocatalyst, humidity sensors, medical implants, as well as in cosmetics, and pharmaceutical products. Recently, titanium dioxide in a form of thin layers deposited on various substrates has been in the centre of interest of tribology. Some tribological properties of titanium dioxide and its composites have been already studied, but in many domains these properties are still not investigated enough. This study focuses on some tribological aspects of titanium dioxide thin films including the influence of such parameters like temperature, surface modification, deposition method and porosity.

First part of this work presents the preparation methods of titanium dioxide thin films deposited on Si wafers. Titanium dioxide was prepared mainly with the use of sol-gel method from the $Ti(-OPr)_4$ precursor and deposited on silicon wafers with the use of dip-coating method. Titanium dioxide thin films were obtained in non-porous and porous form. Porous samples were obtained with the use of surfactants.

Second part of the investigations presents titanium dioxide surface characterization (surface topography, internal structure, surface properties and friction) performed with such techniques like Atomic Force Microscopy (AFM), infra red spectroscopy (FT-IR), X-ray diffraction (XRD), etc. Tribological properties of the samples were characterized with the use of the microtribometer operating in the load range of 0-100 mN. The coefficient of friction and wear obtained in microtribological measurements were main representative parameters giving information about the influence of temperature, deposition method, porosity and surface morphology on frictional properties of titanium dioxide thin films.

Parallel Session
all Symposia exept symposium A
Monday afternoon, 4 September, 15:50

Poster Session
Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Machanical properties of composite films
Tuesday morning, 5 September, 9:00
Chair: Ireneusz Piwoński, Mariana T. Braic

9:00 Invited oral

Mechanical and tribological properties of multilayered-composite solid lubricant films

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In the present investigation, Ti+TiN+TiAlN-MoS_2+MoS_2-Ti solid multilayer-composite solid lubricant coating was deposited by magnetron sputtering from separate Ti, Al, and MoS_2 target on D2 tool steel. EDS, X-ray diffraction, microhardness tester, scratch tester, and pin-on-disc tribo system were used to evaluate coating structural, mechanical and tribological properties. Adhesion was also improved in the multilayer-composite coating. Pin-on-disc measurement at atmospheric condition against WC ball showed much lower wear and friction coefficient.

9:30 Oral

Mechanical Properties of Nanostructured Composite Diamond Thin Films Prepared by Microwave PECVD Enhanced by RF Induced DC Self-Bias

Vilma Bursikova[1], Zdenek Frgala[1], Jiri K. Bursik[2], Ondrej Jasek[1], Lenka Zajickova[1], Monika Karaskova[1], Jirina Matejkova[3], Antonin Rek[3], Daniel Franta, Petr Klapetek

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The aim of the present work was to deposit nano-crystalline dimond films with low surface roughness, high hardness and fracture toughness by microwave PECVD in the ASTeX type reactor from the mixture of hydrogen and methane. The applied microwave power at 2.45 GHz was 900 W, the total pressure ranged from 7.5 to 8.0 kPa, H_2 flow rate was 400 sccm and CH_4 flow rate varied between 8 and 40 sccm, which resulted in CH_4 concentration from 2 to 10%. The substrate used was mirror polished (111) oriented n-doped silicon single crystalline wafer. The substrate temperature and the deposition time varied from 820 to 920 °C and 10 to 35 minutes, respectively. The diamond nucleation process was enhanced using RF (35 W, 13.56 MHz) induced DC self-bias. The time dependence of DC self-bias turned out to be an important characteristic of the growth process. The deposited layers have been analyzed by scanning electron microscopy, transmission electron microscopy, atomic force microscopy, spectroscopic ellipsometry and depth sensing indentation tests.

The analysis of AFM data yielded rms roughness 8.8 nm and autocorrelation length 120 nm. The results of the presented study suggested that the films have a nanocomposite structure and consist of diamond nanocrystals embedded in a amorphous carbon matrix. The films had relatively high hardness (~70 GPa) and elastic modulus (~400 GPa) and exhibited high fracture toughness and excellent adhesion to the substrate.

The work was supported by the Czech Science Foundation (Projects 202/05/0607 and 106/05/0274)

9:50 Oral

Si- and W- containing carbon based nanocomposite thin films: chemical and nanomechanical properties

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Si- and W-containing a-C films were deposited by dual magnetron sputtering onto polished silicon wafers. A DC source was used for carbon sputtering, and a composite RF source was applied for sputtering the Si and W additives. For comparison, carbon, silicon and C-Si films were deposited in separate experiments, using a similar source arrangement. The chemical composition and the bonding states of the constituent elements were characterised by X-ray photoelectron spectroscopy and X-ray induced Auger electron spectroscopy. Mechanical properties (dynamic hardness and reduced modulus) were estimated by using depth-sensing nanoindentation.
The silicon content of the C-Si films varied between 20-40 atomic %, depending on the applied source power. The tungsten content in the C-Si-W films varied between 25-50 atomic % while the silicon content at the same time was between 25-0 atomic %. Despite a slight surface oxidation, the oxygen content of the films was insignificant, typically below 3 atomic %. Predominant C-Si and C-W chemical bonds could be identified by the chemical shifts of the XP spectra and by the Auger parameters.
The values of nanohardness and reduced modulus for the silicon substrate were 10 GPa and 127 GPa, respectively, while those for the deposited layers were much higher: for the a-C film the corresponding values reached 16 GPa and 155 GPa, for the C-Si films 13-16 GPa and 140-185 GPa and for the C-Si-W films 12-19 GPa and 160-210 GPa, depending on the actual chemical composition.
This work was supported by the National Scientific Research Fund through the project OTKA T-043359.
Keywords: DLC-Si-W, nanocomposite, XPS, Auger parameter, nano-hardness, reduced modulus

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10:10 Invited oral

Indentation testing of thermally nano structured metal-containing carbon films

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For metal-containing carbon films a wide range of application is envisaged, ranging from wear protection due to their tribological prop-

erties to electronics due to their electric and optical properties. Our interest is based on having well-characterized material for investigating the reduction of the chemical erosion of metal-doped carbon materials by hydrogen impact and elucidating underlying mechanisms. The chemical erosion is the main drawback for using carbon materials in future fusion devices like ITER.
The characterization on nanometer scale is essential for getting an understanding of the behavior of metal-containing carbon films in their application. Therefore, we characterize films produced by magnetron sputter deposition with floating potential in respect to composition, morphology, phase and structural ordering by thermal treatment and their chemical erosion by hydrogen impact.
Annealing up to 1300K of metal-doped (Ti, V, W, Zr) amorphous carbon layers with metal content up to 20 % leads to carbide formation and grain growth (several nm). Composition, distribution and diffusion of the metal in the carbon are investigated by Rutherford backscattering spectroscopy. X-ray absorption spectroscopy shows how the local atomic environment of the metal and carbon is affected by thermal treatment up to 1300 K.
In order to extent the list of characterized properties, the hardness in dependence of dopant type (V, Zr), dopant concentration (<18 at %) and annealing temperature (<1300 K) were investigated. The experimental boundary condition of film thickness (200-1800 nm) and substrate (Si, SiC, Cu, SiO2) were varied. The pure carbon film has a hardness of 12 GPa, which is always slightly increased up to 15 GPa by the doping. The annealing to 1300 K leads to a slight decrease of the hardness of the pure carbon film (11 GPa) and increase of the metal-containing ones (17 GPa).

Coffee break

Tuesday morning, 5 September, 10:30

Characterization of composite films

Tuesday morning, 5 September, 11:00
Chair: Imre Bertóti, Vilma Bursikova

11:00 Oral

Microstructural characterization of TiN-Ni nanocomposite coatings deposited by reactive ion beam assisted deposition

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Nanocomposite coatings of TiN-Ni in the form of nc-ceramic/a-metal were deposited on (100) Si wafer and AISI304L stainless steel using an ultra high vacuum dual ion beam sputtering system. A composite Ti-Ni target was sputtered with 1,2 keV Ar^+ions and the growing film was continuously bombarded with a mixture of 50 eV Ar^+-N^{2+}-N^+ ions during deposition. Phases, grain size and texture of the coatings were determined by XRD in the θ-2θ and GIXRD geometries. XRD results show that δ-TiN is the only crystalline phase and Ni is expected to be as an XRay amorphous phase. In order to obtain additional structural details on the nanostructure, TEM and XPS experiments were carried out. The TEM observations confirm the formation of a nanocomposite structure with cristallite size of < 10 nm for TiN grains and reveals equiaxed growth morphology. XPS studies show that Ni is only present in the amorphous intergranular phase and exhibits a metallic character. The formation of nickel nitride is not detected and the incorporation of Ti in Ni is negligible.The friction coefficient is slightly lower than for TiN and these coatings exhibit an increased wear resitance in ball on disc tests. Such improved wear resitance is attributed firstly to the development of a nanocomposite structure and increased toughness of nc-TiN/a-Ni coating with respect to pure TiN.

11:20 Oral

Synthesis and microstructural characterisation of (Ti-TiC) and (TixWy)/TiC nanostructured composite films obtained by ARE

Javier A. Montes de Oca V[1,2], Jean-Pierre Manaud[2], Yann Le-Petitcorps[2], Jorge Galaviz Pérez[1], Jorge R. Vargas García[3]

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The use of refractory and hard coatings is increasing due to the development of nanostructured composites which may conveniently combine high hardness and high toughness. As the structure and functional properties of nanostructured composites strongly depend on the synthesis method, it is of utmost importance to select the most appropriate technique for their preparation. So far, several physical and chemical deposition techniques have been used to successfully produce nanostructured composites based in ceramics. In this work, nanostructured titanium carbide films were deposited on tungsten substrates at a high growth rate by Activated Reactive Evaporation (ARE) at 500 and 600 °C in order to protect a tungsten device against liquid uranium. The crystal structure, lattice parameter, preferred orientation and grain size of the coatings were determined by XRD using Cu Kα radiation. The analysis of the film morphology was performed by SEM and AFM and their composition was analysed by AES and EPMA. Experimental results suggest that temperature was one of the most important parameters in the fabrication of nanostructured Ti-TiC composite films using propene as reactive atmosphere. Thus, nanostructured TiC0.6 coatings codeposited with a free-Ti phase were obtained at 500 °C. On the other hand, stoichiometric TiC coatings were obtained at 600 °C due to a higher energy of the carbon ions for reacting with evaporated Ti atoms. After annealing at 1000 °C, the stoichiometric films remained stable but a crack pattern was formed over all the film surface. Moreover, Ti0.6W0.4/TiC0.6 composite thin films were obtained at 500 °C. For these films the presence of a Ti0.6W0.4 ductile phase within the TiC0.6 layer was responsible for the avoidance of the film cracking.

11:40 Oral

TEM characterisation of indented TiN/SiN$_x$ multilayered coatings

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Thin TiN/SiN$_x$ multilayered films composed of 100 repetitions of 5nm TiN and 0.5nm SiN$_x$ with a total thickness of 500nm were deposited by Closed Field Unbalanced magnetron sputtering on Si wafers at 300°C and -70V substrate bias. The TiN layers consisted of equiaxed, crystalline nanograins with a diameter of 5nm. Conversely, the SiN$_x$ layers were fully amorphous, providing a very sharp interface with TiN layers. The films were indented to 100, 250, 500 and 1000nm with a Berkovich indenter tip resulting in initial penetration depth between 20% and 200% of total film thickness. In this study nanoindentation technique was used to deliberately destroy the films, as 10% is the maximum penetration depth commonly agreed for hardness measurements. We investigate the induced defects and deformation mechanisms occurring upon plastic deformation. The microstructural observations were performed *post-mortem* by conventional and high resolution TEM on cross-sections prepared by FIB technique through the indentation imprint. For all indentation depths the TiN layers situated under the indenter tip apex showed evidence of severe compression. It is thus assumed that during indentation of multilayered coatings the deformation mechanism occurring is material flow. In all indented films, except the one with the lowest load, lateral cracks at the bottom of the coatings were observed determining the areas of highest stress concentration. At higher loads the coating delaminated from the substrate. Moreover, at 250 and 500nm indentation depth a median crack was generated in the film. Even for penetration depths exceeding the total film thickness the coating showed few interfacial cracks, while the multilayering remained intact. In the Si substrate deformation damages in form of nanocrystalline and amorphous regions in the vicinity of the film interface and dislocations forming a triangular shape under the indented film were observed.

12:00 Oral

X-ray MicroBeam Characterization of the Near Surface Nanostructure Layer in Ti After Friction Stir Processing

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Spatially resolved white beam Laue X-ray nano- and micro- diffraction at the synchrotron together with scanning electron and orientation imaging microscopy were used to characterize the structural changes in the Ti near surface region after Friction Stir Processing (FSP). It was established that after FSP a special surface layer with nanocrystalline structure is formed within the depth of 300 microns. Probing of this zone with a white microbeam (diameter ~0.5 microns) did not get any detectable signal. However probing of this zone with the white nanosize beam (diameter ~100nm) gave a distinct diffraction patter. Typically several grains were observed within each probing location. Most of the diffraction pattern consisted of long streaked Laue spots. Such streaking is indicating strong plastic deformation in this zone with the formation of strain gradients, geometrically necessary dislocations and boundaries, and resulting in the local lattice curvature in each grain.

Two specific zones are formed underneath the above nanocrystalline layer: thermal mechanical affected zone (TMAZ) and heat affected zone (HAZ). The size and structure of all zones is determined. The grain size increased sharply (by two orders of magnitude) from FSZ to TMAZ zones and reached micron size (5 - 30 microns) in the TMAZ. Intensive streaking of the Laue spots are observed with a microbeam in the TMAZ and HAZ zones. Large densities of geometrically necessary dislocations and strain gradients are found in the TMAZ based on Laue microdiffraction. Dislocation density gradually decreases with depth and reaches the value typical for base material. The geometrically necessary dislocations were inhomogeneously distributed within the TMAZ and HAZ. Inhomogeneity of geometrically necessary dislocations distribution was found at both scales: within the individual grains and between separate grains.

Lunch break

Tuesday afternoon, 5 September, 12:30

Optical properties of composite films

Tuesday afternoon, 5 September, 14:00

Chair: Michael Farle, Rosendo Sanjines

14:00 Invited oral

Metal Nanocluster Composite Silicate Glasses

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Composite materials made by metal clusters embedded in glass matrices are the object of studies for application in several fields. They exhibit striking optical properties, interesting for photonics application, as part of all-optical devices. Moreover, a significant enhancement of the fluorescence properties of rare earths-containing glasses is realized by the introduction of metal nanoclusters in the glass, whichs may exhibit also important magnetic properties and interesting properties in catalysis. Glass-based composites play an important role in nanotechnology application due to the low cost, ease of processing, high durability, resistance and high transparency. These glasses are also studied from more basic viewpoints, the dynamics of clusters nucleation and growth, their stability, and their structure in terms of composition, crystalline phase, size, and size distribution. A great effort has been made to develop novel preparation methods, for example, based on ion implantation techniques, ir-

radiation techniques of metal-doped matrices, and chemical routes such as sol-gel. In particular, by means of the ion exchange technique, it is possible to dope silicate glasses with concentration values well beyond the solubility limit. Subsequent laser, ion or X beam irradiations may then promote in a controlled way the aggregation and the formation of nanoclusters. In this work, a review is presented on some of these novel combined methodologies for metal nanocluster glass camposites preparation, with special emphasis to those based on ion exchange routes.

14:30 Oral

MEH-PPV/SiO_2 and MEH-PPV/TiO_2 nanocomposites with enhanced luminescent stabilities

Sheng Hsiung Yang[2], Philippe Le Rendu[1], Thien Phap Nguyen[1]

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Thin films of composites made by incorporation of silicon oxide (SiO_2) or titanium oxide (TiO_2) nanoparticles into poly[2-methoxy-5-(2'-ethylhexoxy-*p*-phenylenevinylene] (MEH-PPV) were deposited by spin-coating from *p*-xylene solutions and their optical properties were investigated. Several techniques have been used for investigating the composite films: scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) and Raman, and photoluminescence (PL). It was observed that the structure of MEH-PPV films was not affected by blending the polymer with nanoparticles, but their absorption and emission were modified and were assigned to the different extents of aggregates in the composites. Furthermore, the degradation experiments showed that the blended films present a higher stability than that of pristine polymers.

15:10 Oral

Synthesis of organic-inorganic composite films with photonic crystal properties

Pavel E. Khokhlov[1], Alexander Sinitskii[1], Sergey O. Klimonsky[2,4], Tatyana V. Laptinskaya[3], Ming Li[4], Juntao Li[4], Jianying Zhou[4], Yuri D. Tretyakov[1,2]

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Recently, films with three-dimensionally ordered meso- and nanoporous structures have been extensively studied due to their wide applications in catalysis, separations and optics. These materials can be made by using self-assembling systems (*e.g.* surfactants, biological systems and so on). A relatively new field that benefits from porous materials is the field of photonic crystals (PCs) - dielectric composites with a highly periodic structure in which the dielectric constant varies on the scale of visible light wavelength. PCs were predicted to exhibit a photonic bandgap (PBG), for which light within a certain frequency range cannot propagate in any direction inside the crystals, causing the unique properties of PCs, such as localization of light and control of spontaneous emission. The last effect is of great importance for fabrication of low-threshold lasers and can be observed in PCs with luminescent centres.

The aim of the present work was to synthesize PC films infiltrated with organic complexes of Eu^{3+} and Tb^{3+}. Three types of PCs were used as hosts for luminescent centers: ordered films of polystyrene microspheres and inverse PCs based on silica and titania. In each case the position of PBG was accurately fitted to the wavelengths of visible lines of Eu^{3+} or Tb^{3+} photoluminescence by proper choice of PC lattice period. High spatial anisotropy of photoluminescence was observed by spatially- and spectrally-resolved laser spectroscopic measurement.

The work was supported by the Russian Foundation for Basic Research (grants nos. 05-03-32778 and 04-03-39010) and the Program for Fundamental Research of Russian Academy of Sciences.

Coffee break

Tuesday afternoon, 5 September, 15:30

Chemical deposition of composite films

Tuesday afternoon, 5 September, 15:50

Chair: Heidrun Klostermann, Konstantin V. Oskomov

15:50 Invited oral

Protective and Bio-compatible Nanostructured Surfaces by CVD Techniques: Controlled Modulation of Surface and Phase Structures

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Thin film deposition by CVD techniques plays a dominant role in the development of both protective and functional coatings, important for their technological implications. Commonly, multi-component materials are prepared from a mixture of precursors; however the efficiency of such processes is hampered by the mismatch of chemical reactivity such as thermal stability; vapour pressure etc. among the precursor species. As a consequence, phase separation and elemental segregation is commonly observed in CVD deposited materials.

The de-mixing of elements in multi-component systems is thermodynamically driven and sensitive to the chemical behaviour of the precursors. Transformation of precursor compounds possessing bonding features inherent to the solid-state lowers the need of diffusion and counterbalances the thermodynamic impediments. Recently, we have designed several new metal-organic systems and demonstrated their suitability to deposit *corrosion-resistant and bio-compatible coatings* on metallic and non-metallic substrates.

In addition, this talk will address the role of precursor chemistry in plasma-assisted deposition of nanostructured coatings.

16:20 Oral

Cr-C-N single layers and nanostructured multilayer coatings grown at low temperature by Direct Liquid Injection CVD under atmospheric pressure

Aurelia Douard[1], Francis Maury[1], Jean Pierre Bonino[1], Marcel Nadal[2], Herve Glenat[2]

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Atmospheric pressure CVD is an attractive process for on-line strip coating of metal pieces. Moreover, the use of metal organic compounds as molecular precursors allows deposition at low temperature, which extends the applications of the process on various steels. We have developped an atmospheric MOCVD process assisted by Direct Liquid Injection for performing metallurgical coatings. This delivrery system permits accurate, constant and high flow rates of precursor, compared to conventional vaporization and transport method, *i.e.* using bubblers, especially when the metal organic precursor exhibits a low volatility as a powder. We present for the first time the deposition of nanocrystalline chromium carbide (Cr-C) and chromium nitride (CrN) films. CrN/Cr-C multilayer coatings with a typical 25 nm period were grown on stainless steel using alternatively addition of NH3. The main chemical, physical and structural characteristics of these metallurgical coatings have been investigated, as deduced from XRD, SEM, TEM, EPMA and SIMS. Preliminary mechanical properties from scratch test, nanoindentation and residual stress measurements as well as tribological behavior of the mono and multilayer Cr-based coatings are discussed in relation with their microstructure.

16:40 Oral

Design and control of porosity in oxide thin films grown by PECVD

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Control of porosity in oxide thin films is extremely important for a wide set of purposes. The antireflection properties of thin films, their use as optical sensor, the control of the permittivity in low-k materials and others can be quoted among those applications where the tailoring of the porosity is a crucial issue. This paper reports about some approaches developed in our laboratory to get a precise control of the porosity of oxide thin films prepared by plasma enhanced chemical vapour deposition (PECVD). The use of sacrificial layers of organic materials that can be removed during the deposition of the oxide thin film and the adjustment of the deposition parameters (pressure and composition of plasma gas, temperature of support) are two possible methodologies that can render a wide variety of thin film microstructures. Examples will be shown for TiO_2 and SiO_2 thin films grown under different conditions. The microstructure of these thin films is characterised by different classical techniques such as SEM or AFM. In addition, it will be shown the high potential of using the FT-IR spectroscopy and the employ of a quartz crystal monitor to measure gas adsorption isotherms. This latter technique, practically unknown in thin films, relies on the same concepts than the BET procedure for the characterization of powder materials and furnishes the possibility to estimate the total porosity and pore size distribution in thin films. Some examples will be given of properties (optical properties, membrane applications, sensor response, photocatalytic behaviour) of thin films where the porosity is a crucial parameter

17:00 Oral

PMMA/Zinc Oxide Nanocomposites Prepared by in situ Bulk Polymerization

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Hydrophobic ZnO nanoparticles were synthesized by controlled chemical precipitation and incorporated into PMMA matrix by in situ bulk polymerization, giving PMMA-ZnO nanocomposites. The effects of semiconductor nanoparticles on the rate of polymerization, thermal, and optical properties of the nanocomposites were studied with respect to reference sample polymerized in absence of particles.

Particles having a sharp diameter distribution around 25 nm were precipitated via transesterification of zinc acetate dihydrate with 1-pentanol at 130 ºC. After the formation of crystal core, a mixture of tert-butylphosphonic acid (t-BuPA) and tetrabutyl ammonium acetate (TBuAc) was applied. t-BuPA coats the particle surface, quenches the particle growth, and hydrophobizes the particles whereas TBuAc prevents the formation of undesirable lamellar zinc t-butylphosphonate. The surface-coated particles were dispersed in methyl methacrylate and free radical polymerization was carried out at 60 °C using AIBN as initiator. The particles induce the termination of chain growth by degenerative chain transfer. This suppresses both the gel effect and the formation of chains having thermally labile linkages, which inevitably form under conventional polymerization conditions. Hence, the thermal stability of PMMA is improved when the polymerization proceeds in presence of the ZnO nanoparticles. Spin-coated films of these composites exhibit a distinct cut off of transmission at 370 nm towards shorter wavelengths due to the high absorption coefficient of ZnO. The particles contribute to turbidity in the visible region because of scattering originating from the refractive index mismatch of PMMA and ZnO. Detailed data on the optical properties of these nanocomposite films will be reported.

17:20 Oral

Ti_2AlN thin film formation by multilayer and nitriding approaches

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Ti_2AlN belongs to a novel class of nanolaminated materials: the MAX Phases. Up to now, several approaches were used to synthesize MAX phases under bulk forms but their synthesis as thin films remains poorly documented. Two research groups recently demonstrated that Ti_2AlN thin films formation can be achieved by high temperature magnetron sputtering processes. Here, alternative approaches are described. The first one is a two-step technique based on the deposition of a TiAl/TiN multilayer followed by a thermal annealing. Multilayers were deposited by dual ion beam sputtering at room temperature. Theses samples were then annealed (600°C and 780°C) to allow atomic diffusion and Ti_2AlN formation. HRTEM observations and EELS spectra were recorded prior and after the thermal treatment. The progressive formation of Ti_2AlN during the annealing was evidenced both from in situ plane view TEM and XRD observations. Our results suggest that nitrogen diffusion into the TiAl sublayers allows the formation of the MAX phase whereas the Al atoms in excess presumably out diffuse towards the TiN sublayers where they are inserted as interstitials. The second approach consists in a direct formation of Ti_2AlN by plasma nitriding of TiAl bulk substrates or thin films deposited by magnetron sputtering onto Si and TiAl substrates at room temperature. TiAl bulk substrates and thin films were then exposed to nitrogen plasma at 900°C during 2 hours. HRTEM observations and XRD spectra reveal that this procedure allows the MAX phase formation whatever the substrate is. Of interest, TiN formation was never observed. Furthermore, XRD analysis shows that (0002) textured Ti_2AlN thin films are obtained when TiAl layers are nitrided. The Ti_2AlN formation by nitriding will be discussed on the basis of XRD, TEM but also glow-discharge spectrometry experiments.

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break

Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break

Wednesday afternoon, 6 September, 12:30

Physical deposition of composite films

Wednesday afternoon, 6 September, 14:00
Chair: Nikola Radić, Rosendo Sanjines

14:00 Oral

Oxide and nitride nanocomposite coatings deposited by Pulse Magnetron Sputtering

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Pulse Magnetron Sputtering proves its versatility and capability in the production of high quality coatings on 3D-parts of various functions. It is a high-rate deposition technique for films of excellent properties, especially from dielectric materials. Examples of actual state-of-the-art functional coatings deposited by pulse magnetron sputtering will be presented, which are nanocomposite oxide and nitride coatings.

For the deposition of nanocomposite coatings, reactive co-sputtering of different target materials allows a fine adjustment of the film composition to generate the specific two-phase structures that exhibit outstanding mechanical properties. In this way, nc-ZrN/Al, and nc-Al_2O_3/ZrO_2 have systematically been generated with generally good adhesion and maximum hardness in the order of 30 GPa in both systems. In the nitride system Zr-Al-N, structure-related hardness increase is achieved for a small fraction of aluminium in the coating. In the oxide system Al-Zr-O, depending on the composition, crystalline phases of g-alumina or tetragonal zirconia can be stabilised.

Another oxidic nanocomposite system attracting much interest is the system nc-TiO_2/Ti, where crystallites of TiO_2 are embedded in a Ti-matrix. Pulse Magnetron Sputtering allows the tailored generation of anatase or rutile phase through the choice of pulse parameters and deposition conditions. Properties like hydrophilicity and photocatalytic activity are investigated as a function of crystalline phase formation and stoichiometry in the nc-TiO_2/Ti system.

14:20 Oral

Nanostructure Functionally Graded Coatings Based on Multilayered Carbides

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Within the framework of the present study, we produced functionally graded (FG) Ti/TiC and Nb/NbC multilayered coatings using a magnetron sputtering system. The composition gradient was generated by varying the relative thickness of the as-deposited metal and carbide layers. Subsequent short diffusion treatments were aimed at eliminating the interfaces between the adjacent layers yet maintaining an overall carbon composition gradient across the thickness of the coating.

We were able to determine the diffusivities of carbon in the TiC layer at different temperatures and, hence, the activation energy, 132 kJ/mole, which is one third that for bulk activation energy. The diffusion coefficients were $3.5x10^{-18}$- $5.7x10^{-15}$ cm^2/s for the temperatures range of 355-550°C. These values are significantly higher than expected from extrapolation of data obtained at elevated temperatures.

The triboligical properties were carried out on coatings consisting of graded Ti-TiC multilayers with different concentration profiles and that had been deposited on WC-Co substrates in two steps. First, 0.5μm thick graded Ti-TiC multilayers were deposited followed by a 2.5μm outer layer of stoichiometric TiC. A critical load ,Lc >27N in the as deposited and post annealed coating was observed for a Ti-rich profile The normal wear resistance in the Ti- rich profile was higher by one order of magnitude than for the other profiles.

The structural evolution of the Nb/NbC multilayers was carried at the temperature range of 400-800°C. At these temperatures, the changes of the relative amount of the phases present are due to the mobility of the carbon atoms. The Nb_2C structure was found to have an orthorhombic structure, in contrast to the expected from the phase diagram.

14:40 Oral

Nanocrystalline TiN and TiCN biomedical gradient films produced by pulsed laser deposition

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Titanium nitride and carbonitride are regarded as potential biomaterials for contact with soft tissue or blood. TiN and TiCN films were fabricated by PLD method with system working with a Nd:YAG laser onto three substrates i.e. Tiα, Ti6Al4V alloy and polycrystalline silicon. A transition (buffer) layer adjacent to the substrate was applied to improve adhesion of the coating. It comprises gradient nitride or carbonitride layers produced by application of controlled variation of reactive gaseous atmosphere in the chamber, by switching the gas flow from Ar to N_2 or C_2H_2. Atomic force microscopy (AFM) was used to examine morphology of the surface in three crucial stages of deposition process i.e. substrate before deposition, after deposition of transition (buffer) layer and after deposition of the final TiN or TiCN layer. Cross- section of the materials was examined by transmission electron microscopy (TEM) as well as by high resolution transmission electron microscopy (HRTEM) with selected area diffraction (Fourier transform) to study the buffer layer. The structure transformation from amorphous type, existing at the border between the substrate and the buffer layer, to crystallized one formed under the maximal nitrogen flow close to the surface was stated. Crystallographic X-ray texture tomography (XTT) in the near-the-surface area was performed for detection of texture variation in respect to the reactive gas flow. Residual stress measurements on the basis of the X-ray method ($\sin^2\Psi$) were performed for the deposited films. Biological examinations were performed on human fibroblast cells in 48h culture. Fibronectin expression was investigated with application of the confocal microscopy.

15:00 Oral

Hybrid pulsed laser deposition of Ti-Cu-N ternary nitride thin films

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Composite ternary nitrides of the form TM-NM-N, where TM and NM stand for Transition Metal and Noble Metal respectively, are interesting materials for mechanical applications, since their mechanical properties may be tailored based on the existing metal and nitride phases. The nitride and metal phases contribute to the hardness and the ductility of the composite structure, respectively. The very low miscibility between NM and N and the high heat of formation of NM-N promote the formation of composites consisting of TM-N and NM phases. In this work we present the growth of Ti-Cu-N films (an example of TM-NM-N) by hybrid Pulsed Laser Deposition. In this configuration, a composite Ti-Cu target, which was ablated by a high-fluence Nd:YAG laser (2nd harmonic) in N_2 ambient was used. The process was carried out in a homogeneous electric field with the substrate being at a negative DC potential (-50 V) with respect to the target. The partial pressure of the ablated Ti-Cu vapor was $\sim 1x10^{-3}$ Pa, while the base pressure P_b was $<5x10^{-5}$ Pa. The N_2 partial pressure (P_{N2}) varied between $1x10^{-2}$ Pa and 40 Pa. Under these conditions there was no self-sustained plasma generation (even for the highest P_{N2} case) due to the low value of V_b. However, laser-induced plasma generation was observed for a wide range of pressures. Films with the typical gold-like appearance of TiN were grown at $P_{N2} \sim 10^{-1}$ Pa. The effects of P_{N2} to the Metal/N ratio and Ti/Cu ratio into the films, as well as the crystal structure of the films were systematically studied employing *in-situ* Auger Electron Spectroscopy and X-Ray Diffraction, respectively.

15:20 Oral

Growth of thin TiO_x films by high power pulsed magnetron sputtering from a compound $TiO_{1.8}$ target.

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We present a study on the growth of TiO_x films by high power pulsed magnetron sputtering (HPPMS). High power unipolar pulses with a low duty cycle (<20%) are applied to a $TiO_{1.8}$ target resulting in a high ionization of the sputtered material and consequenty an increased energetic bombardment of the growing film. We investigate the effect of pulse duty cycle on the deposition rate, the structure, the surface morphology and the optical properties of the TiO_x films. Samples are sputtered at a constant average current mode on grounded, floated, and negatively biased substrates for different duty cycles (1.5%-20%). X-ray diffraction shows that in all cases nanocrystalline films are obtained. It is found that the peak target current (I_{Tp}) and the target voltage increase with decreasing pulse duty cycle, which has implication on the film properties. X-Ray Reflectometry (XRR) measurements show that the deposition rate for films sputtered at I_{Tp}<14 A increases with I_{Tp} and it is up to 60% higher with respect to the rate achieved by DC sputtering. The HPPMS rate decreases at I_{Tp}>14 A but remains slightly hihger than the DC rate. The existence of the two I_{Tp} regimes is discussed in the light of the change of the plasma characteristics upon increasing the peak current and the self-sputtering effect. Moreover, the XRR analysis shows that the HPPMS films exhibit surface roughness of ~0.5 nm, which is 70% lower than the corresponding DC values. Finally, spectroscopic ellipsometry measurements reveal that transparent films with band gap of ~3.2 eV are obtained in all cases. In addition, refractive indices up to 2.52 are obtained for HPPMS deposited films. These values are higher than the correspoding values for DC films (~2.46) grown at otherwise identical conditions.

15:40 Oral

From exotic implantations to nickel-cobalt alloy for totally silicided gate modulation

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In order to obtain a dual metal gate, workfunction tuning by various exotic implantations (F, O, Ga, In, C, Er, Se, Al, Sb,...) has been investigated for a fully silicided NiSi gate. According to electrical data, there is no significant breakthrough observed in the NiSi workfunction. However, it seems that some species like Al, Sb and Ga have special interactions with nickel silicide. TEM and SIMS analysis indicate that silicidation is strongly slowed down with Sb implant (<3e15 dose), and in a less extent with Al implants (>5e15 dose). In both cases, the silicidation has been blocked and the polySi gate has not been entirely silicided. On one hand, a uniform but thin NiSi layer was observed with Sb.On the other hand, Auger mapping clearly showed clusters and evidenced lateral phase segregation into NiSi matrix with poly-Si domains. Moreover, for species like Ga, the surface composition would consist mainly of Ni_2Si and NiSi domains with local presence of Ga-precipitates, surrounded by Si-rich halo. This implies that implanted Ga has segregated during/after silicidation anneals, locally affecting silicidation kinetics. The same phenomenon might have appeared for fluorine.

As most exotic species implanted hardly modulate the workfunction, nickel-cobalt alloys formed from bilayers were also investigated for further gate modulation. It was observed that a significant modulation from 4,62eV to 5eV can be achieved, which is interesting for PMOS devices. Thorough electrical and physical characterizations such as SIMS and TEM were used to understand this modulation.

Poster Session 2/ Poster Award Ceremony

(continuation of Monday's Poster Session),Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Synthesis of composite films

Thursday morning, 7 September, 9:00
Chair: Arvaidas Galdikas, Francis Maury

9:00 Oral

Preparation and electrochemical investigations of well coated carbon fiber-conducting polymer composites.

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Electrode material based on composite of vapor grown carbon fiber(VGCF) and polyyrrole(Ppy) was synthesized by insitu chemical polymerization using different concentrations of pyrrole in the initial polymerization solution, so as to improve the specific capacitance and power characteristics of polypyrrole. Polypyrrole thin films were chemically deposited on vapor grown carbon nano fibers. Ppy-carbon fiber composite electrodes obtained by coating of PPy on VGCF with controlled thickness, has been investigated as electrode material for supercapacitors. Polymerization reaction was carried out with constant sonication. The PPy/VGCF composite was characterized by FTIR spectroscopy. The surface morphology of the polymer films was characterized by using scanning electron microscopy(SEM) and scanning transmission emission microscopy(TEM). The capacitances of the composite electrodes were investigated with cyclic voltammetry(CV). Below ~10 nm of uniformly deposited nano-thin PPy layer on VGCF(02PV) was effective to obtain fully reversible and very fast Faradaic reaction. Almost the entire mass of the PPy for the 02PV composite could contribute to the pseudo-

capacitive charge storage. The nano-thin PPy layer exhibited higher specific capacitance of ~588 F g^{-1} at 30 mV s^{-1} and ~545 F g^{-1} at 200 mV s^{-1} along with an excellent power capability.

Keywords: Composite electrode, Polypyrrole, supercapacitor, vapor grown carbon fiber.

9:20 Oral

Ion beam-assisted method of nanofabrication

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Sputtering can be thought as an erosion of surface due to primary ions bombardment. In our experiments we have investigated effects of ion beam sputtering on shapes and sizes of nanowires initially produced with e-beam lithography. Nanowires have been sputtered with low energetic (1keV) Argon ion beam. For such a low energy ions can alter only very surface of the wire as ions' projected range in bombarded targets is at the level of 1nm. As a result ion beam sputtering provides powerful tool for gentle, layer-by-layer removal of the bombarded material. In parallel with experimental research we have developed theoretical description of evolution of nanowires under ion beam irradiation. We have shown that it is possible to reduce nanowires' cross-section in predictable and controllable way. Starting with different initial sectional profiles of the wires and using different sputtering angles it is also possible to control aspect ratio (height to width ratio) of wire's cross-sectional profiles. We have used described method to investigate the modification of superconducting transition (due to quantum phase slips) for the same wire with progressively reduced cross-section.

9:40 Oral

Formation of three-dimensional quantum dot superlattices

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We study the formation of Ge quantum dots (QD) within Ge-SiO_2/SiO_2 multi-layers deposited by magnetron sputtering deposition. Within a very narrow range of deposition parameters, we found self-organization of QDs in the three-dimensional QD superlattice with hexagonal symmetry. The phase of the QDs and the superlattice unit cell parameters can be tuned by varying deposition and after-deposition annealing parameters. Formed QDs have narrow size distribution and high spatial density yielding many potential applications.

10:00 Oral

Synthesis of an ultra-thin palladium membrane for hydrogen extraction

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Environmental concerns and energy crisis lead to increase of global interest in the development of "hydrogen economy". The increased demand for hydrogen consumption has led to a revival in methods for separation and purification of hydrogen from gas mixtures. The interaction between Pd and hydrogen has been studied extensively and applied widely in hydrogenation catalysts of chemical engineering because palladium absorbs and adsorbs hydrogen easily. Pd membranes having good chemical compatibility and hydrogen selectivity are widely used for hydrogen separation and purification. So far, electroless, electrochemical and sputtering depositions are used for the fabrication of Pd membranes. Most efforts have been focused on reduction of the membrane thickness in order to maximize hydrogen permeability and reduce the membrane cost. However, the fabrication of an ultra-thin and pin-hole free membrane is a challenging task. Based on the observation of microstructure of Pd deposited on the rough stainless steel surface using electroless process, we find that Pd membrane is made of nanoparticles and the thickness of an ultra-thin membrane is directly dependent on the diameter of nanoparticles. The diameter of Pd nanoparticles can be effectively controlled by the concentration of $PdCl_2$ in the plating bath. The higher concentration of $PdCl_2$ in the plating bath will result in a smaller size of Pd particles deposited on the substrate. The smaller the diameter of Pd nanoparticles is, the thinner the dense Pd membrane is built. Hydrogen permeation tested result from the ultra-thin Pd membrane having the thickness of 400 nm demonstrates that the ultra-thin membrane is solid and it can be used at the temperature of 550^{o}C and hydrogen pressure difference of 50 psi. These results will allow optimizing the design of an ultra-thin Pd membrane for hydrogen extraction.

10:20 Oral

Electrophoretic deposition of naturally derived and chemically synthesized hydroxyapatite on metallic substrates

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Metals and alloys are used in restoration of anatomical structures for centuries owing to their superior mechanical properties. However, after their application, a bond with living bone often does not develop or the integration of the implants with bone tissue takes generally several months. This drawback has recently been addressed by coating the metal with a thin layer of a bioceramic, hydroxyapatite (HA), which is the main component of bone and thus a very good osteoin-

ductor. HA coating accelerates bone formation on initial stages of osseointegration, thus improves implant fixation. In this study, electrophoretic deposition (EPD) method was used for coating of titanium substrates. Chemically synthesized nano-powders and naturally derived sub-micron powders of HA were used as coating materials. 50 Volts and 60 seconds were chosen as coating parameters. Particle size distribution and zeta potential measurements were done for characterization of EPD suspensions. Effects of different powders on coating efficiency were investigated. Also effect of inserting a TiO_2 inner coating layer between titanium substrate and HAP coating were investigated. HAP powders and sintered coatings were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

Coffee break

Thursday morning, 7 September, 10:30

Electrical properties of composite films

Thursday morning, 7 September, 11:00

Chair: Agustin R. González-Elipe, Gennaro Conte

11:00 Oral

Optical and electrical properties of sputtered Zr-Si-N thin films: from solid solution to nanocomposite

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DC reactive magnetron sputtering was used for the deposition of Zr-Si-N thin films. Four series of samples have been deposited at various substrate temperatures T_S: room temperature, 240 °C, 440 °C and 640 °C. The total pressure was 0.6 Pa. Depending on T_S, for obtaining nearly stoichiometric ZrN films, the nitrogen partial pressure was kept constant for each series at 5 %, 12.5 %, 37.5 % and 65%, respectively. Si content (C_{Si}) was varied in each series by changing the power applied on the Si target, whereas that on Zr target was kept constant. Electrical resistivity measurements have been performed between 20 and 300 K. The resistivity measured as a function of temperature reveals variations of the temperature coefficient of resistivity as a function of Si concentration. The results of theoretical fitting using the grain boundary scattering model show that the transport properties change with Si addition from a moderated damping regime to a strong damping regime. The optical properties of the Zr-Si-N films with $C_{Si} \leq 10$ at. % can be well explain by straight forward modeling of their dielectric functions by a set of Drude-Lorentz oscillators under the assumption that the films are single, though defective, phase. In this model, the Drude dumping factor Γp (or time relaxation of the free carriers) is mainly related to the film morphology, i.e. the crystallite size and the nature of the grain boundary barrier for electrons scattering. The electron transmission probability G (obtained from fitting of resistivity measurements), the Si coverage on the ZrN grain surfaces (obtained from structural model calculation) and the Drude dumping factor Γp (obtained from fitting the real part of the dielectric function behaviour) are well correlated.

11:20 Oral

The structural and electron field emission properties of ion-beam-synthesised metallic-dielectric nanocomposites

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Metallic nanoclusters embedded in dielectric matrices have attracted a significant research interest because of their uniquely optical and magnetic properties. In this work, we show how the electron field emission (FE) properties of these nanocomposites can potentially form the basis for the next generation vacuum nanoelectronic electron sources. The study of FE of metallic-dielectric nanocomposites is motivated by creating a local field enhancement arising from the isolated conductive clusters embedded in an electrically insulating matrix. Metallic-dielectric nanocomposites, including Ag-SiO_2, Co-SiO_2 and WC-SiC, were synthesised by metal ions implantation onto a dielectric on Si substrates. The WC-SiC nanocomposites fabricated under appropriate conditions show the best FE properties with thresholds field less than 1 V/μm. Moreover, the optimised threshold fields of Ag-SiO_2 and Co-SiO_2 nanocomposites are determined to be 13 and 5 V/μm, respectively and are still comparable to other FE materials. The structural properties of these samples were systematically studied by atomic force microscopy, Rutherford backscattering spectroscopy, x-ray photoelectron spectroscopy, and transmission electron microscopy. The FE properties of these samples are critically dependent on their structure and can be understood by the electrical inhomogeneity and geometric local field enhancement effects. Finally, the details of the dependence of fabrication conditions on the structure and FE mechanisms in these samples will be presented.

11:40 Oral

Dielectric Properties of Nickel Containing Amorphous Hydrogenated Carbon Films Prepared by Microwave Plasma-Assisted Deposition Technique

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Nickel/hydrogenated amorphous carbon (a-C:H) composite films have been deposited at room temperature on nickel coated (100)-oriented single crystal silicon substrates by combining sputter-deposition of metal and microwave plasma-assisted chemical vapor deposition of carbon from argon-methane mixtures of various con-

centrations. The nickel target voltage was varied from - 600 to - 100 V. The films have been deposited on either grounded substrates or substrates biased to - 10 V. The dielectric properties of films were investigated as functions of the frequency and temperature varying from 0.1 Hz to 1 MHz and from -150°C to 175°C, respectively. Pure a-C:H films exhibited a dielectric constant of 2.8 at a frequency of 1 Hz. The dielectric constant at a frequency of 1 Hz was equal to approximately 300 for Ni/a-C:H composite films of 360 nm in thickness containing 20 at.% of nickel. The value of the dielectric constant of these composite films was observed to decrease from 300 to 200 as the frequency increased from 1 Hz to 100 kHz. The effect of the electric field on the permittivity of films is also discussed in the paper.

12:00 Oral

Development and characterization of ferroelectric semiconducting nanocrystalline films

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Recently, ferroelectric semiconductors, namely barium strontium titanate (BST) and strontium bismuth tantalate (SBT) have drawn considerable attention for micro-electronic applications as memory-devices, sensors, uncooled infrared (IR) focal plane arrays (UFPAs), electro-optic devices and MEMS. The present work deals withsynthesis of nanocrystalline thin films of stoichiometric Ba0.8Sr0.2TiO3 and SrBi2Ta2O9 by radio-frequency magnetron sputtering technique (RF-MS) at different deposition and post annealing temperatures and associated microstructural, electrical and thermo-physical characterization. Among other variables, the influence of substrate and annealing temperatures on the microstructure and degree of crystallinity, the mechanism and rate of charge transport/conduction through BST/Si heterostructure, and ac frequency response of the BST film were studied. Process temperatures significantly affect the crystallinity, microstructure and electrical properties of the BST. However, SBT films are not that sensitive to these parameters. However, effect of temperature is pronounced on electro-optic and storage properties of both BST and SBT films. Usually, low deposition temperature ensures a better crystallinity and compositional uniformity. Marginal ferroelctric hysteresis is observed at low and high substrate temperatures. Memory window as high as 1.8 V is noted in BST films deposited at 4500C. Atomic force microscopy reveals that the growth of nanoclusters is governed by Frank-van der Merwe mechanism at low substrate temperature. Memory window was noted as high as 3.6 V for Al/SBT/Si heterostructure. The UV-Visible transmission spectroscopy reveals a direct band (~ 4.0 eV as per Tauc's relation) transition with no vertical forbidden transition. The SBT film is 100% optically transparent. [Partial financial support from the Department of Science and Technology, New Delhi (Grant: SR/S5/NM-04/2005; SCM) is gratefully acknowledged]

Lunch break

Thursday afternoon, 7 September, 12:30

Magnetic properties of composite films (a)

Thursday afternoon, 7 September, 14:00

Chair: Nicolae D. SULITANU, Styliani Kennou

14:00 Invited oral

Structure and magnetic properties of C/FePt granular multilayers prepared by ion-beam sputtering

David Babonneau[1], Frédéric Pailloux[1], Grégory Abadias[1], Frédéric Petroff[2], Nuno Barradas[3], Eduardo Alves[3]

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Nanoscale magnetic systems are attracting considerable attention due to their potential in the field of ultrahigh density recording media. To achieve high storage densities, assemblies of ferromagnetic particles with uniform and small size below 10 nm are required. However, as the magnetic bit size is further reduced to the superparamagnetic limit, the magnetization is easily perturbed by thermal agitation. Therefore, recent studies have been focused on FePt nanoparticle arrays owing to the existence of chemically ordered phases with exceptional magnetic properties such as large magnetocrystalline anisotropy constant.

We have used ion-beam sputtering to grow FePt thin films and C/FePt granular multilayers at room temperature. We have investigated the effects of thermal annealing on the structural and magnetic properties by combining the sensibilities of different techniques including TEM, RBS, GIXRD, GISAXS, and SQUID. We will show that the as-deposited films and granular multilayers contain FePt grains with a disordered fcc structure and are magnetically soft. Thermal annealing causes partial $L1_0$ ordering and growth of the FePt grains both in the FePt films and in the C/FePt multilayers, but these effects are restrained by the presence of carbon. We will demonstrate that thermal annealing of granular multilayers also results in the preferential graphitization of the carbon matrix and in the chemical interaction of the Fe atoms with the Si substrate. Magnetization measurements indicate that magnetic hardening occurs after annealing and that the saturation magnetization is strongly dependent on the annealing temperature.

14:30 Oral

Effect of hydrocarbon ligands on the magnetism of FePt nanoparticles

Carolin Antoniak[1], Kai Fauth[2], Ulf Wiedwald[3], Hans-Gerd Boyen[3], Fabrice Wilhelm[4], Andrei Rogalev[4], Nina Friedenberger[1], Marina Spasova[1], Michael Farle[1]

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Chemically disordered wet-chemically synthesised FePt nanoparticles [1,2] with diameters around 6nm were self-assembled in 2D islands on Si substrates. The magnetic moments of the Fe and Pt atoms of naturally oxidised particles were measured before and after removal of the oleylamine and oleic acid ligands by means of x-ray magnetic circular dichroism (XMCD). We find by XMCD at the Fe $L_{3,2}$ edges that the particles contain a number of Fe atoms corresponding to less than one monolayer equivalent which are in an oxidic environment like α-Fe_2O_3. No influence of the ligand shell on the formation of oxides or on the magnetic properties was detected. After removal of the oxide shell by a soft hydrogen plasma treatment, the particles are transformed into a pure metallic state with an effective spin (orbital) magnetic moment per atom of $2.48\mu_B$ ($0.056\mu_B$) at the Fe sites and $0.41\mu_B$ ($0.054\mu_B$) at the Pt sites. Compared to the oxidised state, the values of the spin magnetic moments per unoccupied d-state at the Fe sites increased by a factor of 4, whereas at the Pt sites no significant changes are observed - confirming the initial guess of an antiferromagnetic Fe oxide. The possibility to identify a Pt enriched particles surface by high-resolution transmission electron microscopy (HR-TEM) analyses of the lattice expansion and the z-contrast will be discussed.

This work was supported by Deutsche Forschungsgemeinschaft (SFB 445), the Marie Curie Research Training Network "SyntOrbMag", the BMBF (05 ES3XBA/5) and the ESRF.

[1] C. Antoniak, J. Lindner and M. Farle; Europhysics Letters **70** (2005) 250

[2] M. Ulmeanu, C. Antoniak, U. Wiedwald, M. Farle, Z. Frait, and S. Sun; Phys. Rev. B **69** (2004) 054417

14:50 Oral

EPR investigations of hybrid nanocomposites based on silicon carbide nanoparticles and polyaniline

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EPR investigations are carried out on functional hybrid nanocomposites associating silicon carbide (SiC) nanoparticles and polyaniline. The nanoparticle surfaces are functionnalised by the polymer being arranged as thin layer (1-5 nm thick). The conductivity of polyaniline was modulated by camphor sulfonic acids (CSA) as dopants. The EPR spectra in different systems (polymer, SiC, nanocomposites) were investigated as function of the doping rate and versus the sample temperature in the range (4K,450K). Analysis of the EPR spectra evidences the relevance of two contributions. The first occurs from dangling bonds and radicals well localised on the polymer backbone. The second type of paramagnetic centres seems to correlate with delocalised unpaired spins induced by the acid doping. The thermal evolution of the spin susceptibility is satisfactory accounted by the thermal activation of paramagnetic centres. The relaxation mechanism probed by the EPR line widths point out the relevance of short relaxation times for the unpaired spin carriers and longer ones of the polymer chains. The evolution of the EPR spectra intensity after annealing at 450K is marked by a relatively stable spin carriers in composites materials while a net decrease occurs in the solely doped polymer. A discussion of the stability and the features of polarons and bipolarons in these composites will drawn from the features of the EPR results.

15:10 Oral

Magneto-optical and electron transport properties of Co-based amorphous and nanocrystalline ribbons

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The discovery of the so-called giant magnetoimpedance (GMI) effect in Co-based amorphous and nanocrystalline ferromagnetic materials have enabled the development of high-performance magnetic sensors. However, the maximum value of GMI experimentally observed for existing materials has been much smaller than the theoretically predicted value. In order to obtain the suitable value of GMI for practical applications we have used low temperature (77 K) treatment and laser annealing of Co-based amorphous alloys. These combining treatments significantly improve the GMI effect. Co59Fe5Ni10Si11B15 and Co71Fe4Si10B15 amorphous were prepared by the melt-spinning method. GMI measurements were carried out along the ribbon axis with dc magnetic field up to 100 Oe and in the frequency range of 10-1000 kHz. Magneto-optic Kerr effect was used for characterization of surface magnetism in laser annealed Co-based alloys. The magneto-transport properties of these materials were studied by infrared reflection spectroscopy method. Laser annealing of the Co-based amorphous alloys leads to the formation of nanocrystal grain whose size varies between 20 and 50 nm. The value of GMI grows in amplitude about of 2-3 times after low temperature treatment and laser annealing for the abovementioned samples. The measured hysteresis loops as a function of the external magnetic field show the approximately coherent rotation of magnetization. From measurements of the magnetoreflectance in the mid-IR region the relaxation time of conductive electrons was estimated. It was shown that the magnetoreflectance exists in amorphous and nanocrystaline Co-based ribbons due to a change in their conductivity behavior when a magnetic field is applied.

Coffee break

Thursday afternoon, 7 September, 15:30

Magnetic properties of composite films (b)
Thursday afternoon, 7 September, 15:50
Chair: Jean Paul Riviere, Nicolae D. SULITANU

15:50 Oral

Comparison of patterning of highly ordered CoPt alloys with perpendicular anisotropy using two methods: ion etching and ion irradiation

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CoPt alloy layers were prepared by molecular beam epitaxy, deposited directly on a MgO(001) substrate. These layers had the $L1_0$ tetragonal structure, ordered in the growth direction with an easy magnetization direction perpendicular to the layer plane and a 100 percent remanence. The layers were nanostructured using two different methods:

A) A network of dots were classically realized by electron beam lithography and ion etching. Whereas the continuous layers had a labyrinthine magnetic structure after perpendicular demagnetization, all the dots are single domains with randomly distributed up and down magnetization.
B) Ionic irradiation was used to magnetically pattern a CoPt alloy film, using a mask to protect arrays of CoPt dots during a ionic irradiation. The unprotected regions became disordered and magnetically soft, whereas the protected regions remain magnetically hard. This spatially selective irradiation provides a spatial distribution of magnetic anisotropy and hence of the magnetization direction, perpendicular to plane in the ordered zones and in-plane in the disordered zones. The interest of this method is that the film recovers its initial roughness after elimination of the mask which is promising for the data storage applications.

16:10 Oral

Magnetic properties of nanogranular composites CoFeZr -Alumina

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The 5 to 15 mm thick nanocomposite films of CoFeZrin alumina matrix were sputtered in the chamber evacuated with pure Ar gas. It is shown that magnetic state of the films was strongly dependent on concentration *X* of CoFeZr nanoparticles. The films being before percolation threshold *X*c = 40-45 at.% displayed superparamagnetic state of metallic nanoparticles with the lack of sextet in Mössbauer specta, non-hysteresis character of magnetization curves and invariability of real *m1* and imaginary *m2* parts of magnetic permeabilities measured at frequences 20-200 MHz. Beyond the *X*c, the values of *m1* were increased with *X* elevation due to decrease of interparticle distances. As a result, the effects of dipole-dipole and exchange interactions of nanoparticles became apparent resulting in the presence of magneto-ordered regions (magnetoclusters) including a few nanoparticles. The formation of net of magnetoclusters was observed using MFM and Mossbauer measurements. The observed changes in magnetic properties of nanocomposites with *x* fully agree with our earlier DC/AC measurements revealed peculiarities of hopping carrier transport, negative magnetoresistance and impedance at *X*c.

16:30 Oral

Magnetoresistivity of Cobalt/PTFE granular composite film produced by Pulsed Laser Deposition technique

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Giant Magnetoresistance (GMR) was observed in Cobalt-PTFE heterogeneous granular films. The samples were deposited on glass substrates by using the Pulsed Laser Deposition (PLD) technique. The samples prepared exhibit a percentage change in resistance (MR%) of about 6% at room temperature with an applied magnetic field (H) of 10kOe. A significant change of the MR profile was observed when the magnetic field was applied in-plane and out-of-plane to the film. The temperature dependence of resistance (R-T) of the samples was measured from 10K to room temperature. The results show that charge transportation is mainly due to tunneling in the sample. The GMR effect can thus be interpreted by the spin-dependent electrons tunneling through the ferromagnetic metal granules (cobalt) in the insulating matrix (PTFE).

Friday, 8 September

Structure of composite films
Friday morning, 8 September, 9:00
Chair: Adam Tokarz, Magdalena Parlinska-Wojtan

9:00 Invited oral

Atomistic simulations of nanocomposite carbon films

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Nanocomposite carbon (*n*-C) films may be described as hybrid materials, consisting of C nanostructures embedded in a host amorphous carbon (*a*-C) matrix. This form of C offers the unique possibility to intermingle the properties of C nanostructures with those of pure

a-C, and thus tailor the mechanical and electronic properties by controlling the type and size of the embedded structures and the hybrid state of the *a*-C matrix.

Here, I review recent computational work in my group aiming at an accurate atomistic description of *n*-C films. The methods employed are state-of-the-art Monte Carlo and tight-binding molecular dynamics simulations. Focus is given on two vastly different *n*-C materials.

The first one consists of diamond nanocrystals in *a*-C. It is shown [1] that nanodiamonds are stable in only dense, tertrahedral *a*-C matrices. The films exhibit high strength and hardness, and extreme elastic moduli approaching those of diamond [2], making them ideal for ultrahard coatings and MEMS/NEMS devices. Furthemore, our simulations elucidate for the first time the deformation properties and fracture mechanisms of *n*-C films.

The second nanomaterial discussed here consists of C nanotubes embedded in low-density *a*-C matrix [3]. It is shown that van der Waals forces are crucial in shaping up the interfacial geometry, producing a curved graphitic wall surrounding the tubes without direct bonding to the matrix. This might lead to films with excellent thermal and electrical conductivity in the tube direction.

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9:30 Oral

Discussion of a possible third carbon bonding state in PVD-deposited metal carbide / amorphous carbon nanocomposite thin films and its impact on mechanical and electrical properties

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In thin film composite systems of nanocrystalline metal carbide particles or clusters embedded in an amorphous carbon matrix the standard model of the microstructure suggests two bonding states for the carbon: C-C bonds, both in sp^2 and sp^3 configuration, and carbidic C-Me bonds. In spectroscopic measurements of sufficient resolution it is hence expected that two states can be observed. Already since the early 1990:s there have however been several publications where a third bonding state for carbon has been observed for nanocomposite nc-TiC/a-C thin films. Explanations have varied and no consensus of the nature of this third state exists.[1-6]

We have prepared nc-TiC/a-C samples of various chemical composition by non-reactive DC-magnetron sputtering. These samples have been evaluated using XPS and XRD, and either their mechanical or electrical properties have been measured using nanoindentation and four-point-probe methods. Variations in C1s sub-peak intensities for samples of different carbon-content have been observed and the film properties are discussed in the light of these features. The nature of this third carbon state and its possible impact on film properties and role in design of coatings will be discussed. Two models of the nature of this new carbon state will be suggested requiring future, more detailed research studies.

[1] V. Schier et al, Fresenius Journal od Analytical Chemistry 346, 227-232 (1993)
[2] B. Hornetz et al, Fresenius Journal od Analytical Chemistry 349, 233-235 (1994)
[3] J. Luthin et al, Nuclear Instruments and Methods in Physics Research B 182, 218-226 (2001)
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[5] A. Mani et al, Surface and Coatings Technology 194, 190-195 (2005)
[6] E. Lewin et al, Journal of Appplied Physics Submitted (2005)

9:50 Oral

Electronic structure and chemical environment of Si nanoclusters embedded in a SiO_2 matrix.

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Using photon-in photon-out soft x-ray absorption and emission spectroscopy, the electronic structure of silicon nanoclusters embedded in an electrically insulating SiO_2 host matrix is investigated as a function of nanocluster size. Using the new method of cluster preparation the size of the Si clusters is well controlled. The diameter of the nanoclusters was less than 5nm.

We find the nanoclusters to be of a core-shell structure with a crystalline Si core and a thin transition layer of a suboxide. Based on the spectroscopic data the electronic structure of the Si core and the chemical content of the suboxide shell are studied. The presented results are in agreement with photoluminescence measurements.

Effects of electronic quantum confinement are detected in the Si cores. We find that the influence of confined excitonic states and the presence of SiO_2 matrix manifests itself predominantly in the unoccupied electronic states.

10:10 Oral

Structure and properties of ion beam deposited SiO_x doped DLC films

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Diamond like carbon (DLC) films received considerable interest due

to the outstanding mechanical, optical and electrical properties. The properties of the DLC films can be controlled by doping with both metallic and non-metallic elements and compounds. In such a way problems of the adhesion with ferrous substrates, high internal stress and thermal stability can be solved. Particularly SiO_x doping of the hydrogenated DLC films results in reduction of the internal stress, friction coefficient, dielectric permittivity, considerably better adhesion with ferrous substrates such as iron. Increase of the fracture toughness, deposition rate, optical transmittance, hydrophobicity and higher thermal stability were reported for SiO_x doped DLC films as well. Such combination of the properties is particularly interesting for possible microelectronic applications of the DLC such as anti-sticking layers in novel lithographic techniques such as nanoimprint lithography as well as room temperature deposition of dielectric films.

In this research silicon oxide doped hydrogenated amorphous carbon films have been synthesized by direct ion beam deposition. Effects of the deposition process conditions such as composition of the gas precursors, ion current density and ion energy were studied. Structure of the films was investigated by means of the Raman and FTIR spectrometry as well as X-ray photoelectron spectroscopy (XPS). Hydrophobic properties of the SiO_x doped DLC layers were evaluated by measuring contact angle with water. Dielectric properties of the synthesized diamond like carbon films such as breakdown voltage as well as dielectric permittivity were studied.

Coffee break

Friday morning, 8 September, 10:30

Applications of composite films

Friday morning, 8 September, 11:00

Chair: Isaac Dahan, Sigitas Tamulevicius

11:00 Invited oral

Electrocatalysis and bioelectrocatalysis and nanostructured composite films

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We pursue here the concept of protecting and activating of Pt and Ru-Se nanoparticles by coating them with Keggin type phosphododecamolybdate or phosphododecatungstate, the well-defined oxygen-bridged metal clusters related to the parent tungsten and molybdenum oxides. In addition to the changes in morphology, the increased dispersion, as well as to the bifunctional nature of their reactivity, the polyoxometallate stabilized Pt nanoparticles are characterized by a broader potential window where platinum metal is not covered by the inhibiting PtO oxides. In the case of Ru-Se clusters, modification of their surfaces results in diminishing of their size and leads to the lowering of background currents in the potential range where the oxygen reduction reaction is operative. Recently, there has also been growing interest in biofuel cells that can be viewed as analogues of conventional fuel cells except that they typically utilize biocatalysts (enzymes), biofules, and neutral or slightly acidic electrolytes. While oxygen serves as a cathode fuel, glucose, lactate or ethanol can be considered as anode fuels. The possibility of use of carbon nanostructures will be discussed. The concept of fabrication of the phosphomolybdate-stabilized colloidal suspensions of metal particles is extended here to the formation of the analogous dispersions of carbon nanoparticles and multi-walled carbon nanotubes and utilized to the generation of network films of conducting polymer linked carbon nanostructures on electrode substrates. The research is not only of importance to the construction of effectively operating charge storage devices (capacitors) but also charge mediators (relays), chemical and biochemical sensing devices, as well as electrocatalytic systems.

11:30 Oral

Structure and Catalytic Activity of Nanocrystalline Nickel Thin Films

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The effects of phase composition of the nc-Ni thin films upon their catalytic activity is examined.

Nickel thin films (400 nm) were deposited by dc magnetron sputtering onto fused silica, mono-Si and alumina ceramic substrates. The base pressure in the process chamber was 10^{-6} - 10^{-5} Pa, and the working gas was argon at 1.3 Pa with a small admixture of oxygen. The substrate temperature was varied in the 20 - 700 °C range in order to examine the effects upon the film structure.

The film structure has been investigated using the XRD method and the Grazing Incidence Small Angle X-ray Scattering (GISAXS). The oxygen content was determined by the ERDA method using 35 MeV Cl^{7+} ions. The electrocatalytic activity of the nc-Ni electrodes for the Hydrogen Evolution Reaction (HER) was studied in 1.0 mol dm^{-3} NaOH solution at 25 °C using polarization and electrochemical impedance spectroscopy techniques.

It was found that the prepared nickel films exhibit nanocrystalline structure in the whole range of examined substrate temperatures. However, with the increase of substrate temperature up to 700 °C the Ni-grain size increases from 12 to 130 nm, while characteristic length of the intergranular matter decreases from 8 to 2 nm. The oxygen content strongly decreases with increase in substrate temperature.

Electrocatalytic activity of the nc-Ni films for the HER significantly decreases (by more than 20 times) with the increasing substrate temperature and corresponding increase of the Ni-grain size. Thus, improved catalytic activity of nc-Ni is in accordance with an increase

in the intercrystalline volume fraction at the very small grain size of the sputter deposited nickel.

11:50 Oral

Synthesis and characterization of hard / soft nano-layered composite carbon films: implications for gas barrier applications

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The synthesis of thin amorphous carbon films, consisting of periodically stacked hard (a-C:H) and soft polymer-like carbon (PLC) nanolayers is reported. The a-C:H layers were deposited by sputtering of a graphite target in an Ar-H_2 plasma and the PLC ones by plasma-enhanced chemical vapor deposition using CH_4 as main gas precursor in the gas mixture. The film growth mechanism was first studied for the single layer materials grown on silicon and then for one kind of carbon grown on the other, the latter taken as the substrate for the former. The chemical properties of the films were studied by x-ray photoelectron spectroscopy (XPS) and Fourier-transform infra-red spectroscopy (FT-IR). Angle-resolved XPS was also used for the depth profiling of the films in their early growth stages while Auger electron spectroscopy (AES) was employed for that of the layered structures. The film internal stress was determined from the substrate curvature measurements and the role of the different stress contributors, i.e. interface, bulk and surface, was investigated. The gas barrier properties of the composite films against He were studied and the implications of both the gas permeation and the mechanical properties are discussed for flexible gas barriers.

12:10 Oral

A novel alloy (Tantalum Carbide) for metal gate deposited by Ion Beam Sputtering for CMOS 45 nm device

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Since the MOSFET gate lengths scale down to 50 nm and below, it appears 2 major problems: the first one is the increase of leakage current and the second one is the polysilicon gate electrode depletion induces an additional capacitance [1]. To solve the first issue high-k dielectric maybe a solution since for the same electrical thickness. To solve the problem of depletion (valid for the semiconductors) the solution is containly the use metal gate. In this study, we will use Tantalum carbide (Ta_xC_y). Ta_xC_y [2] electrode is a ve metal candidate. In this work, the influence of the sputtering conditions was studied in order to optimise the value of work function.

Ta_xC_y films were prepared by Ion Beam Sputtering with a tantalum target and a mixture of argon and methane. The material is tantalum carbide Ta_xC_y of 10 nm thickness deposited on dielectric gate. All the samples are deposited at room temperature. The physicochemical characterizations used for Ta_xC_y alloys are: X-rays difraction, SIMS, RBS and XRR.A relation between the carbon rate, the phase of Ta_xC_y formed and the work function was established.

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Lunch break

Friday afternoon, 8 September, 12:30

Sensors and devices based on composite films

Friday afternoon, 8 September, 14:00

Chair: Bogusław Major, Nadhira Laidani

14:00 Invited oral

Nanostructured metal oxide thin films for gas sensors

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This paper will deal with aspects of selected metal oxide thin films with respect to gas sensing. There will be a presentation of the main methods employed for the fabrication of ZnO and InO, i.e. reactive sputtering, spray pyrolysis and pulsed laser deposition (PLD), followed by a study on how the above growth techniques result to polycrystalline films with lateral grain sizes ranging from 20nm to 70nm and thickness between 40nm and 1μm. The study will continue on how the broad range of morphological parameters affect the thin film response to gases. It is noted that the change in the order of magnitude between the post and the pre-gas exposure response (usually this is the DC conductivity) constitutes a figure of merit of the device sensitivity. In the case of ozone, for high ppm concentrations in air background, the response reached values as high as 8, while for low ppb concentrations it was of the order of 1, enough to detect a 1ppb ozone concentration. It was found that the sensitivity is related in an inversely proportional manner to crystallite size and film thickness and is influenced by the growth method. However the sensitivity of a thin film is not just a function of the sample morphology. Environmental parameters, such as the operational temperature, as well as the type of excitation, influence the response significantly. Results obtained both with the measurement of the DC conductivity or the use of surface acoustic waves (SAW) have shown that it is possible to further enhance the thin film response to state of the art levels, which in the case of ozone, could be as low as 1ppb at room temperature.

14:30 Oral

Polypyrrole Based Nano-Electrode Arrays

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The implementation of sensor platforms providing high sensitivity of detection is a crucial step for the design of the new analytical device generation for biosensor developments. Designing platform with active/non-actives region at nanoscale has shown already a drastic increase of sensitivity. Besides, the electrochemical sensitivity can be as well enhanced by using nano-electrode arrays that increase mass transport rate. Polypyrrole (PPy) is a good candidate to fulfil these requirements. Its preferential material for bio-analytical electrochemistry based sensor thanks to its good environmental stability, excellent biocompatibility and higher conductivity, together with the possibility of being functionalised with biological relevant functional groups. In this work surfaces with PPy nano pillars were fabricated by electrochemically growing PPy in a nano-template of gold nano-seeds in a silicon oxide (SiO_x) matrix. Atomic force Microscopy and Scanning Kelvin Microscopy demonstrated that PPy grown only inside the conductive gold seeds, creating nano pillars of conductive material surrounded by an insulating material. The nanostructured surfaces were studied by Cyclic Voltammetry using hexacyanoferrate and the typical sigmoidal shape voltammogram of nanoelectrodes was obtained . Square Wave Voltammetry was tested in the surface and a well defined peak was obtained which indicates that the nanoarray is a promising surface for use in trace analytical analysis.

14:50 Oral

The baric coefficient of a quantum dot

Roman Peleshchak, Olesya Dankiv

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The studies of physical processes in zero-dimensional heterostructures occupy a particular position, because the unique fundamental properties which are not inherent to massive crystals can be observed in such systems. Nanostructures were successfully used in opto- and microelectronics during last years.

The basic physical characteristics of nanoobjects (the baric coefficient, Young's modulus, Poisson's ratio) are accepted in the majority of theoretical models to coincide with the corresponding characteristics obtained from macroscopical experiments. However, if the described structures contain a few nuclear layers, the physical characteristics of nanostructures appreciably differ from the corresponding characteristics of bulk crystals. In particular, a discrepancy between the values of the baric coefficient of the InAs quantum dots (QDs) in an InAs/GaAs heterostructure and in a bulk InAs crystal is observed. The results of experimental researches show that the value of the InAs-QD baric coefficient differs from that of the bulk InAs crystal by about 30-40%. The aim of this work is therefore to calculate the dependence of the QD baric coefficient on its dimensions in the framework of the deformation potential model.

The results of calculations for the InAs/GaAs heterosystem with InAs QDs:

The value of the baric coefficient for the spherical QD of the radius 4.5nm equals 9.45meV/kbar provided the plate thickness of the surrounding matrix 50nm. This means that the value of the InAs-QD baric coefficient is smaller than that of the bulk InAs crystal by 21%. An increase of the energy of the transition into the ground state results in a linear growth of the baric coefficient. The increase of the QD radius stimulates the opposite effect: the baric coefficient diminishes.

15:10 Oral

Evolution Of Quasistationary Electron Spectra In Nanofilm Systems: Quantum Wells And Quantum Dots

Mykola V. Tkach, Julia O. Sety, Oxana M. Voitsekhivska, Maxym Y. Rudko, Rostyslav B. Fartushynsky

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The modern experimental abilities of the creation of complicated nanoheterosystems (quantum dots, wires and wells) allow to produce and study the closed and opened systems as well. The important peculiarity of the latter is the posibility to guide the additional chanell of quasiparticles (electrons, holes excitons) relaxation due to their penetration into the external medium. Since, it is able to create the super fast radiating devices in the needed region of the spectrum. There is observed the spectrum and wave functions of quasiparticles in the closed multifilm nanosystems where the increasing of the magnitude of the external well thickness brings to the almost opened nanosystem. Herein there arises the question at what sizes of the closed system external well and with which exactness one can assume such system as the opened one. The other principal question is how does the stationary discrete quasiparticle spectrum in closed nanosystem transform into the quasistationary one at the increasing of the external well thickness. Also it is explained the arizing of band semiwidth and quasiparticle life time in quasistationary states observed in the closed systems with very big thickness of the external well. The properties of electron spectra in the closed two well QD and three well QW are studied. Using the rectangular potential wells and the effective mass approximation there is obtained the exact solution of the Schrodinger equation with the fitting conditions of wave functions and density of currents continuuty. The electron energy spectrum and wave functions are established. The dependences of probability of electron location in both nanosystems on the thickness of the external well and both wells of plane nanosystems are calculated.

Coffee break
Friday afternoon, 8 September, 15:30

Parallel Session
Friday afternoon, 8 September, 15:50

15:50 Oral

Investigations of GaN-Based Heterostructures with Scanning Capacitane Microscopy

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As the dimensions of devices have minimized, two-dimensional dopant profiling of semiconductor devices has become an important technique for obtaining the required electrical characteristics. As a result, various analytical techniques have been developed to obtain two dimensional information. Scanning capacitance microscopy (SCM) has been shown to be useful for quantitative two dimensional dopant profiling.

In this study, GaN Light emitting diodes were grown by MOCVD and the samples were prepared using a cross-section polisher for SCM measurements. SCM was performed on Veeco MultiMode SPM. We show that SCM enables quantitative two-dimensional dopant profiling of p- and n-GaN at concentrations around $10^{19}cm^{-3}$. It is also possible to analyze a sub-um p-GaN and investigate the dopant distribution in the InGaN/GaN heterostructure device. We compare SCM signal with secondary ion mass spectroscopy profile.

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2. M.L. O'Malley, G.L. Timp, S.V. Moccio, J.P. Garno and R.N. Kleiman, Appl. Phys. Lett., 74, 272 (1999).

16:10 Oral

The criterion of appearance of an n-n+ junctions in a crystal with dot defects

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The interaction of dot lattice defects with self-consistent deformation field, caused by dot defects, results in a formation of ranked defect-deformation (DD) structures: clusters and periodic structures. The reason of the appearance of non-uniform deformation, which results in local change of the band spectrum, is the presence of DD-structures. As a consequence, the spatial redistribution of electrons takes place, which gives birth to the electrostatic potential. The aim of the present work is to study the conditions of the appearance of an n-n+ junction in a crystal with dot defects in the framework of the model of electron-deformation potential.

The main results. The criterion of the appearance of an n-n+ junction is concentration of lattice defects. At concentration of defects $n_{d0}<n_{dc1}$ (n_{d0} - medial concentration of defects) the processes of self-organizing of defects are absent and, accordingly, a n-n+ junction is absent. Within the interval of concentration of defects $n_{dc1}<n_{d0}<n_{dc2}$ the clusters in a crystal are formed, which are the reason of non-uniform deformation. In a result, there is a shortage of electrons in of one part of a crystal, and surplus - in another. Thus, the double electrical layer is formed in a crystal with dot defects. At medial concentration of defects $n_{d0}>n_{dc2}$ the defect periodic structures are formed. Accordingly, redistribution of electrons will have periodic character, and the sequentially joint n-n+ junctions appear.

Posters

Monday, 4 September

Poster Session

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster A/PI.01

Synthesis and tribological properties of nanostructured WC/a-C coatings deposited by magnetron sputtering

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Nanocomposite coatings made of crystalline hard phases in combination with an amorphous soft matrix represent a good solution for achieving a compromise between mechanical and tribological properties. In this work, nanocrystalline tungsten carbide (WC) was combined with amorphous carbon (a-C) in different proportions by magnetron sputtering technique at near room temperature. By controlling the power applied to WC and graphite targets it is possible to obtain nanocomposite coatings with variable hard/lubricant phase composition. The microstructure and chemical composition of the samples were studied by transmission electron microscopy (TEM), electron (ED) and X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and electron energy-loss spectroscopy (EELS). The crystalline phases were identified as WC_{1-x} and W_2C by XRD dispersed in a disordered carbon matrix. The C1s photoelectron peak was used to estimate the distribution of carbon in WC and a-C phases inside the composite (C_{wc}/a-C) and this parameter was correlated with the tribological properties measured in a pin-on-disk tester in dry conditions. The friction coefficients decreased from 0.8 to 0.15 when diminishing the C_{wc}/a-C ratio from »1.7 to 0.7. The endurance of the coatings is increased following the same trend reaching optimum values of 1×10^{-7} mm^3/Nm. In view of the results, relationships among synthesis conditions, microstructure, chemical bonding and compositions and tribological performance of the WC/a-C nanocomposites were established.

17:20 Poster A/PII.01

Analytical methodology development for SRO chemical physical characterization.

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Silicon reach oxide (SRO) layers and silicon nano-crystals nc-Si have been gaining particular attention for their optoelectronic properties. Lasers and electro-optical modulators are interesting examples of their promising applications. State of art of complementary analytical techniques, with an accurate and precise characterization methodology, are recognized as a keystone to correlate the growth process of these materials and their optoelectronic properties. Photo-electron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS) have been focused in this work as helpful ways to obtain the required characterization of these materials. XPS and SIMS combine the chemical physical analytical techniques requested to provide quantitative and accurate results. The SIMS quantification of elements in silicon reach oxide with variable concentration of oxygen, silicon and nitrogen is not trivial. Besides the high precision of SIMS profiles, performed by a Cameca SC-Ultra apparatus, the accuracy of the data in SRO is a difficult task, because of different matrices. In fact the ratio between SiH_4, N_2O and NH_3 was varied during SRO deposition by PECVD. A fit equation for silicon SIMS depth profiles quantification in SRO is proposed. The physic-chemical data provided by the developed methodology have been related to electro-optical properties of SRO layers, characterized by electroluminescence (EL) emission. The opportunity of a cyclic feedback from the growth parameters to the electro-optical properties, passing through the materials characterization by XPS and SIMS is highlighted. Furthermore an original approach for the determination of structural properties of silicon nano-crystals by atomic force microscopy (AFM) has been investigated.

17:20 Poster A/PI.02

Amorphous carbon layers on polymeric substrates.

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Optical plastics are gradually replacing glass in many commercial applications. They now cover a broad field of possible uses in automobile headlamps, corrective lenses, compact discs ad protective windows etc. The problem is however that in many cases a widening of the area of the applications is limited because of low wear and scratch resistance of the materials. This is why they share the need for application of ceramic coatings. The cerefully desighed layers deposition on polycarbonate surface are expected to improve their mechanical and tribological properties. One of the primary technologies used in the processing of the ceramic layers of tailored properties is the plasma enhanced Chemical Vapor Deposition (PE CVD).
In this work we report the results of studies on the technology of a-C:H and a-C:N:H layers for applications on polycarbonate. The layers have been deposited using plasma sustained by radio-wave-frequency excitation (RF CVD), at the temperatures not higher than 80 ^oC. In the experiment H_2, CH_4 and optionaly N_2 have been used as gasous precursors. The optimal processing parameters have been indicated for which good adhesive layers can be obtained.
With applications of FTIR, EDS, and AFM techniques the chemical composition, atomic structure and microstructure od the layers have been studied. Additionally the friction coefficient and the tribological properties of the obtained layers have been cerfully analysed.
It has been found that the a-C:H and a-C:N:H layers deposited on polycarbonates with RF CVD method are good adhesive and possess many promising properties. They have high hardness, high wear resistance nad low friction coefficient. Simultaneously they are smooth nad transparent. This allow beliwing that they are good coatings for a wide area of applications.

17:20 Poster A/PII.02

GISAXS study of temperature evolution in nanostructured $CeVO_4$ films

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Cerium vanadate films on glass substrate were obtained by sol-gel process. The morphology of these nanostructured and porous films was studied by grazing-incidence small-angle X-ray scattering (GISAXS). The aim of the GISAXS study was to investigate the changes in grain sizes due to the temperature evolution with three different time intervals (5min., 15 min. and 30 min.) of annealing at 673 K. We found that the effects of the different times of annealing are diverse for surface and bulk properties of this V/Ce oxide. Although the increase in size is common to all the samples, it is far more pronounced in the bulk. The result is that for short annealing time grain sizes are bigger close to the surface, while this is reversed after long annealing. Generally, the annealing time is critical parameter in sol-gel preparation of the nanostructured vanadium oxide films, which are used as electrodes in new optoelectronic devices. This particular morphology is quite suitable for application in electrochromic devices, in an advanced electrochemical cell concept and efficient new solar cells.

17:20 Poster A/PII.03

Preparation and Characteristics of the Fe_3O_4 Nanoparticle/Chitosan Composite Microspheres

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The Fe_3O_4 nanoparticles are prepared by hydrolyzing the co-solution of $FeCl_3 \cdot 6H_2O$ and $FeCl_2 \cdot 4H_2O$ with NH_4OH solution. Fe_3O_4/ chitosan composite microspheres are prepared using the ionotropic gelation method by adding a proper amount of TPP solution to chitosan solution with Fe_3O_4 nanoparticles dispered in. The size of composite microspheres distribute between 0.1m to 1μm. This composite microsphere is magnetically responsive and biodegradable, and thus can be used as functional material or carrier for the delivery of drugs.

Key words: Fe_3O_4 nanoparticle□ionotropic gelation reaction□composite chitosan microsphere

17:20 Poster A/PI.03

Stress State and Mechanical Properties of Arc Evaporated Ti-Al-N Thin Films

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We present recent results on the residual stress and mechanical properties of TiN/TiAlN composite thin films grown on WC-Co, tool steel and high-speed steel substrates using arc-evaporation method. From XRD measurements it was found that for TiN/TiAlN multilayer the residual stress in the TiN sublayers were higher than in the TiAlN sublayers, as a consequence of the lower efficiency of atomic peening in TiAlN. The difference in the residual stress between the sublayers has strong implications on the texture development in multilayer thin films. For instance, it was found that TiN monolayers present a (111) texture, while TiN/TiAlN multilayers exhibit a texture that is inclined with respect to surface of the samples. Microbending tests in SEM and nanoidentation experiments were conducted in order to investigate the influence of the residual stress in fracture behaviour of thin films.

17:20 Poster A/PI.04

Dynamic Nanoindentation Spectroscopy and Microscopy on thin polymer films

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The mechanical characterization of polymeric films is an important issue in many industries such as automobile, microelectronic or packaging where films are used as paint coating, photoresists, dielectric insulators and permeable coating. In particular, reliable and reproducible quantitative values of E' (storage) and E" (loss modulus) are difficult to achieve.

Nanoindentation, the traditional and well established instrumental technique to have access to the Young Modulus and hardness, is however inappropriate for thin and low compliant materials. Indeed, such *dc*-nanoindentation experiments give a convolution of both the film and the substrate which is not straightforward to interpret.

In contrast to quasi-static measurements, the instrument can be used in *ac* mode by modulating the force; this mode enables quantitative studies of dynamic mechanical properties like viscoelasticity.

We have designed a dynamical nanoindentation setup based on sample modulation, using a stacked ceramic sample actuator instead of standard bimorph, allowing spectroscopy and quantitative imaging measurements.E' and E" were imaged with a dual phase lock-in amplifier (R and f) from which E' and E" have been calculated.

The input signal was the actual probe displacement relative to the sample and the reference the sample excitation voltage.Polymeric samples have been encapsulated with phenol formaldehyde resin.

Dynamical Mechanical Analysis and classical nanoindentation measurements on the resin where used to calibrate E' and E" for the other samples.

Thin film of PolymethylMethacrylate/Polystyrene, a non miscible polymer blend, has been deposited on various substrates and mechanically characterised after different thermal and light sample excitation.

17:20 Poster A/PII.04

Surface investigations of TiN layers on different substrates

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The subject of this presentation are TiN layers deposited on steel, silicon and glass substrates. TiN coverages have been prepared by the arc physical vapor deposition (PVD) technique. TiN are well known material used in many industrial applications. Particularly the TiN is a very attractive material for creation of hard protective coat-

ing layers. The final useful mechanical properties of TiN coating layers are strongly determined by surface morphology. It depends on many factors i.e topography of substrates, deposition technology, mechanical or thermical treatments. For this reasons the optical, AFM and SEM measurements have been done. The optical methods comprised: integrating sphere, double beam reflectometer, XY optical profilometer measurements in the range from 190 nm to 2500 nm. Additionally for refraction *n* and extinction *k* coefficients the ellipsometric study has been performed. As a complemented to AFM and SEM method, the BRDF (bidirectional reflection distribution function measurements have been performed. It is based on analysis of scattered radiation intensity from samples for different scatter angles. The power spectral density is proportional to BRDF signal and then it allow to evaluate surface parameters on much bigger than AFM and SEM surface area. The statistical surface parameters of substrates and layers before and after mechanical and thermal treatment have been obtained. The statistic surface make up: PSD (power spectral density), roughness, correlation length and distribution of heights. It allowed to estimate correlation functions of surface statistic before and after deposition and measure surface parameters after mechanical and thermo-mechanical treatments.

17:20	Poster	A/PII.05

Preparation and microstructure of Al-Mg thin films deposited by PLD

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In recently years hydrogen has been attracting attention as a clean energy. For its storage, as a tank for liquid hydrogen Al-Mg alloy has been chosen. Hydrogen is throught to permeate most metallic materials and causes hydrogen embrittlement. Therefore in the present examination the Al-Mg thin films has been made using pulsed laser deposition (PLD) technique. The Al-Mg alloy was employed as a target (substrate). The thin films have been deposited on silicon and glass wafers at room temperature. In the investigations have been used the scanning electron microscope and the transmission electron microscope. After pulsed laser deposition observed the changes of the microsturctures, the chemical composition and the thin films have been characterized using X-ray diffraction. The microstructure of the deposited thin films especially depended on the exposure time.

17:20	Poster	A/PI.05

The influence of the structure of the nanocomposite Ni-PTFE coatings on the corrosion properties

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The nanocrystalline composite electrochemical coatings prepared with nickel matrix and PTFE particles were investigated. Nickel plating bath of low nickel ion concentration (0.75 M) with organic compounds and dispersed PTFE particles content was used for coatings electrodeposition. PTFE particles content was determined gravimetrically. The roughness of the nickel foils was evaluated using a profilograph. The microhardness of the deposited layers was measured using a Vickers' method at a load of 0.01 and 0.05 kG. Following dependencies were obtained:

$$R_a = 0.44 * C_p + 0.0065 * D_{111}$$

$$HV_{0.01} = 25.65 / D_{111}^{1/2}$$

X-ray diffractometer with CuKα radiation was used to determine the preferred orientation, dimension of nickel [111] crystallites and microstress. The crystallographic structure of the composite coatings was investigated by TEM. Size of [111] nickel crystllites in the Ni-PTFE coatings varied from 11 to 39 nm. All coatings have approximately random orientation. Decreasing of the crystallite size in the nickel coating influenced on the increase of microstress. Voltammetric method and a three-electrode cell were used for examination of the samples of composite coatings in 0.5 M NaCl, pH 7. Results were used for calculation of the free corrosion potential and current. The corrosion resistance of all of the composite coatings was superior to that of the nickel coating. Non-conducting PTFE particles incorporated into a nickel coating also occur on its surface, which should affect the corrosive behaviour of composite coating. Following dependence was obtained:

$$P = 26.3 * P_{111} + 1.5 * D_{111}$$

The corrosion rate decreases with increasing of the preferred orientation (111) content and with increasing of the dimension of nickel [111] crystallites.

17:20	Poster	A/PII.06

Structural, electronic and chemical properties by simultaneous combination of X-Ray diffraction (XRD) and Hard X-Ray Photoelectron Spectroscopy (HAXPES, up to 15KeV)

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In this contribution we present for the first time simultaneous combination of X-Ray diffraction (XRD) and Hard X-Ray photoelectron spectroscopy (HAXPES, photoelectrons with kinetic energy up to 15 KeV). Thanks to the simultaneous capability to detect the diffracted photons and the ejected photoelectrons, the developed experimental set-up offers a unique opportunity to obtain, on the same sample region and under identical experimental conditions, structural, electronic and chemical properties of the studied systems. Due to the high penetration depth of X-rays and the large escape depth of high energy photoelectrons (15 KeV kinetic energy) surfaces, bulk and buried interfaces are accessible in a non-destructive way. Its implementation at the Spanish CRG beamline (SpLine) at the European synchrotron radiation facility (ESRF) offers an exceptional tool cap-

able to correlate in a direct way composition and structural profiles over a depth of several 10s of nanometers. An ultra-high vacuum set-up, which incorporates an Helium crysotat, a Mini-LEED, a UV-discharge lamp, an ion gun, precision leak valves and a removable sample transfer system, has been constructed that simultaneously fulfils the requirements for HAXPES and XRD. Special effort has been devoted to develop a novel electron analyzer, capable of working at very high as well as low kinetic energies (from few eV up to 15 KeV). The electron analyser can be operated at high spatial magnification, i.e., micrometer sample area, which combined with a micrometer analyser-lens displacement enables the possibility of performing sample microscopy. The first results on metal/metal and metal/semiconductor multilayers will be presented demonstrating the high efficiency of the proposed technique for structural, electronic and chemical properties determination.

17:20 Poster A/PI.06

Residual stresses in TiN-Ni nanocomposite coatings deposited by reactive ion beam assisted deposition

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Often, the enhanced hardness reported for nanostructured coatings is associated with high residual compressive stresses. In this work, we report on the hardness enhancement in TiN-Ni nanocomposite coatings wihtout increase of stress in the coatings. Nanocomposite coatings of nc-TiN/a-Ni were deposited using an ultrahigh vacuum dual ion beam sputtering system from a composite Ti-Ni target sputtered with 1,2 keV Ar^+ ions and the growing film was bombarded during deposition with a mixture of 50 eV Ar^+-N^{2+}-N^+ ions . The residual stress in the coatings was determined using XRD analysis and crystallite group method . The results are compared with the conventional $\sin^2\psi$ and radius of curvature methods. For the films deposited at 300°C the progressive addition of Ni in TiN produced a decrease of coating residual stresses and for films deposited at RT they remain nearly constant. For both temperatures, the hardness increases and exhibits a maximum in the range 5 to 10 at% Ni.

17:20 Poster A/PI.07

Young modulus and adhesion coefficient of Ni+Mo composite layers on metallic substrate

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Nickel-based composite layers electrochemically deposited on steel are known as materials of special properties like very good corrosive resistance or high catalytic activity in hydrogen evolution. Especially interesting are those containing metals like Ti, Mo, W (e.g. Mo plays a role of an activator in the hydrogen electroevolution).
The aim of the paper is to study Young modulus and adhesion coefficient (defined as a fraction of mechanical energy transferred from substrate to layer) for Ni+Mo composite layers electrochemically deposited on stainless steel. The layers (20 and 50 μm) were prepared by electrodeposition of nickel from a nickel bath containing Mo particles. The structural and electrochemical examinations of these kinds of layers were published in [1].
Young modulus E and adhesion coefficient of the composite layers were determined versus temperature (300-600 K, heating rate 3 K/min) using a vibrating reed apparatus. Samples in form of a plate (70x7x0.5 mm3) with the Ni+Mo layer on one side were excited into flexural vibrations with the resonant frequency of about 100 Hz. The measurements of resonant frequency vs. temperature allow determining the Young modulus of the layer material. It was shown that E continuously decreases with temperature and the Ni+Mo layers are thermally stable up to about 600 K. The Young modulus of the layer strongly decreases with increasing Mo content. The relative adhesion coefficient was determined vs. temperature for layers with the same Mo content and different layer thickness.

1. M.Popczyk, J.Kubisztal, A.Budniok, Materials Science Forum, 514-516 (2006) 1182.

17:20 Poster A/PII.07

Stability of a-CN_x:H layers deposited by plasma enhanced CVD

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Diamond-like layers doped with nitrogen (a-CN_x:H) on metallic, ceramic and polymeric substrates receive particular attention as new-generation material for applications in engineering industry, microelectronics and medicine. They are known to be chemically resistant and to have attractive mechanical and electric properties.

The research problem is that the properties of the a-CN_x:H layers may differ in accordance to their composition (nitrogen and hydrogen content) and to atomic/micro-scale structure. Thus, any applications of a-CN_x:H in advanced technologies are conditioned by detailed knowledge of relationships between synthesis conditions and constitution of the layers.

This work reports the results concerning stability and structure of a-CN_x:H layers deposited on Si (001) using plasma enhanced RF CVD method. The series of the layers have been obtained from CH_4 and N_2 gas precursors under modified synthesis conditions. In experiment the following technological parameters have been controlled: CH_4/N_2 ratio, substrate temperature, gas pressure in the chamber and RF plasma power. The studies of the surface morphology, microstructure, chemical composition and the thickness measurements have been performed using typical tools: EDS, SEM and AFM. The atomic-scale structure has been modelled from IR spectra at 1250-4000 cm^{-1}. With application of the obtained results an influence has been evaluated of the technological parameters on the pro-

cessing efficiency, including deposition ratio and quality of the material. It has been confirmed that high temperature may exert destructive effect. It may be partially compensated by increase of either gas pressure or RR power or both. Taking all found relationships as a basis the diagrams of stability of the layers in (p,T), (p, P_{RF}) and (T, P_{RF}) systems have been drawn.

17:20 Poster A/PI.08

Preparation and Properties of Chitosan and Silicon Dioxide Nano-composite Film

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Abstract: Chitosan is a kind of natral polymer with antibacterial activity. In this study, chitosan and silicon dioxide composite films was prepated using Sol-Gel method. The microstructure and mechanical properties of the films was examined. The results disclose the existance of strong hydrogen bond between chitosan and silicon dioxide. The crystal structure of chitosan was disordered by introduction of silicon dioxide and there wasgood compatibility between the two component with silicon dioxide content below 7 percent that appear best michanical properties.

Key words: Sol -Gel method; Chitosan; silicon dioxide

17:20 Poster A/PII.08

Electrochemical production and characterization of Ni-based composite coatings containing Mo powder

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Composite Ni+Mo coatings were obtained by electrodeposition of Ni with Mo particles on a steel substrate from the nickel bath in which metallic powder was suspended by stirring. Deposition was conducted under galvanostatic conditions. Deposits were characterized by the presence of Mo microsize particles embedded into the nanocrystalline nickel matrix. The influence of metal powder amount in the bath, as well as deposition current density on the chemical composition of coatings was investigated. It was stated that the content of incorporated Mo increases with the increase in the amount of metal powder in the bath, and diminishes with the increase in deposition current density. The mechanism of metallic particles embedding was explained on the base of Ni^{2+} ions adsorption process. Incorporation of Mo powder into electrolytic nickel matrix causes an increase in the real surface area of deposits.

Thermal treatment of deposited coatings leads to chemical reactions in the solid state and in a consequence exerts significant influence on their phase composition and surface morphology. As a result of interaction between the nickel matrix and incorporated Mo particles Ni_3Mo intermetallic phase and Ni-Mo solid solution are arising.

The obtained composite coatings were tested as electrode materials for hydrogen evolution in alkaline environment. Electrochemical characterization of the composites was carried out by steady-state polarization method. It was ascertained, that as-deposited Ni+Mo coatings are characterized by enhanced electrochemical activity for this process, which was confirmed by considerable decrease in the hydrogen evolution overpotential, by a nearly 170-260 mV compared to nickel electrode. Thermal treatment decreases electrochemical activity of investigated materials, as the values of hydrogen evolution overpotential on heated coatings are much higher.

17:20 Poster A/PI.09

Magneto-optical Faraday effect in composite films containing semiconductor nanoparticles

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As the size of metal or semiconductor particles decreases to a nanometer scale there can appear new optical and magneto-optical properties. For composite materials consisting of dielectric matrix and the embedded semiconductor nanoparticles the Faraday rotation should depend not only on particle size and shape but also to be a function of interparticle distance. Our previous findings suggest of large enhancement of the Faraday effect for CdMnTe nanoparticles in glass composites when compared to the bulk crystals. This experimental result has to be still confirmed for other systems and for nanoclusters embedded in different dielectric matrices. In this work we present results of magneto-optical studies of composite films containing II-VI based semiconductor nanoparticles prepared by different techniques. The composite films IIMnVI were prepared by growth of the nanoparticles embedded in SiO_2 matrix with using pulsed laser deposition, magnetron sputtering, embedding of semiconductor nanoparticles into polymer matrix and growth by melting of the semiconductor doped fine powder borosilicate glasses. New chemical approach to synthesis of CdMnS nanoparticles in polymer matrix has been proposed. There were found peculiarities in spectral, temperature and magnetic field dependences of the Faraday effect for the studied composite films which can be attributed to the influence of the reduction of dimensionality on spin exchange parameters for this class of materials. Possible application of the studied materials is discussed. The work was supported by grants No. M/128-2004 and No.D3/152-2005 from Ministry of Education and Science of Ukraine.

17:20 Poster A/PII.09

Study of Organic-Inorganic nanocomposite materials and nanostructured metal oxides as efficient adsorbants for water treatment.

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Organic- inorganic nanocomposite materials and nanostructured metal oxides have been used for water purification purposes. By immersing such materials in polluted water, pollutants are adsorbed and retained by the adsorbers. In particular, nanocomposite materials based on a silica backbone covalently connected through urea bridges with poly(oxyalkylene) chains (called Ureasils) are efficiently adsorbers of dyes from aqueous solution. Anionic, cationic as well as hydrophobic dyes are very efficiently adsorbed. Very impressive adsorbing capacity has been registered for anionic dyes bearing a sulfonate group.

In the case of mesoporous oxides, transparent nanocrystalline TiO_2 films were used as dye adsorbers. Their regeneration has been achieved by the photocatalytic treatment of the films under UV and visible light. Possibility of selective sensing of dyes and heavy metal ions adsorbed by TiO_2 nanostructured films from polluted water has also been examined by monitoring the changes of the electrical conductivity of the films.

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17:20 Poster A/PII.10

Aqueous Corrosion Behaviour of Fe-Ni-B Nanostructured Alloys

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Metallic glasses (MGs) based on the Fe-Ni-B system are promising materials regarding the requirements of various industrial applications. The surface structure and composition of ribbons of isoatomic Fe-Ni with varying percentages of B, was investigated by X-Ray Diffraction (XRD), Auger Electron Spectroscopy (AES) and Energy Dispersive X-Ray Spectroscopy (EDS). The resistance of Fe-Ni-B MGs in general and localised corrosion, in various aqueous corrosive environments (3.5% NaCl, 1N HCl, 1N NaOH, 7N NaOH, d. NaOH, 1N HNO_3, 7N HNO_3, d. HNO_3, 1N H_2SO_4), at ambient temperatures, was electrochemically studied. A three electrode corrosion cell (reference electrode: Ag/AgCl (3.5 KCl), counter electrode: Pt) was employed and cyclic potentiodynamic polarization tests were performed.

The surface of the MGs (prior to corrosion testing) was found to be Fe enriched, whereas exposure to 200 eV N^+ and Ar^+ plasma slightly modified the top surface layer possibly inducing localized crystallization. The nanostructured alloys presented very low uniform corrosion rates in 3.5% NaCl, low corrosion rates in 1N NaOH and very high corrosion rates in 1N HNO_3 and 7N HNO_3. They showed a strong passive-pseudopassive behaviour in 1N NaOH, 7N NaOH, d. NaOH, d. HNO_3, and 1N H_2SO_4, whereas they were not found susceptible to pit corrosion. The Fe-Ni-B glasses were not susceptible to pitting in the 3.5% NaCl solution but they yielded localized corrosion when exposed to 1N HCl. The experiments indicated that B has a positive role on the corrosion resistance of the MGs.

17:20 Poster A/PI.10

Optically Transparent Coatings of Al-Si-N for Wear Protection

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Based on the success of metallic nanocomposite hard coatings, we have developed new nanostructured thin films based on the Al-Si-N system combining similar wear resistance with excellent transparency in the visible range of light. The films were deposited by reactive DC magnetron co-sputtering of Al and Si targets in Ar/N_2 at 200°C. They were characterized by XRD, XPS, TEM, UV-Vis spectrometry and nanoindentation. Chemical compositions from pure AlN to Al-Si-N with 30 at.% of Si were investigated. Under conditions of sufficient nitridation, an average optical transparency approaching 100% was obtained. The hardness of these films shows a weak maximum around 25 GPa and a resilience increased by about 60% at silicon concentrations of 12-15 at.%. However, in contrast to known silicon-containing ternary nitrides, this material shows no phase segregation during deposition into *nc*-AlN/*a*-SiN_x, as would be expected from thermodynamical data. XRD analysis proves the presence of h-AlN (wurtzite), with a decreasing lattice parameter and a texture change as silicon is introduced into pure AlN. This suggests the formation of a metastable $Al_{1-x}Si_xN$ solid solution, formed by the substitution of Si atoms on Al sites in the AlN crystalline lattice. TEM images reveal a columnar growth throughout the entire film thickness of about 1 μm. Each column is composed of elongated nanocrystallites whose length decreases down to 25 nm with addition of silicon into AlN. The films are stable upon annealing at 800°C under N_2 for 2h. Above 13 at.% of Si, the coatings are X-ray amorphous, which may be interpreted as the solubility limit of Si in AlN. The observed properties make this material promising for wear protection applications on a variety of surfaces which require optical transparency.

17:20 Poster A/PI.11

Self-organization and optical response of silver nanoparticles dispersed in a dielectric matrix

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Because of their reduced size, the nanostructures have specific properties different from those of bulk materials, which not only depend

on their nature or morphology but also on their spatial arrangement. In particular, the optical spectra of noble metallic nanoclusters embedded in a dielectric matrix (nanocermet thin films) are characterized by an absorption band in the visible spectrum, known as surface plasmon resonance (SPR). In this context, we develop an activity that aims to understand and control the growth of Ag nanoclusters (NCs) dispersed in a Si_3N_4 matrix. The nanocermets are elaborated by dual ion beam sputerring in the form of $(Si_3N_4/Ag)_n$ multilayers deposited on plane oxide surfaces and $Si_3N_4/Ag/Si_3N_4$ trilayers deposited on vicinal oxide surfaces. The morphology and the organization of the Ag NCs are determined by transmission electronic microscopy (TEM) and atomic force microscopy. In parallel spectroscopic ellipsometry and light transmission spectroscopy are used to determine the optical response of the films. First, we show that on a plane surface the Ag NCs are organized in an isotropic way with a short-range order. Then, for multilayer systems, the analysis by autocorrelation of the cross-section TEM images shows that the NCs are organized preferentially in the hollows formed by the intermediate matrix layer and that the degree of order depends on the thickness of this transition layer. In addition, it is possible to use vicinal surfaces of Al_2O_3 to obtain a preferential organization of the NCs along the steps, which results in a shift of the position of the SPRs according to the polarization of the incident electromagnetic field.

17:20 Poster A/PII.11

Amorphous a-Si_xN_y:H layers on silicon substrate for application in solar cells.

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Limited natural reserves and a necessity of environment protection are the reasons of growing interest in renewable power resources,e.g.the sun power.According to these trends the last decades have seen a rapid development of photovoltaics.A promising progress in this area is expected across applying a-SixNy:H layers.They have a number of functions,e.g. they exhibit optimal antireflection parameters,a content of the hydrogen in the layer is expected to ensure effective passivation of the defects present in the substrate.This is why an application of the a-SixNy:H layers in the technology of the solar cells should allow replacing expensive monocrystalline Si plates with much cheaper polycrystalline ones. The research problem is however that the properties of the layers depend on their chemical composition as wel their microstructure and atomic level structure. This means a crucial role of the choice of the proper technology. The most promising technologies of a-SixNy:H materials are based on Plasma-Enhanced Chemical Vapour Deposition (PE CVD).

In this work we present the result concerning details of RF CVD (Radio Frequency Chemical Vapour Deposition) method applied in the technology of the a-SixNy:H layers on polycrystalline Si substrate.The layers have been synthesized from SiH_4 and N_2 gaseous precursors.A special attention has been paid to a choice of the experimental conditions,such as:gas mixture composition, i.e.SiH_4/N_2 ratio and SiH_4/H_2 ratio, heater temperature, gas pressure and the RF plasma power.An influence of the technological parameters on the ratio of the deposition process as well as the quality, chemical composition and atomic-scale structure of the layers has been carefully determined.In the analysis SEM, EDS and FTIR methods have been applied.Finally a role of hydrogen diffusion into the silicon has been analysed and the passivation ability of the layers has been evaluated.

17:20 Poster A/PI.12

Characterization of structural and optical properties of nanopatterned polar thin films

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Nanopatterned $Ba_{0.8}Sr_{0.2}TiO_3$ (BST), $SrBi_2Ta_2O_9$ (SBT), and $NaNbO_3$ (NN) ferroelectric thin films were deposited by the PLD on various substrates $Si/SiO_2/Ti/Pt$, $Si/SrRuO_3$ and Si. Structural properties of the films were analyzed by the RENISHAW micro Raman spectrometer in the temperature range from 80 - 573 K. The surface morphology and piezoresponse were analyzed by the AFM with conventional Si and TiN tips in the contact and taping modes. The Carl Zeiss brand model EVO 50 XVP SEM was used to characterize the local composition and structure of the BST, SBT and NN thin films. The optical measurements were performed by means of a J. A. Woollam spectral ellipsometer operating in rotating analyzer mode. The main ellipsometric angles ψ and Δ were measured in the spectral range from 250 to 1000 nm at the incident angles 65, 70 and 75 degrees. For optical constant calculation, Lorentz, Cauchy, Urbach and other models were used. Surface roughness were also taken into account using effective medium aproximation theory. Dependence of the Raman spectrum, refractive and extinction coefficients, optical band gap energy, and surface roughness on film structure was analyzed.

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17:20 Poster A/PII.12

Electrical characterisation of magnetoresistive sensors based on AMR and GMR effects used for lab-on-a-chip applications

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The magnetoresistive sensors made from Permalloy ($Ni_{80}Fe_{20}$) thin films and Permalloy based multilayered structures are very attractive for detection of low magnetic fields in lab-on-a-chip applications. We exploit the anisotropic magnetoresistance effect (AMR), which appears in these structures, to build a microcompass for detection of

the position and rotation rate of a micro rotor used to study the dynamics of the biological fluids. For this reason, we used the planar Hall effect (PHE) to get direct access to the anisotropic part of the resistance with the advantage of a reduced thermal drift of the output signal. The multilayer structure presents, in addition to the AMR effect, the giant magnetoresistance effect. The samples have circular shape geometry with approximately 5 mm in diameter. The four-lead setup consists of 4 Cu strips forming a square of 4 mm each side. We used a computer controlled measurement system. Because of the contacts misalignment the angular behaviour of the PHE voltage is distorted. To compensate these errors and to increase the sensor sensitivity we used a special setup in which we made, sequentially, two measurements of the PHE for each angle over two orthogonal directions. Also, we performed micromagnetic simulations to discuss the effect of the magnetic field strength on the shape of the angular dependence of PHE. To make these simulations we used complex structures of Permalloy single domains which interact between them and with the applied magnetic field. From experimental measurements and micromagnetic simulations, made on the multilayered structure, results a distortion of the angular dependence of the PHE for low magnetic fields (less than 200 Oe) because the magnetization vector can not follow the direction of the applied magnetic field.

17:20 Poster A/PI.13

Structure and Optical Spectra of Cu Nanoparticles in Silica Matrix

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The copper nanoparticles have been grown in silica matrix by annealing of the matrix impregnated with the copper nitrate. The annealing has been performed in air, successively in air and hydrogen, and in hydrogen. The transmission electron spectroscopy and optical spectroscopy studies of the copper nanoparticles in silica matrix have been performed. Depending on the conditions of annealing, the Cu nanoparticles in size range 2-65 nm are grown. At annealing in air the nanoparticles of the copper oxide (Cu_2O) are grown as well. The copper nanoparticles of two types are grown: spherical large "mature" particles and elliptical small "seed"-particles. The surface plasmon peak has been observed clearly in absorption spectra of Cu nanoparticles. The peak demonstrates slight blue shift with decrease of particle size. The half-width of the surface plasmon peak decreases appreciably at the lowering of temperature from 293 K to 77 and 4.2K that is due to strong electron-phonon interaction. The low-frequency Raman scattering data are in agreement with electron microscopy and absorption data. The photoluminescence from the copper nanoparticles has been observed. It is shown that the efficiency of the luminescence increases appreciably at the decrease of particle size. The observed increase is explained, probably, by the coupling of the excited incoming and outgoing emitted photons with surface plasmon.

17:20 Poster A/PII.13

A low-voltage-operative nanocrystal memory made with high-k control oxide

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Recently, the metal-nanocrystal embedded in gate dielectric has attracted much attention because of its potential for low power, high speed and high density non-volatile memory application. In order to further improve the program/erase speed and reduce the power consumption, high-dielectric-constant (high-*k*) materials were adopted to replace SiO2 as the control oxide in nanocrystal floating gate memory. A higher electric field can be induced across the tunnel oxide by using the high-*k* dielectrics as the control oxide under the same programming voltage. A large hysteresis loop results in the capacitance-voltage (C-V) relation at a low operating voltage. In addition, the gate injection can be avoided due to the lower electric field across the control oxide.

In this work, thermal oxidation and RF magnetron sputtering, sequentially, were used to fabricate MOS capacitors with a layer of Au nanocrystals embedded within a SiO_2/BST ($Ba_{0.5}Sr_{0.5}TiO_3$) stack. A 2.5 or 5-nm-thick dry oxide was grown at 900 °C on *p*-type, 5-10 Ωcm, (100) silicon substrate. Subsequently, a Au ultrathin film were deposited at room temperature and then BST thin film was deposited at 550°C *in situ* by RF magnetron sputtering.

The MOS structure was examined by high resolution transmission electron microscopy (HRTEM) to observe the morphology and distribution of nanocrystals. High frequency C-V measurements were used to investigate the charge trapping and detrapping processes in floating gate metal-oxide-semiconductor memory based on Au nanocrystals.

17:20 Poster A/PII.14

Influence on the precursors nature on the morphology and optical properties of Ru(II) containing thin film used as oxygen sensor

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SiO_2-based thin films with immobilized Ru(II)-tris(4,7-diphenyl-1,10-phenanthrolyne) dichloride/$Ru(dpp)_3$/ have been produced in order to elucidate the influence of the precursors nature and the production method on their morphology as significant factors for the effectiveness and service time of oxygen

sensors.

The morphology of the films deposited in various conditions (varying the number of immersions, withdrawn speed and $Ru(dpp)_3$ content) is studied by scanning and transmission electron microscopy. The specific surface area and the porosity are determined by krypton adsorption.A smooth, glass-like surface is characteristic for the layer, produced from TEOS. The ormosil-type precursors lead to the formation of "structured" surface and the morphology strongly depends on the hydrocarbon chain length. The presence of the Ru(II) complex changes the morphology. The crystals, specific for the complex are seen on the surface of all the produced layers, appearing above the surface but also "immersed" in the matrix volume. The data from X-ray energy-dispersive microanalysis show that the mole ratio Si/Ru in the dots is~24 and ~121 outside them. The X-ray photoelectron spectroscopy results confirm the presence of Ru(IThe influence of storage of the films in distilled water, HCl or HNO_3 solutions (pH 2 and 4), NaOH solution (pH 8) and in beer for 2160 hours (90 days) on the films morphology is studied also. As can be expected the alkali solution disturbs the surface rather significantly.

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17:20 Poster A/PI.15

Color dependency on optical and electronic properties of TiN thin films

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In this work we present in-situ and real-time Spectroscopic Ellipsometry (SE) spectra analysis as a tool for investigation of the relation and the underline physics between visual appearance and the optical and electronic properties of TiN_x nanocrystalline coatings. The films were fabricated employing Reactive Magnetron Sputtering in an unbalanced configuration. We also present a comparison between the electronic properties of these thin films and TiN_x coatings fabricated with balanced magnetron sputtering, which their results have been reported in our previous work. Deposition was carried out at various values of substrate bias voltage and N_2 pressure. Insights on the optical and electronic properties were arisen from the analysis os SE spectra, using the combined Drude-Lorentz model, which describes the optical response of the conduction and valence electrons. The energy, strength and the conduction electron density, were studied with respect to the bias voltage. The observed color variations can be resolved in terms of the Drude-Lorentz model. The onset of the interband transitions is responsible for the color of the nanocoatings and occurs in lower energies than the calculated transition energies. This weak absorption, which is the fundamental interband transition, cannot be experimentally identified and discriminated from the strong, dominating contribution of the intraband absorption of the SE spectra at low photon energy. For the determination of this weak interband transition we used the Tauc-Plot method. Finally, we associated this energy with the screened plasma energy, therefore with the stoichiometry, and with the grain size of the coatings as well.

17:20 Poster A/PII.15

A comparative studies of atomic-layer-deposited Hf-based thin films for high-k gate dielectric applications

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In this work, we use the atomic-layer-deposition (ALD) method to prepare the extra thin Hf-based high-k dielectrics on HF dipped p-type Si substrate. From the typical 100k Hz C-V characteristics and ncsu-CVC model simulation results, we can find that the experimental data matches quite well with the simulation curve which indicates the good quality of the thin films and interface with Si substrate. The equivalent oxide thickness (EOT) of 4.2nm thick HfO2 reaches about 1.39nm with the correction of quantum mechanical effect. On the other hand, the equivalent oxide thickness of the Hf-aluminate (HfAlOx) and HfO2/Al2O3 thin films with the same physical thickness reach about 1.37nm and 1.29nm. The reason for the lower EOT of the HfAlOx and HfO2/Al2O3 thin films comes from more stable interface with Si substrate which is also confirmed from the XPS results. Very weak hysteresis (<20mv) are observed from all the thin films. The leakage current density is around 1.35*10-1□1.55*10-5 and 1.01*10-4 (A/cm2) for Vg at (Vfb-1) volts for the HfO2□HfAlOx and HfO2/Al2O3 thin films. With the incorporate of Al2O3, the crystalline temperature enhances and the less crystalline thin films results in the lower leakage current density. The HfAlOx thin films have less EOT increment with increasing post-deposition temperature than HfO2/Al2O3 thin films which means HfAlOx has the more stable structure than HfO2/Al2O3 on Si substrate. In conclusion, the HfAlOx and HfO2/Al2O3 thin films have more advantages than HfO2 used for high-k MOS applications. The difference between HfAlOx and HfO2/Al2O3 thin films is not so large but still has some interesting results.

17:20 Poster A/PI.16

Template Synthesis of Nanomaterials Inside the Pores of Track-Etched Membranes

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The template synthesis method based on deposition of materials inside well-defined
uniform pores of isoporous membranes offers unique possibilities of manufacturing one-dimensional, high aspect ratio cylindrical species with lateral dimension below 100 nm, practically from any solid material (carbon, metals, metal oxides, polymers) in the form of rods, wires, tubules, multiwall tubules and multilayer rods. Growing interest in template-based synthesis is the result of search for novel methods of manufacturing materials with predefined shapes and dimensions in nanorange, preferentially based on assembling them directly from molecules.

Two types of available commercially templates are commonly used for nanomaterials synthesis - Track-etched polymeric Membranes (TM) and Anodic Alumina Oxide (AAO) membranes. Due to the method of preparation of TM (irradiation of thin polymer films by heavy ion beam following by the chemical etching) the distribution of pores over film area is stochastic. The pore diameters depend on chemical etching conditions; the smallest available diameters are usually of about 50 nm. In contrast AAO membranes obtained by anodic oxidation of aluminium foil in appropriate solution, are characterised by highly ordered, closely packed array of columnar, nearly hexagonal cells. The porosity of these membranes is about 66 %. The diameter of pores can be varied in the range 100-500 nm.
The kinetics of deposition process into pores of nanoporous membrane is still not well-understood. In many experiments on deposition into the pores, an observation has been made that chemical properties of pore walls (chemical groups present on the surface) play an important role in kinetics of the deposition process, in particular, during its initial step. In the present paper the kinetics of deposition of conducting polypyrrole polymer inside TMs is described.

17:20 Poster A/PII.16

Mg-doped $Ba_{0.6}Sr_{0.4}TiO_3$ gate dielectrics for low voltage operating ZnO transparent transistor on polymer substrate

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Transparent ZnO based thin film transistors (TFTs) have received intensive interest due to their potential of replacing hydrogenated amorphous or polycrystalline silicon (a-Si:H or poly-Si) TFTs. Znic oxide (ZnO) is a transparent compound semiconductor with a wide band gap (3.37 eV) which can be grown as a polycrystalline film at low or even room temperature. ZnO is, therefore, considered to be an ideal material for serving as the channel layer in transparent and flexible TFTs. As an important element, gate insulators for ZnO based TFTs have received increasing attention because ZnO based TFTs switching voltage can be reduced by using high-K gate dielectrics which can lead to high capacitance value. In this presentation, we report on the role of Mg doping in conspicuously reducing leakage currents in $Ba_{0.6}Sr_{0.4}TiO_3$ (BST) high-K gate dielectrics utilized in ZnO based TFTs fabricated on plastic substrates (PET). The 3% Mg-doped BST thin films, deposited by rf magnetron sputtering at room temperature on Pt/Ti/SiO_2/Si substrates, showed a relatively high dielectric constants of ~21. The 3% Mg-doped BST films exhibited remarkably improved leakage current densities less than $5x10^{-9}$ A/cm^2, as compared to that ($5x10^{-4}$ A/cm^2) of undoped BST films at an applied electric field of 250 kV/cm,. All room temperature processed ZnO based TFTs using the 3% Mg-doped BST gate insulator demonstrated a high optical transparency (> 80%, for wavelength > 400 nm), a high field effect mobility of 13.2 cm^2/Vs and low voltage device performance of less than 7 V. This result indicates that ZnO based TFTs with 3% Mg-doped BST gate insulator will open up a promising route for a wide variety range of applications in transparent, flexible, and portable electronic devices.

17:20 Poster A/PII.17

The formation of homogenous ultradence arrays of PbTe quantum dots on strained BaF_2 surface

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Nanostructured composite optoelectronic devices based on quantum dots (QD's) of an IV-VI semiconductors are very interesting for various applications. The nucleation and growth of PbTe QD's from vapor phase on BaF_2 (111) substrates strained by an extern load under conditions close to thermodynamic equilibrium have been studied by using atomic force microscopy. The size distribution of the QD's was statistically analyzed as dependent on the character of straining of the substrate. The PbTe dots were grown epitaxially under Volmer-Weber growth mode. The maximum values of density of self-assembled QD's grown on the unstrained BaF_2 (111) substrates did not exceed $(6 \div 8)\cdot 10^{10}$ sm^{-2}. The size distribution of this dots is quite broad (standard deviation ~ 20%-50%). Ultradence (above 10_{11} sm^{-2}) arrays of faceted PbTe QD's with a average height ~ 3,5 nm (8% - 9% std) are obtained on strained BaF_2 (111) substrates under conditions of small vapor supersaturation in the condensation zone. Its uniformity and high QD's density is based on the formation of self-assembled QD's on the bent (stretched) BaF_2 (111) surface, where the effect of localization of the plastic deformation was observed on the nanometer length scale. This is due to the self-organization of the dynamic dissipative system of an interacting dislocations in a certain range of temperatures and stresses. We find that a lateral periodicity along the direction <110> amounts to 300 Å due to the heterogeneous nucleation of QD's at straight surface slip steps and anisotropic surface migration of the adsorbed PbTe molecules. The influence of straining on the kinetic processes on the substrate surface was investigated.

17:20 Poster A/PI.18

Al-Fe-Cu surface layers obtained by PLD technique

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In the present paper the morphology and microstructure of Al-Fe-Cu surface layer are presented. Al-Cu-Fe alloys has low electrical conductivity, low surface energy and coefficient of friction, high hardness and brittleness at room temperature. Al-Fe-Cu alloys are suitable for several applications, for.ex. wear resistant coatings, thermal barrier coatings. The laser ablation was performed using Nd:YAG pulsed laser (355 nm emission wavelength, 10 ns pulse duration, 10 Hz repetition rate). The higest laser fluences was 10 J/cm^2. The SEM, EDS, AFM and RTG techniques are used for characterise the

surface layers obtained.

17:20 Poster A/PII.18

Room temperature fabricated ZnO thin film transistors with MgO coated $Bi_{1.5}Zn_{1.0}Nb_{1.5}O_7$ gate insulator

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ZnO is a transparent compound semiconductor with a wide band gap (3.37 eV) which can be grown as a polycrystalline thin film at room temperature. ZnO thin films, therefore, have received intensive interest for applications as a channel layer in transparent and flexible thin film transistors (TFTs). But, the high operating voltage of the ZnO based TFTs fabricated at a reduced temperature hinder its application in portable devices. The operating voltage can be reduced by using high dielectric constant materials as gate insulators. In this presentation, we report on dielectric and leakage current characteristics of room temperature deposited $Bi_{1.5}Zn_{1.0}Nb_{1.5}O_7$ (BZN) films on the basis of crystallographic structure and suitability of MgO coated BZN gate insulators as key building blocks in the fabrication of low voltage operating ZnO based thin-film transistors (TFTs). The MgO coated BZN gate insulator exhibited relatively high dielectric constant of 32 and low leakage current densities below 10^{-8} A/cm^2 at an applied voltage of 5 V, which was an operating voltage of our TFTs. All room temperature processed ZnO based TFTs with MgO coated BZN gate dielectricshowed good saturation characteristics and low operation voltages (< 5 V) due to high dielectric constant (> 30) of MgO coated BZN gate dielectric. The field effect mobility and the on-off ratio were 5.5 cm^2/Vs and 5×10^5, respectively. The threshold voltage and subthreshold swing were 2 V and 0.35 V/dec, respectively. The room temperature processing, low operation voltage and high mobility of ZnO based TFTs with MgO coated gate insulator offer a promising route for the development of transparent and flexible electronics.

17:20 Poster A/PI.19

Composite nanofibers formed at low temperature by plasma deposition

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Ag-TiO_2 nanofibers (three μm long, 30-150 nm thick) constituted by a single-crystalline silver wire (20-30 nm thickness) and an external layer of amorphous TiO_2 are prepared at 403 K by oxygen plasma activation of a silver substrate followed by plasma deposition of TiO_2. Thicker fibers of crystalline anatase around the silver wire were prepared when plasma deposition occurs at 523 K. The formation mechanism of these micro and nano-fibers supported on the substrate is discussed and a nobel volcano-type mechanism proposed for the formation of these composite nanofibers. The effect of plasma and the high mobility of the silver are key factors determining the formation of the fibers. Preliminary results on the formation of nanofibers of other oxides (SiO_2, ZnO) by plasma deposition are shown and their formation mechanism discussed.

17:20 Poster A/PII.19

Microstructure and micro mechanical properties of multilayer Cr/CrN, PLD coatings

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The CrN shows superior high temperature corrosion resistance over TiN. The paper describes investigation of possibilities of improving mechanical properties of wear resistant CrN coatings through their regular slicing with increasing number of Cr layers. It was realized by deposition of the Cr/CrN coatings built of 2, 4, 8, 16, 32 and 64 Cr/CrN bi-layers using reactive Pulsed Laser Deposition (PLD) technique. The application of transmission electron microscopy helped to determine coatings bi-layer super-lattice thickness at 1000 ,500 ,250 ,125 and 62 falling with increasing number of deposited bi-layers. The columnar microstructure for multi-layered Cr/CrN coatings was determined. The inter-crystallite defect density in multilayered coatings was otherwise comparable with that in CrN monolayer coating. The X-ray diffractometry measurements confirmed that the multilayer coatings are retaining main texture parameters of CrN once. The micro-mechanical properties were assessed by microhardness measurements and scratch test using Rockwell penetrator of 200mm diameter loaded from 0.03N up to 20N and finally by measuring their wear resistance through ball on disc test. The performed measurements indicated that switching from single layer to multi-layered metal/nitride coatings resulted in small hardness drop from 17 to 10 GPa. Simultaneously the it was noted that the scratch resistance rose from 4.4N for CrN mono-layer to a maximum of 7.7N for Cr/CrN- 8 layered one. The wear test showed advantage of multilayer coatings over mono-layer CrN as the 35% lowest wear was observed for Cr/CrN-8 layers. The present investigation confirmed that a significant improvement of mechanical properties of CrN type coatings by their sandwiching with Cr layers might be achieved.

17:20 Poster A/PI.20

Annealing ambient effect of the phase formation in the Ni(10nm)/C(2nm)/Si(001) thin film system

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Annealing ambient effect on the phase formation in the Ni(10nm)/C(2nm)/Si(001) thin film system obtained by magnetron sputtering was under investigation. Specimens were annealed for 30 sec in the vacuum at $1.3*10^{-4}$Pa and in the flow of nitrogen in the temperature range 400-1000°C. Temperature stimulated solid-state reactions that occur as the result of interdiffusion processes between layers under investigations were examined by metods of x-ray and electron diffraction, resistivity measurements and Rutherford backscattering. It was established that an annealing ambient influences on the development of the solid-state reactions in the Ni(10nm)/C(2nm)/Si(001) thin film system. The sublayer of carbon and oxygen of a residual atmosphere are slow down solid-state reactions of the formation silicide phases of the nickel. Result of it is displaced up the area of existence NiSi to 950°C.

17:20 Poster A/PII.20

Self-organized nanoscale multilayer growth during the deposition of hyperthermal species

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The quasi-simultaneous deposition of low energy mass selected C^+ and either Au^+ or Fe^+ ions resulted in the formation of alternately metal-rich and metal-deficient layers in an amorphous carbon matrix with periods in the nm range. Transmission electron microscopy reveals that the metal-rich layers consist of rather densely distributed crystalline particles while the metal-deficient layers are amorphous or contain only smaller numbers of crystalline clusters. The concentration variation is confirmed by Rutherford backscattering spectroscopy and Auger electron spectroscopy depth profiling. A similar structure was found in films grown by reactive dc-magnetron sputtering of Cu, Pt, and Ni targets in an Ar/CH_4 plasma. Also films deposited with reactive rf-magnetron sputtering of Fe targets in an Ar/CH_4 plasma show hints of a layered structure. The processes during mass selected ion beam deposition and magnetron sputtering deposition are far from thermodynamical equilibrium. Therefore, the formation of such periodic concentration variations cannot be attributed to mechanisms like Liesegang pattern formation. The multilayer formation can be described by an interplay of sputtering, surface segregation, ion induced diffusion, and the stability of small clusters against ion bombardment.

17:20 Poster A/PI.21

Non-monotonous surface morphology of YSZ thin films deposited by e-beam technique: experiment and modeling

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In present study, YSZ thin films were grown on the Alloy-600 and optical quarts substrates by e-beam deposition. By changing deposition time and e-beam gun power (from 0.48 to 1.2 kW) the growth mechanism of YSZ thin films were investigated. YSZ thin film structure and surface morphology were investigated by X-ray diffraction, scanning electron microscopy (SEM), and atomic force microscopy (AFM). It was found that electron gun power and substrate temperature has influence influencing the thin film deposition mechanism. The structure of deposited YSZ thin films on the optical quartz is tetragonal (101). Experimental results show that surface roughness of thin films pass through the minimum with the change in substrate temperature in interval from 200°C to 600°C and e-beam gun power in interval from 0.75kW to 1.05kW. The aim of this work is to explain and analyze this non-monotonous behavior of surface roughness by proposed kinetic model. The model is based on rate equations and includes processes of surface diffusion of adatoms, nucleation, growth and coalescence of islands in the case of thin films growth in Volmer-Weber mode. It is shown by modeling that non-monotonous dependence of surface roughness on the factors influencing energy of adatoms (e.g. temperature, assisted beam irradiation, accelerating voltage) occurs as a result of interplay between diffusion length of adatoms and size of islands, because both parameters depend on energy of adatoms. Variation of island size and diffusion length results in atomic jumps from islands forming rougher or smoother surface. The functions of surface roughness, island size, island density on diffusion length of adatoms and on other parameters are calculated and analyzed in this work.

17:20 Poster A/PII.21

Structure, Hardness and Thermal Stability of Electrodeposited Cu/Ni Nanostructured Multilayers

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Nanostructured materials like CMA (compositionally modulated alloys) multilayers with bilayer period thickness less than 100 nm have very interesting properties, which are unattainable in bulk materials. The primary interest is in magneto-electrical properties

(GMR effect) with the secondary goal of improving the tribological and mechanical properties of the surface.

Cu/Ni multilayers have been grown by electrodeposition from single solution with potentiostatic control. Several Cu/Ni samples deposited on (001)-oriented Si and polycrystalline cooper plates with different sublayer thickness in the range of 1 to 20 nm were investigated. One series of the multilayers with the constant bilayer thickness and two other series with constant Cu or Ni sublayer thickness (with varying Ni or Cu sublayer thickness respectively) were electrodeposited on cooper substrates. Some Cu/Ni multilayers were annealed in a vacuum furnace at temperature increasing from 150

The X-ray diffraction investigations and SEM observations were used to analyse the modulated structure and determine the bilayer thickness of the nanomultilayers.The SEM and XRD investigations confirmed the layered structure of all deposited samples. The multilayered Cu/Ni coatings exhibit the bigger hardness than single Ni and Cu layers when the thickness of the bilayers is approximately a few nanometers. A maximum of the hardness was measured when the bilayer thickness was 10 nm and was about 25 % bigger than the Ni single layer hardness. Subsequent anneals led to a significant change in the preferential crystallographic orientation, quality of modulation structure and size of crystallites.

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17:20 Poster A/PI.22

Coverage dependent reaction of yttrium on silicon

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In this work, the reaction of yttrium (Y) on Si(001) is investigated using in-situ X-ray photoelectron spectroscopy (XPS) for different coverage of Y with low temperature annealing. We report on the 3 general reaction phases of yttrium on Si that are coverage dependent. Firstly, for 1ML of Y coverage, a strong Y-Si bonding is formed at room temperature. The strong bonding could be seen from the significant dipole induced band bending effects evidenced by Si2p and Y3d core-level binding energy shift. The bonding stability is manifest itself as being resistant to both oxidation in ultra-high-vacuum (UHV) or any further Si interdiffusion after a 300ºC annealing. For higher coverage of 2-4ML, spontaneous room-temperature mixing of Y-Si is observed. This is consistent with most rare-earth metal's reaction with Si at these coverage regimes. Upon a 300ºC annealing in UHV, selective oxidation of Y in the 2-4ML of film is observed while no significant diffusion of Si occurs. Finally, for coverage of >4ML, pure metallic Y forms during room temperature deposition. Subsequent annealing at 300ºC shows substantial Si-diffusion accompanied by a reduction in the intake of oxygen. The diffusion is attributed to a metal-weakened Si bonding effect and the competition of Si with O for the metallic Y limits the amount of oxygen intake in UHV.

17:20 Poster A/PI.23

Pulsed laser deposition of nanostructured tungsten oxide thin films for optical gas sensor applications

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Nanostructured ungsten oxide thin films were deposited by pulsed laser deposition (PLD) (Nd: YAG, λ=1064 nm) for optical hydrogen gas sensor applications. Deposited was performed onto heated glass substrate kept at 400 °C in HV (5×10-5Torr) and 100mTorr oxygen partial pressure. Deposited films in HV showed blue color with 3.1 eV optical band gap and amorphous structure. Samples turned to transparent films with band gap of 3.0 eV and crystalline structure after annealing in air at 400 °C. Films that were deposited in oxygen partial pressure were amorphous and transparent with 3.3eV band gap and changed to dark blue after annealing in vacuum at 400 °C. The structure of these films remained amorphous after annealing and the band gap of these films was reduced to 2.7eV due to creation of oxygen vacancy. To observe hydrogen gas sensing properties a very thin layer of palladium was coated by electro-less method. Optical switching effect due to hydrogen exposure was observed only for samples deposited in oxygen environment. XPS data of the samples present W6+/W5+ ratio higher than those deposited in vacuum. Regarding to Atomic Force Microscopy (AFM) data, deposited samples in oxygen environment showed RMS surface roughness of 13.6 nm and grain boundary of 60 nm while those prepared in HV showed smother surface (2.87nm) and grain boundary of 150nm. Higher surface roughness and lower oxygen vacancy of the films prepared at oxygen partial pressure made it suitable for gas sensing response.

Keywords: *tungsten oxide, pulsed laser deposition (PLD), gasochromic, hydrogen sensors.*

17:20 Poster A/PII.23

SIMS depth profiling of thin nitride- and carbide-based films for hard coating

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Many new materials have been developed for material coating films. Recent investigations show that Ti/Cr-nitride and Ti/Cr-carbide

based complex layers provide corrosion-protection of metals and improve mechanical properties. Secondary ion mass spectrometry (SIMS) technique has proven to be an essential tool for depth profiling of such thin coating films and offers high in-depth precision only if ultra-low impact energy is used for sputtering. However the quantitative depth profiling of such materials is not straightforward in presence of surface contaminations and matrix effects.

This is the case of multilayered TiN/CrN, CrC/CrN and CrC/CrN and TiC:Si, TiCrN films (10 nm - 3000 nm thickness range) created on steel substrates using various deposition methods like condensation from a plasma phase in nitrogen atmosphere, reactive magnetron sputtering and reactive arc deposition, respectively. Additionally TiC films were Si implanted.

The aim of this work is to perform distribution of elements in the films and to obtain chemical characterisation allowing description of nitridation and carbonisation mechanisms to monitor the coating production. In order to achieve a more precise quantification we have been monitored the multilayered films using ultra-low energy of primary ion beam (below 1 keV).

The measurements have been performed using SAJW-05 instrument with Ar ion beam of about 100 micrometers in diameter. Auger investigation of the structures provides additional information on quantitative distribution of main components. SIMS depth profiles of these films show higher oxygen contamination in the case of TiN layers than in CrN, while higher nitrogen concentration was recorded in CrN films. The silicon profile implanted into TiC layer indicates that the implantation range is about 600 nm. Results of depth profiles have been compared to process parameters of coating technology.

17:20	Poster	A/PI.24

AC transport properties of Nanocrystalline $CsPbBr_3$

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$CsPbBr_3$ produced by co-evaporating CsBr and $PbBr_2$ purified powders are attracting much interest for their interesting optical characteristics. Indeed, nanocrystallites are produced in the thin films as evidenced by optical spectroscopy and X-ray diffraction. These nanoaggregates are wide-gap semiconductors with direct band-to-band transition. Identification of the nanocrystallites in the film is based on a similarity of the observed spectroscopic properties with those of the bulk $CsPbBr_3$ purified by the Bridgman technique. The materials object of this study have been deposited by PVD inside an UHV apparatus. These materials show an exciton absorption also at RT. The FWHM of the exciton peak has been used to evaluate, together with X-ray analysis, the quality of the deposited material. The paper reports on the AC transport properties investigated by admittance spectroscopy techniques in a wide frequency and temperature range aimed to identify the role of the surface located defects. AC electrical measurements have been carried out in the 0.1 Hz-10 kHz range by using a Solartron 1250 FRA equipped with a 1296A dielectric interface, whereas a HP4192A LF impedance analyzer has been used in the 10 kHz-10 MHz range. Results show as the Fermi level is pinned by the surface states which induce an electron accumulation layer at the crystallites' surface influencing the current transport at lower and intermediate temperature while, at the higher, an activate transport mechanism is found. The possibility to apply the time-temperature superposition principle gives the opportunity to present and discuss the results as a function of the f and T on the basis of master curves obtained by normalizing f to the most probable hopping frequency.

17:20	Poster	A/PII.24

Metastable Phase and Structural Transitions of Superthin Ti/Si and TiN/SiN Multilayers

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Composite structures based on Ti - Si and TiN_x - SiN_x materials are interesting for photonic, optoelectronic and sensor techniques. The TiN_x - SiN_x multilayers were deposited on Si(100)-wafers by laser ablation of the Ti and Si targets in ambient nitrogen gas in range of pressures ranging from $3x10^{-3}$ to $5x10^{-3}$ mbar. The layer thickness and composition was controlled by parameters of the excimer laser pulses (wavelength at 193 nm), ambient gas species and gas pressure.

Processing of Ti - Si and TiN_x - SiN_x multilayers at high temperature (HT) lead to uniform metastable phase of composite films. The effect of HT treatment (typically for 10 min by rapid thermal process, RTP) at 870 K on structural transformation in multilayers were investigated by X-ray reflectrometry (XRR), X-ray diffractometry (XRD), Scanning Electron Microscopy (SEM) and ellipsometric measurements. Morphology of samples was characterised by atomic force microscopy (AFM).

The Ti - Si multilayers show the superlattice with bilayer period Λ = 31 nm and Si thickness t_{Si} = 2.9 nm. Due to the very thin layers, the interfacial and surface energies dominate both the bulk and the strain-energy terms, which encourages the formation of metastable phase with a low interfacial energy. Then the inter facial energy term becomes less important and bulk formation energies dominate. These energetic arguments can only give a preference for phase formation and are most valid when growth occurs at near-equilibrium conditions.

17:20 Poster A/PI.25

Electronic properties of graphite-like ion tracks in insulating tetrahedral amorphous carbon

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We investigated the formation of quasi one-dimensional conducting filaments in diamond-like carbon (DLC) films by swift heavy ion irradiation. Various DLC films with thicknesses of several 100 nm were grown using mass separated ion beam deposition on highly conducting Si and Ni substrates. After deposition the films were irradiated with 1 GeV ^{238}U ions with fluences between 10^9 and 10^{11} ions/cm^2. Due to their high electronic energy loss of about 30 keV/nm the swift heavy ions graphitize the predominantly (80%) sp^3-bound carbon film along their trajectories yielding conducting nanowires embedded in an insulating matrix. Using atomic force microscopy (AFM) with conducting cantilevers and applied bias voltage the presence of conducting tracks was confirmed and their conductivities were determined to be several orders of magnitude higher than of the host matrix. Temperature dependent electrical measurements were performed on the irradiated samples at 300 K - 10 K with fields up to 5 V/µm. We will discuss the results with respect to contact resistances and possible one-dimensional conduction mechanisms within the tracks.

17:20 Poster A/PII.25

Multilayer coatings of TiNiNb shape memory alloys

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TiNiNb shape memory alloy has been used extensively as a material for medical implant devices due to its shape memory effect, superelasticity and biocompatibility. Due to its relatively high amounts of nickel it is not adequate for implants and prosthesis. Therefore, in the last years, the PVD coating methods have become a good choice to prevent the nickel release from substrate composition, as the obtained protective films may present high protection against corrosion and good wear properties.

In this paper, samples of TiNiNb alloy were coated with two types of multilayer coatings (TiN/NbN and TiC/NbC) in order to improve their corrosion resistance and drop the nickel release in artificial physiological solution, without sacrificing their shape memory effect and superelasticity. The Ni ions release of the coated and uncoated samples was investigated. The films were deposited onto TiNiNb alloy by vacuum arc deposition technique under various deposition conditions. In order to have a more complete characterization of the investigated coatings, other properties such as elemental and phase composition, morphology, texture, microhardness and adhesion were investigated. The corrosion tests revealed that all the coated samples were characterized by very low anodic currents in the passive region and by an exceedingly low metal ion release rate. The obtained results suggest that nanostructured multilayered coatings based on Ti and Nb nitride and carbide obtained by vacuum arc deposition can improve the performance of TiNiNb alloy for biomedical applications.

17:20 Poster A/PI.26

Electrochemical and Electrochromic Properties of Layer-by-Layer Films from TiO_2 and Poly(vinyl Sulfonic Acid)

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Self-assembled electrodes formed from TiO_2 nanoparticles and poly(vinyl sulfonic acid) (PVS) were prepared using the Layer-by-Layer (LbL) technique. The thickness of the 5-, 10-, 15-, and 20-bilayer films was 58 nm, 92 nm, 174 nm and 296 nm, respectively. The electrochemical and chromogenic properties of LbL films of TiO_2/PVS have been examined in an electrolytic solution of 0,5 M $LiClO_4$/propylene carbonate. It is noted the presence of two sites during the positive potential scan, which were attributed to ionic trapping effects. The absorbance change associated to oxidation of trapping TiO_2 sites is attributed to electronic transitions involving energy states in gap band, which are formed due to strong distortion of the $Li_{0.5}TiO_2$ sites. Using the Quadratic Logistic Equation (QLE), it was possible to analyze the electronic transfer as a function of the number of lithium ions intercalated into the LbL film. From the parameters obtained from the fitting, it was also possible to determine the amount of trapping sites produced during the insertion of lithium ions. The electrochemical impedance spectroscopy (EIS) data show the presence of diffusion and trapping sites. The diffusion coefficient of lithium ions changed from $1.2x10^{-10}$ cm^2.s^{-1} to $3.1x10^{-11}$ cm^2.s^{-1} during the discharge process. Finally, the trapping effects associated to the amount of TiO_2 nanoparticles are discussed.

17:20 Poster A/PII.26

Stress and related mechanical properties of multilayer coatings deposited by vacuum arc method

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The multilayer coatings composed of different thin films with thicknesses in the nanometre range (1-20 nm), known as super lattices, have received considerable attention due to their increased hardness, wear and corrosion resistance. As it is known besides its composition and structure, the properties of a coating are also related to the induced stress during growth. The aim of this paper is to investigate - by computer simulation and experimental approach - the variation of the stress in different transitional metals nitrides MeN (Me=Ti,Zr,Nb) and in the related multilayered structures as Me_1N/Me_2N (e.g. TiN/NbN). We have performed finite element method simulation in order to observe the behaviour of the multilayered material under intrinsic stress and in order to achieve the response of the structure under different varying loads. The mesh was build with shell elements and we have used the initial stress commands to apply the right amount of stress into the layers. The simulations were performed for different thickness values of the individual layers in the superlattice structure. Films with a total thickness of 2 microns were deposited by the cathodic arc method on Si and HSS substrates; the stress was determined by XRD. The bilayer period was 500 nm for MeN/Me multilayered coatings and 50 nm, respectively 10 nm for Me_1N/Me_2N multilayer. Both theoretical and experimental results showed the decrease of the stress in the multilayered coatings. These results were also correlated with the observed increased microhardness, adhesion onto the substrate and the corrosion resistance of the deposited films.

17:20 Poster A/PI.27

wetting behaviour under light irradiation of Ta_2O_5 thin films

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Ta_2O_5 thin films have been prepared by evaporation from a tantalum oxide precursor and by assisting the growth of the films by oxygen and nitrogen ion bombardment (IBAD). The obtained thin films depict different microstructures and optical and dielectric properties. In general, the films prepared by evaporation have lower refraction indices that those prepared by IBAD. In this case a clear correlation exists between the ion current and energy and the properties of the films. The trend of dielectric properties of these thin films agrees with that defined by their optical properties. They also agree with the microstructrue of the films as determined by SEM. A particular type of films was found when prepared by evaporation under grazing conditions. These films depict a columnar microstructure with oblique columns and an extremely high porosity. Refraction indices ranging from 1.7 to 1.2 have been obtained for these thin films.

Another interesting property of these thin films refers to its wetting behaviour under UV light. In literature there are many papers dealing with the conversion of the TiO_2 surface from partially hydrophobic into fully hydrophilic by UV irradiation. In this work we also show that Ta_2O_5 thin films present a similar behaviour changing from hydrophobic into hydrophilic by illumination. A careful study of this type of transformation is being carried out as a function of the microstructural and structural properties of the films. Doping of Ta_2O_5 thin films with foreign cations and with nitrogen has revealed to be an effective way to induce a partial change in wetting angle by using visible light for the illumination.

17:20 Poster A/PII.27

Characterisation of nanostructured NbN/TiAlN multilayers deposited by cathodic arc method

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While Nb and Ti are well known as biocompatible materials, the use of NbN coating is reduced, regardless its higher microhardness, due to its increased brittleness compared to TiN or TiAlN films.

The objective of the current work was to take advantage of the known biocompatibility of Nb and Ti layers, by depositing on stainless steel a new superlattice coating consisting of alternate multilayer NbN/TiAlN. Different nanostructured NbN/TiAlN multilayer films, with various bilayer periods (4 ¸ 80 nm), were deposited on 316L stainless steel specimens by the cathodic arc method. The influence of bilayer period of this superlattice coating on the wear behavior in artificial physiological solution was investigated. The coated samples were investigated with respect to elemental (AES, XPS), phase composition and texture (XRD), Vickers microhardness, thickness, adhesion and wear resistance. It was found that the coatings' properties depended both on the bilayer period and on the elemental composition on the individual layers. As compared with the NbN and TiAlN monolayers, the multilayered structures led to an increase of the microhardness with about 3 ÷ 5 GPa. While all coatings had a good adhesion, the highest adhesion, as well as the highest wear resistance were obtained for films with bilayer period in the nanometer range.

17:20 Poster A/PI.28

Synthesis and magnetic properties of Nickel-Iron oxide nanostructures in zeolites

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One-dimensional (1D) nickel iron oxide nanostructures were grown in zeolite-4A, $Na_{12}[Al_{12}Si_{12}O_{48}]$ by a chemical precipitation method from aqueous solutions. Aqueous solutions of nickel sulphate and iron chloride in appropriate volumetric amounts, were used as the starting materials in the synthesis of $NiFe_2O_4$ nanostructures. Precipitation of the desired 1D nanostructures in zeolite-4A,were then achieved by the technique of acid-base titration. Crystal chemical aspects of the resulting nanostructure were studied by Rietveld analysis, as a function of the amounts of Ni present in the crystal structures and the heating temperature (1350 to 1400°C). Qualitative chemical analysis of the samples was performed by EDXS. The microstructure and morphology of the 1D nanostructure composite were characterized by XRD and TEM. All these results indicate that the 1D-$NiFe_2O_4$-nanostructures were synthesized in Zeolite-4A, $Na_{12}[Al_{12}Si_{12}O_{48}]$. The final $NiFe_2O_4$ nanoparticles exhibit special magnetic properties with a remnant magnetization of 6.3 emu/g, coercivity of 66 Oe and saturation magnetization of 38.8 emu/g. properties with a remnant magnetization of 6.3 emu/g, coercivity of 66 Oe and saturation magnetization of 38.8 emu/g.

17:20 Poster A/PII.28

Application of position-sensitive silicon strip detector for X-ray diffraction of thin films and multilayers

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The silicon strip detector represents a new class of one-dimensional position-sensitive single photon counting devices. It allows us to reduce time in XRD measurements of thin films compared to instruments with a single proportional counter. Our detector was mounted at the Philips X'Pert diffractometer with 230 mm instrument radius. Experiments were performed with use of Ni-filtered Cu radiation.

The angular resolution about 0.04^o at low angles was demonstrated for classical Bragg-Brentano geometry measurements of standard reference material (SRM 660) LaB_6. Obtained values were very close to the results achievable for classical proportional counter and X'Celerator.

θ-2θ-scan, w-scan and 2θ-scan of spin valve multilayers were performed by means of the strip detector and the proportional counter. Collected diffractograms showed comparable characteristic features and numbers of counts for acquisition time of the strip detector by two orders of magnitude shorter compared to the proportional counter.

θ-2θ and w-scan of spin valves with the stack composition: substrate Si(100)/SiO_x 47 nm/ buffer layers /IrMn 12 nm/CoFe 15 nm/Al-O 1.4 nm/NiFe 3 nm/Ta 5 nm, with buffers: a - Cu 25 nm, b - Ta 5 nm/ Cu 25 nm, c - Ta 5 nm/Cu 25 nm/Ta 5 nm/Cu 5 nm and d - Ta 5 nm/ Cu 25nm/Ta 5 nm /NiFe 2 nm/Cu 5 nm allowed us to determine the degree of texture of the samples and its dependence on the buffer features.

θ-2θ profiles of the Ni/Au - glass/[Ni/Au]×15 periodic superlattice and non-periodic multilayers: substrate Si(100)/SiO_x 500 nm/Ta 5 nm/Co 5 nm/spacer/IrMn 10 nm/ Ta 5 nm with different spacers of platinum 0, 0.1, 0.2, 0.3, 0.4, 0.6, 0.8, and 1.2 nm were interpreted using the model of non-ideal supperlattice structure based on the Monte Carlo simulation. Interplanar distances in the growth direction of layers were determined.

17:20 Poster A/PI.29

Effect of O_2 partial pressure on magnetic properties of Co-Fe-Hf-O nanocrystalline thin films

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Amorphous alloys of CoFe-rich magnetic amorphous films are well known as typical soft magnetic alloys. They are used for many kinds of electric and electronic parts such as magnetic recording heads, transformers and inductors. In order to get superior soft magnetic properties of the CoFe-based nanocrystalline thin films, the Co-Fe-Hf-O nanocrystalline thin films have been investigated. The soft magnetic properties and electrical property of these films show a dependence on the partial pressure of reactive gases, which presumably changes the microstructure of the films and related magnetic anisotropy. After optimum of pressure O_2, these films exhibit excellent soft-magnetic properties: saturation magnetization ($4\pi M_s$) of 29 kG, magnetic coercivity (H_c) of 0.02-1Oe, anisotropy field (H_k) of 50-70 Oe, and an electrical property is also shown to be as high as 900 mWcm. The combination of high $4\pi M_s$ and relatively high H_k in these films are believed to be partly responsible for the excellent ultra-high-frequency behavior.

17:20 Poster A/PI.30

Influence of reactive gases on the properties of CoFeZr-Alumina nanocomposites

Yury E. Kalinin[2], Alexander V. Sitnikov[2], Julia A. Fedotova[1], Alexander K. Fedotov[1]

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The influence of gas mixture during ion-beam sputtering on electric

and properties of the films containing CoFeZr clusters embedded into alumina matrix has been investigated. The films with the fraction *x* of metallic alloy between 30 and 65 at.% were sputtered in the chamber evacuated either with pure Ar or in gas mixtures Ar + O_2 and Ar + N_2. It was revealed by Mössbauer spectroscopy, permeability, MFM and magnetization measurements that magnetic state of metallic nanoparticles at 300 K strongly depends on gas mixture in the sputtering chamber. In the samples deposited in pure argon CoFeZr nanoparticles displayed ferromagnetic state beyond the percolation threshold *X*c = 40-45 at.% whereas at sputtering in Ar + O_2 or Ar + N_2 the *X*c shifted to 50-55 at.% and the region of superparamagnetic state was expanded far beyond *X*c. The last resulted in non-hysteresis behavior of magnetization curves and lowering of real and imaginary parts of magnetic permeability (m1 and m2 correspondingly) far beyond *X*c in comparison with the films sputtered in Ar. Mössbauer spectroscopy have shown that such peculiarities were due to oxidation of nanoparticles preventing magnetic interaction and the direct electric contact between them. Note also that impedance of the samples studied displayed also appreciable inductive contribution at intermediate frequences 1-100 kHz.

Wednesday, 6 September

Poster Session 2/ Poster Award Ceremony

(continuation of Monday's Poster Session),Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium B

Welcome

Particle size is crucial in catalysis - this statement had been known much earlier than the term of *nano* became trendy. Heterogeneous catalysis is connected with the surface effect and active surface is the function of particles' size. Some nanocatalysts are much more active than their coarse-grained counterparts, because of electronic effects playing important role at nano-dimensions. It has been recently shown that gold, previously considered as inactive, is nowadays proved to be a very effective nanocatalyst. In some cases of supported catalysts the size of support particles can also be important.

The growing demand for fine chemicals industries and increasingly stringent ecological standards require new technologies as well as new, more effective and more selective catalysts. To satisfy this demand the current combinatorial techniques enabling an optimisation of elemental composition are not sufficient and much more scientific effort would be required.

The foreseeable future for nanocatalysts is very promising: the worldwide market is foreseen to increase from $3.7 bn in 2004 to $5 bn in 2009.

All the people interested in contribution to this growth and to the development of knowledge in catalysis are cordially invited to participate in this symposium devoted to nanocatalysts.

The main topics of the symposium related to nanocatalysts will be:

- Catalysis by nanostructured materials: the gap between expectation and reality
- Theory of catalytic processes
- Kinetics and mechanism of catalytic reactions
- Effect of catalyst particle size on the activity
- Geometric and electronic effects in catalysis
- New catalytic processes
- New methods of catalyst characterisation
- Quantum chemical and molecular mechanics modelling of surface phenomena
- Nanocatalysts for environmental protection
- Nanocatalysts for fuel cells
- Nanocatalysts for hydrogen storage
- Biocatalysis
- Photocatalysis
- Zeolite catalysts

Scientific Committee:

- Walerian Arabczyk, Szczecin University of Technology, Poland
- Mohammed M. Bettahar, Université Henri Poincaré, Nancy I , France
- Avelino Corma, Universidad Politecnica de Valencia, Spain
- Jerzy Haber, Institute of Catalysis and Surface Chemistry PAS, Poland
- **Barbara Grzybowska-Świerkosz,** Institute of Catalysis and Surface Chemistry PAS, Poland
- Laszlo Guczi, Institute of Isotopes and Surface Chemistry, Hungary
- Klaus Hermann, Fritz-Haber Institute, Germany
- Ronald Imbihl, University of Hannover, Germany
- Maya Kiskinova, Sincrotrone Trieste, Italy
- Erich Knoezinger, TU Wien, Austria
- Zoltan Paal, Institute of Isotopes and Surface Chemistry, Hungary
- Rutger A. van Santen, Eindhoven University of Technology, The Netherlands
- Jacques Vedrine, Ecole Nationale Superieure de Chimie de Paris, France
- Malgorzata Witko, Institute of Catalysis and Surface Chemistry PAS, Poland
- Helmut Weiss, Otto-von-Guericke-Universitaet Magdeburg, Germany
- I **nvited lectures**
- A. B. Jarzębski: "Modified siliceous mesostructured cellular foams - effective catalysts for applications in liquid phase processes"
- J.-F. Hochepied: "How precipitation techniques can be useful for catalysts preparation?"
- Z. Karpinski: "Hydrodechlorination of 1,2-dichloroethane over active carbon supported palladium-nickel catalysts"
- N. Kruse: "Surface reaction kinetics studied with atomic-scale lateral resolution"
- P. Massiani: "Spectroscopic investigation of co-hosted metal and basic nanospecies in Pt/CsBEA catalysts"
- S. Müller: "Ab-initio thermodynamics at surfaces: Relaxation, segregation, substitutional ordering and adsorption"
- D.K. Paul: "Photocatalytic Oxidation of Acetaldehyde over Rh-doped SrTiO_{3} Nanoparticles"
- R. Rosei: "Direct spectroscopic measurements of surface chemical reactivity"
- G. Rupprechter: "In Situ Studies of Surface Catalytic Processes on Nanomaterials"
- J. Ryczkowski: "Application of infrared photoacoustic spectroscopy in catalysis"
- R. Schlögl: "The relevance of nanostructuring for the function of heterogeneous catalysis"
- E.M. Serwicka: "Catalysis by metalloporphyrins supported on mesoporous molecular sieves - steric effects induced by nanospace constraints"
- Yu. Suchorski: "Catalytic reactions on platinum metals nanofacets: Spectroscopy on an atomic scale"
- J.Vedrine: "Nano-oxides for selective oxidation of light alkanes: catalyst preparation, characterization, reaction mechanism and high throughput studies"

Organisers

- Urszula Narkiewicz, Faculty of Chemical Engineering, Szczecin University of Technology, Szczecin, Poland, urszula.narkiewicz@ps.pl
- Jacek Kijenski, Institute of Industrial Chemistry, Warsaw, Poland, jacek.kijenski@ichp.pl

Proceedings

The invited and keynote papers will be published in *Catalysis Today* (http://www.elsevier.com/locate/cattod).

Sponsors

Some PhD students have their conference fees reimbursed by the Scientific Network: « Nanomaterials as Catalysts for New, Environmentally Friendly Processes », represented by Professor Małgorzata Witko.

Programme

Monday, 4 September

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Parallel Session
Monday afternoon, 4 September, 14:00
Chair: Norbert Kruse

14:00 Invited oral

The relevance of nanostructuring for the function of heterogeneous catalysis

Robert Schlögl

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Heterogeneous catalysts modify the rate of a chemical reaction by changing the activation barrier for at least one critical elementary step. This kinetic operation requires catalysts to be reactive themselves. The main difference to other reactive materials is their cyclic operation returning them after having performed a turnover of reaction in their initial state of geometric and electronic structure. This property is referred to as “dynamical”.

Solid surfaces can behave dynamically most easily when they are not in a highly ordered state in order to minimize collective activations of the geometric structure. Collective excitation of the electronic structure would lead easily to over-reaction and hence preclude selective reactions (concept of site isolation). These requirements call for a hierarchical structure of a working catalyst in which a stable matrix contains adaptive sites as clusters which change their properties according to the chemical potential of the gas phase to accommodate for adsorption of reactants and desorption of products.

It is obvious that the design of such a structure is extremely demanding and has not yet occurred in any sustained fashion. It is merely the art of manufacture of catalysts that incorporates suitable dimensions of nanostructure even when the material is a bulk solid. Some insight into the still very rudimentary methods of structuring complex materials will be given.

14:30 Invited oral

In Situ Studies of Surface Catalytic Processes on Nanomaterials

Günther Rupprechter

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Model Catalysis has come a long way. The development of planar nanoparticle model catalysts, consisting of well-defined metal particles grown in ultrahigh vacuum (UHV) on thin oxide films, provides a route to mimic an increasing number of technical catalysts [1,2]. The mean particle size (~2-10 nm) as well as the particle morphology and defect structure can be accurately controlled. Furthermore, the planarity and electrical and thermal conductivity of the model systems allows applying a wide range of surface sensitive imaging (STM) and spectroscopic (XPS, IRAS, SFG, TPD, etc) techniques. In parallel, significant advances have been made in carrying out surface characterization under non-UHV conditions, i.e. under mbar to atmospheric pressure. Photon-based methods such as sum frequency generation (SFG) vibrational spectroscopy or polarization-modulation infrared reflection absorption spectroscopy (PM-IRAS), together with high-pressure X-ray photoelectron spectroscopy (HP-XPS) are among the prime techniques for in situ studies of surface processes or catalytic reactions on model systems. A number of case studies will be presented, including CO adsorption and hydrogenation, partial methanol oxidation and olefin and diene hydrogenation, carried out on Pd-Al_2O_3, Pd-Nb_2O_5 and Pd-Fe_3O_4 model catalysts.

[1] G. Rupprechter, Annu. Rep. Prog. Chem. (C) 100 (2004) 237. [2] H.-J. Freund, M. Bäumer, J. Libuda, T. Risse, G. Rupprechter, S. Shaikhutdinov, J. Catal. 216 (2003) 223.

15:00 Keynote lecture

Modified Pt-Fe catalysts for cinnamaldehyde hydrogenation

Jacek A. Kijeński[1,2], Piotr Winiarek[1], Elżbieta Fedoryńska[1], Agata Purzycka[1]

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A new method has been presented for the preparation of catalysts for selective hydrogenation of cinnamaldehyde to cinnamyl alcohol

(compound used in the synthesis of drugs and flagrances). The following steps have been included in the catalysts preparation procedure: a. reaction of the support (g-Al_2O_3, TiO_2, SiO_2, MgO) with transition metal alkoxide ($Zr(OBu)_4$, $Ti(OBu)_4$, $(BuO)_3VO$); b. thermal decomposition of the obtained surface complex; c. dry impregnation with an aqueous solution of Pt and Fe salts; d. drying; e. reduction with gaseous hydrogen. The products of each step were characterized by means of AAS, XRD, FTIR-PAS, SEM, SIMS and TPR. The monolayer of transition metal oxide (TMOM) (ZrO_2, TiO_2 or V_2O_5) has been formed on the surface of support as a result of surface complex decomposition (b.) at 623 K. Small crystallites (<10 nm) of these oxides have been observed after the decomposition of mentioned complex at 823 K. Ferroane phase (PtFe) has been detected among others on the surface of reduced Pt-Fe catalysts.

A hydrogenation of cinnamyl aldehyde was performed in the flow system (T = 423–573 K, p = 1 atm). The catalyst's selectivity in cinnamyl alcohol synthesis increased in the following order: Pt/support << Pt-Fe/support < Pt/TMOM/support < Pt-Fe/TMOM/support. In the optimized reaction conditions hydrogenation of cinnamaldehyde over Pt-Fe/ZrO_2/MgO yielded cinnamyl alcohol as a main product (78%, selectivity over 90 %).

Coffee break

Monday afternoon, 4 September, 15:30

Parallel Session

Monday afternoon, 4 September, 15:50

Chair: Jacek A. Kijeński

15:50 Invited oral

Nano-oxides for selective oxidation of light alkanes: catalyst preparation, characterization, reaction mechanism and high throughput studies

Jacques Vedrine

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Light alkanes (C_1 to C_5) constitute rather cheap raw materials coming from natural gas fields or from naphta. Dehydrogenation reactions have been widely used to get the corresponding olefins of much higher commercial value. However such reactions are endothermal and the catalysts, often chromia based, deactivate due to carbonaceous residues fouling. Another way is to proceed by partial oxidation, which overcomes easily the last two problems, although alkanes are weakly reactive and can lead to total oxidation due to higher reaction temperature necessary. One then gets the corresponding olefins or oxygenated compounds (alcohols, aldehydes, acids) of even higher commercial value. The oxidation of butane to maleic anhydride is a well-known industrial process on VPO catalyst. MoNbTe(Sb)V-O mixed oxide catalysts of different chemical compositions, calcined and activated before reaction under different conditions, have been developed for ethane or propane oxidation to ethene or acrylic acid respectively and VSb mixed oxide/Al_2O_3 for propane ammoxidation.

In this lecture we will present some recent results from us or from the literature dealing with the preparation of different mixed oxides of nano size particles and some of their catalytic properties. Catalyst characterization will be presented to determine the catalyst behaviour and the role of different phases present.

The studies of reaction kinetic, reaction mechanisms and catalyst characterisation were complemented by a combinatorial approach to try to determine if high throughput technology could be applied for such complex systems.

16:20 Keynote lecture

Nano-Au particles and oxide M_xO_y (M=V, Mo, Cr) clusters dispersed on oxide supports in oxidation reactions

Barbara A. Grzybowska-Świerkosz

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Catalytic and physicochemical properties of Au nanoparticles and MxOy (M=V,Mo,Cr) nanoclusters dispersed on oxide supports will be described. The reactions studied included total and selective oxidation reactions, related to environmental protection: oxidation of CO (also preferential PROX oxidation in the presence of hydrogen) and of C3 and C2 hydrocarbons. Correlations between molecular structure, physicochemical properties of the catalysts and catalysts' activity and selectivity will be shown and discussed. The accent will be put on the effect of nature of the oxide support on dispersion and properties of the active phase: metallic (nanoAu) or oxidic (MxOy clusters). For catalysts based on Au nanoparticles, the activity in CO oxidation increases with the support reducibility. Activity and selectivity in oxidation of hydrocarbons depends also on the support reducibility, nano-Au dispersed on non-reducible oxides of main group elements being more selective. For MxOy/oxide support catalysts an increase in activity and selectivity to partial oxidation products and decrease in the acidity, as compared with bulk MaOb oxides, have been found. Structure and properties of dispersed bidimensional MxOy clusters depend on the number of M atoms in the clusters and on nature of the support. Specific catalytic properties of MxOy nanoclusters as compared with bulk oxides are discussed in the light of the current theories of oxidation.

16:50 Oral

Pt-supported nanocrystalline ceria-zirconia doped with La, Pr or Gd: factors controlling syngas generation in partial oxidation/autothermal reforming of methane or oxygenates

Vladislav A. Sadykov[1], Natalia V. Mezentseva[1], Galina M. Alikina[1], Anton I. Lukashevich[1], Yulia V. Borchert[2], Tatyana G. Kuznetsova[1], Vyacheslav P. Ivanov[1], Sergei N. Trukhan[1], Eugenii A. Paukshtis[1], Vitalii S. Muzykantov[1], Julian Ross[3], Erhard Kemnitz[4]

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These work considers effect of doping of nanocrystalline CeO_2-ZrO_2 supports with La, Pr or Gd cations on the oxygen mobility, surface features and properties of supported Pt as related to CH_4 or acetone transformation into syngas at short contact times. Samples surface features were studied by SIMS and FTIRS of adsorbed CO or NO. The oxygen mobility was characterized by the oxygen isotope exchange and H_2 TPR. Catalytic activity was studied in the flow installation using diluted feeds. Within each series, for both reactions, catalytic activity correlates with the Pt dispersion controlled by the oxidized samples ability to stabilize Pt^{2+} cations as precursors of small reactive Pt clusters formed under reaction conditions. Concentration of these cations correlates with the density of Lewis acid sites -Me^{4+} cations as well as domain boundaries. Among samples of different series, at comparable Pt dispersion, the highest performance was demonstrated by La-doped system, which correlates with the highest surface/near-surface oxygen mobility. This, is turn, correlates with the lowest strength of Ce-O and Zr-O bonds in the surface layer as judged by the ratio of CeO^+/Ce^+ and ZrO^+/Zr^+ ion currents by SIMS. This work is in part supported by INTAS 05-1000005-7663, RFBR-CNRS 05-03-34761 and ISTC 2529 and 3234 Projects

17:05 Oral

Nanostructured catalysts from laser pyrolysis

Nathalie C. Herlin-Boime[1], Hicham Maskrot[1,2], Yann Leconte[1], Cecile Reynaud[1], Monique Gervais[2], Sabine Valange[3], Erwan Guelou[3], Joel Barrault[3], Jean Noel Rouzaud[4]

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Catalysis is one of the privileged applications in nanoscience because it takes advantage of the high surface/volume ratio of nanomaterials. The high dispersion of the active phase in nanomaterials leads to a better catalytic efficiency. However the synthesis of such materials by traditional methods is not straightforward and new methods, such as laser pyrolysis, are therefore under exploration.

Laser pyrolysis is a method allowing the synthesis of various nanoparticles, with well defined chemical composition, size and structure. It is based on the interaction of a powerful IR laser beam with a mixture of gaseous or liquid precursors. This interaction leads to an increase of temperature with decomposition of the precursors followed by nucleation and growth of nanoparticles in an incandescent flame.

This work reports the laser synthesis of titania based powders containing a noble metal (in particular nanoPt/TiO_2) using a spray of TTIP (Titanium tetra isopropoxide) mixed with organometallic precursors. TEM pictures show that the obtained TiO_2 nanoparticles can have a diameter in the range 6-7 nm. The total amount of Pt can be controlled in the range 0.5-5 wt%. The volatile organic compounds elimination tests demonstrate that these nanoparticles are efficient for the total methanol oxidation at a temperature as low as 50°C.

In conclusion, these results show that the laser pyrolysis method allows the synthesis of well dispersed titanium-based nanoparticles which act as efficient catalysts.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Parallel Session
Tuesday morning, 5 September, 9:00
Chair: Günther Rupprechter

9:00 Keynote lecture

Surface composition and reactivity of catalytic systems at micro- and nano- scales

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Most of the attempts to quantify the reactivity and selectivity of catalytic systems have been focused on characterization of specific surface structures, where electronic perturbations are induced by varying the particle size or by adding small amounts of substances. Addressing specific systems the lecture will illustrate the important contribution of chemical specific imaging combined with laterally resolved structural characterizations to understanding key factors controlling the processes at complex surfaces and interfaces related to catalysis. The local composition and reactivity of the phases that can be formed and coexist during oxidation reactions will be described comparing the results obtained with single crystal metal surfaces, micrometer-sized metal particles and nano-crystalline films supported on different substrates.[1] The complexity of the realistic reaction systems, where mass transport processes can introduce lateral heterogeneity in the composition and structure of the interface

creating local micro-reactors with different catalytic activity, will be discussed on the bases of recent results with bimetallic systems.[2] The effect of electron confinement will be shown for oxidation of ultrathin metallic films, where varying the film thickness the sensible differences in the local oxidation rate are observed and correlated to periodic changes in the density of electronic states induced by quantum-well states crossing the Fermi level.[3] 1. P. Dudin et al, J. Chem. Phys.B 109, 2005, 13649. 2. A. Locatelli et al, J. Am. Chem. Soc. 127, 2005, 2351. 3. L. Aballe et al, Phys. Rev. Lett. 93, 2004, 196103.

9:30 Invited oral

Surface reaction kinetics studied with atomic-scale lateral resolution

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This presentation reviews the recent progress made in imaging catalytic and non-catalytic surface reactions and in providing information on the local chemical composition of reactive layers. The methodical approach is based on video-Field Ion Microscopy (FIM) and atom-probe Pulsed Field Desorption Mass Spectrometry (PFDMS). The usefulness of the approach will be demonstrated in several case studies.

First, the carbonylation of Nickel to $Ni(CO)_4$ will be presented. This reaction involves subcarbonyls, $Ni(CO)_{2,3}$ as intermediates. Time-resolved PFDMS studies - in this method short field pulses of variable repetition frequency are used to field-desorb reactive species during the ongoing reaction - reveal $Ni(CO)_2$ formation to be the slowest step in the overall reaction. In FIM this process is seen to coincide with Ni kink sites detaching in a repetitive manner so that an originally hemispherical Ni crystal ("field emitter") is transformed into a polyhedral one. Second, we show that strong morphological changes do also occur during the reaction of CO and mixtures of CO/H_2 with Co crystals. In this latter case video-FIM allows mapping of the catalytic activity and revealing the competition between morphological reshaping and chemical restructuring due to carbon deposition. C_xH_y species are detected by PFDMS during the ongoing CO/hydrogen reaction. The ion intensities of these species depend on the reaction time (ms time scale at 450 K and 10^{-3} mbar, $H_2/CO=2$) and methane is the first product of this hydrogenation. The data will be directly compared to transient kinetic build-up measurements using Co supported model catalysts.

Finally, video-FIM data on the interaction of ethylene with Ni and Co crystals will be shown. In both cases step sites cause a strong promotion of ethylene decomposition. In the Ni case, graphitic overlayers seem to form at intermediate temperatures (~600 K). These layers undergo an explosive clean-off reaction (with hydrogen) in which chemical fronts ignite in (001) planes before travelling in a self-accelerating manner toward the central (001) plane of the crystal. In a similar study, a Ni crystal is heated to ~1000 K in the presence of 10^{-2} Pa ethylene. Subsequent in-situ quenching and FIM imaging at 500 K reveals string-like structures arranged in a concentric manner in the outskirts of the crystal. These structures point away from the crystal centre. A tentative explanation of this observation is based on the formation of carbon nanofibers and their repulsive interaction with the positive electric field present during imaging.

10:00 Invited oral

Catalytic reactions on platinum metals nanofacets: Spectroscopy on an atomic scale

Yuri Suchorski

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The technical importance of heterogeneous catalysts has motivated an intensive development of model systems emulating the behaviour of dispersed metals on oxidic supports. One of the successful approaches is the creation of arrays of the small metal particles, grown on a planar oxidic support. Most surface-sensitive spectroscopies, however, average the collected data (in the best case over few catalytic pellets of the sample), thus the individual characteristic of the single particles are often "smoothed out". This creates difficulties in addressing the details of the reaction mechanisms on the single metal nanoparticles.

Field Ion Microscopy (FIM), Field Emission Microscopy (FEM) and Lithium Field Desorption (Li-FDM) Microscopy can in principle allow investigation of local reaction kinetics on the structurally heterogeneous surface of a single apex of a Pt or Rh tip. The surface of such a tip can be characterized with atomic resolution by FIM and catalytic reactions on well-defined nanosized facets can be monitored in situ with a resolution $\leq$ 2 nm using the mentioned above microscopies [1].

By using the probe-hole techniques the field ions of reacting entities emitted from few selected surface sites can be collected and analyzed in a retarding potential experiment (field ion appearance energy spectroscopy, FIAES [2]). FIAES provides the binding energy of the neutral molecules adsorbed on these selected sites. The probe-hole approach can be also combined with the mass analysis by magnetic separation (mass-to-charge resolved potential analyses) or with the time of flight (ToF) measurements.

[1] V.V. Gorodetskii, V.I. Elochin, J.W. Bakker, B.E. Nieuwenhuys, Cat.Today 105 (2005)
183; Y. Suchorski and J. Beben, Prog. Surf. Sci. 74 (2003) 3, and references therein
[2] Y. Suchorski, W.A. Schmidt, N. Ernst, J.H. Block, H.J. Kreuzer, Prog. Surf. Sci. 48
(1995) 121

Coffee break

Tuesday morning, 5 September, 10:30

Parallel Session

Tuesday morning, 5 September, 11:00
Chair: Jacques Vedrine

11:00 Invited oral

Application of infrared photoacoustic spectroscopy in catalysis

Janusz Ryczkowski

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Photoacoustic spectroscopy (PAS) measures a sample's absorbance spectrum directly with a controllable sampling depth and with almost no sample preparation. This technique is nondestructive, noncontact, applicable to macrosamples and microsamples, insensitive to surface morphology. In the contrary to the traditional transmission methods, PAS is carrying out the information from the surface of the measured sample. In the area of infrared PAS is a supplementary one to the other spectroscopic and classical physicochemical methods of the catalysts surface characterization. It is capable of measuring spectra of all types of solids without exposure to air or moisture. Photoacoustic spectroscopy is detecting a sample's IR spectrum by "listening" to the sound made when the sample absorbs infrared radiation. A highly sensitive microphone is used as a detector, and the spectrum is similar to absorbance spectra. One of the major advantages of PAS is its relative immunity to scattered light. Consequently, this methodology can provide absorption spectra of highly light-scattering materials such as powders (e.g. silica, alumina) or highly-absorbing materials (e.g. different type of carbonaceous adsorbents, materials, species, etc.). Several of the problems associated with the transmission techniques may be overcome utilizing photoacoustic detection (in particular, changes in the spectral region were inorganic supports are strong absorbers). Several practical and valuable examples will be presented covering various areas of catalysts investigations (catalysts preparation, adsorption, surface acidity, characterization of MCM-type materials, modified carbons, carbon deposits, etc.). Moreover, selected examples will be devoted to the comparison of data for the same studied material but obtained by the different techniques in the area of IR. Finally, there will be summarized advantages as well as the limits of the FT-IR/PAS.

11:30 Invited oral

Photocatalytic Activity of $SrTiO_3$ Nanoparticles: An in situ FT-IR Study

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$SrTiO_3$ was prepared by using sol-gel method which involve hydrolysis of alkoxide mixtures in an alcohol-toluene solvent. The product was then characterized by XRD and other techniques. The adsorption and photocatalytic oxidation reaction of acetaldehyde was studied on these aerogel prepared $SrTiO_3$ nanoparticles using *in situ* FT-IR spectroscopy in a specially designed stainless steel UHV cell. The photooxidation involved UV-Vis irradiation in the range of 2.1 to 5.0 eV. It has been found that a large fraction of acetaldehyde physisorbed onto the surface at 173 K which upon oxygen exposure in the dark did not undergo further oxidation. However, in presence of UV light exposure, the aldehyde underwent oxidation forming some CO_2 and other oxidized species. The infrared assignments of all adsorbed species were used to explore the reaction mechanisms of photochemical reaction.

12:00 Oral

UV activity of titanium dioxide films and TiO_2 embedded in ordered mesoporous SBA-15 structures for CO_2 photo-reduction reactions

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TiO_2 films coated on hollow glass beads via a sol-gel procedure were tested for the photo reduction of CO_2 in the gas phase. Pt was incorporated on the films either by adding the precursor salt in the sol, Pt(in), or by wet impregnation of calcined film with an aqueous solution of the precursor salt, Pt(on). TiO_2 films could be prepared with thicknesses ranging from 300 nm to 3000 nm, measured by a surface DekTak profilometer. The synthesized samples were characterized by powder X-ray diffraction and Photo Luminescence spectroscopy. Under UV illumination, the methane yields of platinized TiO_2 films decreased in the following order: Pt(on).TiO_2>Pt(in).TiO2>TiO_2. Photoluminescence spectroscopy indicated that the spectrum was blue shifted for Pt(in) catalysts, while no such shift was observed for Pt(on) catalysts. These results are interpreted in terms of charge separation effect of the external Pt. The band-gap modification as revealed by the photoluminescence spectroscopy indicated no effect on the photocatalytic activity of the films. In the second part of the study, titanium dioxide containing SBA-15 structures have been prepared by a sol-gel method using the self organizing polymer pluronic 123 and adding titanium(IV) isopropoxide into the solution in the preheating and precalcination stage. XRD pattern showed the SBA-15 characteristic peaks at 2q= 1.5 and 1.8 indicating long range order in the SiO_2 framework.

12:15 Oral

Photogenerated charges in TiO_2 nanostructures

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Metal oxide nanocrystals, and in particular TiO_2, are employed for many technologically relevant surface applications. Because chemical and photochemical reactivity critically depends on the size and the shape of the crystals, control over these properties represents a key issue in materials' synthesis. Morphologically well-defined nanostructures will be introduced as model systems to establish a correla-

tion between chemical and photochemical reactivity, on one hand, and the abundance of specific surface sites, on the other.

TiO_2 absorbs photons with energies greater than 3.2 eV which leads to the generation of excited electrons and holes. These can either (a) recombine under photoluminescence emission or heat generation, (b) become persistently trapped or (c) undergo redox reactions with molecules at the particle surface. The particle surface largely determines the efficiency of these processes. For this reason, photoexcitation processes have been investigated on different TiO_2 based model systems: TiO_2 anatase nanoparticles produced via chemical vapour deposition[1], titanate nanotubes[2] and TiO_2 nanorods. Persistent charge trapping and oxygen radical formation were studied by electron paramagnetic resonance. The presence of O_2, an electron scavenger, results in charge transfer to the oxygen molecule to generate adsorbed O_2^- radicals.

Quantification in terms of trapped charges per particle was carried out and the influence of the surface structure and properties on the obtained figures will be discussed. Tracking UV-induced reactions on nanomaterials of different size and shape in comparison with studies on the standard TiO_2 P25 represents a valuable approach towards the identification of active sites on nanocrystalline samples.

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Lunch break

Tuesday afternoon, 5 September, 12:30

Parallel Session

Tuesday afternoon, 5 September, 14:00
Chair: Malgorzata Witko

14:00 Keynote lecture

Theoretical Description of Molybdena Based Catalysts; DFT Cluster Model Study.

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Molybdenum oxide-based catalysts are active and selective in many catalytic reactions. The catalytic properties of such catalysts strongly depend on acid-base properties of catalysts surfaces, which determine the strength of surface-substrate interactions and facilitate the desorption of products from the surface. MoOX systems are particularly good examples for studying the role of different electronic and geometric factors on catalytic properties of the system due to the richness of surface active sites. This richness results from the possibility of different coordination number and oxidation states of Mo and O ions as well as geometrical and chemical environment of surface sites.

For theoretical description ab initio density functional theory (StoBe code) is applied. First, the nucleophilic properties of differently coordinated surface O atoms, which are present at pure (010)MoO_3 surface are discussed. In the next step the results of the creation of local mono- and di-vacancy of O type are studied. In addition, process of surface re-oxidation that leads to the formation of very active surface oxygen species is also investigated. Further, metal reduction states (MoO_3, MoO_2) are studied in order to examine their influence on electronic parameters of oxygen sites. The influence of the additional atoms (Mn, Co) on chemical properties of active sites is examined by the comparison of the electronic structure of cobalt and manganese molybdates ($CoMoO_4$, $MnMoO_4$) with the pure Mo-O (MoO_3) system.

14:30 Keynote lecture

Excitation and reaction at metal and oxide surfaces: cluster models help to interpret experiments

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This talk reviews recent theoretical work on electronic and structural properties of metal and oxide surfaces including adsorption and reaction of small molecules where density functional theory (DFT) and embedded surface cluster models have been used. Differently coordinated surface oxygen in vanadium oxides exhibits pronounced differences in its charging and binding, which influences the surface geometry and indicates different chemical behavior as identified by surface sensitive spectroscopies. Results from recent theoretical spectroscopy studies on vanadium pentoxide, V_2O_5, will be discussed in detail and compared with experimental data.

Surface oxygen binds very strongly to the substrate as determined by corresponding vacancy energies for the V_2O_5 surface. Further, surface vacancies are found to serve as chemically active centers inducing chemical reduction of nearby metal sites. This is obvious from atom projected densities of states and has been confirmed by experiment.

Extended cluster studies on different phenylpropene adsorbates on Cu(111) substrate yield equilibrium geometries which are consistent with experimental findings based on angle-resolved NEXAFS measurements. In particular, the theoretical angle-resolved spectra evaluated in the cluster approach are in excellent agreement with all details of the experimental NEXAFS data. The comparison between theory and experiment can explain the different epoxidation rates of the adsorbates by simple geometric effects.

15:00 Oral

Study of the influence of impurities on reactant distribution functions in a heterogeneous catalytic reaction model

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Extensive Monte Carlo simulations have been performed on a generalized version of the Ziff, Gulari and Barshad model (1986), for CO oxidation on a Pt catalytic surface to account for surface impurities.

Specifically, we show the impact of immobile inert impurities on the kinetics of the catalysis, particularly on the positioning of the CO poisoned dynamical phase transition and also on the O_2 poisoned dynamical phase transition.
We also show the influence of the concentration of impurities on the efficiency of the active catalytic state.
To this end, we tailored a series of simulations involving different impurity concentrations to characterize the reactant cluster structure in the presence of impurities by using a modified Hoshen-Kopelmann algorithm.
We find that impurities tend to prefer to lie at the periphery sites of the clusters rather than in their interior.
Finally, we want to stress the relevance of our study for people working on heterogeneous catalytic reactions and/or environmental issues.

15:15 Oral

Homogenization in catalysis processes.

Georges Griso[1], Maria Teresa Sanchez[2]

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A catalytic converter in an automobile's exhaust system provides an environment for a chemical reaction where unburned hydrocarbons completely combust, in such a way that pollution is reduced. An enormous effort is being made with the purpose of developing appropiate supports and the catalyst itself. In some cases the design of supports geometry is what makes a process more optimal. Our objective is to model catalysis processes which occurs in an automobile's exhaust system. Due to the great difficulty of the problem, the first step is to study the asymptotic behaviour of catalysis supports in a linear elasticity problem. Since the computational domain in catalysis processes are beams with reticulated structure and the finite element method is not suitable for this type of structures, it is necessary to obtain an equivalent mathematical model defined over the domain without holes. This model has to approach as far as possible the global supports behaviour. This is the main objective of homogenization theories.
In this work, a new procedure, called the *unfolding method*, developed by Cioranescu, Damlamian and Griso (see [1]), is applied to solve this first approach. The catalysis support is a structure made of beams, placed periodically and with inner holes. We introduce a decomposition of the displacements in such a structure and, by proving some convergence results, we obtain three one-dimensional uncoupled limit problems: The first problem defines the longitudinal displacement and the second one gives the transverse bending of the structure, while the third one defines the torsion angle. The general form ot these problems is well-known in the classic theory of beams, the only difference appears in the new homogenized coefficients.

References

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Coffee break

Tuesday afternoon, 5 September, 15:30

Parallel Session

Tuesday afternoon, 5 September, 15:50
Chair: Klaus Hermann

15:50 Keynote lecture

Can one tailor a catalyst with particular properties?

Malgorzata Witko

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A main goal of catalytic science is to tailor the most active and selective catalyst for a particular reaction. Such a goal demands synergy between two parallel and complementary approaches: experimental and theoretical, and gives the opportunity to manufacture catalyst for a particular reaction. In the following the role of theory will be will be illustrated on the example of V-O systems.

Vanadia-based catalysts are used in many different processes that belong to various types of chemical reactions. Their wide application follows from a fact that V_2O_5 crystallites may exhibit two structurally different types of faces: surface built of chemically saturated atoms and those built of unsaturated cations and anions. Both show different behavior in catalytic reactions by performing a complex multi-step operation on the reacting molecule through activation of some of the bonds within reactant and hindering those interactions, which could result in unwanted product.

In the lecture the energetic stability of low-indices (010), (100) and (001)V_2O_5 surfaces will be compared based upon periodic DFT calculations. The electronic structure and activity of structurally different surface O sites will be discussed using both cluster and periodic approaches. Adsorption of H leading to the formation of surface OH and H_2O species as well as hydrogen migration through the surfaces will be considered. Creation of surface oxygen vacancies will be undertaken and followed by their re-oxidation through the gaseous oxygen.

16:20 Invited oral

Ab-initio thermodynamics at surfaces: Relaxation, segregation, substitutional ordering and adsorption

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A prerequisite for studying catalytic processes at surfaces is a detailed knowledge of the surfaces' structural properties. Thereby the subject "structure" cannot be restricted to atomic relaxations alone, but also must consider substitutional ordering phenomena which often take place on a mesoscopic scale. Two examples whose model-

ling is fundamental for studying chemical processes at surfaces, are surface segregation and multi-site adsorption. It will be shown that today it is possible to study these properties using a first-principles based Hamiltonian constructed form the energetics of geometrically fully relaxed structures. This Hamiltonian can then be used for Monte-Carlo simulations in order to investigate the temperature-dependence of the surface properties. It will be shown that our new UNCLE code allows for a quantitative prediction of segregation, adsorption. and short-range order in excellent agreement with experimental data. Our focus will be on segregation at metal alloy surfaces and nanostructures stabilized by hydrogen adsorption.

16:50 Oral

How adsorption and reaction influence on the equilibrium shape and surface morphology of the metal nanoparticles.

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The peculiarities of reaction performance over nanocatalysts dictate the special requirements to the models aimed at the simulating of catalytic properties of nanoparticles [1,2]. The goal of our study is the elaboration of the stochastic model of the supported particle taking into account the change of the shape and surface morphology of the particles under the influence of the reaction media. The analysis has been provided by means of the lattice model based on the Kossel crystal located on the inert support [3]. The morphology of the particle's surface is determined by the heights of the metal atom columns. The change of morphology caused by the diffusion of the surface atoms (the metal atoms attract each other and the atoms of support).

The influence of adsorption on the particles equilibrium shape and surface morphology has been studied. By taking into account of attraction "adsorbate-metal" the reshaping of the initial hemispheric particle into cone-shaped one occurs induced by adsorption, similar to the experimentally observed reversible reshaping of active nanoparticles [4]. The isotherms simulated with taking into account the attraction between atoms of metal and adsorbate differ noticeably from the ideal Langmuir isotherm. The simulation of oscillatory CO oxidation reaction over Pd nanoparticles has been provided. The influence of the particles shape and surface morphology, as well as spillover effects, on the characteristics of oscillations has been studied.

Acknowledgements: The study was partly supported by the NWO grant # 047.015.002

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Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break

Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break

Wednesday afternoon, 6 September, 12:30

Parallel Session

Wednesday afternoon, 6 September, 14:00
Chair: Yuri Suchorski

14:00 Invited oral

Hydrodechlorination of 1,2-dichloroethane over active carbon supported palladium-nickel catalysts

Anna Śrębowata[1], Wojciech Juszczyk[1], Zbigniew Kaszkur[1], Zbigniew Karpinski[1,2]

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Two series of 2 wt.% active carbon (Sibunit)-supported Pd-Ni catalysts were prepared using chloride and nitrate precursors. They were characterized by CO chemisorption, temperature programmed palladium hydride decomposition and XRD, and investigated in the hydrodechlorination of 1,2-dichloroethane in the gas phase at a relatively low reaction temperature (230^{o}C). All catalysts showed rather moderate metal dispersion (below 10%). They appeared very stable during a 20 h testing. Ni-rich bimetallic samples exhibited the highest selectivities towards ethylene (desired reaction product). Small amounts of added palladium, the metal which is known for its high activity in hydrogenation, resulting in a massive production of ethane. The results showed a smooth correlation between turnover frequency towards ethylene and Pd-Ni phase composition, irrespective of the type of metal precursor used in catalyst preparation. The 2 wt.% Ni/C catalyst prepared from chloride precursor showed some nonnegligible selectivity toward vinyl chloride monomer, the selectivity which gradually increased in time on stream. All other catalysts did not show such a propensity.

The overall results indicate that palladium segregates to the surface of Pd-Ni, shaping the overall catalytic behavior of bimetallic Pd-Ni catalysts. The temperature programmed hydrogenation of deposits left after reaction show only the presence of surface carbon, without

considerable amounts of chlorine.

14:30 Keynote lecture

Oxide Nanocrystals as Model Systems for Surface Chemistry

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Recent advances in morphology control of unsupported oxide nanostructures allow for the deliberate enhancement or depletion of microstructural characteristics. This opens up new opportunities to relate topographic surface features to their chemical properties. Chemical vapor deposition is an efficient technique for the generation of nanometer-sized MgO particles with characteristic surface defects. After thermal annealing the crystallites adopt cubic shape and the ratio between corner and edge ions depends on the average particle size. Thus, MgO nanocubes represent a powerful model system for molecular spectroscopy studies which aim at the investigation of surface processes on polycrystals.

While on highly dispersed earth alkaline oxides ultraviolet light with energies below 6.2 eV (l>200nm) exclusively addresses the surface, light induced charge separation is initiated in the bulk of TiO_2 nanostructures. Under high vacuum conditions, electron and hole trapping processes can be tracked by electron paramagnetic resonance and IR spectroscopy on a time scale of minutes. The generation and chemical reactivity of trapped charges as well as their quantification using the photoadsorption of O_2 will be discussed for anatase particles and titanate nanotubes.

15:00 Oral

Catalytic Activity and Selectivity of Pt/MWCNTs System in Hydrodechlorination of Freon CFC-12

Kuan-Nan Lin[1], Magdalena Bonarowska[2], Leszek Stobinski[2], Zbigniew Karpinski[2], Hong-Ming Lin[1]

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Carbon nanotubes exhibit unique physico-chemical properties and they may be used as a basis for other nanostructured materials, such as nano-powder catalysts. For example, multiwalled carbon nanotubes (MWCNTs) can be used as a catalyst support where catalytic active metal nano-particles may be deposited onto the functionalized MWCNTs surface.

It is known that activity and selectivity of metallic catalyst significantly depend on its preparation and subsequent modification, such as sintering, calcination and/or reduction. Pt/MWCNTs catalyst with 23 wt. % content of Pt was prepared by polyol process using H2PtCl6 as Pt precursor and ethylene glycol (EG) as a reducing agent.

Uniform distribution of Pt nano-particles on the surface of functionalized MWCNTs was obtained. The size of Pt nanoparticles varied from 3 to 10 nm. Further annealing and reducing of Pt/MWCNTs catalyst in hydrogen atmosphere were performed, obtaining four various samples. The activity and selectivity of each Pt/MWCNTs sample in the hydrodechlorination reaction of freon CFC-12 (CCl2F2) was determined. TEM, XRD and Raman spectra were used to characterize each Pt/MWCNTs sample.

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony
(continuation of Mondazqs Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Parallel Session
Thursday morning, 7 September, 9:00
Chair: Barbara A. Grzybowska-Świerkosz

9:00 Invited oral

Spectroscopic investigation of co-hosted metal and basic nanospecies in Pt/CsBEA catalysts.

Pascale Massiani[1], Chiara Bisio[2], Katia Fajerwerg[1], Gianmario Martra[2]

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In the field of metal supported heterogeneous catalysis, one advantage of zeolites among other aluminosilicate supports is to be characterized by strongly organized microporous channel systems in which both a high dispersion and a regular distribution of metal nanoparticles can be obtained. Another interest of these supports is the possibility to easily tune their acid-base properties by simply changing their chemical composition, thus possibly modifying the characteristics of the dispersed metal particles through metal-support interaction effects. This will be exemplified in this presentation in which we will show how the particle size and electronic properties of dispersed platinum vary when the basicity of Cs-containing support with BEA zeolite structure increases. Particularly, in samples containing caesium in excess as compared to the exchange capacity of the zeolite, Pt nanoparticles with sizes in the 2-1 nm range (detected by TEM) and even below 1 nm (detected by EXAFS) are formed. Besides other physicochemical techniques, we will focus on the characterization of the supported species in these systems by IR spectroscopy of adsorbed CO, CO_2 and N_2 probe molecules. This will allow us to describe the processes of formation and interaction upon thermal activation treatments of co-hosted Pt nanoparticles and Cs-oxide like nanospecies with strong basic character.

9:30 Invited oral

Catalysis by metalloporphyrins supported on mesoporous molecular sieves - steric effects induced by nanospace constraints

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Metalloporphyrins are the most widely studied catalysts for homogeneous selective oxidation and hydroxylation of hydrocarbons. An important area of research into their catalytic properties is associated with immobilization of these species onto a solid support, in order to make the catalyst easier to handle and to separate from the reaction medium, as well as possibly stabilize and/or modify the catalytic performance. However, the conventional microporous supports, including zeolites, fail as possible host structures, due to the incompatibility between the small pore size and large dimensions of catalytically active macromolecules. Discovery of mesoporous molecular sieves with well-defined pore sizes of 2 to 50 nm opened new opportunities in the area of immobilization of organometallic macrocycles. Our recent works demonstrated that Al,Si-mesoporous molecular sieves represent an extremely interesting class of supports for cationic metalloporphyrins, allowing for a strong, electrostatic binding of the species and, what is of immense importance in catalysis, for tailoring of the catalyst selectivity. The talk will present the examples of catalytic oxidation of cyclic alkenes and alkanes by metalloporphyrins supported on mesoporous aluminosilicate solids of HMS, MCM-41, FSM and SBA-15 types. In particular, it will be demonstrated that the catalyst selectivity is modified by constrained space around the metalloporphyrin centre located within the internal system of nanosized pores. Catalytic results imply that a simple reaction, such as oxidation of cyclohexene over metalloporphyrins supported on mesoporous aluminosilicates, may be used to probe the spatial distribution of Al sites within the silica framework. Quantum chemical calculations, shedding light on the mechanism of the observed steric effects, will be presented.

10:00 Oral

Mesoporous organosilica containing niobium for environmentally benign oxidation reactions with hydrogen peroxide

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A synthetic protocol for the preparation of a variety of high-quality periodic mesoporous organosilicas containing niobium (Nb-PMOs) is presented. The nanostructured Nb-PMOs have been synthesized either by the hydrolysis and condensation of bridged silsesquioxane precursors ($(EtO)_3Si\text{-}R\text{-}Si(OEt)_3$,R - ethylene or octylene) or by the co-condensation of tetraethyl orthosilicate and organosilanes. Functionalized materials were prepared by using precursors containing an incremental increase in methylene [$(-CH_2-)_n$, n = 2-8)] or vinylene or phenylene groups. Nonionic or cationic templates were employed as the structure directors using the surfactant approach. The evidence for the successful preparation of the Nb-PMOs was based on X-ray diffraction and transmission electron microscopy (high periodicity), nitrogen adsorption (high surface area, monomodal pore size distribution), DRUV-Vis (Nb in framework band) and FTIR spectroscopies (C-Si band). As the Nb-PMOs are expected to find applications as a water-tolerant solid catalyst for those reactions requiring weak acidic sites and low temperatures, they were tested in the oxidation reactions (epoxidation and hydroxylation). The increase in the chain length of precursor was a contributing factor for the increased hydrophobicity and thus a better selectivity. It is interesting to note that all catalysts with the bridged or functionalized framework exhibited higher activity than the pure niobiosilica catalyst, although they had similar structural/textural properties.

10:15 Oral

Highly Ordered Mesoporous Silicon Carbide - a New High Temperature Stable Catalyst Support

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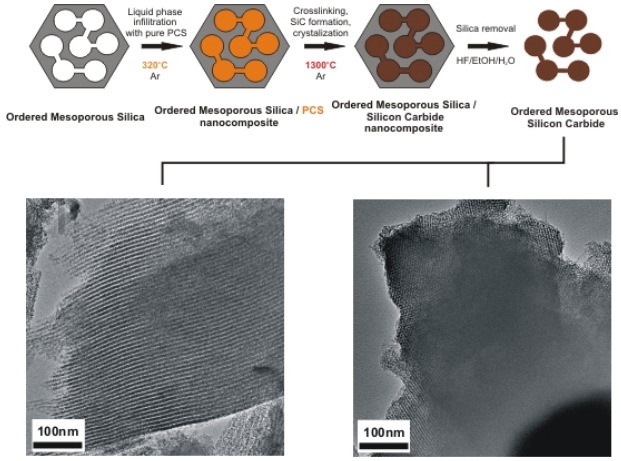
Ordered mesoporous materials which were first discovered in 1992 are promising catalysts and catalysts supports.[1] They posess high surface areas (up to 1400m^2g^{-1}) and precisely defined pore structure (diamater and 3D arrangement). Here we present how a new high surface area hexagonally ordered mesoporous silicon carbide (SiC) supports are prepared via nanocasting of the ordered mesoporous silica.[2-4] Chemical vapor deposition from simple silanes,[2,3] autogenic pressure reactions[3] and polymer precursors infilitration[4] were used to prepare SiC materials with highest surface areas reported untill now (up to 830 m^2g^{-1}) and ordered mesopore structure. Moreover these materials are thermally stable up to 1300^{o}C and have high thermal conductivity. These properties of SiC supports makes them especially interesting in reactions where hot-spot formation is to be avoided and high temperature stability of support is required.[5]



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Coffee break
Thursday morning, 7 September, 10:30

Parallel Session
Thursday morning, 7 September, 11:00
Chair: Ewa M. Serwicka

11:00 Invited oral

How precipitation techniques can be useful for catalysts preparation ?

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Oxide particles can be obtained by various precipitation methods. The reaction of a metallic precursor with a base can be monitored by chemical, physico-chemical and process parameters, with variable impact on the nucleation/growth/agglomeration stages. Parametric studies are the experimental tools for developing kinetics models explaining the observed behaviors and allowing in some cases a fine control or tuning of size and monodispersity.

Some examples will be given. Thermal hydrolysis of acidic titanyle precursors leading to titanium dioxide (anatase) monodisperse particles, generally dense and small agglomerates of nanoparticles, was studied in a batch reactor and the effects of temperature, concentrations and seeding were analyzed to lead to a precipitation kinetics model. The coupling of complexation and template agents can allow a fine tuning of monodisperse nanostructured nickel hydroxide cylinders thanks to slow and homogenous precipitation by decomplexation; the particles can be calcined into NiO with size, morphology and nanostructure retention. The perturbation of a dissolution-reprecipitation phase transformation by external action (addition of a new compound, pH or temperature jump) may also lead to final particle size tuning as will be shown in the case of magnetite. The final objects may be dispersed submicronic or nanoparticles or, as mainly observed with homogenous precipitation, multi-scale objects. This multi-scale objects may result from the use of some agents as surfactants as illustrated by nickel hydroxide/oxide, but they may also result from agglomeration and/or oriented attachment without templates as in the case of thermohydrolysis of titanyl compounds. Hence such techniques can be used for the making of new architectures for catalysts.

11:30 Invited oral

Modified siliceous mesostructured cellular foams - effective catalysts for applications in liquid phase processes

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Advantages and potentials of application of silica mesostructured cellular foams (MCFs) as catalysts and catalyst supports in liquid phase processes are discussed. Due to quite open structure (V_p ca. 2.5 cm^3g^{-1}), large specific surface area (up to 1000 m^2g^{-1}) embedded in cage-like large mesopores (20-40 nm) interconnected by 10-15 nm windows these materials appear to be exceptionally suited for catalytic applications in liquid phase processes. MCFs functionalised with: aminopropyl, aminoethylaminopropyl and glicydoxypropyl groups were found to be very effective carriers for enzymes (invertase, glucoseamylase), far superior to the conventional silica gels and also Eupergit C, a specialty enzyme polymeric support. MCFs grafted with sulfonic acid groups showed activity similar to that of sulphuric acid in the reaction of phthalic anhydride esterification, whereas those post-synthetically treated with titanium alkoxide appeared to be the most efficient amongst a number of catalysts tested in the selective oxidation of large organic molecules using hydrogen peroxide. The latter was due to the presence of numerous site-isolated titanium species in tetrahedral coordinated form. Properties of the materials obtained were investigated using nitrogen adsorption method, IR and UV spectroscopy, thermogravimetry and two step ammonia adsorption to determine the specific properties standing behind the unique behaviour of MCF-based catalysts.

12:00 Oral

Application of ordered mesoporous supports as model systems for studying the effects of catalyst preparation on the ultimate metal particle size.

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Recent research has shown a large impact of cobalt particle size on the activity and selectivity in the Fischer-Tropsch (FT) synthesis. The best performance was reported for particles of 6 nm. Preparation of such catalysts demands full control over the effects of the individual preparation steps on the dispersion and particle distribution. In this respect, we explored the use of ordered mesoporous supports as model systems. Because of the well-defined pore system detailed information can be derived from 3D-TEM and N_2-physisorption. With 3D-TEM the particle size and distribution over the support can be monitored, while with physisorption the degree of pore blocking

by the active phase or its precursor can be quantified.

We studied the preparation of cobalt on silica catalysts for FT synthesis, prepared by impregnation and drying using cobalt nitrate as precursor salt. Generally, catalysts obtained via this method display a poor dispersion, but the reason for this is not yet clear. Using SBA-15 we found that cobalt nitrate was evenly distributed over the support after drying. However, during calcination in air severe precursor redistribution and particle growth took place. Because of the use of SBA-15 as model support we were able to identify that calcination in a diluted stream of nitric oxide prevented redistribution. This enabled us to obtain mono-disperse and homogeneously distributed cobalt oxide particles of 5 nm on both SBA-15 and silica gel. Catalytic tests (1 bar, 220 °C) after reduction showed that a catalyst was obtained that combined a high loading (18 wt%) with an excellent activity.

In conclusion, using SBA-15 as model support yielded detailed information on the preparation steps that enabled us to control the cobalt particle size resulting in a highly active FT catalyst prepared by simple impregnation and drying using cobalt nitrate.

Lunch break

Thursday afternoon, 7 September, 12:30

Parallel Session

Thursday afternoon, 7 September, 14:00
Chair: Maya Kiskinova

14:00 Keynote lecture

Poisoning of iron catalyst with sulfur

Walerian Arabczyk, Dariusz Moszynski, Urszula Narkiewicz, Rafał Pelka

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Sulfur has a strong affinity to metal surfaces and acts as a poison of many metal catalysts, blocking active sites on the catalyst surface.
In this paper the effect of sulfur on the activity of identical iron catalysts used in three different processes, namely: methane decomposition, ammonia synthesis and its decomposition, is compared. In all cases the adsorption of sulfur on the catalyst surface decreases the reaction rate. However, the influence of poison on the apparent activation energy of each process and the dependence of the activity on the temperature differ. The observed differences are attributed to the various character of the active sites involved in each process.

14:30 Invited oral

Direct spectroscopic measurements of surface chemical reactivity

Renzo Rosei

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It has been firmly established that the atom-projected d-band center of mass of transition metal systems, is a good descriptor of surface local chemical reactivity. Any surface modification, like alloying, atomic coordination changes and adsorption processes, brings about shifts of the d-band center of mass. Surface Core Levels (SCLs), as measured by High Resolution X-Ray Photoelectron Spectroscopy, shift almost rigidly with the center of the d-band, and provide therefore a measure of local surface reactivity changes. I will present a number of examples which illustrate this paradigm. In particular, I will present SCL shifts measurements originating from Rh atoms, with coordination number varying between 3 and 12, and SCL shifts originating from Rh, Pt and PtRh surfaces, upon adsorption of O, H, N and CO species. I will show that the energy shifts and intensity changes of time-lapsed SCL spectral components, contain information from which the kinetics and dynamics of surface processes can be direcly determined. For each of the presented systems, DFT calculations show that SCL shifts provide a good spectroscopic descriptor of local surface chemical reactivity changes.

15:00 Oral

Impact of the Surface Carbonates on the Interaction between CO and ZnO Nanomaterials

Xinyu Xia[1], Jennifer Strunk[1], Raoul Naumann[1], Yuemin Wang[1,2], Christof Woell[2], Martin Muhler[1]

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The adsorption of CO on ZnO nanomaterials samples after different pretreatment procedures was investigated with microcalorimetry. The interaction between CO and ZnO nanomaterials became much stronger after the adsorption and partial desorption of CO_2. On carbonate-free ZnO surfaces, the CO equilibrium coverage is less than 0.1 micro mol/m^2 for p = 100 Pa, and the differential heat of adsorption (q^{diff}) is only 40 kJ/mol for this coverage (at coverage < 0.05 micro mol/m^2, q^{diff} may be extremely high due to the active sites).

After the adsorption sites on ZnO were partly covered by CO_2 (after CO_2 adsorption, the sample is evacuated at room temperature overnight), the equilibrium coverage for CO reaches 0.8 micro mol/m^2 at p = 100 Pa, and q^{diff} is higher than 60 kJ/mol. The adsorption rate also became faster. This strong adsorption is due to the existence of surface bidentate carbonates on the mostly exposed ZnO (10-10) faces, which increase the Lewis acidity of Zn^{2+} on this faces, as also shown by TPD and HREELS studies on ZnO (10-10).

Corresponding to similar results for CO adsorption on sulphate-doped TiO_2, it can be concluded that these acid anions generally increase the Lewis acidity of the transition metal ions exposed on metal oxides.

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Coffee break

Thursday afternoon, 7 September, 15:30

Friday, 8 September

Parallel Session
Friday afternoon, 8 September, 14:00

Coffee break
Friday afternoon, 8 September, 15:30

Parallel Session
Friday afternoon, 8 September, 15:50

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster

Microcalorimetric Studies to Investigate the Effect of Sn Addition to Elucidate CO Poisoning on Pt-Sn Direct Ethanol Fuel Cell Catalysts

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Better understanding of the interactions of pure and alloy catalysts are pertinent for designing the next generation direct alcohol fuel cell catalysts with improved performance. In this study microcalorimetric measurements of carbon monoxide, hydrogen, oxygen, and ethanol were performed on 5%Pt/γ-Al_2O_3 with Pt:Sn ratios varying between 1:0 to 15:1. The catalysts were prepared by incipient wetness impregnation of $PtCl_2(NH_3).2H_2O$ and $SnCl_2.2H_2O$salts. Microcalorimetric measurements were performed at 323 K by using a Tian-Calvet type heat flow calorimeter (Seteram C-80) connected to a gas handling system and a volumetric adsorption apparatus employing Baratron capacitance manometers in the range of 10^{-4}-10 Torr for precise pressure measurement. The amount of Sn was adjusted to obtain Pt:Sn ratios varying between 1:0 to 15:1. It was observed that the addition of Sn caused the loss of adsorption sites on (111) planes more severely than on (100) planes of the bimetallic clusters as monitored by CO adsorption differential thermograms. For hydrogen an opposite trend was observed, probably caused by chemisorption in subsurface layers of (111) planes. Alloying effects could be observed from the initial heats of adsorption of all of the adsorbates. The saturation coverages of all of the adsorbates decreased with increasing Sn loading while the defect site concentrations were not influenced much. Microcalorimetric studies on carbon supported Pt-Sn and Pt-Ru bimetallics will also be presented.

17:20 Poster B-1

Synthesis Of Polyethylene With Various Morphologies Using Cp_2ZrCl_2 Supported On Pure and Al-containing MCM-41.

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In recent years, several reports have shown that polymerisation catalysts supported on mesoporous silicas are able to produce polyolefins with unusual morphological properties. Nevertheless, the method of catalyst preparation, the chemical composition of the support and the experimental conditions used, were not always clearly correlated with the catalyst activity and polymer morphology.

In this work, polyethylene was synthesised using zirconocene supported on pure siliceous MCM-41 and Al-containing MCM-41 variants. The mesoporous supports were characterized by powder X-ray diffraction and nitrogen adsorption. The heterogeneous catalytic systems were prepared using controlled Cp_2ZrCl_2 loads below the maximum of adsorption limit for each mesoporous support. The metallocene was present in the polymerisation reactor in known amounts. This procedure allowed a quick and direct comparison of the performances of the various catalytic systems in selected experimental conditions. It was observed that the Si/Al ratio affects the zirconocene load and the corresponding catalytic activity. Small Al amounts greatly improve the polymerisation activity of the catalytic system, but high Al levels become detrimental. On the other hand, modification of supports, by prior impregnation with MAO modifies the surface properties, leading to a change on polymerisation activity.
SEM analysis showed different polyethylene morphologies depending greatly on the catalyst support and the experimental conditions used. Polyethylene nanofibers were clearly observed for the polymers synthesised with low activity catalytic systems. The DSC thermograms for the polymers synthesised with heterogeneous and homogeneous catalysts were compared.

17:20 Poster B-2

Structure studies on nanocrystalline MgO powders prepared by sol-gel method dried under different conditions

Grzegorz Dercz[1], Krystian Prusik[1], Lucjan Pająk[1], Roman Pielaszek[2], Janusz Malinowski[3], Wojciech Pudło[4]

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The structure studies were performed on tree nanocrystalline MgO powders prepared by sol-gel technique, which produce high purity, chemically homogeneous materials with relatively high specific surface areas. Magnesium methoxide was used as MgO precursor. The wet gels were dried under different conditions to prepare a hydrated form of MgO. The applied dry conditions yielded two aerogels and one xerogel with periclase phase, the only crystalline form of magnesium oxide.

The X-ray diffraction and scanning electron microscopy were used as the tools of structure analysis. The Toraya PRO-FIT procedure and Rietveld refinement method were applied at X-ray data analysis. PRO-FIT procedure enables the determination of parameters of individual diffraction lines and applies Pearson VII function for the description of line profiles. This function appeared to be the most useful also at Rietveld refinement procedure.

The average crystallite size of the studied MgO powders estimated from Scherrer formula was in the range 5.5 - 7.5 nm. The crystallite size distributions were also determined using *FW(1/5* and *4/5)M* method proposed by R. Pielaszek. The obtained at Rietveld refinement R_{wp}, R_{exp} and *S* fitting parameters, seem to be satisfactory due to the nanosize of MgO crystallites and since the presence of small amount of amorphous phase is very probable.

17:20 Poster B-3

Effects of Organic Modifiers on Mesoporous Silicas

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Mesoporous silicas are high surface area materials that are promising candidates as supports for heterogeneous catalysts. It is often found that the surface properties of the silica i.e. hydrophobicity, have a significant effect on the pathways that occur in reactions. Here, we examine the addition of methyl groups to the surface of mesoporous silica. The results show that the mesoporous structure is maintained after the addition of organic groups, and that a lattice contraction is observed with increasing modifier content.

17:20 Poster B-4

Zeolite films prepared by the Langmuir-Blodgett technique

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The syntheses of high quality zeolite thin films often involve arrangement of preformed zeolite seed nanocrystals onto a surface followed by crystallisation. The quality of the resulting films depends on the properties of the seed layer and efforts have focused on developing techniques for depositing improved seed layers. Methods such as dip/spin-coating, electrostatic, electrophoretic and ultrasonic deposition have been reported in the literature. Here, we report on the arrangement of preformed Silicalite-1 crystals on silicon wafers by the Langmuir-Blodgett method.

Silicalite-1 crystals with a size of about 100 nm were prepared from clear synthesis solutions containing tertaethoxysilane, tetrapropylammonium hydroxide and distilled water by hydrothermal treatment at 100ºC for 24 h. The zeolite suspension (ca. 1 wt.% concentration) was used after purification by three-time centrifugation and redispersion in distilled water. A film of cationic surfactant was prepared in a Langmuir-Blodgett trough by spreading a solution of octadecylamine (ODA) in chloroform on water subphase containing NaCl. Silicalite-1 suspension was then spread directly onto the subphase covered with a layer of octadecylamine. The influence of Silicalite-1 amount, equilibrium time of interaction between Silicalite-1 and ODA, subphase pH and ionic strength was studied by recording the pressure-area isotherms. Langmuir-Blodgett films were transferred to silicon wafers precleaned in piranha bath under different surface pressures. Denser Silicalite-1 films were prepared by steaming the seeded silicon wafers in autoclaves at 150ºC. The Langmuir-Blodgett seed films and the films after steaming were studied by scanning electron microscopy, atomic force microscopy, X-ray diffraction and attenuated total reflection.

17:20 Poster B-5

Novel molecular catalyst candidates for H_2 evolution from hydrogen storage materials

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Thermal stability in a strongly reducing environment of Ni(II) and Cu(II) sulphate complexes with cyclam (1,4,8,11-tetraazacyclotetradecane), or its N-methylated derivatives, was examined using TGA and DSC techniques. Samples were prepared by mechanochemical doping of NaH or $LiAlH_4$ with ca. 2-4 mol % (20-30 wt. %) of a complex. Samples of NaH doped with Ni(II) complexes decompose in two steps. The I-st step corresponds to one-electron reduction of Ni(II) with an evolution of small amounts of hydrogen. During the II-nd step a progressive degradation of the organic ligand takes place. Cyclam complex of $CuSO_4$ - very stable thermodynamically - is very resistant to reduction by NaH (slow degradation is only above 120 ^oC), while its addition to $LiAlH_4$ results in a decrease of the temperature of the first step of alanate's decomposition (by ca. 30 ^oC). These results suggest that a range of catalysts for H_2 evolution from hydrides might now be expanded, and include cations of electropositive metals (provided the presence of strongly chelating ligands). Complexes studied here form molecular crystals which are much easier to disperse (at a molecular level) wihin a hydrogen storage material, as compared with commonly used extended solid catalysts (TiO_2, SiO_2, $FeCl_3$). This allows to reduce time for the high-energy milling from days to less than a minute, and to make use of every single metal center as a catalytically active species. The drawback of molecular catalysts is in their large molecular mass; this decreases the active hydrogen

content of a H_2 storage material.

I thank for financial support of my participation in E-MRS Fall 2006 from a Nationwide Polish Catalytic Network "Nanomaterials as Catalysts for New, Environmentally Friendly Processes" (WD).

17:20 Poster B-6

Structural peculiarities of nanocrystalline Ce-Sm-O catalysts with Ruthenium modified surface

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In the present work, we report on successful preparation of the Ce-Sm-O/Ru catalysts based on an organic-free modification of the sol-gel technique, since the ceria-based materials are essential as components of promoters in the many catalyst systems. A series of samples with Sm 5-15 % mol content were characterized by TEM, XRD, BET, FTIR, XPS, and ESR. Also, the catalytic properties of some Ce-Sm-O and Ce-Sm-O/Ru samples were studied. We examined powdered Ce-Sm-O samples derived from thermally treated sol-gel products - xerogels (*X*) and precipitates (*P*) of Ce-Sm hydroxides after the ammonia-promoted hydrolysis of aqueous Ce-Sm nitrates. The crystallization of amorphous materials obtained started at 600 ^oC resulting in cubic $Ce_{1-x}Sm_xO_{2-y}$ (x = 0.05-0.15) solid solution. According ESR data after samples Ru-treatment, the concentration of Ce^{3+} centers became 100 times higher for the samples prepared by thermal treatment of xerogels. In contrast, for the samples prepared from precipitates this concentration increased only 50 times. According to XRD analysis, the sudden change of unit cell parameter for $Ce_{1-0.1}Sm_{0.1}O_2$ were observed. This very sample with maximum paramagnetic centers concentration after Ru-containing solution impregnation possesses the highest catalytic activity in methane conversion reaction.

17:20 Poster B-7

Nanocrystalline $Ce_{0.45}Zr_{0.45}La_{0.10}O_{2-y}$ for CH_4 oxidation: characterization and the origin of improved catalytic performance

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Defective structure, high thermal stability and dispersity are the most essential peculiarities of materials for application as redox catalystsit and is very important to develop the methods alloying to control these structural features. We report on preparation of the nanocrystalline $Ce_{0.45}Zr_{0.45}La_{0.10}O_{2-y}$/Ru(Pt) catalysts via organic-free modification of the sol-gel technique, and their characterization by TEM, XRD, DTA, TG, FTIR, EPR, XPS and TPR by CH_4. The incorporation of La(III) into Ce-Zr-O structure leads to stabilization of highly dispersed (4-5 nm) *c*-$Zr_xCe_{1-x}O_2$ solid solution with developed surface up to 1000^oC. La, Ru and Pt doping of the $Zr_{0.5}Ce_{0.5}O_2$ lattice increases the concentration of Ce^{3+} defects detected by ESR. The cause of $Ce_{0.45}Zr_{0.45}La_{0.10}O_{2-\delta}$/Pt-anodes catalytic activity in partial oxidation of dry CH_4 in a SOFC-type reactor will be discussed.

17:20 Poster B-8

Influence of Pd(0) nanoparticles size on the catalytic activity in methoxycarbonylation of iodobenzene

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Recently, palladium nanoparticles have often been considered responsible for the activity of phosphane-free catalytic systems, especially for C-C coupling processes [1]. It has also been proved that palladium nanoparticles are formed during reduction of the $PdCl_2$(cod) complex under carbonylation reaction conditions [2].

Three kinds of palladium colloids were prepared by chemical reduction of an aqueous solution of $PdCl_2$ in the presence of PVP as the protecting polymer. Pyrogallol, chromium (II) acetate, and hydrazine were employed as the reducing agents.

All the obtained palladium colloids are characterized by high activity in methoxycarbonylation of iodobenzene. The size and the shape of palladium nanoparticles, as well as the molecular weight of PVP, play the decisive role from the point of view of their catalytic activity [3].

Oxidative addition of PhI to Pd-PVP as the first step of catalytic reaction was confirmed by TEM and XPS measurements. TEM studies showed a reduction of nanoparticle size after reactions with PhX (X = I, Br) and $[Bu_4N]X$ (X = Cl, Br, I). The formation of $[Bu_4N]_2[Pd(Ph)Br_3]$ or $[Bu_4N]_2[PdBr_4]$ type complexes was evidenced by XPS and UV-vis spectra [4].

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17:20 Poster B-9

Synthesis and characterization of $La_{1-x}Sr_xCoO_{3-d}$ perovskites prepared by spray-pyrolysis

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Perovskite-type mixed oxides have long been recognized as active oxidation/reduction catalysts with potential applications in environmental catalysis. These complex oxides have been studied as potential substitutes of noble metal supported exhaust control catalysts. Among the wide range of samples investigated, Co and Mn perovskites have shown the best catalytic activities and oxidation stabilities. These compounds can modify its standard ABO_3 structure by partial substitution of cations in A or B positions.

In this work we have prepared $La_{1-x}Sr_xCoO_{3-d}$ cobaltites with different Sr^{2+} content ($0<x<0.75$). A spray pyrolysis method has been applied to synthesize the samples. This method is found to be feasible and powders with proper stoichiometry are obtained from nitrate liquid precursors, at lower temperatures than solid methods (i.e. 873 K) after calcination.

Single crystal phases with rombohedral distorted structures are determined by XRD analysis. FE-SEM, TEM, XAS and XPS techniques are also used for bulk and surface characterization of powders. Finally, different temperature programmed combustion experiments carried out with the perovskites have confirmed the high catalytic activity of these oxides towards oxidation processes.

17:20 Poster B-10

Microstructure of Mo-V-Te-Nb mixed metal oxides for selective oxidation of propane to acrylic acid

Almudena Celaya Sanfiz[1], Thomas W. Hansen[1], Frank Girgsdies[1], Rolf Jentoft[1], Olaf Timpe[1], Dangsheng Su[1], Annette Trunschke[1], Robert Schlögl[1], Ming-Hoong Looi[2], Sharifah Hamid[2]

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Natural gas is an abundant natural resource, consisting of mainly light alkanes. The utilization of these alkanes by selective oxidation has been widely investigated, and selective oxidation of propane has been one of the recent challenges. Among the most promising catalysts for this reaction are Mo-V-Te-Nb mixed metal oxides. The preparation method and the calcination procedure seem to be crucial to achieve high catalytic performance. In this work we prepared Mo-V-Te-Nb mixed oxides using hydrothermal synthesis. The phase composition of the crystalline product was controlled by varying the metal stochiometry and the thermal activation procedure. In this way, a series of single-, bi-, and multi-phase materials has been prepared. Samples were characterized before and after thermal activation using XRD, SEM-EDX, TEM, and BET measurements. The materials have been tested in the selective oxidation of propane to acrylic acid. The hydrothermal synthesis of Mo-Te-V-Nb mixed oxide results in amorphous materials with differing chemical homogeneity as revealed by SEM-EDX. During heat treatment, crystallization occurs. XRD and HRTEM indicate the formation of orthorhombic phases, referred to in the literature as M1 and M2, respectively.Other phases observed are M_5O_{14} (M=Mo, V, Nb) and $V_{0.95}Mo_{0.97}O_5$. Phase composition and homogeneity of the heat treated product is related to the composition of the precursor material. The specific elemental composition of the M1 phase is already reflected in fairly homogeneous precursors of single-phase M1 materials, which show improved catalytic performance compared to multi-phase materials prepared by hydrothermal synthesis.

17:20 Poster B-11

Direct synthesis of bulk VPO catalysts by barothermal, solid-state and mechano- chemical reaction

Svitlana Khalameida[1], Volodymyr Sydorchuk[1], Valeriy Zazhigalov[1], Krystyna Wieczorek-Ciurowa[2], Katarzhyna Gamrat[3]

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Vanadium-phosphorus oxide (VPO) catalysts are used in many processes of hydrocarbons oxidation, ammonium-containing VPO for ammoxidation of substituted toluenes. For preparation of more effective VPO catalysts it was investigated alternative approaches. Barothermal synthesis permits to diminish of synthesis duration to 7-10h. Organic compounds are applied in role of reducing agent and solvent. VHP was prepared with following parameters: specific surface area S=5-20 m2/g, relation of intensities of (001) and (220) crystalline planes 1-10, general acidity according to pyridine adsorption C=0,1- 0,33 mmol/g. First solid-state interaction of V2O5, VO2, NH4VO3 and (NH4)2HPO4 was investigated in autoclave without solvent. Ammonium-containing VPO phases NH4VP2O7, (NH4)2(VO)3P2O7 are formed at 200-250C. VHP is crystallized at 300C already for 2 h with size of particles D=21-23 nm. Under higher temperature either structure disorder or formation of intecalated compounds and vanadyl pyrophosphate occurs. The same systems were undergone mechanochemical activation (MChA) in planetary mill with agate grinding (700 rpm) in various mediums. At MChA conditions analogous to barothermal and solid-state reactions can be realized. However phases that are formed under MChA possess some peculiarities namely unusual thermal transformations, higher solubility in water. New VPO phases first of all ammonium-containing appear. MChA of mixture V2O5-(NH4)2HPO4 in water and in air at addition of citric acid obviously leads to creation distorted structure of VHP. Obtained by means of MChA samples have S=2-25 m2/g, D=20-40 nm and C=0.3-3,0mmol/g. All suggested methods allows to prepare catalytic active VPO phases with nanosized crystallites (nanodots).

17:20 Poster B-12

Catalytic activity of supported $LaMnO_3$ for methane oxidation

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$LaMnO_3$ reveals very high catalytic activity for methane combustion. Samples with the highest specific activity had the surface area 20-30 m^2/g and were obtained by nano-technology methods (sol-gel processes, flame pyrolysis and flame hydrolysis). The alternative technique for synthesis of nano-size oxides consists in dispersion of active material on support with high surface area. This procedure is easier and more efficient in comparison with methods of nano-technology. In the present work, we compared catalytic characteristics of $LaMnO_3$ (5-20 mol%) supported on lanthana, Mn_2O_3, MgO and the same parameters of individual substrates. Mn-perovskite formed on support as a result of chemical interaction between carrier and metal oxide(s).The last one(s) formed at heat treatment of powder support impregnated by La and Mn nitrate solutions.

The rate constant for sample $LaMnO_3$/MgO with 5% at 550°C achieved 845 μmol/bar·s·g_{LaMnO3}(845 rc-unites) and exceeded the same characteristic for individual $LaMnO_3$ (20 m^2/g), having the highest catalytic activity (700 rc-unites). It was revealed that increase of concentration leaded to decreasing of the rate constant down to 476 rc-unites as a result of agglomeration of nano-size particles supported $LaMnO_3$. Samples $LaMnO_3/Mn_2O_3$ showed similar behaviour but the values of the rate constant in this case were lower. This fact correlated with support surface area. The experimental data concerned to lanthana in contrast to MgO and Mn_2O_3 showed that activities of supported catalysts and pure support were comparable and there were some difficulties to calculate activity referred to $LaMnO_3$.

17:20 Poster B-13

Field-induced electron density redistribution at carbon surface: many body effects

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Increasing interest to the field emission applications of the carbon nanotubes and other nanosized carbon formations stimulates the theoretical studies of the electron density distributions near the carbon surfaces. In present contribution a new approach is proposed for the calculations of the field-free and field-modified electron density distributions at a semi-infinite carbon crystal using the functional integration method [1]. This approach allows correct considering of the exchange-correlation effects and makes possible the proper field-effect account for broad field ranges without to use the perturbation theory. The electron-ion interaction is described by a nonlocal model pseudopotential of Kleinman-Bylander type.

The results of calculations are compared to the field-ion microscopic measurements of local electrostatic fields in the immediate vicinity of individual surface atoms. The implications of the obtained results on the particular fields of nanotechnology and heterogeneous catalysis, are discussed.

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17:20 Poster B-14

Catalytic activity for methane flameless combustion and thermal stability of nano-sized lanthanum cobaltites doped with Ce, Pr and Tb

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High surface area nano-sized catalysts with general formula $La_{0.9}M_{0.1}CoO_3$ (M=Ce, Pr, Tb) were synthesised by a novel flame-spray-pyrolysis method, based on spraying a solution of the corresponding metals salts (acetates in the present case, dissolved in propionic acid), together with oxygen, in a nozzle burner, where the mixed oxides particles form, followed by collection of the latter by means of an electrostatic precipitator.

All the prepared samples possessed the $LaCoO_3$ perovskite-like structure and consisted of 30-60 nm particles, lumped into larger (80-200 nm) agglomerates. Their surface area ranged from 45 to 60 m^2/g. All of the catalysts showed a very high activity for the methane flameless combustion, attaining 100% conversion at a temperature T_f (temperature of full conversion) ranging between 495 and 515°C, depending on the nature of the M doping ions. The results of life-tests did not show any decreasing of catalytic activity after 50 h under reaction conditions at T_f. Thermal stability under high temperature exploitation was investigated by overheating the catalysts two times for 1h at 800°C in flowing reacting gas mixture (fast deactivation cycles) and then measuring again methane conversion at T_f. All the catalysts showed a more or less considerable decrease of activity, depending on chemical composition. For example, conversion after the second cycle decreased from 100% down to 80% for $LaCoO_3$ and to 48% for $La_{0.9}Tb_{0.1}CoO_3$. Taking into account the results of SEM analysis, this was attributed to sintering, connected with Gibbs instability of nano-sized materials.

17:20 Poster B-15

Spectroscopic and magnetic properties of gadolinium macroacyclic and macrobicyclic complexes

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The macroacyclic and macrobicyclic complexes are able to select charged and neutral molecules; based on this concept they are often used in the activation and catalysis processes. These kind of complexes arising as an effect of adding of lantanide ion to the ligand, being also homogeneous catalysts, are often applied, e.g., as electron transfer agents in cleavage of DNA and RNA. They can be conjugated to RNA or DNA oligomers forming some kind of artificial enzymes. Undoubtedly, these specific artificial enzymes could be essential tools for biotechnology in the future.

Current interest is focused on the design of new active catalysts that one can also easy undergo to derivatization. The ideal catalyst should possess one or more metal ions in the active site. Lanthanide ions that are able to cut nucleic acids, can form, moreover, a variety of coordination compounds in aqueous solution.

In this paper discussion on the structure and interactions between donor lantanide ions inside the podants and cryptates is presented. The analysis of EPR and IR spectra of gadolinium macroacyclic and macrobicyclic complexes was used to deduce the environment of the host metal site in the terms of local symmetry and coordination number.

I thank for financial support of my participation in E-MRS 2006 given from a Nationwide Polish Catalytic Network" Nanomaterials as Catalysts for New, Environmentally Friendly Processes" (Grzegorz Leniec).

17:20 Poster B-16

The Study of Growth Mechanism of Gold Nanorods

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A new approach to fabricate long length of gold nanorods by controlling the volume of growth solution will be reported. The shape evolutions ranging from fusiform nanoparticles to 1-D rods were observed. Increasing the addition of growth solution can control the length of nanorods. The length of rods can be extended to 2 μm, and nanorods with aspect ratios of up to ~ 70 could be obtained. Moreover, X-ray absorption spectroscopy (XAS) is applied herein to elucidate the growth mechanism of gold nanorods. The gold ions were directly reduced to gold atoms by ascorbic acid during the reaction, and then gold atoms were deposited on the surface of gold seeds that were introduced into the reaction. Extended X-ray absorption fine structure (EXAFS) confirmed the growth of gold and the environment around Au atoms over the reaction. The XAS are expected to have wide applications in the growth of gold and other related materials.

17:20 Poster B-17

Comparative Study of Different Preparation Routes for Obtaining Nanoporous Titania Nanocrystals

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The preparation of titania (TiO_2), which owing to its photoactivite properties is a very promised material also for catalysis, was studied. The work was focused on the mutual correlation between individual preparation steps and tailoring of titania nanostructure.

The texture of prepared titania powders (specific surface area, micropore volume, pore-size distribution(PSD)) was evaluated from adsorption-desorption nitrogen isotherms by application of determined titania standard (master) isotherm. Another textural information was obtained via high-pressure mercury porosimtry, helium pycnometry, SEM images and XRD phase analysis.Three different preparation routes of nanoporous titania powder were chosen: a) surfactant-mediated sol-gel synthesis controlled in inverse and lamellar configuration [1],

b) reaction of liquid metal alkoxide with H_2O_2 [2],

c) acid hydrolysis of titanium(IV) chloride [3].

In all cases the correlations between the change of reaction conditions (e.g. different length of a polar chain in the surfactant molecule, different types of metal alkoxides, different extent of hydrolysis, replacement of organic solvent in micellar system, different molar ratio of water/alkoxide and water/surfactant, different reaction temperature, changes of calcination temperature and calcination time) and position and width of TiO_2 PSD were found.

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17:20 Poster B-18

Calcined NiMgMn LDH Modified with Noble Metals in Total Oxidation of toluene

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Volatile organic compounds (VOCs) in industrial gases represent a serious environmental problem. The VOCs in the air can be reduced applying the process of total catalytic oxidation. Catalysts with noble metals are currently preferred as they are highly active and stable but expensive. Metal oxide alternatives are cheaper but less stable. Combination of both groups of active materials could give highly active catalysts with sufficient stability. In our previous study we found high activity of the calcined NiMgMn LDH in total oxidation of VOC in air. Now we attempted to improve its catalytic properties by introducing of small amount (0.1 wt%) of noble metals from different precursors and to reveal their effect on physical chemical properties and the activity in total oxidation of toluene. The Ni-Mg-Mn LDH precursor was prepared by coprecipitation. The calcined product was either impregnated by solutions of various noble metal salts or by their microemulsions. The catalysts were characterized by S_{BET}, XRD, TPR and XPS. Activity in toluene total oxidation was examined. A well-crystalline Ni-Mg-Mn LDH was obtained after precipitation, with small fraction of crystalline $MnCO_3$. After calcination, mixed oxide phases were formed. XPS analysis showed enrichment of its surface by Ni and Mn (surface concentrations Ni:Mg:Mn-41:11:48 at.%). Temperature T50 (corresponding to 50% toluene conversion) was 153°C and after 15 h increased to 180°C. Modification of the parent catalyst with noble metals did not change initial catalytic activity too much (T50 152+-2°C). However, substantial difference was observed in catalyst stability. After 15 h examination, the T50 value for the NiMgMn catalyst (180°C) decreased for the modidied catalysts in the following order: AgNO3 (174), Pd(NO3)2 (170), H2PtCl6-Me (170), (NH3)4Pt(NO3)2-Me (166). Acknowledgement This work was supported by the Grant Agency of the Czech Republic (grants No. 104/04/2116 and GD203/03H140).

17:20 Poster B-19

Synthesis of porous manganese oxides and carbon monoxide oxidation with them

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Manganese compound, todorokite, having one dimensional microporous structure is known. Todorokite is made up with MnO_6 octahedra connected to each other by its corner and edge. The rectangular pore mouth consists of 3x3 array of the MnO_6 unit and its pore size is 0.69 nm x 0.69 nm. Although todorokite is expected as a functional material for catalysts or battery materials, not many minute researches on the synthesis are reported so far. We studied the synthesis, characterization of todorokite and its catalytic performance for carbon monoxide (CO) oxidation.

Several todorokites were synthesized by changing the aging time during the synthesis of its precursor, birnessite, of layered structure. Then, the birnessite was ion-exchanged to buserite and the buserite was finally transformed to todorokite by the hydrothermal treatment. The products were analyzed by several methods such as XRD, Iodometry, FE-SEM and TEM. CO oxidation was studied using a conventional fixed bed reactor.

It was revealed from XRD that crystallinity of todorokite depended on the aging time of birnessite and high crystallinity necessitated aging for 3 to 7 days. Primary particle size of todorokite calculated by Sherrer equation increased with the aging time to about 25 nm. The valence of manganese in todorokite coincided with that in birnessite and was almost constant irrespective of aging time between 5 min and 7 days. The morphology of todorokite observed by FE-SEM changed from amorphous for shorter aging time to plate-like structure for longer aging time. On the other hand, TEM revealed that even in 5 min aging, small plate-like particles of birnessite were observed together with dominant amorphous particles. CO oxidation was studied as a model reaction. Higher CO oxidation activity was observed on the todorokite obtained by aging birnessite for 3 hrs. The relation between the activity and crucial properties of todorokite will be discussed.

17:20 Poster B-20

Catalytic decomposition of ethylene on nanocrystalline cobalt

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Nanocrystalline ferromagnetic metals (such as Fe, Co and Ni) have many applications in magnetic data storage, magnetic toners in xerography, magnetic inks and ferrofluids. However, nanocrystalline metals are very sensitive to rapid environmental degradation. This attribute significantly limits the scope of their potential industrial applications. Encapsulation of nanocrystals with graphitic layers is a way to protect metal crystals from negative environmental influence. Also, carbon coatings can endow these particles with biocompatibility and make carbon-encapsulated particles useful in bio-engineering applications, such as drug delivery, biosensors, magnetic hyperthermia and magnetic contrast agents for magnetic resonance imaging.

Carbon-coated cobalt nanoparticles were successfully synthesized by catalytic ethylene decomposition. Nanocrystalline cobalt was prepared by cobalt hydroxide precipitation, followed by calcination and reduction. A small amount of structural promoters was added. The process of cobalt oxide reduction and cobalt carburisation was carried out in a thermobalance. The carburisation process was performed under pure ethylene flow in the temperature range 340 - 400°C. After carburisation process the samples were cooled under helium flow or reduced under hydrogen flow in the temperature

range 500 - 560°C. The phase composition of the samples was determined using XRD technique. The phases in carbon-coated nanocapsules turned out to be fcc-Co and hcp-Co. The HRTEM method was used to study the morphology of the samples after carburisation. Both carbon-coated cobalt nanocapsules and carbon nanotubes were found in the samples.

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17:20 Poster B-21

Iron-carbon nanofillers for polymers

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Iron-carbon nanofillers for polymer composites were obtained by catalytic decomposition of ethylene on nanocrystalline iron. The carburisation was performed under ethylene/hydrogen mixture or under pure ethylene. Three kinds of samples were prepared: cementite/carbon, iron/cementite and iron/carbon. After carburisation the samples were characterised using XRD and SEM method. The obtained nanofillers were applied for polymer nanocomposites prepared by polycondensation reaction (*in situ*) in poly(ether-ester) matrix. The nanofillers were dispersed in monomers (diols) using sonificator and a high - speed rotary mixer. The obtained nanocomposites were characterized using SEM method. The addition of iron-carbon nanofillers increases the stress-strength of composite (ca. 30% at break) and the elongation at break.

17:20 Poster B-22

Microstructural and Auger Microanalytical Characterization of Cu-Hf and Cu-Ti Catalysts

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Degradation processes occurring at the surface and in the bulk of Cu-based amorphous alloys during cathodic hydrogen charging were used for promoting the catalytic activity of such alloys. These processes modifying the structure, composition, and morphology of the substrate proved to be useful methods for transforming Cu-Hf and an inactive Cu-Ti amorphous alloy precursors into active and durable catalysts. Indeed their catalytic activity for dehydrogenation of 2-propanol increased up to a conversion level of ~60% at selectivities to acetone of about 99% for Cu-Ti and to conversion of ~90% at selectivities of ~95% for Cu-Hf. Previous attempts carried out by ageing in air or hydrogen charging from the gas phase, resulted in a maximum conversion level up to 15% for Cu-Hf and up to 3% for Cu-Ti. High resolution Auger spectroscopy allowed changes occurring during the activation process to be identified, namely, the formation of small Cu particles on the HfO2 surface and the formation of highly porous particles containing mostly Cu and some Ti and O (Cu-Ti-O) on a Cu-Ti substrate. Differences in the chemistry and structure of both catalysts are discussed, and the implications for catalytic function are considered. A probable configuration of active sites on the Cu-Ti-O/Ti-O-Cu catalyst for dehydrogenation of 2-propanol is proposed.

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17:20 Poster B-25

Silver Clusters in Molecular Sieves

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Molecular sieves have unique abilities to stabilize small metal clusters which are the active sites for many catalytic processes. The most important question concerning metal clusters is their size and structure. For characterization of small paramagnetic clusters of magnetic nuclei electron paramagnetic resonance (EPR) spectroscopy is a method of choice.

For the last few years we had been characterizing by EPR cationic silver clusters in different zeolites reduced by gamma irradiation. Depending on Ag^{+} loadings we identified Ag_3^{2+} trimers and Ag_6^{n+} hexamers in dehydrated AgNa-4A zeolites and Ag_8^{n+} in AgNa-sodalites. In AgCs-rho zeolite at low temperature Ag^{0} atoms and $Ag2^{+}$ dimers are observed. After annealing to RT Ag_3^{2+} trimers and Ag_4^{3+} tetramers are stabilized. When irradiated AgCs-rho containing Ag_4^{3+} clusters is exposed to ammonia the hyperfine splitting decreases due to shift of spin density from Ag nuclei to the ammonia ligands.

The EPR study on silver zeolites clearly showed two different mechanisms of silver agglomeration. In zeolite rho agglomeration process initiated by Ag^{0} formation involves the reaction of silver atoms, dimers and trimers with silver cations to form Ag_4^{3+}. In zeolite A and sodalite pre-existing assembly of Ag^{+} cations traps electron during radiolysis forming Ag_6^{5+} or Ag_8^{7+} clusters, respectively.

In mesoporous molecular sieves like MCM-41 silver nanoparticles are formed inside channels showing characteristic singlet of conduction electron spin resonance (CESR). It was proved experimentally that the CESR linewidth depends on cluster nuclearity.

17:20 Poster B-26

Characterization of Nd-MCM41 obtained by impregnation

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The mesoporous silica MCM41 has properties such as a high specific surface area, tuneable pore size and a narrow pore size distribution. Rare earth oxides supported MCM41 is a promising optically functional material. MCM41 was synthesized adding the silica precursor tetraethoxysilane to an aqueous solution containing cetyltrimethylammonium bromide and hydrochloric acid. The obtained solid was calcined at 600°C for 4h. The impregnation of MCM41 with neodymium was conducted by incipient wetness method, using neodymium nitrate solution with different Nd/Si weight ratio, followed by calcination at 550°C for 5h. Samples were characterized by several techniques. The presence of neodymium was confirmed by Energy Dispersive X-rayanalysis. X-Ray Diffraction analysis indicated that the unit cell parameter a_0 increase with Nd/Si weight ratio, but MCM41 structure is not disrupted by the neodymium presence. The specific surface area decreases from MCM41 to Nd-MCM41 with increasing of neodymium content. It is probably due to the partially blocking of the mesostructure pores during impregnation.

17:20 Poster B-27

New method of carbon nanofibers synthesis for nanocatalysis applications

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Due to their strong metal/support interaction, their high specific surface area and the absence of pores reducing the diffusion the carbon nanofibres (CNFs) are considered to be promising nanosized catalytic support materials [1]. In present contribution a new method of CNFs synthesis based on a catalytic pyrolysis of butane-propane mixture is proposed. Individual 3d-metals as well as their binary mixtures and alloys were used as catalysts. The CNFs synthesized in this way have the diameters of 8 to 80 nm, and the lengths up to 1 micrometer. All the fibres have curved shapes and the amount of inclusions does not exceed 2-3% after appropriate purification. Additionally to the nanocatalysis application, the use of the CNFs in the field emission cathodes and in the field ion sources is proposed and discussed.

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17:20 Poster B-28

Catalytic properties of polypyrrole-platinum nanocomposites

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In the work polypyrrole (PPy) - Pt nanocomposites have been obtained by: 1) doping of PPy with Pt ions in PtCl4 aqueous solution followed by reduction with NaBH4, 2) reduction of Pt ions from PtCl4 aqueous solution with NaBH4 in the presence of PPy, 3) incorporation of Pt particles into PPy from Pt aqueous sol. The prepared composites have been characterized by X-ray diffraction as well as scanning and transmission electron microscopies. This has allowed us to establish that in all the materials prepared, crystalline metallic Pt phase has been present. Size of the crystallites depends on the method of synthesis: the smallest ones (4-10 nm) have been in the composites prepared by method 3. Catalytic properties of PPy-Pt nanocomposites have been investigated using isopropyl alcohol conversion as the test reaction. It has been found that PPy-Pt nanocomposite prepared by method 3 has shown the highest activity, giving mainly the redox product, i.e. acetone. In the presence of this nanocomposite cyclohexene oxidation has been also investigated. This process leads predominantly to the formation of the epoxy compound. *Agnieszka Sniechota thanks the Scientific Network: "Nanomaterials as Catalysts for New, Environmentally Friendly Processes" for financing her participation in E-MRS Fall Meeting 2006, Symposium B.*

17:20 Poster B-29

Gold supported on ceria and ceria-alumina promoted by molibdena for complete hydrocarbons oxidation

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The detoxification of the hydrocarbons pollutants is one of the global environmental problems. Considerable efforts have been made to design the highly efficient catalysts for complete oxidation of hydrocarbons. Nanosized gold supported catalysts are widely used as active catalysts at low temperatures in a lot of important reactions. The interesting properties of ceria as a support for the noble metals catalysts are well known to concern mainly its function as oxygen buffer. Efforts to increase the oxygen storage capacity by introducing of different cation dopants with valence lower than 4+ have been put forward. Most of these materials have shown promotion of both: oxygen vacancy concentration and oxygen storage capacity as well

as redox activities, compared to undoped ceria. An accent was put also on the synthesis of nanosized ceria as an important factor for the preparation of highly active gold supported catalysts.The object of this study is to synthesize new gold catalysts supported on ceria and ceria-alumina non-promoted and promoted by molybdena for complete benzene oxidation. The catalysts were characterized by means of XRD, TPR, XPS and Raman spectroscopy. High and stable catalytic activity was established in the temperature region 200-2400C. The presence of gold causes a modification in ceria structure leading to an increase of Ce3+ and oxygen vacancies formation. The differences in the activities within the temperature range 150-1800C and in the region of 100 % conversion (200-2400C) could be explained by supposing that in the LT region the electron transfer between nanosized gold and ceria particles via oxygen vacancies has a crucial role. In the HT region the oxygen mobility, provoked by the defective structure of ceria due to the presence of Al3+, becomes of prevailing importance.. It was also concluded that alumina prevents the gold and ceria agglomeration, which is the main factor to avoid deactivation under extreme reaction conditions.

17:20 Poster B-30

Determination and fine tuning of the cerium oxidation state in cerium calixarene complexes

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Interesting structures and properties as well as perspectives of practical use motivated intensive studies of various calixarene complexes in recent years. The development of new catalysts increases also the interest in the design and synthesis of supramolecular metallocalixarene arrays. The novel cerium calixarene complexes [p-tBu-calix[4](OMe)$_2$(O)$_2$Ce(acac)$_2$] (1) and [p-tBu-calix[4](OMe)$_2$(O)$_2$Ce(hfac)$_2$] (2) were recently synthesized in an equimolar reaction between p-tBu-calix[4](OMe)$_2$(OH)$_2$ and cerium complexes Ce(acac)$_4$ (acac = acetylacetonate), and Ce(hfac)$_4$ (hfac = 1,1,1,5,5,5-hexafluoro-acetylacetonate), respectively [1].

The cerium oxidation state in such complexes is discussed intensively, whereas contradictory results are reported [2]. In the present study, X-ray photoelectron spectroscopy (XPS) with monochromatic Al Kα excitation (1486.74 eV) was applied in a "fast transfer" mode [3]. The experiments revealed an irradiation-induced decrease of the Ceox values of both complexes dependent upon X-ray exposure. The detailed study of the dynamical response of the Ce_{ox} to X-ray irradiation allowed the determination of Ce_{ox} at "zero irradiation" [3.60 for complex (1) and 3.65 for complex (2), respectively]. Neither vacuum-induced reduction, nor re-oxidation of the irradiated compounds in air were observed suggesting the possibility of the long-lasting X-ray induced tuning of Ce_{ox} in a range of at least 3.65 to 3.26.

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17:20 Poster B-31

Preparation of VPO-catalysts on supports of different nature

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Vanadium-phosphorus oxide systems (VPO) are catalysts for oxidative transformations of hydrocarbons. Vanadyl hydrophosphate (VHP), precursor of catalyst, is not formed as a rule at deposition of VPO on supports. It was offered following new pathways for preparation of mesoporous supported VPO-catalysts by means barothermal synthesis (BTS) in organic and aqueous medium under 170 C: 1) introduction of nonporous fumed oxides or carbon nanotubes in reaction mixture on various stages of VPO synthesis; this route permits to obtain needed phase and to regulate its porous structure in wide limits; first it was prepared supported VPO-catalysts aerogel type on base of C-nanotubes; 2) precipitation on porous solids of different nature: silica gel, phosphates of Ti and Zr, active carbon; in latter case synthesis was carried out in aqueous medium without reducing agent; 3) deposition on preliminary modified silica with organic grafted layer which is potential reducing reagent at VPO preparation; such supported catalysts have residual organic groups on surface which are thermostable on air to 350 C; synthesis of VPO on oxidized carbon allows to increase both louding of active phase and its dispersity in two times; 4) mechanochemical deposition of ready VHP on fumed oxides and phosphates. All suggested approaches permit to prepare on surface of supports necessary phase of VHP in nanodisperse state; size of crystallites according to XRD data has following values: 33-35 nm for bulk samples, 30 nm for method 4, 23-26 nm for method 1, 20 nm for method 3, 6-15 nm on carbon surface. VHP is formed with various relation for intensities of crystalline patterns of (001) and (220) planes: 0,39-1,69. This relation determines primary surface orientation of planes and hence catalytic properties of VPO in diverse processes.

17:20 Poster B-32

Synthesis and Electrochemical Characteristics of Highly Dispersed Platinum Nanoparticles on Different Carbon Supports for PEMFC

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Commercial XC-72 carbon black, multi-wall carbon nanotube (MWCNT), and bamboo charcoal were used as carbon supports for Pt catalyst for application in proton exchange membrane fuel cell (PEMFC). Due to the unique morphology of carbon nanotube, there are less geometric barriers than XC-72 for deposition of Pt. Accu-

mulation of platinum particles could be decreased by using carbon nanotube as support. Surface treatment on MWCNTs has been studied for many years. In order to get more active sites on the surface of MWCNTs, a sonochemical treatment with sulfuric acid and nitric acid was made. By this process, functional groups such as carbonyl (-CO), hydroxylic (-COH), and carboxylic (-COOH) can be created on the surface of carbon nanotubes. Bamboo charcoal has a very large surface area. There are many pores on the surface to make easier gas diffusion. Chloroplatinic acid was used as the precursor, and ethylene glycol was used as the solvent and reducing agent to get well-dispersed platinum nanoparticles on the carbon supports. The particle size and dispersion of platinum particles are two key factors to determine the performance of catalysts. In this experiment, platinum particle size was controlled by changing the loading percentage of platinum. The deposition of Pt was characterized by X-ray diffraction, thermal gravimetric analysis, and transmission electron microscopy. It was found that platinum particles were well-dispersed on all carbon supports. The average diameter of platinum particles was lower than 3nm. The performance of single-cell PEMFC using the Pt deposited on various supports was evaluated.

17:20 Poster B-33

Magneto-chemical properties of some new Ni and Co tungstates formed as NiRE2W2O10, Co2RE2W3O14 and CoRE4W3O16 where RE=Nd, Sm, Eu,Gd and Er and M=Ni, Co

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Producing H2 from the splitting of water with using oxide-type semiconductors under light irradiation is an attractive way to establish a clean energy system without relying on fossil fuels. Several types of semiconductor photocatalysts such as Bi2RENbO7 (RE=Y, rare-earth) [1], In0.8M0.2TaO4 (M=Ni,Cu,Fe) [2], M-doped InTaO4 (M=Mn,Fe,Co,Ni and Cu) [3] were synthesized by the solid state reaction method. Moreover, a group of mixed oxides with the wolframite structure (ABO4 where A=Ni(II), Co(II), etc, B=W) has been studied for its interesting catalytic properties [4]. These compounds are so-called "mechano-catalytic" materials and are used for overall water splitting. The water splitting process consists in continuously converting mechanical energy into chemical energy with help of mentioned powder as catalytic materials.

In our investigations we have obtained as a result of the solid-state reaction between MWO4 (M=Ni,Co) and RE2WO6 (RE= Nd,Sm,Eu,Gd and Er) new series of compounds with the following formulas: NiRE2W2O10, Co2RE4W3O14 and CoRE4W3O16 [5]. It seems that these nickel and cobalt tungstates with rare earth ions seem to be good catalysts in many processes. In the present work, we presented the results of IR-spectroscopy, X-ray and EPR measurements showing some structural features of the compounds. The Co2RE2W3O14 as well as CoRE4W3O16 compounds crystallize in the orthorhombic system and only the basic crystallographic parameters have been determined up to now. Wide, unstructured EPR powder spectrum of these compounds and their temperature evolution have been analyzed and interpreted. The optimization of the spin-Hamiltonian parameters and EPR data simulation was performed using the software package EPR-NMR.

I thank for financial support of my participation in E-MRS 2006 given from a Nationwide Polish Catalytic Network" Nanomaterials as Catalysts for New, Environmentally Friendly Processes" (Adam Worsztynowicz)

17:20 Poster B-34

Nanosized Pt and Pd containing catalysts immobilised on pillared montmorillonite in n-hexane isomerisation

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The present work is devoted to Pt- and Pd- containing catalysts, immobilised on Al-pillared montmorillonite (Al-MM) in the n-hexane hydroisomerization. Al-pillared montmorillonite was prepared with various ratio of Al^{3+}/clay. Pt and Pd content was equal to 0,35%. Pt and Pd were introduced by impregnation using the H_2PtCl_6 and $PdCl_2$ solutions. The metal species size control was carried out using the electron microscope EM-125K. It have been shown that the Pd- and Pt- containing catalysts with high dispersion of metallic species were obtained. The high dispersion is remained even after treatment at 450^0C. The size of Pt species varies in the range 30-80Å and the majority of this species has the size of 30-40Å. The size of Pd-catalysts species is bigger (50-100Å). Conversion of n-hexane over the Pt- and Pd-catalysts was 58,1% and 29,3% at 350^0C, respectively. Catalysts demonstrated the high selectivity to C_6-isomers at temperatures from 250 to 400^0C. The products of cracking were absent.

17:20 Poster B-35

Temperature dependence of the FMR spectra of polymer composites with nanocrystalline α-Fe/C filler

Nikos Guskos[1,2], Michał Maryniak[2], Janusz Typek[2], Iwona Pelech3, Urszula Narkiewicz[3], Zbigniew Roslaniec[4], Magdalena Kwiatkowska[4]

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Two different concentrations of nanocrystalline material: α-Fe/C have been prepared by carburization of nanocrystalline iron and characterized by using XRD and SEM methods. The nanoparticles

were next applied as fillers in polymer nanocomposites using the polycondensation reaction (*in situ*) in poly(ether-ester) matrix with two different concentrations: 0.1 wt. % and 0.3 wt. %. The temperature dependence of the ferromagnetic resonance (FMR) spectra has been investigated to study magnetic interactions in the compounds. The introduced FMR parameters (intensity and position of the right peak) describe well the temperature dependence of FMR spectra of strongly interacting magnetic nanoparticles. The FMR spectra depend strongly on the concentration of magnetic nanoparticles, which influence the magnetic interactions between them. Two main critical points of the matrix (glass state and freezing of benzene rings) influence the behaviour of the FMR spectra.

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Mondazqs Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium C

Doped nanopowders: synthesis, characterization, applications

Welcome

- Production and applications of nanopowders is an important field of nanotechnology. It was covered by a range of conferences and workshops. Also the fact that the properties of nanopowders are strongly influenced by their shape, size distribution and surface chemistry is well known. In this symposium we want to focus our attention **on doping nanopowders.** Doping nanopowders with rare earth ions, or with transition metals, and other may influence a range of their physical properties as well as their structure. Some questions that arise in this context are:
- Are higher doping levels possible to achieve in nanopowders than in micron sized powders or single crystals of the same material?
- Are the properties of materials made from doped nanopowders different comparing to conventional materials?
- Are solubility limits achievable higher when nanopowders are doped comparing to conventional materials?
- Is doping nanopowders and their consolidation or embedding in a matrix a convenient method to produce **functional** materials?
- How the physical properties of nanopowders are modified with doping?
- How doping influences phase diagrams of nanopowders comparing to bulk materials?
- How to characterize the structure of doped nanopowders?
- Where are the dopands situated, how are they distributed?
- Methods of doping: which are the better methods depending on application?
- Applications of doped nanopowders to make functional materials.
- Safety
- Scale up of production
- Consolidation

Organisers

- Markus Winterer, Nanoparticle Process Technology, Department of Engineering Sciences, University Duisburg-Essen, Duisburg, Germany
- Jianzhong Jiang, Laboratory of New-Structured Materials, Department of Materials Science & Engineering, Zhejiang University, Hangzhou, P.R. China
- Witold Lojkowski (Chairman), Institute of High Pressure Physics, Polish Academy of Sciences, Warsaw, Poland
- Aharon Gedanken, Department of Chemistry, Bar-Ilan University, Ramat-Gan, Israel

- Local organiser: **Janusz D. Fidelus,** Institute of High Pressure Physics, Polish Academy of Sciences, Warsaw, Poland

Proceedings

The manuscripts submitted to this symposium will be reviewed and published in *Solid State Phenomena.*

Sponsors

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Programme

Sunday, 3 September

COST Action D30 WGM/1
Sunday afternoon, 3 September, 13:00

Welcome Reception

Faculty of Physics, WUT
Sunday evening, 3 September, 19:00

Monday, 4 September

COST Action D30 WGM
Monday morning, 4 September, 9:00

Coffee break
Monday morning, 4 September, 10:30

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

COST D30 WGM/2
Monday afternoon, 4 September, 14:00

Coffee break
Monday afternoon, 4 September, 15:30

COST Action D30 WGM/3
Monday afternoon, 4 September, 15:50

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Session Industry

Tuesday morning, 5 September, 9:00

9:00 Invited oral

Properties and applications of doped nano-structured materials in coatings and films

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The use of engineered nanomaterials provides the potential to impart new and improved functionalities into coatings systems, polymer films, and adhesives. The impact and effectiveness of doped nanoparticulate additions into formulations is determined by many factors, including the composition, morphology, crystallinity, refractive index, and surface chemistry of the particles. Along with these particle characteristics, their degree of dispersion in the matrix or formulation is also a critical factor in obtaining enhanced functionality. Additionally, surface treatment and post-processing of the doped nanostructured particles allows for customization leading to compatibility with specific systems. With proper preparation and formulating, systems can be imparted with new benefits such as UV protection, IR filtering, conductivity, accelerated curing, and enhanced mechanical properties. The selection of nanomaterials to provide such features often coincides with a desire for transparency to visible light in the coatings and films, which requires careful engineering of both the particles and the formulations. Such transparent formulations often find their way into high value applications where several functionalities are combined, thereby enhancing the value proposition of the supplier to the potential customer. This presentation will cover synthesis of doped nanomaterials; properties and performance enhancements achieved through doping; customization for specific formulations; proper dispersion into matricies; and examples of applications for multi-functional nanostructured coatings, films, and adhesives.

9:30 Invited oral

Doped nanostructured materials for electronic applications

Frank Martin Petrat[1], Stipan Katusic[2]

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Nanotechnology already plays an important role in our daily life and will form the foundation for revolutionary discoveries and advancements in the decades to come. Nanotechnology will profoundly influence the competitiveness of companies in every important industry. The chemical industry in particular is working on translating the potential of nanotechnology into products.

There are several parameters to improve product performance of nanostructured materials e. g. aggregate structure, primary particle size and surface chemistry. Doping is a common method to influence the properties of bulk semiconductor materials. Doping is a key step in semiconductor industry to introduce traces of impurities in order to align the electronic properties of semiconductors. While doping of bulk materials is well understood there is little known about the properties of doped nanoparticles.

Examples will be given for doped nanoparticles with relevance to electronic and electrochemical applications. Indium tin oxide (ITO) is an important material for photovoltaic application. We present results on indium tin oxide doped with platinum. The morphology of the material as well as its performance in dye sensitized cells (DSC) will be discussed. Another example covers doping of nanoscaled silicon particles. These particles are of special interest in printed electronics as they offer a potential route to formulate semiconducting inks for low cost printed circuits.

10:00 Invited oral

Nanoparticle Characterization (Size and Zeta Potential) Using Novel Light Scattering Techniques

Michael Kaszuba[2], Adam Próchniak[1]

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Particle size and zeta potential are important parameters for the characterization of nanoparticles. The technique of dynamic light scatteringis well suited to the measurement of the size of nanoparticle dispersions. Traditionally, measurement of very small and/or poorly scattering particles or samples that are very dilute were difficult unless high powered lasers were used. These problems have been addressed in a light scattering instrument incorporating novel non-invasive backscatter optics (NIBS®). This novel optic arrangement maximises the detection of scattered light while maintaining signal quality.

Laser Doppler electrophoresis is an accepted method for the measurement of particle mobility and zeta potential of dispersions of colloidal size materials. A novel sheme using a combination of Phase Analysis Light Scattering (PALS) and a pathented method of measurement called M3 to improve the accuracy and ease of use of the technique will be discussed.

These technologies have been incorporated in the Zetasizer Nano Series of instruments from Malvern Instruments Ltd (Malvern, Worcestershire, UK). Examples of the application of these novel light scattering techniques to a wide range of nanoparticle samples will be presented which will illustrate the capabilities of the technology.

* Dr Michael Kaszuba is the product technical specialist in Malvern Instruments Ltd, UK

** Dr Adam Prochniak is the exclusive representative of Malvern Instruments Ltd in Poland

Coffee break

Tuesday morning, 5 September, 10:30

Session 2

Tuesday morning, 5 September, 11:00

11:00 Invited oral

The Role of the Surface in the Doping of Semiconductor Nanocrystals

David J. Norris

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While a variety of doped semiconductor nanocrystals have been synthesized, no detailed understanding has existed about how this process works or how it can be controlled. Moreover, it was not clear why it is possible to introduce impurities into some semiconductor nanocrystals but not into others. These difficulties have hindered the development of a whole class of new materials, including n-type and p-type semiconductor nanocrystals. Here we discuss a new model based upon kinetics that addresses the doping problem. In particular, we use Mn-doped II-VI semiconductor nanocrystals as a model system to show that: (i) the binding of the impurity to the nanocrystal surface plays a key role in the doping process and (ii) the surfactants in the growth solution can inhibit doping by competitively binding with the impurity. After verifying the central principles of this model, we then use its predictions to incorporate Mn into previously undopable CdSe nanocrystals. Thus, this model should provide future guidance for obtaining a variety of doped semiconductor nanocrystals.

11:30 Oral

Theoretical studies on magnetic impurity doped semiconducting clusters

Stephan Buschmann, Sanjeev K. Nayak, Markus E. Gruner, Peter Entel

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Theoretical investigations on structural, electronic and magnetic properties of passivated III-V and II-VI diluted magnetic semiconducting clusters are performed with density functional theory. Magnetic impurities are doped into the semiconducting host, where the impurity atom substitutes the host metal atom. The magnetic dopant (Mn, Co) in III-V and II-VI semiconductors (GaN, ZnO), which acts as a substitutional impurity replacing the host metal atom exhibits an enhanced magnetic moment per impurity atom. In the present work, we report the results of magnetic impurity doped semiconducting clusters and study the changes in the properties with respect to concentration of dopant, distribution of the impurity atom in the cluster and the effect of cluster size. The results are compared to bulk.

11:45 Oral

Colloidal Diluted Magnetic Semiconductor Quantum Dots

Daniel R. Gamelin

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The generation and manipulation of electron spins in magnetic semiconductor nanostructures is a central theme of the emerging field of spintronics. Carrier-dopant magnetic exchange interactions in diluted magnetic semiconductors provide the basis for many important magneto-electronic phenomena, including carrier-mediated ferromagnetism, magnetic polaron nucleation, and proposed spin-based quantum information processing schemes. This talk will describe our group's recent investigations into the use of photochemical carrier generation and magneto-optical spectroscopies to probe carrier-dopant interactions in colloidal diluted magnetic semiconductor quantum dots.

Related references:

Liu, W. K.; Whitaker, K. M.; Kittilstved, K. R.; Gamelin, D. R. *J. Am. Chem. Soc.*, **2006**, *128*, 3910.

Norberg, N. S.; Kittilstved, K. R.; Amonette, J. E.; Kukkadapu, R. K.; Schwartz, D. A.; Gamelin, D. R. *J. Am. Chem. Soc.*, **2004**, *126*, 9387.

Schwartz, D. A.; Norberg, N. S.; Nguyen, Q. P.; Parker, J. M.; Gamelin, D. R. *J. Am. Chem. Soc.*, **2003**, *125*, 13205.

12:00 Oral

Influence of Doping and Co-doping on the Optical and Electronic Properties of Si Nanocrystallites

Luis E. Ramos, J Furthmüller, Friedhelm Bechstedt

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Quantum confinement in Si nanocrystallites (NCs) leads to interesting optical and electronic properties that can be useful for optoelectronic applications. Silicon is largely used in device technology but it is an indirect-gap material, which limits its application optoelectronics. In contrast to Si bulk and as a result of the confinement of holes and electrons, luminescence can be observed in Si NCs. Recent experiments confirm that co-doping with group-III and group-V can enhance the intensity of luminescence in Si NCs. The co-doping can in principle prevent
radiationless Auger recombinations, which is the main problem in shallow-impurity doping in Si NCs.
Since measurements in Si NCs usually correspond to an ensemble of nanoparticles instead of a single Si NC, theoretical investigations become important to understand and interprete the mechanisms of optical transitions.[1] We perform ab initio calculations for doped and co-doped Si NCs, which are based on density-functional theory and generalized-gradient approximation to investigate their electronic

and optical properties. We verify that the doping with group-V impurities have significant influence on the calculated optical absorption spectra of the Si NCs.

[1] L.E. Ramos, J. Furthmüller, and F. Bechstedt,
Appl. Phys. Lett. **87**, 143113 (2005); Phys. Rev. B **72**, 045351 (2005); Phys. Rev. B **70**, 033311 (2004).

12:15 Oral

Thermochemical synthesis of Mn doped CdS nanoparticles

Maziar Marandi[1], Nima Taghavinia[1], Azam Iraji zad[1], Seyed Mohammad Mahdavi[1], Zohreh Sedaghat[2]

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We report a new method to synthesize CdS:Mn nanoparticles. CdSO4 and Na2S2O3 were used as the precursors while the latter is sensitive to heating process. Na2S2O3 has been considered as a UV sensitive material that finally results in releasing S species in the solution. Here heating process can cause a similar mechanism for dissociation of Na2S2O3. Therefore by heating the precursors in the Ar Atmosphere and in presence of the thioglycerol as the capping agent, CdS nanoparticles could form. The reaction was investigated at different pH, and different temperature from room temperature to 100ºC. The results showed that by increasing the pH the rate of the growth decreases and finally go to a regime that the number of the particles gets constant and only the existing particles grew in size. Also the pH of the solution had an important role in doping process of the nanoparticles. The results of the experiments performed at 100ºC showed that the incorporation of Mn ions inside the CdS nanoparticles is more considerable at lower pHs than the higher pHs. For doing these experiments MnNO3 with different ratios of [MnNO3]/[CdSO4] in the range of 0.01 to 0.1 were used. All experiments carried out at 100 ċ showed that samples moved toward a saturation point in size after a short time. The samples were refluxed for different times to enhance the incorporation of Mn ions inside the particles. The results of the experiments were carried out for the different Mn concentrations demonstrated that increasing the refluxing time increased the intensity of 590 nm Mn photoluminescence peak compared to the intrinsic luminescence peak of undoped CdS nanoparticles. Transmission electron microscopy was the analysis that exhibited crystalline spherical nanoparticles with the sizes around 3nm. X- ray diffraction patterns also demonstrated the crystalline CdS nanoparticles and confirmed the sizes achieved from the TEM images.

Lunch break
Tuesday afternoon, 5 September, 12:30

Session 3
Tuesday afternoon, 5 September, 14:00

14:00 Invited oral

Doping and Characterization of Nanocrystalline Materials

Thomas Wichert, Zhiqun Q. Guan

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Synthesis and characterization of nanocrystalline materials is presented. The materials synthesised are ZnO and InP doped with shallow donors and acceptors, respectively, and Ni-Cu alloys. The characterization is performed with radioactive isotopes using the perturbed γγ angular correlation technique (PAC), thereby, yielding local information on an atomic scale. The characterization is supplemented by different techniques, such as XRD spectroscopy, TEM, UV-VIS, photoluminescence spectroscopy, and EXAFS. By using the radioactive probe atom ^{111}In/^{111}Cd, information about its local surrounding is obtained via the electric and magnetic hyperfine fields. The technique typically uses 10^{11} probe atoms and the signal amplitude does not depend on the Debye-Waller-factor, both making this technique well suited for the investigation of nanocrystalline materials. The information obtained in this way is discussed for the nanocrystalline materials ZnO, InP, and NiCu alloys.

ZnO nanoparticles synthesized by electrochemical deposition under oxidizing conditions (EDOC) are doped with radioactive ^{111}In and stable In. The range of the relative concentrations of dopants is varied between 10^{-9} and 10^{-3}. The as-prepared ZnO particles have a crystal size of 5 nm whereby the dopant atoms are incorporated into disturbed crystalline surroundings that are attributed to imperfect crystalline structures. It turns out that annealing at 473 K represents a good compromise for incorporating the dopant atoms into nanocrystals of high crystalline quality and, at the same time, avoiding a significant crystal growth.

InP nanoparticles with average particle size of 2.4 nm are synthesized via the reaction of dry $InCl_3$ with $P(Si(CH_3)_3)_3$ in trioctylphosphine (TOP) at 530 K. The InP crystals are doped with the acceptor Cd using the radioactive decay of the ^{111}In/^{111}Cd. After preparation no cubic lattice environments around the dopants are detected. Annealing the caped InP nanoparticles at 770 K results in a particle size of 4 nm and yields 10 % of the dopants to be incorporated into an undisturbed lattice environment, and at 870 K the corresponding values are 33 nm and 42 %, respectively. Supported by theoretical calculations, the results suggest the presence of distortions of the lattice structure in the InP nanocrystals, which decreases with increasing particle size.

In nanocrystalline Ni-Cu alloys, synthesized by pulsed electrodeposition (PED), the presence of microscopic inhomogeneity, e.g. caused by pure Ni precipitates, is controlled using the characteristic hyperfine field of nanocrystalline Ni. In this way, precipitates that are invisible by XRD are still detectable by PAC. By increasing the temperature of the electrolyte or lowering the current density of electrolysis, the formation of Ni precipitates is strongly suppressed but, at the same time, the grain size is increased. The addition of saccharin inhibits grain growth and improves the homogeneity of the Ni-Cu alloy on a nanocrystalline scale.

The financial support by the Deutsche Forschungsgemeinschaft within the Sonderforschungsbereich (SFB) 277 is gratefully acknowledged.

14:30 Oral

Atomic Structure, Assembly and Novel Properties of Nanowires

Dorothy Duo Duo D. Ma

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This talk describes our recent research on nanowires. We introduce the oxide-assisted growth (OAG) as a general method for producing silicon nanowires, as well as a range of other nanostructures (wires, cables, chain, ribbons, etc). The nanowires have been assembled to form a nanowire lattice or fiber as well as designed to form a new system. We have developed the techniques to probe the properties of nanostructures. The single-nanowire measurements correlate the properties (electronic and optical) and the structure of individual nanowires. The nanowires have been systematically characterized and shown to have high potential for applications. The available preparation and advanced techniques as well as the wide range of interesting properties underscore nanowires as exciting nanomaterials for fundamental research and technological applications.

14:45 Oral

Nano-Blast Synthesis and SP Sintering of CeO_2-Gd_2O_3 Nanosize Powder.

Oleg Vasylkiv[1], Yoshio Sakka[2], Valeriy Skorokhod[3]

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Engineering CeO_2-Gd_2O_3 ceramic nanopowder with precise morphology by engineering of intermediate nano-reactors followed with 'nano-blast' calcination/deagglomeration was applied. Multiple 'nano-blasts' of $C_3H_6N_6O_6$ embedded to engineered nano-reactors broke apart the agglomerates due to the highly energetic impacts of the blast waves. Extremely rapid detonation (10^{-8} sec/gram) forms gaseous products with a temperature of 2000 to 5000 ºC compressed into a volume equaling the initial volume of each explosive RDX particle. The instantaneous power of each explosion (i.e., the expansion of compressed gases) is 500 MW/gram. The impacts of the blast waves lead to the fragmentation of the surrounding matter. The impulse evolution of a large volume of gaseous products dissipates the heat of the process and limits the temperature increase, thus reducing the possibility of premature local partial sintering among the primary particles. The solid-solubility is enhanced by the extremely high local temperature generated during the 'nano-explosions'. We produced CGO powder with an average primary crystallite size of 11 nm, an aggregate size distribution of 33 ~ 70 nm , with uniform morphology and precise stoichiometry. Spark plasma sintering study was conducted to receive full dense nanostructured ceramic with uniform grains. The properties of SPS processing as far as ionic conductivity of produced ceramic will be described.

15:00 Oral

Incorporation of SiO_2 nanoparticles to 1,3 dialkyl imidazolium ionic liquid

Urša Opara Krašovec[1], Marko Berginc[1], Nina Hauptman[2], Marta Klanjsek Gunde[2], Marko Topic[1]

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Ionic liquids (IL's) are salts having unique property of being liquid at room temperature. They exhibit negligible vapour pressure, non-flammability and good electrochemical stability which makes them interesting for different applications. They can exchange flammable organic solvents in organic synthesis well as being used in electrolytes for different electrochemical systems (lithium batteries or dye-sensitised solar cells (DSSC)). Recently, they have been explored with their potential as reaction media for the fabrication of nanoscale inorganic matter (metal oxide aerogels). The 1,3-dialkyl imidazolium IL can be described also as well organized hydrogen-bonded polymeric supramolecules in solid, liquid and to some extent also in gas phases. Incorporation of different nanosized inhomogenities disturb the hydrogen-bonded network and in some cases generates nanostructures with polar and non-polar regions where inclusion type compounds can be found. We report on the electrolyte system based on 1,3-dialkyl imidazolium IL, iodine and inorganic nanoparticles developed for dye-sensitized solar cell (DSSC). Exchanging volatile electrolytes in DSSC with ILs and their solidification is one of the main research topics in order to bring the DSSC technology closer to the market. The solidification of IL electrolyte with inorganic nanoparticles results in an improvement of the overall DSSC efficiency for up to 20%. This can be explained with the unique ordering of the electrolyte structure being beneficial for the charge transport in the electrolyte. Imidazolium cations are adsorbed on the surface of the nanoparticles, and surrounded with a continuous chain of I- and I3- anions. Beside physical diffusion exchange reaction along the iodide-triiodide chain contributes to the charge transport in the electrolyte.

15:15 Oral

Doped Mesoporous Silica Spheres

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In high-performance liquid chromatography porous uni-sized particles, between 3-7μm in diameter are used as the stationery phases. Improvement in the detection limit and speed of analysis has

been achieved by particle miniaturisation. As pumps and detection equipment improve then particles below 2μm will allow on-line analysis. However, regular and size monodsiperse particles of this size are difficult to prepare. Here we report methods by which small porous microspheres may be prepared by combining advances colloidal particle and mesoporous material synthesis. The synthesis of mesoporous silica spheres with uni-directional and tuneable mesoporous diameters have not previously been reported. Organic micelles (e.g. formed block copolymer surfactants) and silica hydrolysis are used to generate ordered mesoporosity. Careful reaction control allows size-monodisperse spherical particles, ± 5 RMS with tuneable diameters between 1-5 μm to be formed. We show that the mesopores are aligned through the spherical structure to provide an open porous surface with high surface areas, typically between 1000-1500 m^2g^{-1}. Doping of silica microspheres with ceria is also carried out in attempt modify chromatographic performance (more selective to bioentities). It is shown that using different reaction routes that the ceria can either be distributed homogeneously through the structure or in the form of discrete and thermally stable nanoparticles at the surface. These nanoparticles can increase the rate of sintering by necking. We also demonstrate that relatively large diameter mesopores can be obtained by a unique method, between 6-11 nm, using supercritical carbon dioxide (sc-CO_2) as a swelling agent during synthesis. Finally, materials were packed into columns for chromatographic testing. Results are presented that illustrate the application of these microspheres in UHPLC.

Coffee break
Tuesday afternoon, 5 September, 15:30

Session 4
Tuesday afternoon, 5 September, 15:50

15:50 Invited oral

Luminescence of doped nanoparticles of wide band gap II-VI compounds

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Nanoparticles of wide band gap II-VI compounds doped with transition metal or rare earth ions are perspective fluorescence labels for applications in biology and medicine. For this application nanoparticles should efficiently emit light, which was rather unexpected for parity and often spin forbidden 3d-3d and 4f-4f intra-shell transitions. However, in nanoparticles efficiency of intra-shell transitions is enhanced. Mechanisms of emission enhancement will be discussed, based on our recent results for Mn doped nanoparticles of ZnS and CdS. Two effects will first be discussed - quantum confinement imposed on free carriers, which enhances spin dependent interactions of free carriers with impurities and increased rate of host to impurity transfer.

Passivation of surface states is important to limit rates of nonradiative transitions. We show that this can be achieved by doping of nanoparticles. Nanopowders of ZnO n-type doped with aluminium were studied to demonstrate such possibility. We show that intensity of light emission increases with an increasing Al fraction in ZnO nanopowders.

This work was partly supported by grant no. 1 P03B 090 30 of MEiN granted for the years 2006-2008.

16:20 Invited oral

The luminescence properties of ZnO:Al nanopowders obtained by sol-gel and vaporization-condensation methods

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ZnO is attractive material for many practical applications. Doping with Al enhances luminescecne of this mateiral. ZnO nanopowders (undoped and Al doped) were prepared by sol-gell method (SG). The SG made powders were used as substrates for vaporisation-condensation process (VC) using the SPVD (Solar Physical Vapor Deposition) method. The luminescence spectra were measured in spectral range 1.55 eV-3.75 eV under 266 nm pulsed laser excitation (pulse duration 10 ns). The luminescence decay process was analysed in time range 2ns - 1ms with time resolution 2 ns. SEM, FTIR, XRD were used for samples characterisation. The luminescence spectra for SG and VC nanopowders are different mainly in the range 2.6 -3.1 eV. The defects responsible for luminescence within this region disappeared during VP process. However both sets of ZnO:Al samples (SG and VP) show defect state luminescence intensity larger than that fresulting from excitons recombination. The well-resolved excitons luminescence band observed in VC nanopowder indicate that in these powders there are regions with defect free ZnO structure. The defect luminescence decay kinetics shows an initial fast (<10 ns) decay followed by a slower one (ms range). This slow decay of luminescence might be due to electron tunneling. Possible processes responsible for luminescence observed will be discussed.

Wednesday, 6 September

Plenary Session
2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break

Wednesday afternoon, 6 September, 12:30

Session 5

Wednesday afternoon, 6 September, 14:00

14:00 Invited oral

Nonaqueous Synthesis of Metal Oxide Nanocrystals: A Versatile Approach to Control Size, Shape, Assembly and Composition

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Nonaqueous solution routes to metal oxide nanoparticles are a valuable alternative to the well-known aqueous sol-gel processes, offering advantages such as high crystallinity at low temperatures, robust synthesis parameters and ability to control the crystal growth without the use of surfactants.

The talk will present the synthesis of crystalline metal oxide nanoparticles based on nonaqueous soft-chemistry routes involving the reaction of metal oxide precursors such as metal halides, alkoxides or acetylacetonates with organic solvents like benzyl alcohol, benzylamine or various ketones. Control over particle size and shape is achieved solely by the solvent without any additional structure-directing agents. This synthesis methodology enables the preparation of a large variety of binary as well as ternary metal oxides on the nanoscale, often with crystallite sizes well below 10 nm. Furthermore, the simultaneous use of two or more chemically different precursors allows the controlled preparation of doped metal oxide nanoparticles, thus providing a powerful tool to tailor the chemical as well as the physical properties. As an illustrative example the synthesis of indium tin oxide nanoparticles with varying tin oxide content will be presented.

14:30 Oral

Ceria-zirconia nanoparticles doped with La or Gd: effect of the doping cation on the real structure

Vladislav A. Sadykov[1], Vladimir V. Kriventsov[1], Ella M. Moroz[1], Yulia V. Borchert[2], Dmitrii A. Zyuzin[1], Vera P. Kolko[1], Tatyana G. Kuznetsova[1], Vyacheslav P. Ivanov[1], Andrei I. Boronin[1], Natalia V. Mezentseva[1], Elena B. Burgina[1], Julian Ross[3]

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This work presents results of studies of the real structure of nanocrystalline CeO_2-ZrO_2 (Ce:Zr=1:1) systems prepared via Pechini route and doped with La^{3+} or Gd^{3+} cations (x up to 30 at.%). X-ray diffraction, EXAFS, UV-Vis and Raman spectroscopy were applied for the bulk characterization, while the surface features were studied by XPS and SIMS. For both systems, the increase of Ln^{3+} content was accompanied by the decline of domain size and increase of the specific surface area and lattice parameter of fluorite-like phases. While for bigger La^{3+} the system becomes bi-phasic at xLa > 10 at.%, for smaller Gd^{3+} cation single-phase solid solution exists within all studied range. For both systems, depletion of the surface layer by smaller Zr^{4+} cations was revealed, while the surface content of a doping cation is either close to that in the bulk (La) or lower (Gd). Such a spatial distribution of components results in some ordering of cations within the particle. It is reflected in different modes of rearrangement of oxygen coordination polyhedra with the Gd or La content (by EXAFS), and redistribution of the intensity of reflections in XRD patterns not conforming to a simple model with statistical distribution of oxygen vacancies. Associated variation of average Ce(Zr)-O bond strength (SIMS) appears to affect the lattice oxygen mobility. This work is in part supported by INTAS 05-1000005-7663, RFBR-CNRS 05-03-34761 Projects and Integration Project 4.15 SB RAS.

14:45 Oral

Nanocomposite powders by evaporation driven self assembly during spray drying: Some new insights on the structure evolution

Debasis Sen[1,2], Olivier Spalla[1], Antoine Thill[1], Patrick Haltebourg[1]

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Spray drying is an interesting process to synthesize nanocomposite powders of technological interests. However, structure and organization of the particles depend strongly on the thermodynamic/hydrodynamic conditions and interaction of the basic particles. Structural/compositional investigations on dried powders can reveal the mechanism of the organization during drying. In-situ scattering experiments can reveal such information. Small-angle X-ray scattering (SAXS) is a novel technique to investigate mesoscopic structure over a wide length scale. As volume fraction of droplets is not high enough to be probed in-situ by laboratory X-ray source, experiments at synchrotron source becomes essential. In-situ SAXS experiments on structural/correlation evolution of grains, during drying have been performed at ID2 beamline of European synchrotron radiation facility. An indigenously developed spray dryer has been used. Some salient results will be presented. In brief, depending on thermal gradiant of the tube, in intermediate drying stage, the arrest/drag of the nanocolloids at the droplet surface produces core-shell type grain with jammed particle concentration in shell and core with lower concentration. With the passage of droplets through thermal atmosphere, gradually the core also mimic the jammed concentration followed by the overall compression of initial droplet size. In addi-

tion, spray drying of mixed inorganic/organic colloids followed by post thermal treatments lead to production of porous nanopowders. The evolution of the structure, the correlation and the composition of such nanoporous powders [1] during the post thermal treatments will also be elaborated. [1] D. Sen, O. Spalla, L. Belloni, T. Charpentier, and A. Thill; Langmuir 22 3798-3806 (2006)

15:00 Oral

The potential use of doped nanoparticles from hydrothermal synthesis in cell signalling

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Whilst the chemistry of supercritical water (scW) synthesis is relatively simple, the 'engineering' of these continuous reactions is much more difficult. The development of the nozzle reactor at Nottingham has overcome the problem of poor mixing with additional benefits as well: the ability to make nanoparticles continuously, doped and undoped; continuous online capping, post production; the ability to alter particle size and morphology by altering the operating parameters of temperature, flow rate and metal salt concentration; the ability to produce stable suspensions of nanoparticles in water. In contrast to other methods particle production in supercritical water is a relatively simple one stage route, chemically more benign, and produces product dispersed in water. The ability to create suspensions of particles in water, of controlled size and chemistry, using 'biologically benign' precursors such as iron nitrate or silver acetate has presented a new and exciting opportunity for biological applications.

The paper will describe how the nozzle was developed and how it can be used as a rapid prototyping system for a range of different doped nanoparticles for medical applications, particularly in the field of cellular imaging.

15:15 Oral

Production of doped and non doped ceramics nanoparticles for optical applications

Ed Lester[1], Tadeusz Chudoba[2], Jun Lee[1], Paul Blood[1], Agnieszka Opalińska[2,3], Witold Lojkowski[2], Martyn Poliakoff[1], Albertina Cabanas[3]

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The synthesis of yttrium aluminium garnet (YAG or $Y_3Al_5O_{12}$) has received much attention on account of its use in structural and functional materials. Among its functional properties, YAG possesses a high efficiency of energy transfer and is resistant to radiation damage, which therefore makes it ideal for laser lenses. The usable transmittance range of YAG extends from the UV to the mid-IR range, making it very useful for IR and laser windows, with a very low absorption coefficient . Furthermore YAG displays a cubic crystal structure, which imparts optical isotropy (unlike sapphire). Doping YAG with different trivalent ions changes the optical properties of the doped materials. Whether manufacturing YAG for phosphor applications or ceramic fabrication, fine-sized particles with no agglomeration are desirable.

The paper presents collaborative work between UNIPRESS and The University of Nottingham in the production of transparent ceramic lenses from doped and non doped Zirconia and YAG nanoparticles. Nanoparticles of ZrO2 and YAG have been made using two different hydrothermal techniques. UNIPRESS use a microwave assisted batch reactor and Nottingham has a nozzle reactor which operates continuously. Both systems are currently small scale and can produce materials sub 20 nanometres on g/hr scale.

The paper will describe the various production techniques, the relationship between operating conditions and particle quality, the similarities and differences between the two synthesis techniques, as well as the effect of the raw powders on the quality of the final lens material.

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony
(continuation of Monday's Poster Session)Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Session 6
Thursday morning, 7 September, 9:00

9:00 Invited oral

Evolution of grain boundaries and interfaces during sintering and grain growth in doped oxide ceramics: from nanosized powders and atomistic simulations to transparent ceramics and tailored interfaces

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Ceramic properties are inherently linked to their chemical composition and microstructure. The transformation of a ceramic powder from a loose collection of particles into a sophisticated ceramic piece, e.g. dental implants, hip implants, multilayered capacitors, cutting tools or transparent ceramics for laser applications depends heavily on our capacity to control and modify the interfaces during processing and sintering. The use of dopants, which can segregate to both surfaces and grain boundaries during powder synthesis and sintering, are often used to influence these interfacial regions. The significant improvement in analytical techniques namely high resolu-

tion transmission electron microscopy (HRTEM, EELS) has given us much insight into the general location of dopants in ceramic microstructures but we still lack the atomistic knowledge which should enable us to design grain boundaries and interfaces for specific properties and applications. A key feature in being able to tailor microstructures from powder processing routes is our capacity to synthesise well controlled powders. The powder characteristics of key importance are size, size distribution, morphology and purity. A brief description of an innovative tubular reactor, the segmented flow tubular reactor (SFTR), that produces powders with better characteristics for ceramics will be presented and the applicability demonstrated with examples of scale-up, powder quality and reproducibility for $BaTiO_3$.

One route towards the goal of tailored microstructures is to use the ever increasing computer power to model at an atomistic level these key interfacial regions. Controlling grain growth and grain boundary composition is a key factor for the production of transparent polycrystalline ceramics such as yttrium aluminium garnet (YAG) and alumina. For applications in high performance lasers YAG is doped with neodymium (Nd), single crystals are limited to around 1%, whereas polycrystalline YAG can accommodate more. To increase laser power but still be able to produce transparent ceramics the location and effect of increased Nd levels is a key issue for progress. The effect of dopants in alumina such as Y for creep resistance, Mg for grain growth control is well established but the detailed mechanisms behind the effects are poorly understood. The level of dopant whereby there is segregation to grain boundaries and eventually the formation of a secondary phase is linked to the type of grain boundary present and their relative surface area, which in turn is linked to the grain size - one of the parameters we are trying to control. Using energy minimisation techniques the location, dopant concentration, grain boundary structure, surface and interfacial energies have been investigated for a number of crystallographic surfaces in YAG and alumina giving us further insight in our quest towards tailored interfaces. Validation of the modelling approach will be demonstrated by comparison with available experimental data and in particular grain boundary segregation/precipitation maps.

9:30 Oral

Nanopowders of yttrium aluminum garnet for transparent ceramics

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Transparent $Y_3Al_5O_{12}$ (YAG) ceramics is very attractive material for many applications in optics as laser gain host material, laser windows etc, and as structural materials for high temperature apparatus and devices. The main point in YAGceramic production is preparation of mono dispersive nano sized powders. We produce Nd doped YAG powders via co-precipitation route using mixed water solutions of yttrium nitrate and ammonium aluminum sulfate dodecahydrate or aluminum nitrate hexahydtrate as mother solution which were dropwised into solution of ammonium hydrocarbonate as precipitant. Different ratio of yttrium to ammonium hydrocarbonate was used. The small amount of ammonium polyacrilate or ammonium polycarbonate was added to precipitant solution during precipitation or aging stages. Washed and dried precursor was calcined at various temperatures in the range of 800 - 1300 ^oC. Precursor and oxide powders obtained in this route were investigated using SEM, XDR, BET and photoluminescence methods. Substantial influence of precursor dewatering process and calcination temperature on size, shape and agglomeration of YAG particles were found and discussed. In optimal conditions well- dispersed well-sinterable Nd doped YAG powders with ball-shape particles were obtained. This powder was used for preparation of high transparent Nd doped YAG ceramic.

9:45 Oral

Host size effects on optical properties of rare earth and transition metal doped materials

Gilles Ledoux[1], David Amans[1], Christophe Dujardin[1], Yann Leconte[2], Lionel Combemale[2], Nathalie C. Herlin-Boime[2], Cecile Reynaud[2]

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Rare earth or transition metals doped materials have numerous applications in our day life (TV screens, solid lasers, scintillators …) thanks to their efficient and robust luminescence properties. In the recent year growing interest has focused on the changes in their optical properties with size of the host particle. In the case of many semi-conductors, the intrinsic luminescence is blue shifted when the size decreases. But, the size effect is not so clear with traditional doped insulators. In this case the dopant has an "atomic" character and does not "feel" so much the size of the host. We have elaborated this kind of materials for the first time by using laser pyrolysis. This technique, based on the decomposition of a liquid or gaseous precursor by a powerful CO_2 laser, allows the production of weighable amount of powders with very high purity. In the present work, yttrium and lanthanum nitrates, as well as several rare earth nitrates were dissolved in water and then carried as an aerosol by an argon flow to the reaction zone. A weak flow of ethylene was added as a sensitizer gas to absorb the laser emission. Many different compositions have been achieved from La2O3: Bi to Y2O3:Ce. Our aim is to check how some optical properties like autoionization processes or thermal quenching processes are modified by the size of the particle. The latest development on this subject will be presented at the conference. Comparisons with bulk materials are presented and the observed differences are discussed.

10:00 Invited oral

Polyol-mediated synthesis and high-throughput screening of the gas sensing properties of rare-earth doped metal oxide nanoparticles

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In this work we present the gas sensing properties of rare-earth doped nanoparticular metal oxides which have been prepared by the polyol mediated synthesis. The materials are studied using a high-throughput equipment, applying multi electrode substrates and high-throughput impedance spectroscopy in a temperature range from 500 to 200°C and under various gas atmospheres (synthetic air, H_2, CO, NO, NO_2, ethanol and propylene). We will report about the development of this new experimental technique and its potential to be applied for gas sensor development.

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Coffee break

Thursday morning, 7 September, 10:30

Session 7

Thursday morning, 7 September, 11:00

11:00 Oral

Surface and Bulk Modifications of Nanostructural ZrO_2 Powder Induced by Doping with Vanadium and Molybdenum Ions

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Zirconia and ZrO_2-based materials have been widely used in many fields due to their high melting point, low thermal conductivity, high resistance to corrosion and remarkable oxygen transport properties.

In the present studies a nanostructural tetragonal zirconia (*t*-ZrO_2) of enhanced thermal stability with developed surface area and unimodal pore structure, was obtained from $ZrOCl_2$ by modified forced hydrolysis method followed by aging at 100°C for 48 h. Collective XRD, RS, EPR and microscopic SEM/TEM investigations, complemented by N_2-sorption measurements, showed that surface and bulk properties of ZrO_2 as well as its phase composition are strongly dependent on the presence, nature and concentration of dopants such as vanadium and molybdenum ions. Those additives were deposited on the surface of *t*-ZrO_2 using the classical or slurry impregnation techniques.

As revealed by variable-temperature XRD and EPR measurements, even at temperatures below the Hüttig temperature (*i.e.* < 800°C), the V and Mo dopants can migrate into the bulk of *t*-ZrO_2, forming the corresponding solid solutions. The observed structural changes were further confirmed by Rietveld refinement, basing on the XRD data. It was found that spontaneous reduction of the surface V^{5+} and Mo^{6+} ions and concomitant phase transition from the tetragonal to the monoclinic polymorph of ZrO_2, opened channels of facile diffusion of the resultant idiovalent dopant ions into the bulk of the ZrO_2 matrix. Final phase composition of the doped ZrO_2 materials was determined by the amount of incorporated additives. Vanadium loadings below 3 mol. % stabilized the low-temperature tetragonal polymorph, whereas higher loadings favored its transformation into the monoclinic form.

11:15 Oral

Comparison of Eu-doped effects on the photocatalytic activity of nanoparticulate TiO_2 and Ag-deposition TiO_2 under visible and UV light irradiation

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We have investigated the photocatalytic degradation of methyl orange (MO) dye in the aqueous suspensions of Eu-doped TiO_2, Ag-deposited TiO_2 nanoparticles under visible and UV light irradiation. To evaluate and distinguish various effects of the Eu-TiO_2 and Ag@TiO_2 photocatalystic activity, the Eu-doped TiO_2 and Ag@TiO_2 photocatalysts were characterized by XRD, TEM, XPS, BET, PL and UV-visible absorption. For comparison, the MO photodegradation was carried out in P25 TiO_2 and Ag@P25 suspensions under the same condition. In the MO/ Eu-Ag-TiO_2 system, Eu doping significantly enhanced the MO photodegradation under visible and UV light irradiation. The significant enhancement in photocatalytic activities due to doping of foreign elements can be ascribed to simultaneous effects of Eu and Ag on the electron trap and electron-transfer efficiency.

11:30 Oral

Synthesis improvement of Yb^{3+}-activated SnO_2 nanocrystals

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Rare-earth doping of nanocrystalline oxides is a current topic of interest for opto-electronic application. To achieve maximum homogeneity and solubility of the dopant ion in the host structure, various processes have been proposed. On this regard, SnO_2 nanocrystals, where the band gap energy is blue-shifted in respect to bulk SnO_2, are attractive for rare earth ion activation, but the solid state solubility of rare earth ion represents a serious limitation. Besides, solution processing may lead to separation of solid SnO_2, leaving in solution most of the rare earth ions. To overcome this, we synthesized Yb^{3+}-doped SnO_2 nanocrystals, using the hydrolytic route in the presence of starch as size stabilizer, a process we recently reported for other oxides. Due to small quantum defect and simple electronic structure, Yb^{3+} shows promising power and efficiency for laser and amplifiers operating around 1 mm. Starting from salt precursors in a starch solution, stable powders with various Yb loads were prepared and characterized by XRD, TEM, ICP and TG-MS techniques. Preliminary assessments of the spectroscopic features of nanocrystals (d <6 nm) were performed by absorption, luminescence, and Raman measurements: The Yb^{3+} typical absorption peak centred at 977nm and intense $^2F_{5/2}$ ® $^2F_{7/2}$ Yb^{3+} emission band were observed. Fabrication of silica films loaded with Yb^{3+}-activated SnO_2 nanocrystals is in progress.

11:45 Oral

Manipulation of chemical and optical properties of MgO nanocubes via surface functionalization

Slavica Stankic[1], Thomas Berger[1], Oliver Diwald[1], Johannes Bernardi[2], Erich Knoezinger[1]

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We use chemical vapour deposition (CVD) for the synthesis of pure and doped MgO nanoparticles[1]. In this presentation we demonstrate how the admixture of a second metal component alters the electronic surface properties of MgO nanocrystals. When MgO nanoparticles are doped with monovalent Li-ions, traces of a dopant already induce dramatic changes in the optical and chemical surface properties. Isovalent Ca^{2+} cations can be distributed homogeneously in MgO nanocrystals although CaMgO mixtures are thermodynamically forbidden on a macroscopic scale. Subsequent thermal activation leads to calcium ion segregation into the MgO surface. These CaMgO nanocrystals represent novel materials with enhanced surface basicity and a higher thermal stability in comparison to pure MgO[2]. Furthermore, an unexpected photonic behaviour of those mixtures is reflected by dramatically increase in photoluminescence emission which is also red-shifted with respect to pure MgO. On the contrary, the admixture of also isovalent but larger Sr^{2+} cations induces a blue-shifted emission irrespective of the excitation wavelength.

It is known that by mixing MgO (band gap 7,8 eV) with ZnO (band gap 3,4 eV), one can tune the band gap[3]. However, the effect of ZnO on the surface electronic structure of MgO in terms of optical absorption and emission effects is so far unexplored. Therefore, surface studies on powdered $Mg_xZn_{x-1}O$ samples are currently carried out in our lab in order to elucidate whether such materials represent interesting candidates for light-induced conversion of an insulating metal oxide into a persistent electronic conductor.

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[3]I. Takeuchi et al. *J. Appl. Phys.***94** (2003) 7336

12:00 Oral

Chemical Vapor Synthesis and Structure of Chromium Doped Nanocrystalline Zinc Oxide

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Zinc oxide is a direct wide band gap semiconductor. The introduction of magnetic elements can produce semiconductors which are ferromagnetic close to room temperature [1]. Potential applications of such materials are e.g. magnetic sensors and actuators, high-density memory. The properties of ZnO can be tuned chemically and by size variation. However, the efficient doping of nanocrystals is difficult [2] and the location of the dopant atoms is important for their function. Atoms on substitutional sites in the wurtzite structure of ZnO behave differently compared to dopants on interstitial sites or on particle surfaces.

Doped Nanocrystalline zinc oxide powders are produced by evaporation of mixed precursors in a laser flash evaporator and subsequent particle generation by chemical vapor synthesis (CVS) [3, 4]. The powders are characterized by means of X-ray diffraction, nitrogen absorption, and transmission electron microscopy. XAFS spectra at the Zn- and Cr-K X-ray absorption edges supply information on the local structure of the nanocrystalline zinc oxide materials. The XAFS spectra are analyzed by Reverse Monte Carlo Methods (RMC) [4, 5]. A careful analysis of the partial pair distribution functions for the different cations provided by RMC reveals the location of the dopant atoms within the host structure.

References

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12:00 Oral

Zirconia-based nanomaterials for oxygen sensor - generation, characterisation and optical properties

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We found out that zirconia-based nanomaterials can be used in optical
sensor for detecting oxygen partial pressure. Generation of those
materials with varying phase comphosition, type and concentration of
dopands, their characterisation and selected optical properties are reported
concerning best using in luminescence oxygen sensor.

12:15 Oral

Localization of rare-earth dopants in the lattice of nanocrystalline ZrO_2 - EXAFS study

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Doping with rare earth ions is a well known process to add optical properties to oxides, like YAG or ZrO_2. Such materials are used to make lasers, LEDs, scintilators, phosphors. Lighting efficiency depends on the dopant concentration but also on interactions between the doping ions and their position in the lattice and interactions with lattice defects. During high temperature processes of growing single crystals or sintering microcrystalline grains, such negative effects are difficult to be avoided.

Nanocrystalline host materials show great flexibility of growth from rare-earth-rich solutions and their use to make optical ceramic oxides is an attractive field of technology. However, the optical properties depend on the distribution of the dopants in the nanocrystal: whether it interstitial, or replaces a host ion, and whether it is at the surface of a nanocrystal or segregate as oxides (which makes them optically ineffective).

The present work is an XRD-EXAFS study of the rare-earth dopant location in the zirconia lattice. Samples grown in 1% through 20% solutions of rare earth were examined using both methods. Localization of the luminescence centers in nano-zirconia lattice was investigated.

Authors wish to thank to ANKA Synchrotron (Karlsruhe, Germany) for infrastructure and professional assistance in high quality XRD/ EXAFS measurements. We thank to the Polish Ministry of Science for the financial support (grant 3-T08A-029-27).

Lunch break
Thursday afternoon, 7 September, 12:30

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster C1

The influence of ZrO_2 containing 10% Eu^{3+} on the polyurethane hard domain structure of nano-composites with luminescence properties

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Organic polymers that emit light have commanded increasing attention in the last decade, both for their interest to fundamental studies and electro-optical/opto-electronic applications.

In order to produce elastic and resistant foils with luminescent properties inorganic luminophors of nanometric sizes were introduced into the polyurethane elastomer. Nano-composites were prepared by in-situ polymerization. As the best performances of nanocomposites requires good dispersion of the nano-fillers, stirring of the components have been carried out under different values of such parameters as stirring time and speed. Various solvents and process temperatures were also investigated. Nano-composites with different amounts of nanofiller were manufactured to investigate the nanofiller distribution and the structure of hard domains of polyurethanes designed for opto-electro applications.

For PUR synthesis the following chemicals were used: polycaprolactone diol (PCL) Mn=2000,
4,4'-dicyclohexymethane diisocyanate (H'MDI), chains extenders (DIOL A, DIOL B). As a nanofiller zirconium oxide containing 10% Eu^{3+} was used. The filler was added to the polyurethane matrix in the quantity ranging from 0.05 to 0.2% wt. The synthesis was performed via pre-polymer route.

The microstructure of the obtained materials was examined by SEM. The size analysis of nanofillers was investigated with HRSEM and TEM. The mechanical and thermal properties of polyurethane nanocomposites were also investigated in addition to the analysis of transmittance and luminescence of obtained materials was performed.
The results obtained indicate possibility of fabrication of polymeric nano-composites for opto-electro applications via relatively inexpensive processing route.
This research was financed by the Polish State Committee for Scientific Research (PBZ-KBN-095/T08/2003).

17:20 Poster C2

Particle Size Distribution of $ZrO_2:Pr^{3+}$- Influences of pH, High Power Ultrasound, Surfactant and Dopant Quantity

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Nanoparticles seize the scientific world and have long found their fields of application. However, synthesis and handling of these small particles remain a big challenge: They tend to agglomerate, not only in dispersions but also during the production process.

In this work, various factors governing agglomeration were examined. The influences of pH, surfactants and the application of high power ultrasound (HPUS) have been investigated for nanoscaled praseodymium doped zirconia. In order to devise efficient strategies for stabilising a dispersion of zirconia nanoparticles, the topological distribution of dopants within the particles is important to know.

Nanoscaled $ZrO_2:Pr^{3+}$ powders were obtained via a hydrothermal microwave driven process. [1] The Pr^{3+} content varied between 0.05 and 10 mol%. The particle size distribution as a function of pH and dopant quantity was measured to unravel the impact of the isoelectric point (IEP). Pure zirconia has an IEP at pH5, whereas pure Pr_2O_3 has its IEP at pH9. Due to missing electrostatic interactions, the agglomeration tendency is strong around the IEP of the sample. Measurements of the particle size distribution reveal a strong variation of the maximum average particle size depending on Pr^{3+} content and pH. At low Pr^{3+} doping levels, particle agglomeration occurs close to the IEP of zirconia, whereas at higher amounts of Pr^{3+}, strong agglomeration appears at pH values close to the IEP of pure Pr_2O_3. Due to the significant changes, it is assumed that the Pr^{3+} ions are predominantly located in the particle surface region.

In order to obtain stable water-based dispersions of Pr^{3+} doped zirconia, it is thus important to set the pH according to the content of praseodymium. The addition of Sodium Dodecyl Sulphate (SDS) as a surfactant was examined and resulted in dispersions with a small tendency to form agglomerates. Another efficient way to obtain dispersions with small particles is the application of HPUS for a limited time. It was shown that the application of HPUS can improve the luminescence properties of $ZrO_2:Pr^{3+}$, especially for the samples with a small quantity of praseodymium. Both, excitation and emission are affected by the application of HPUS.

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17:20 Poster C3

Luminescence of Europium doped ZrO_2 nanopowder

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Luminescence of ZrO_2:Eu free-standing nanocrystals (nanopowders) containing different concentration of Eu (from 0.1 up to 5% in the raw material for nanocrystal synthesis) was studied. The luminescence was excited by a pulsed laser beam (4.66 eV, 8 ns). The luminescence spectra and decay kinetic were recorded. Eu^{3+} emission lines within 1.4 and 2,5 eV ("red luminescence") were observed in all samples studied, whereas trace from Eu^{2+} emission was not found. The intensity of Eu^{3+} luminescence was higher for samples with larger Eu concentration, however above 1% concentration the intensity of luminescence tend to the saturation. The luminescence decay kinetic shown dependence on Eu^{3+} concentration also. It was concluded only Eu^{3+} state was incorporated in ZrO_2 and above 1% (in raw material) concentration interaction between Eu^{3+} ions take place. The luminescence decay kinetic shown more than one decay component indicating that Eu^{3+} was incorporated in different positions. More appropriate position for Eu^{3+} is Zr^{4+} site and the charge compensation by oxygen vacancy take place, since one vacancy serve as charge compensator for two Eu^{3+} the different spacing between oxygen vacancy and Eu^{3+} is suggested. Thus, the oxygen surrounding of Eu^{3+} was not the same for all ions and different distorted sites appear. Possible configurations of Eu^{3+} sites will be discussed.

17:20 Poster C4

Characterization of nanocrystalline ZrO_2 doped with Rare-Earth elements synthesized via High Pressure Hydrothermal Method

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Zirconia is an important ceramic material with useful mechanical, thermal and electrical properties. Nanocrystalline zirconia powders doped with rare-earth (RE) elements have been successfully prepared via microwave hydrothermal process. The microwave driven hydrothermal synthesis permits to precisely control the reaction re-

gime and in consequence provides expected properties of the resulting nanocrystalline powders. Using this method powders with the average grain size 5-10 nm with different dopant (e.g. Eu, Nd, Lu, Gd, Tb) in the range 1 -10 mol% can be obtained.

The aim of this investigation is to evaluate a fundamental characteristics of the nanopowders: grain size, grain size distribution, density, phase composition (relative fraction of the monoclinic and tetragonal phases), lattice parameters for both phases and luminescence properties as a function of element and concentration of the RE dopant in ZrO2 nanopowders.

17:20 Poster C5

Optimization of conditions of preparation of YAG nanopowders for sintering of translucent ceramics

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Agglomeration of YAG (Ytrium-Aluminium Garnet) nanopowders doped with various rare earth ions obtained by coprecipitation and calcination route is a serious problem when one wants to exploit nano-size properties like transparency of dispersions of the powders or low temperature sintering. YAG nanopowders doped with rare earth ions can be used as a starting material to make laser ceramics. We optimized the process of preparation of the powders for High Pressure-Low Temperature Sintering of semi-translucent pellets. Proper time of the milling to decrease agglomeration was evaluated with the help of Zeta-potential measurement. The dependence of the agglomerates size vs. time of the hand-milling permitted to optimize that process.

17:20 Poster C6

Luminescence and structural properties of rare-earth doped YAG nanoceramics

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Transparent YAG nanoceramics composed of nanosized grains were fabricated by high pressure low temperature sintering technique. Absorption and luminescence spectra were measured. It was fount that the emission properties of Nd^{3+} ions in YAG nanoceramic was different than in single crystal. An increase of the applied pressure during sintering cause some special effects like broadening of full-wide at haft maximum of emission lines and decrease of luminescence lifetimes. XRD analysis indicate presence of microstrain in nanoceramics and no significant grain growth during sintering process. Influence of microstrains on luminescence properties of rare-earth ions was analysed.

17:20 Poster C7

Concentration dependent fluorescence lifetime in nanocrystalline Y_2O_3:Eu phosphors

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Nanocrystalline Y_2O_3:Eu powder was synthesized via chemical vapor reaction. X-ray diffraction revealed the structure of cubic yttria with crystallite sizes of about 5 nm. The Eu-dopand concentrations for the samples were determined by EDX-spectra and resulted in values in the range from 0.3% up to 16.5%.

Luminescence of the nanopowders was investigated by continuous and timeresolved UV fluorescence spectroscopy and compared to a microcrystalline Y_2O_3:Eu phosphor as a reference. Emission spectra show increasing intensity for higher doping concentrations. Compared to the microcrystalline material, however, the yield was significantly lower. The lifetime τ of the $^5D_0 \rightarrow {}^7F_J$ transition in the nanocrystalline Y_2O_3:Eu was strongly enhanced in the lowest doped sample (3.71 ms for the 611 nm line) compared to the microcrystalline reference (1.07 ms). By increasing the Eu-content the lifetime τ decreased until reaching a value of 1.20 ms for the highest doping concentration.

The concentration dependent lifetime behaviour was interpreted by energy transfer between Eu ions among each other and between Eu ions and impurities as a competing process to the radiating $^5D_0 \rightarrow {}^7F_J$ transition.

17:20 Poster C8

Investigation of luminescece of Nd: Y_2O_3 ceramics

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In present paper the pulsed cathodoluminescent (PCL) spectra of Nd: Y_2O_3 ceramics fabricated from nanopowders were investigated in the range of 350-800 nm . The synthesis technology and the results of structure research were presented elsewhere [1].

For the ceramics quality determination a nondestructive method based on the analysis of the PCL spectra in the visible range was used. Pulsed cathodoluminescence was excited and investigated by CLAVI-device [2].

There are three intensive neodymium bands at 399.2, 425.7 and 459.6 nm in the PCL spectra of Nd: Y_2O_3 nanopowders, from which the ceramics is obtained . In these ceramic specimens two types of PCL spectra were registered. The first type spectra almost coincide with the nanopowders spectra. There is the difference in the intens-

ity. In the spectra of the second group there is the broad substrate and some bands of Nd ions didn't appear. The luminescence of all Nd: Y_2O_3 ceramic samples with both types of spectra was unstable. At the irradiation of the samples by electron beam the luminescence doesn't appear in the first pulses. The luminescence intensity varies randomly in different excited pulses upon spectra shape maintaining. The reason the PCL instability can be the electron traps, which capture secondary electrons produced by the electron beam. The presence of the traps was confirmed by the thermoluminescence, which was observed in all of the ceramic specimens; and these traps were explained by the presence of the anion vacancies.

17:20 Poster C9

Time-Resolved Luminescence Characteristics of Doped YAG and YAP Nanopowders

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Nanopowders (NP) of yttrium-aluminium garnet (YAG) and/or yttrium aluminium perovskite (YAP) doped by rare-earth ions were studied as a prospective material for transparent scintillating ceramics. The YAG and YAP NP with the grain size ~20 nm and higher were obtained by coprecipitation method. Nominally pure, Ce3+, Nd3+ and Eu3+ doped NP have been studied by means of time-resolved luminescence spectroscopy. Pulsed electron beam (energy of electrons was 280 keV, pulse duration was 8 ns) was applied for luminescence excitation in 80-300 K temperature range. Intrinsic luminescence and energy transfer from the host lattice to rare-earth ions are competing relaxation channels. Time-resolved characteristics were measured for both intrinsic and dopand emissions. Results obtained may be summarized as follows: 1) Intrinsic luminescence bands were detected in all samples studied except cerium doped YAG NP. These bands are similar to well known emission for single- and poly- crystals. It means that centers (defects) responsible for unwanted intrinsic luminescence persist in NP. On the other hand no new (if compare with crystals) intrinsic luminescence bands appeared due to nanocrystalline structure. The reason of the absence of intrinsic luminescence in cerium doped NP will be discussed. 2) Luminescence decay kinetics of rare-earth ions strongly depends on dopand concentration and NP grain size. It is shown that the relaxation time for rare-earth related emission for NP is faster than for crystals with similar impurity concentration. 3) At low temperature after an excitation pulse a significant rise front of rare-earth related luminescence was observed. The rise front is strongly dependent on rare-earth ions concentration and temperature. The experimental facts allow us to suggest that energy transfer from the host lattice to the rare-earth ions differs in NP and crystals and an appropriate model will be suggested and discussed.

17:20 Poster C10

Vapor phase synthesis of nanocrystalline YAG:Ce

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Nanocrystalline YAG:Ce ($Y_3Al_5O_{12}$:Ce) is a highly efficient phosphor used for light conversion in white LEDs. YAG:Ce with an average particle size of 10…50 nm is a promising candidate for this application. We report about synthesis of YAG by vapor phase reaction (CVR) in a hot-wall reactor. In a first step the formation of the YAG phase by CVR has been studied. Structural analysis of the phases formed using different synthesis parameters was done by x-ray diffraction. It is found that up to a temperature of 1200 °C other Y-Al-oxides form instead of YAG. Scanning calorimetry experiments (DSC) have been performed in order to investigate the phase formation at different temperatures and heating rates. These results serve as a basis for the modeling and the optimization of the parameters of the CVR synthesis. Samples with Ce concentrations ranging from 0,1 at.% to 4,2 at.% have been prepared to investigate the influence of the doping concentration on the luminescence of the YAG:Ce. Photoluminescence has been used to investigate the optical properties of the YAG host lattice doped with Cerium. We will report on the results of as-prepared and annealed samples.

17:20 Poster C11

The luminiscent response on the structure imperfection of Nd: YAG crystals, nanopowders and nanoceramics

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The possibility of fabrication of the active elements from the Nd:YAG singlecrystals and ceramics is determined by the perfection degree of their crystal structure. One of the imperfection reasons is the presence of the impurity crystal phases. The X-ray analysis for their determination, which is carried out on the finished substances is commonly used. Since some of them appear to be useless for active elements, the problem of express structure analysis of singlecrystals and the ceramics in the synthesis process is actual. In present paper the luminescent analysis is presented, which can be used for determination of synthesized substances quality. The luminescence was exited by CLAVI-device [1]. The singlecrystals, nanopowders and nanoceramics with the composition similar to Nd:YAG stoichiometry were investigated. The general forms of the samples spectra are similar, but there are some differences in the intensities of the certain bands. The differences were observed upon the Nd3+ radiation bands in the 450-475nm (S1) and 550-575nm (S2) ranges. These bands have a common upper level 2D5/2 [2]. As intensities of these bands light-amounts s1 and s2 are accepted, they are normalized to common light-amount S0(350-650nm). The dependence of intensities of all spectral bands on yttrium content in the crystals are

characterized by the indetermination in the rage of yttrium content C0=44.93% corresponding to YAG stoichiometry. There is the linear dependence between s1 and s2. The experimental points of s1 and s2 of nanopowders form the individual group, which corresponds to yttrium content of C>C0. These points are situated above the correlation curve of the crystals, but out of indetermination range. In this range the linear correlation between s1 and s2 is also observed. The nanoceramics was synthesized by the technology described elsewhere [3]. The experimental points of the ceramics form the individual group with the yttrium content of C<C0 at s2 is about constant.

17:20 Poster C12

Effects of thermal treatment on Eu:YAG nanopowder structure

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The role of thermal treatment at different time and temperature on the structure of *Yttrium Aluminium Garnet* nanopowders doped with europium ions (Eu:YAG) will be presented in order to understand the structure evolution. Nanopowder of Eu:YAG has been obtained by co-precipitation method treating aluminum, yttrium and europium nitrates solution with ammonia. The hydroxides were calcined at different temperature (900, 950 and 1050°C) for different times (1, 2 and 6 hours). The presence of Eu was confirmed by Energy Dispersive X-ray analysis. By X-Ray Diffraction has been found that the sample treated at 900°C for 1h is constituted essentially by YAG phase, but a minor hexagonal $YAlO_3$ phase is present also. At longer time treatment and higher temperature, the samples are constituted by only YAG phase. Cubic garnet lattice parameters increase with both temperature and time. For the sample treated at 900° for 1h, Electron Microscopy showed spherical particles agglomerates whose size is smaller than 40 nm. Particles sintering has been observed at higher temperature and longer time.

17:20 Poster C13

Preparation of ZnO-Based Mixed Metal Oxide Nanoparticles via Polymer Precursor

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Two kinds of nanometer-sized mixed metal oxide (MMO) particles ($Zn_{1-x}Mg_xO$ and $Zn_{1-x}Co_xO$) with very precise stoichiometry are prepared employing a versatile polymer-based method. The precursor is formed by loading different metal ions in polyacrylates and purified via repeated precipitation/redissolution cycles. The calcination of polymer precursor at relatively low temperature (550 ^oC) gives metastable solid solution of oxide systems in a particular range of composition. The MMO crystal particles are typically 20-50 nm in diameter. Doping of Mg in ZnO lattice causes a shrinkage of the lattice parameter *c*, while it remains unchanged with Co incorporated. Effects of band gap engineering on the optical band gap are seen in the Mg:ZnO system. The photoluminescence in the visible is enhanced by incorporation of magnesium on zinc lattice sites, while the visible emission is suppressed in the Co:ZnO system. This points toward the usefulness of our approach to create photo emissive materials with tuneable sensitivity. Magnetic properties of cobalt doped-ZnO were investigated as well. Ferromagnetic ordering was not found in our sample.

We believe that this method has a wide scope of application for the preparation of powders of mixed metal oxide alloys of metastable type from a polymer metal ion complex as the precursor, which is converted to the MMO at relatively low temperature where sintering is not yet prevailing.

17:20 Poster C14

The effect of yttrium and indium doping on structure of zinc-ferrite nanoparticles

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Yttrium- and indium-doped zinc-ferrite nanoparticles ($Zn_{1-x}M_xFe_2O_4$ for $0 \le x \le 0.6$) were prepared using a coprecipitation method. The nanoparticles were charactarized by conventional powder X-ray diffraction, energy dispersive X-ray spectrometry, transmission electron microscopy, Raman and Mössbauer spectroscopy. The influence of doping level on the structure of the as-synthesized nanoparticles were investigated. It was found that Y-doped $ZnFe_2O_4$ nanoparticles, with single spinel phase and size of few nanometers, are formed in a whole range of yttrium addition. On the other hand, In-doped $ZnFe_2O_4$ nanoparticles can be formed for all *x* investigated, but only the samples $Zn_{1-x}In_xFe_2O_4$ with $x \pounds 0.15$ are single phase spinel (for $x^3 0.20$ a second phase is formed).

17:20 Poster C16

Cathodoluminescence of Al doped ZnO nanopowders

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ZnO powders are well known material for fluorescence displays. The luminescence band peaking at 2.5 eV one increases after firing in a reducing atmosphere. The origin of this band is not clear, but the defects such as Zn interstitial or oxygen vacancies are involved. The interstitial oxygen is one of the possible defect center responsible for the luminescence band at yellow-red region. In present investigation pulsed electron beam excitation (energy of electrons was 280 keV, pulse duration was 8 ns) was applied for cathodoluminescence spectra and luminescence decay studies on ZnO nanopowders with different Al concentration. The two types of powders were studied: obtained by hydrothemal (HY) synthesis and by vaporisation - condensation (VC) in solar reactor from the HY powders . The differences between of HY and VC powders have been studied. It is shown that along the differences obtained in SEM and TEM images (whiskers and spherical type powders) the differences in cathodoluminescence spectra were observed: VC samples show higher level of defect states luminescence and luminescence in exciton (blue) region. After VC the blue luminescence rise more than 10^3 times and luminescence maxima of defect band shifts from yellow (1.9 eV) to green (2.4 eV). These results confirm the change of defect type and take place the crystalline structure ordering during VC process.

The work was supported by SPUB-COST of Polish Ministry for Science and Higher Education and Project SOLFACE-LUMINANOS

17:20 Poster C17

Synthesis and XRD/PL studies of pure and Sb_2O_3 doped ZnO nanophases

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The synthesis of pure and Sb_2O_3 (0 to 5% molar fraction) doped ZnO has been realized by sublimation-condensation method in a solar furnace. The characterization of the initial and final powders was done by means of XRD and PL techniques.

The XRD obtained results showed: the formation of a new phase $Zn_7O_2Sb_{12}$ in the micropowders which disappears in the nanopowders. These results showed also a small increase of the grain size as a function of Sb_2O_3 concentration in the nanopowders. The analysis of the photoluminescence spectra allowed us to establish the following features: a decrease of D°X and D^+X and an increase of A°X peaks when the Sb_2O_3 concentration increases for the micropowders and an increase of D^+X and no change in A°X and no peak shift and a disappearance of X peak as far as nanopowders are concerned for all considered compositions.

17:20 Poster C18

Nanostructured ZnO: from Monodisperse Nanoparticles to Nanorods

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A simple method was used to prepare monodispersed ZnO nanoparticles, which can be used as seeds for the synthesis of [0001]-oriented ZnO nanorods by refluxing. Based on the observation of shape and size of ZnO nanocrystals at different refluxing times and surface and attachment energies for various crystallographic planes calculated from the BFDH and HP models, a possible growth mechanism for ZnO nanorods was put forward to explain the experimental observations. The synthesized ZnO nanocrystals show green emission, which are correlated with the degree of oxygen vacancies in the samples.

17:20 Poster C19

Morphology of Al doped Zinc Oxide Obtained using Hydrothermal and Vapour Condensation Methods

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The aim of our work is the description of the morphology of Al - doped zinc oxide obtained by the hydrothermal and vapour condensation methods as well as changes in its alttice cosntant with Al ions addition. The shape and grain size were analysed by Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD). The helium pycnometer method was applied for density measurement. The dependence of density and lattice parameters on Al concentration is presented.

17:20 Poster C20

Characterization of Phosphorus Spin Resonance in Silicon Nanowires

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Several sets of self-assembled silicon nanowires grown using two different catalytically enhanced methods are characterized using electron spin resonance, scanning electron microscopy, and x-ray diffraction. Some of the samples were highly doped by paramagnetic atoms (P^{31}) during growth. Undoped samples of unattached to substract nanowires show the resonance signal of dangling bonds with g=2.0055 and line width DH=7.2 G. In the doped samples a single line of P^{31} strongly narrowed was observed. The anisotropy of the P_{31} g-factor was found, indicating compressive stress T in the nanowires. The dependence of the g-factor on stress was analyzed, and the value of T along the [100] direction was estimated to be T_{100} » 0.2-0.1 GPa. All spectral lines from nanowires except the signal of dangling bonds are characterized by a small value of the line width 1. Oe≤ DH ≤ 2.6 Oe, which suggest long spin life time.

17:20 Poster C21

Prepared Multi-Element Magnetic Nanoparticles

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MnFe, MnFeCo, MnFeCoNi,and CrMnFeCoNi magnetic nanoparticles are synthesized by superhydride reduction of CrCl3, MnCl2, Fe(acac)2, Co(acac)2 and Ni(acac)2 at high temperature, and nanoparticles are dispersed by oleic acid in hexane. Adding the LiB(Et)3H to the phenyl ether solution of metal precosor in present oleic acid, and 1,2 hexdecanediol at 180□, followed by refluxing at 250□, to prepare the multi-element nanoparticle. Anneal the particles at 400□, 500□, 600□ by RTA. The compositional analyses of nanoparticles are performed by EDS of TEM. The crystal structures are decided by XRD, electron diffraction pattern, and HRTEM. The microstructure of annealed particles are observed by SEM. Magnetic properties of nanoparticles are charactered by VSM.

17:20 Poster C22

Microwave asssited hydrothermal synthesis as a rapid route toward manganites preparation

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The manganites $A_{1-x}A'_xMnO_3$ with A a rare-earth metal and A' an alkali-earth metal are materials of great importance owing to their magnetic and electronic properties, particularly the phenomenon of colossal magneto resistance, whereby a decrease in resistivity of several orders of magnitude is seen on application of magnetic field close to the Curie temperature of the material. The manganites have also been studied as oxidation catalysts and as cathodes in solid-oxide fuel cells, and their potential application in electronic devices are highly dependent upon their atomic structures.

The synthesis of these class of perovskites is a challenge since control of manganese oxidation state. Main preparation methods of these compounds consist on solid state reaction and also sol-gel route, which require drastic or complex conditions. Hydrothermal method in combination of microwave technology is a advantageous tool for the efficient obtainment of inorganic compounds like manganites.

In this work attempts to prepare strontium-doped lanthanum manganites $La_{1-x}Sr_xMnO_3$ using microwave-assisted hydrothermal synthesis are performed starting from a mixture of lanthanum nitrate, strontium nitrate, manganese(II)nitrate, potassium permanganate and potassium hydroxide as mineralizer. For x = 0.5 the relative perovskite is obtained not pure. While with x = 1 for the first time hexagonal strontium manganite is prepared as tablet shaped crystallites with narrow particle size distribution (ca. 6 mm) at 210°C in 1 hour treatment. Convetional hydrothermal synthetic routes required at least 24 hrs treatment time.

17:20 Poster C23

Effect of the A-site randomness on magnetotransport properties of $PrBaMn_2O_6$ manganites.

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A-site ordered manganites of the $PrBaMn_2O_6$ system have been obtained using a two-step synthesis and characterized with respect to the chemical composition, crystal structure, magnetic and magnetotransport properties. The physical properties of the A-site ordered $PrBaMn_2O_6$ manganites obtained using this method significantly differ from the properties of A-site disordered $Pr_{0.50}Ba_{0.50}MnO_3$ synthesized by means of the conventional ceramic technology and depend on the degree of ordering of the Pr^{3+} and Ba^{2+} cations. In

particular, the A-site disordered $Pr_{0.50}Ba_{0.50}MnO_3$ has a cubic perovskite-like unit cell, while A-site ordered $PrBaMn_2O_6$ has a tetragonal unit cell. Cation states in the system under study are reversible. The A-site ordered $PrBaMn_2O_6$ state remains stable upon heating in an oxidizing medium up to 1300 ^{0}C. The ordering of the Pr^{3+} and Ba^{2+} cations leads to a significant increase in the critical temperatures of phase transitions. In particular, $PrBaMn_2O_6$ with the maximum degree of ordering is a metallic ferromagnet with the Curie temperature T_C ~ 320 K, whereas T_C of a fully disordered sample is on the order of 140 K. The samples with intermediate degrees of ordering contain two magnetic phases. Slightly below T_C, all such samples exhibit a metal-insulator transition and a peak of the magnetoresistance, which amounts to approximately 10 and 65 % in a magnetic field of 9 kOe for the fully ordered $PrBaMn_2O6$ and disordered $Pr_{0.50}Ba_{0.50}MnO_3$, respectively. The results are interpreted in terms of the Goodenough-Kanamori empirical rules for indirect exchange interactions with allowance for the degree of ordering of the Pr^{3+} and Ba^{2+} cations.

17:20 Poster C25

Synthesis of Nano-Powder Materials Using Spray-Pyrolysis Method.

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In this work the laboratory installation for synthesis of powder materials with nanosize grains is presented. Nano-sized zinc and tin were obtained using the spray-pyrolysis (SP method). The zinc oxide was derived from zinc acetate at 300, 430, 580, 800 i 950^{o}C. The tin oxide had been produced by the thermal decomposition of tin tartrate at 500^{o}C. The SP synthesized zinc oxide was used for processing of methane sensor. The methane sensor made of SP derived zinc oxide was characterized with much wider range of operating and substantially (10 times) higher sensitivity when compared to that made of commercially available zinc oxide.

17:20 Poster C26

Nanopowders of hydroxyapatite doped with NH_4NO_3 and $CaCl_2$

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The nanopowder of hydroxyapatite (HAp) synthesized by means of precipitation demonstrates a very high specific area and a good ability to adsorb different ion from solution. The aim of the present work was to investigate properties and thermal behavior of HAp with NH_4NO_3 (by-product of HAp synthesis) and $CaCl_2$ and densification of nano-sized hydroxyapatite (HAp) powder compacts. Powders of HAp were fabricated via wet-precipitation technique from $(NH_4)_2HPO_4$ and of $Ca(NO_3)_2$ solutions at 60^{o}C with pH=9 and pH=7. Ratio of Ca/P was 1.67, 1.61 and 1.48. $CaCl_2$ was added to the powder of HAp in quantity of 10 % wt. The powders and ceramics were tested with XRD, FTIR, TGA, TEM, SEM/EDX and dilatometry (up to 1000^{o}C). DTA experiments showed that increasing of concentration of salt solution leads to higher adsorption of NH_4NO_3 by-product by HAp precipitate from the mother liquor. According to dilatomeric curves NH_4NO_3 can be considered as a densification additive for the HAp compacts since it undergoes melting in the interval of 150-250^{o}C. Maximum rate of linear shrinkage for the powder of HAp (Ca/P=1.67, pH=9) was located in the interval of 850-950^{o}C. Reduction of Ca/P value at pH=7 resulted in changing of composition of adsorption layer due to presence of extra phosphate-ion in solution. While heated, extra $(PO_4)^{3-}$ can react with the main phase, by-product and with an additive affecting the sintering process. When adding $CaCl_2$, the maximum of the rate linear shrinkage was in interval of 500-700^{o}C.

17:20 Poster C27

Title: Factors Influencing Dispersion of Cobaltoxide Nanoparticles in Mesoporous Silica SBA-15.

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Cobalt containing mesoporous molecular sieve SBA-15 with different silicon to cobalt mol ratios have been synthesized by various methods. The samples were characterized by XRD, TEM, BET, FT-IR and ICP-AAS. The effect of cobalt salt addition sequence to solution, such as after polymer and after silica source were studied to observe stability of mesoporous structure. Same composition of cobalt containing SBA-15 materials were prepared by wet impregnation method.The sol-gel derived composite with cobalt salt added after polymer is more stable in the structural regularity of mesoporous material than the cobalt salt added after silica source and the wet impregnated sample. Although the powder XRD pattern of Co containing SBA-15 samples display characteristic peaks of Co_3O_4 TEM measurements did not indicate presence of nanaoparticles. This behaviour of samples proves the high dispersion capacity of cobalt oxide nanoparticles in SBA-15 mesoporous silica material.

17:20 Poster C28

Wurtzite-to-rocksalt structural transformation in nanocrystalline CoO

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Hexagonal CoO nanocrystals are synthesized by hydrothermal reaction. Structural stability and phase transformation of the hexagonal CoO phase have been investigated by x-ray powder diffraction with Rietveld refinement, transmission electron microscopy, x-ray absorption fine structure and differential scanning calorimeter. It is found that hexagonal CoO phase is a metastable phase, which in-

creases its grain size from 50 to 250 nm for refluxing times from 1 to 6 h at 200 °C. After 12 h, cubic-structured CoO grains with an average grain size of 20 nm are observed, which spread around big hexagonal CoO grains. After about 24 hours, only cubic CoO phase with an average grain size of 25 nm is detected. The onset temperature of hexagonal-to-cubic phase transformation in CoO is estimated to be 378 °C by DSC using a heating rate of 20 K/min. The results obtained indicate that the hexagonal-to-cubic phase transformation in nanocrystalline CoO is by nucleation and growth mechanism, starting from surface to center of the hexagonal grains.

17:20 Poster C29

Photoluminescent properties of oxide nanopowders synthesis by pulsed laser evaporation

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In the paper the method of producing of complex nanopowder materials and barrier structures on their basis by means of pulse laser reactive evaporation of metal targets is offered. It enables to effectively produce nanopowders with mean geometrical diameter up to 5-10 nm and specific surface more than 80-190 m^2/g at productivity up to 50 g/h. The carried out researches of photoluminescent properties of nanopowder materials ZnO and TiO_2 (after laser anneling including) with the purpose of studying the opportunity of creation on their base a gas sensor. The influence of adsorption of molecular gases O_2, H_2, N_2, and CO on change of intensity of photoluminescence of nanopowder ZnO is investigated. On the basis of the carried out researchers the method for detection, recognition and estimation of gas concentration in the analyzed enviroment is proposed. With the purpose of increasing the selectivity of the method it is offered to analyze the radical-recombination luminescence, which spectral characteristics are determined by the given superficial condition of granules of nanopowder and chemical structure of the analyzed gas.

17:20 Poster C30

Growth and properties of ytterbium doped $KY(WO_4)_2$ nanocomposites

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Nanocomposites (nanocrystals) of KY(WO) and KY(WO)+1% mol Yb have been synthesized using a Complex Sol-Gel Process (CSGP). The chemical treatment with concentrated nitric acid and hydrogen peroxide was used to decrease the decarbonisation temperature.

The expected monoclinic phase C2/c of the KYW of the nanocomposite powder has been confirmed using XDR technique. From the X-ray diffraction measurements, the unit cell parameters and the size of a nanoparticles have been determined. Electron spin resonance studies in the X band have been performed on KYW and KYW:Yb nanocrystals.

The sintered samples were made with using the high pressure technique in the temperature up to 600^oC. The chemical analysis, the X-ray diffraction measurements and ESR investigations were made for sintered samples too.

17:20 Poster C31

Spectroscopic study of sol-gel SiO_2-SnO_2 glass-ceramics with controlled nanocrystal size

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In the past few years, nanostructured materials have focused much attention due to their potential technological applications. In particular when doped with RE-ions they are attractive for photonic applications [1]. With appropriate choice of host matrix and RE doping optical properties can be enhanced notably. In this respect, properties of semiconductor nanoparticles are interesting when their sizes are comparable or below the exciton Bohr radius. Tin oxide is a versatile n-type semiconductor with a wide band gap of 3.6 eV at 300K. Materials based on tin oxide have a wide range of applications like gas sensors or transparent conducting electrodes [2]. Tin oxide nanocrystals should have better optical properties than the bulk ones owing to quantum confinement.

In this work nanostructured samples based on SiO_2-SnO_2 have been obtained by a thermal treatment of sol-gel precursor glasses. Structural analysis has been performed by means of X-ray diffraction. The average size of the obtained nanocrystals can be predetermined using well controlled concentration of tin precursor. The mean radius of the nanocrystals is comparable to the exciton Bohr radius in an insulator SiO_2 glass. Optical absorption and photoluminescence spectra have been measured as a function of tin oxide concentration. The nanocrystal sizes have been estimated as function of process parameters using the Brus and Scherrer equations. The band gap increase is in conformity with the theorical result based on the effective mass model. A mechanism has been proposed to explain the observed photoluminescence properties.

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17:20 Poster C32

What are the limits of Co and/or Pb loading on SBA-15 structures?

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SBA-15(Santa Barbara amorphous-15) is one of the types of mesoporous silicas with highly ordered hexagonal mesochannels, large pores, thick walls and high thermal stability which make it a promising catalyst support. In this study, SBA-15 samples are modified by doping of Co and/or Pb with different weight loadings. Pure and Co and/or Pb incorporated SBA-15 were prepared by direct synthesis. The synthesized samples were characterized by powder X-ray diffraction, N_2 adsorption isotherms and TEM images. The BET surface area of pure SBA-15 mesoporous structure is observed to be slowly decreasing by the addition of metal oxides. No characteristic peaks of metal oxide are observed in the large angle XRD pattern by the loading of 5, 10, 15 wt %Pb into SBA-15 indicating metal particles are dispersed in the SiO_2 structure without accumulating and forming a crystal in a certain part of the framework. But loading of 20 wt%Pb in SBA-15 showed more appreciable characteristic peaks, indicating small quantity of crystallites of metal oxide existing on the surface of silica. The introduction of high amount of cobalt and lead oxides into SBA-15 resulted in the long range order according to the low angle XRD patterns. The hexagonal structure of pure SBA-15 and 1-D channel structure in hexagonal type can be observed in TEM images. When SBA-15 structures were overloaded with Co, it is observed that Co extends outside the structure.The performance tests of the materials as methane partial oxidation catalyst for hydrogen generation is in progress.

17:20 Poster C33

High Throughput Screening for New Hydrogen Storage Materials

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Arrays of hydrogen storage materials were synthesized in an air and moisture free environment. Several innovative techniques to synthesize, characterize and screen materials for reversible hydrogen sorption will be reported, these include:

- high-energy ball milling - 96 solid state or slurry based reactions for synthesis or catalyst doping.
- diffusion multiple - metal and intermetallic alloy synthesis.
- reactive thin film diffusion multiple - metal and intermetallic alloy synthesis.

High throughput techniques for evaluating hydrogen sorption / desorption properties include, optical imaging, thermography and ToF- SIMMS. Materials identified with promising performance were scaled -up and analyzed with traditional thermal desorption techniques.

17:20 Poster C34

The effects of oxidized and non-oxidized boron on the Mg-B-H nanohydrides transformation in the nearly nanosized powders

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In this work oxidized and non-oxidized amorphous boron (a-B) powder was mixed with the elemental Mg in the atomic ratio 2:1 with the intent to mechanically synthesize the $Mg(BH_4)_2$ complex hydride. The blends were subjected to the controlled reactive mechanical alloying (CRMA) under hydrogen in a magneto-mill up to 200h. In effect, the particle size was refined to the 100-200nm range. Nanocrystalline MgH_2 (~6nm crystallite size) has been formed within the particles when an oxidized a-B is used. In contrast, a mixture of MgB_2 and an amorphous hydride MgH_x (a-MgH_x) has been formed when a non-oxidized a-B is used. The XRD pattern for the nearly-nanopowders containing non-oxidized a-B demonstrates the complete disappearance of the MgH_2 phase. Instead, the peaks rising above the background can be attributed to MgB_2. We are inclined to attribute it to the hydrogen evolution since desorption conducted in a Sieverts-type apparatus revealed ~1.4wt.% of hydrogen release. The XRD pattern for the powder after DSC test exhibited strong peaks from the MgB_2 phase. Oxide doping effect on the observed phase transformations is discussed.

17:20 Poster C35

Comparative analysis of ZnS:Mn^{2+} nanophosphors prepared by low-temperature precipitation and hydrothermal methods

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Manganese-doped ZnS nanophosphors were synthesized through two different methods: low-temperature precipitation method and hydrothermal method. Both ZnS:Mn^{2+} nanophosphors had the size of 20 nm and the spherical shape, and the cubic-dominant double phases of cubic and hexagonal. Both showed the yellow emission

peaks around 600 nm originated from ^4T-^6A transition of Mn^{2+} ions. The emission intensity of the nanophosphor prepared by low-temperature precipitation method was optimized at 3 mol% of Mn^{2+} concentrations whereas that by hydrothermal method was optimized at 10 mol% of Mn^{2+} concentrations. The former nanophosphor showed the blueshift in the excitation spectrum and the redshift in the emission spectrum in comparison with the latter nanophosphor. The decay time of the former nanophosphor was longer than that of the latter nanophosphor. These phenomena can suggest that the higher quenching concentrations, the blueshift and the shorter decay time generally observed in nanophosphors are originated not from the nanostructure but from the difference in the synthesizing processes.

17:20 Poster C36

Optical properties of ZnS:Mn^{2+} yellow nanophosphor prepared by spray pyrolysis

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Spray pyrolysis was applied to prepare ZnS:Mn^{2+} yellow nanophosphor. As an increase of pH of spraying solution, the ZnS:Mn^{2+} nanophosphor showed the hexagonal-dominant phase, whereas, as an increase of firing temperature for spray pyrolysis, it showed the cubic-dominant phase. The emission intensity of the ZnS:Mn^{2+} nanophosphor was optimized at the pH of 3.5 and the temperature of 900 □. The ZnS:Mn^{2+} nanophosphor showed the broader excitation spectrum but no difference in the emission spectrum in comparison with the microphosphor synthesized by solid-state reaction. The temperature-dependence photoluminescence showed the different spectroscopic behavior from the microphosphor. The time-resolved photoluminescence show slightly longer decay time than the microphosphor. These behaviors can be explained in terms of the interaction of Mn^{2+} state and impurity level of nanophosphor.

17:20 Poster C37

Doping of ZnO nanopowders with Mn, Ni and Cr In an ultrasound and microwave driver hydrothermal reaction

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Nano-sized powders of Mn, Cr, Ni and Co ions doped ZnO have been synthesized in a sonochemical reactor and in a microwave domestic oven.

The density, grain size and lattice parameters of the samples have been investigated as a function of the ions concentration in the reaction substrates.

Our result indicate that in Mn ions doped (or alloyed) powders no precipitates are observed even 20 mol% doping level. Phase analysis of the powders obtained shows that at 10% concentration of ions in the reaction substrates only for Mn doping pure ZnO phase was observed. For the other elements a peak corresponding to their oxides appears, indicating a lower solubility limit in the ZnO lattice then the case of Mn.

For Mn doped samples, luminescence and magnetic properties of the samples were measured.

The result obtained indicate that both the ultrasound and microwave driven reactions are suitable for doping ZnO with transition metals ions. However, the microwave reaction was more efficient, most likely because of the higher average temperature comparing to the ultrasound driven one.

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session)Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium D

Polymer materials modified by nanoparticles

Welcome

The topics of the Symposium will include: methods of nanoparticles characterization and manufacturing, modification of polymers with those nanoparticles in order to develop new constructional and functional materials, finding correlations between structure and properties of nanoparticles and their activities, technology of new polymer materials modified by nanoparticles, and development of preliminary technologies suitable for usage by Small and Medium Sized Enterprises (SMEs). The objects of research are e.g. layered, fibrous and solid constructional polymers as well as solid functional polymers, all modified with nanoparticles.

Different types of nanoparticles, like aluminosilicates, oxygen based nanofillers (silicon dioxide, titanium white, zinc oxide and others), phosphate and aluminorganic nanofillers will be discussed.

Nanomaterials have been studied extensively over the last decade. The research, initially of basic character, now is concentrated on technologies of production of such materials and on broad investigations of their properties. Various techniques for production of nanometals, nanoceramics and nanocomposites have been developed and superior properties of nanomaterials have been reported.

Experience of the scientists from the old countries of European Union (EU) in the research area of nanosciences and nanotechnologies is still wider than that of their colleagues from Poland and from other countries of the Eastern Europe. Researchers from the old EU countries could share the experience from their National Projects with the researchers from the new member countries. The Symposium will be a good occasion for the scientists from the new member countries and from the other countries of the Eastern Europe to start collaboration in the trans-national research projects. The Symposium will also be fruitful for Small and Medium Sized Enterprises (SMEs) from Poland and from other Eastern Europe Countries. It will make nanoscience and nanotechnology more comprehensible for the people of SME-s. The programme of the Symposium will ensure its importance for students, PhD students and post docs.

Scientific Committee:

Prof. Ludomir Slusarski(Chairman), Technical University, Lodz
Prof. Andrzej Galeski, Center of Molecular and Macromolecular Studies, Polish Academy of Science, Lodz, Poland
Prof. Zbigniew Roslaniec, Szczecin University of Technology, Poland
Prof. Tadeusz Spychaj, Szczecin University of Technology, Poland
Prof. Stefan Wojciechowski, Warsaw University of Technology, Poland

Organisers

- **Prof. Ludomir Ślusarski,** Institute of Polymers,Technical University of Lodz, Poland, sludpolp@mail.p.lodz.pl
- **Dr Joanna Ryszkowska**, Faculty of Materials Science and Engineering, WUT, Poland, jrysz@meil.pw.edu.pl

Proceedings

After a regular review, the accepted manuscripts will be published in a special issue of **POLIMERY**.

Programme

Monday, 4 September

Opening Ceremony
2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Poster Session 1
Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Parallel Session
Tuesday morning, 5 September, 9:00

9:00	Invited oral

Elastomer/Synthetic Clay Nanocomposites : Preparation, Structure and Reinforcement

Alain Vidal

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Polymer/clay nanocomposites are investigated using mainly thermoplastic polymers such as polyamide-6, polyethylene, polypropylene, … Much less attention has been paid to rubbers and rubber-like materials. Our objective is to define parameters controlling clay exfoliation in elastomer matrices by direct blending. The latter condition implies no solvent use in the blend together with preserving maximum exfoliation to a single layer level.
Barriers hindering rubber incursion inside clay structure are identified as due to the characteristics of the clay on the one hand (hydrophilicity, surface free energy, interlayer gap, ion exchange capacity,…), and of the polymer on the other hand (hydrophobicity,

surface free energy, molecular weight and molecular weight distribution,…).
In this work we present an attempt to overcome such obstacles by three major means :
- clay synthesis
- chemical modification of the synthesized clay
- chemical modification of the polymer and MW variation
Since natural clay, as all natural products, suffers from a lack of reliability, and therefore exhibits an experimental irreproducibility, we chose to synthesize our clay in the laboratory. Thus, the sodium form of a montmorillonite-type [$Na_{0.3}(Al_{1.7}Mg_{0.3})Si_4O_{10}(OH)_2$] clay (Mmt) was prepared by hydrothermal synthesis in hydrofluoric acid medium at 20 °C for 48 hours in a stainless steel PTFE lined autoclave. Further selection and control of the particle size and its influence was investigated.
Clays are basically hydrophilic materials, as a consequence they cannot be blended properly with hydrophobic matrices such as elastomers. Two ways of material modification can be anticipated to overcome this dilemna:
- grafting the rubber with species having natural affinity for the clay and/or
- converting the clay into an organophilic material by cation exchange treatment.
Both ways were investigated, chemical structures were controlled by elemental and TGA analysis, exfoliation was monitored by X-ray diffraction TEM and rheological measurements and hybrid composites were tested by dynamic mechanical thermal analysis.Results will be discussed in term of modification/exfoliation relationship as well as polymer/clay-monolayer adsorption and hybrid three-dimensional network formation.

9:40 Oral

Rheological and thermal properties of waste polycarbonate/nanoclay composites

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The influence of organically modified montmorillonite on rheological and thermal properties of waste polycarbonate was investigated. For the study, nanocomposites of PC and natural montmorillonite (Nanocor Nanomer PGW) or chemically modified clay (Nanocor Nanomer I.30P or Nanocor Nanomer I.44P) were prepared by melt blending in HAAKE roller-type mixer. Both used organoclays belong to group of quaternary ammonium modified montmorillonites. In all PC/Nanomer composites the concentration of clay was 5 wt%. The thermogravimetric analyze (TG) results show that thermal decomposition of organically modified montmorillonite starts in the range of 220-230ºC, while the thermal stability of PC/clay nanocomposites is much better and begins at 320ºC. The linear dynamic viscosoelasticity measurements shows that the PC/clay nanocomposites containing organically modified montmorillonite reveal strong thermal degradation, which may result from quaternary ammonium decomposition during the mixing processing and further initiates of matrix thermal degradation.

10:00 Oral

Surface and interface effects of nanoparticles in polymer materials

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The optimal adhesion parameters i.e. the increased work of adhesion, lowered interfacial free energy and positive coefficient of wetting, was revealed as parameters for the effective adhesion at the interface between the selected nanoparticles and polymer matrix in comparison to its microcomposite counterparts. In this paper we will make an overview of our results about the surface effects of the selected calcium carbonate ($CaCO_3$) nanofillers vs. microfillers in polyvinyl acetate (PVAc) and/or polyurethane (PUR) matrix. The previous results about the relatively new approach in compatibilizing the immiscible blends by addition the selected stearate pretreated $CaCO_3$ nanofilers and its specific surface effects on polymer matrices in blends (SAN/EPDM, PP/PVC) will be also presented. The changes in surface functionality of calcium carbonate nanoparticles by the specific surface pre-treatments, for example with silanes, resulted in higher work of adhesion with PU matrix due to the increased interactions at the interface. On the other hand some pretreatments of calcium carbonate by irradiation in a presence of monomer and/or with stearic acid might lowered the interactions with PVAc matrix. Significantly higher coefficients of interactions in nanocomposites vs. microcomposites was indication of the mechanical properties improvements. The homogeneous morphology without agglomerates was found as important factor for the increased interactions with significant amount of immobilized polymer chains in the interfacial area. We found that *in situ* technology of preparation the PVAc+$CaCO_3$ nanocomposites in comparison to the *mixing* procedure result in the more homogeneous morphology and significantly improved properties even with a smaller amount of nanofillers.

Coffee break
Tuesday morning, 5 September, 10:30

Parallel Session
Tuesday morning, 5 September, 11:00

11:00 Oral

Nanocomposite Manufacturing using Ultrashort-Pulsed Laser Ablation in Solvents and Monomers

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Even though many techniques for the dispersion of nanoparticles in-

to polymers are avaiable up to now, the enhancement of the material performance is still restricted due to inefficient homogenization and re-dispersion of available nanoparticle materials into the poylmers or masterbatches. Moreover, improvements are still required in terms of their purity, and costs.

In this study, a novel route no nanocomposite manufacturing is presented, based on the in-situ dispersion of nanoparticles into monomers and subsequent polymerisation. The capability of a novel method for nanocomposite manufacturing is demonstrated in examples specific to biomedical applications such as medical devices (implants) and a bionic clamping device for micro-parts.
Direct ultrafast laser ablation of solids is used to design and fabricate nanoparticle-based nanomaterials. Without using any chemical precursors, nanoparticles are generated from different materials and directly dispersed in a liquid, such as acrylic acids, acetone, and water . This direct (in-situ) dispersion of the nanoparticles into prepolymers allows for the manufacturing of highly homogenous nanocomposite materials. In addition, compared to conventional laser ablation, the ultrashort-pulsed ablation gives access to stoichiometric nanoparticles such as shape memory alloys (NiTi) generated from the bulk material.

11:20 Oral

Modification of polyethylene with high energy ions and electrons. Structural, micromechanical and dielectric studies of the surface layer.

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Engineering applications of polyethylene is limited due to its low mechanical durability and wear resistance. Increase of the degree of crystallinity or crosslinking of polymer is not always effective, due to associated increase of its brittleness. This observation makes the modification to be limited to the surface layer of material. Application of ions and electrons for bombardment of the surface of high density (HDPE) or ultra high molecular weight (UHMWPE) polyethylene produces significant changes to the materials in the range of depth not exceeding some hundreds of nanometers. The paper presents nanoindentation data for HDPE and UHMWPE. Hardness and mechanical modulus generally increase due to ion bombardment. The effect is the higher the lower the mass of nanoparticles used, however diminishes for electrons. Figure 1 confirms that HDPE differs from UHMWPE according to interactions with a beam. The data is discussed from the point of view of structural modification of materials, studied by Grazing Incidence X-Ray Diffraction (GIXRD) and dielectric measurements. Degree of crystallinity of the surface layer of polyethylenes increases due to bombardment with light He^+ ions, whereas remains constant when heavy Ar^+ ions are applied. Thickness of crystallites practically does not change in both cases. The surface conductivity of the polymers studied significantly increases due to Ar^+ ions treatment, whereas the effect is less pronounced for He^+ ions and starts for higher beam densities. It suggests that modified layer for the latter is created in the subsurface of materials, gradually propagating towards the surface.

11:40 Oral

Stability and optical properties of gold nanoparticles in different liquids produced by laser ablation

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We study the preparation, stability and optical properties of pure gold nanoparticles prepared by pulsed laser ablation from gold target in water and ethanol as liquid carrier media. Nd:YAG laser at wavelength of 1064 nm, 130 mJ/pulse energy, 10 ns duration and reparation rate of 10 Hz has been used for ablation. Optical properties and structures of nanoparticles were investigated by UV/visible and TEM. The shapes of particles are rather spherical in all of the carrier media. Gold nanoparticles in water are stable and exhibit narrow size distribution with mean diameter of about 7 nm. But in ethanol broad size distribution with mean diameter of 18 nm was observed. Some of nanoparticles gathered to form chain like structures. Gold nanoparticles in ethanol completely precipitated after 2 days. Optical extinction spectrum of nano gold in water is sharp with single maximum at 522 nm, whereas in ethanol a red shift with broadening in spectra has been observed. In addition a rather weak peak at 545 nm was appeared which is related to surface plasmon resonance.In present paper we discussed size and stability of nanoparticles by considering the polarity of different media. This study provides a simple method for synthesis and control the properties of gold nanoparticles produced by laser ablation method.

12:00 Oral

Synthesis and optical properties of silver nanoparticles/ polystyrene nanocomposite

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Silver nanoparticles/polystyrene nanocomposites were prepared via casting the solution of polystyrene in a mixture of carbon tetrachloride and acetone containing silver nanoparticles. Colloidal silver nanoparticles in acetone were synthesized by pulsed laser ablation of pure bulk silver. Nd:YAG laser at wavelength of 1064 nm, 130 mJ/ pulse energy, 10 ns duration and reparation rate of 10 Hz has been employed to synthesis of nano silvers. TEM images show rather spherical nanoparticles with mean diameter of 3 nm. Silver nano-

particles in acetone are stable even after 8 months. Optical measurements of nano silver in acetone exhibit single maximum of optical extinction at 399 nm which is related to surface plasmon resonance. Casting the solution of nano silver in a mixture of carbon tetrachloride, acetone and polystyrene results a transparent polymeric sheet in light yellow color. Maximum of optical extinction of the sheet appeared at 428 nm and exhibited a red shift due to change of refractive index of the medium. The peak optical extinction spectra are in agreement with that calculated from the Mie theory for 5 nm spherical silver particles in polystyrene. This study demonstrates that hybridizing of pulsed laser ablation in liquids with casting method provides a simple, applicable and flexible technique in order to fabricate nanocomposite from different metals and polymers.

Lunch break

Tuesday afternoon, 5 September, 12:30

Parallel Session

Tuesday afternoon, 5 September, 14:00

14:00 Oral

Nanofillers as flame retardants

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Since the late 1990s nanocomposites (polymer-layered silicates based on aluminosilicate clay minerals like montmorillonite, composed of layers with gaps (gallery spaces) in between) have been gaining increasing attention in flame retardancy area. Nanocomposites particularly prevent dripping and promote char formation. Therefore, they have been used as synergists in some polymer/flame retardant combinations. Previously nanocompositeflame retardants have been tested in the plastics like polypropylene, polymethylmethacrylate (PMMA), polystyrene, and polyamides.It is shown that silicate nanocomposites can be successfully used as a flame retardant additive in the case of biodegradable matrix (poly(lactid) acid).Nanocomposites prepared by melt blending were investigated. The materials were composed of poly(lactid acid) with addition of 2, 3, 5 and 8 wt.% of nanofillers (two grades of montmorillonite and calcium carbonate). Melt strength and fire resistance of the composites have been presented.

14:40 Oral

The Nucleating Effect of Carbon Nanotubes on Crystallinity in Thermoplastic Polyimide

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Thermoplastic polyimides (PIs) present several advantages as matrices for advanced composite materials. PIs. Particularly, the thermoplastic PI that can crystallize from the melt, is of high interest. A specific feature is their ability to crystallize from the melt to form crystalline matrices for composite materials. The generation of PI crystallinity after melting is therefore an essential quality for a thermoplastic PI for its use in advanced composites. Here, we report of a further step in the study of crystallization of the PI matrix. It is based on the nucleating action of CNT that are embedded in very low concentrations in the PI matrix. Only a small quantity of CNT is required to induce crystallization, while their ultrahigh properties promise significant mechanical advantages too. The DSC results, show that only in the presence of CNT does the PI recrystallize, exhibiting melting enthalpy of 14 J/g, which amounts to around 15% crystallinity. The polarized light microscopy images of a PI/CNT film, show clearly crystallization around CNT clusters.

15:00 Oral

Synthesis of a Pd-functionalized macroporous polymer material: application to the heterogeneous catalysis of the Suzuki-Miyaura reaction

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We have synthesized and used a new polymer support for the catalysis of the Suzuki Miyaura reaction. The process is based upon the use of the so called polyHIPE mixed material. A macroporous polymer support can be obtained by the formulation of a water-in-oil emulsion. For this purpose we introduce a large quantity of water into a mixture of a polymerizable solvent (styrene in this case) with an adapted surfactant. Since the proportion of water is very high in the system, the final mixture is equivalent to a biliquid foam in which the water cells are separated one from the other by a thin organic film. During the polymerisation process (triggered by a radical initiator and the increase in temperature), the styrene films toughen and small holes appear at the same time, leading to the formation of a fully connected material. Water can then be evaporated and the obtained porous polymer support is further used for the in-situ growth of palladium nanoparticles. First, the polymer surface is functionnalized by various specific organic functions which are known to favour the stabilization of palladium nanoparticles. The support is then put into a palladium salt solution. The palladium salt reduction process occurs at the surface of the polymers leading to the in situ growth of metallic palladium nanoparticles. A particular attention should be given to the used reduction trigger in order to get nanoparticles having a narrow size distribution. The obtained mixed material is then used for the catalysis of the Suzuki-Miyaura reaction and its performances are compared to what can be obtained with more classical types of palladium supports (such as Pd/C) and ho-

mogeneous catalysts.

Coffee break
Tuesday afternoon, 5 September, 15:30

Parallel Session
Tuesday afternoon, 5 September, 15:50

15:50 Oral

Polyoxymethylene modified by montmorillonite - preparation, characterisation, properties

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Polyoxymethylene is one of the important engineering polymers which is characterised by high mechanical and thermal stability, as well as barrier properties. The recent developments are focused on POM-based nanomaterials that incorporate nanofillers homogeneously distributed in the polymer matrix. One of the most promising is montmorillonite (MMT) which consists of layers made up of two silicate tetrahedron fused to an adge-shared octahedral sheet of either aluminium or magnesium hydroxide. Contrary to conventional microcomposites that require few tens wt % of filler to rich desirable improvement in mechanical properties, nanocomposites offer significant improvement at filler content of only few wt %. Such low-filled polymeric compositions have processing properties similar to this of pristine resin and typical problems of microcomposites' processing, such as changes in melt viscosity or surface roughness of moulded parts, do not occur [1,3].

In our work we have obtained by injection molding a series of organomodified POM-MMT nanocomposites which were then subjected to structural, morphological, thermal analysis and mechanical properties investigations which will be reported in the presentation.

Acknowledgements

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16:10 Oral

Nonwoven textiles modified by deposited nano-layers

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A layer-by-layer technique was used for deposition of nano-layers of polyelectrolytes onto nonwoven textiles.

Polypropylene and polyester nonwovens grafted by acrylic acid were used as starting material for deposition of polymer complex layers. It was found that a layer-by-layer technique makes it possible to produce materials with diversified properties. Depending on the layer structure, hydrophilic properties, electro-kinetic potential and absorption of dyes were changed. Water-tightness and remission of methylene blue were measured for samples with different types of deposited layers.Using deposited layers as nano-reactors polymerization of aniline was carried out in order to obtain electro conducting materials. Moreover, nano-particles of gold, silver and metallic oxides were deposited onto such layers. Properties of the obtained materials were tested. It was found that particles are distributed without aggregation.

16:30 Oral

Studies on the crystalline phase of iPP/organo-modified montmorillonite composite fibers

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The isotactic polypropylene (iPP) fibers are among the most important synthetic fibers. To improve their mechanical properties many attempts have been made through last years. One manner of overcoming this problem is presented in this paper. Our modification introduced into the traditional technology of manufacturing iPP filament, consisted in physical blending of iPP for textile use with a special clay reinforcement CloisiteÒ 30B obtained from Southern Clay Products Inc. in the melt. Our iPP/Cloisite 30B composite fibers (0-5% of the nano-filler) were formed from the melt by means of a laboratory spinning machine. The main goal of investigations presented in this paper was to determine the influence of the content of nanoclay on the supermolecular structure of fibers formed from such modified iPP by means of X-ray (WAXS and SAXS) and the differential scanning calorimetry (DSC) methods. In our DSC study we also took an attempt to describe the transformation of the crystalline phase of modified undrawn iPP fibers occurred under the influence of conditions of the formation process. The crystallinity index was evaluated on the basis of the melting enthalpy determinated by the extrapolation from the melt. The "pseudo-c_p" DSC measurements were performed.

16:50 Oral

Nano-identification of the bonds between the concrete elements

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Scientists are discovering a host of new properties in everyday materials by manipulating, measuring and introducing minuscule bits of matter a million times smaller than a pin-head. Any activity concerned with particles at a scale around 0.1-100nm (nanometres) is classified as Nanotechnology. Characterisation of the bonds between elements of the concrete microstructure by means of nano-identification and SEM image analysis will help us better understand this microstructure of the concrete material on nano-level. This valu-

able information obtained, will in turn help industry improve the bonds on this level and maybe new properties might immerge for concrete. This paper summarises a literature survey conducted as part of a study to characterise the bonds between the elements of the concrete microstructure, which will comprise of the following points of relevance. Firstly, the method of preparing the specimens for the SEM image analysis. Secondly, the method of conducting the SEM image analysis. Thirdly, the five point analysis to be performed on potential representative spots on the border line between the two different materials under question. Fourthly, the performing of the spectra analyses to obtain spectra graphs at these points by an EDS detector.

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

Parallel Session
Wednesday afternoon, 6 September, 14:00

14:00 Oral

Physical properties of carbon nanomaterials filled condensation polymers

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Carbon nanomaterials (CNM) possessing exceptional mechanical and electrical properties are promising new class of materials for special applications. Particular importance seems to have a possibility of introducing them into polymer. An incorporation of carbon nanotubes or nanofibres in a polymer system gives a promise to improve material properties as a result of physical and chemical interphase interactions. This new kind of composites based on both thermoplastics and thermosets could find a range of applications in many branches of industry. One of the manners of nanocomposites preparation is to introduce nanofiller into polymer during its synthesis (the *in situ* method). In order to ensure a uniform distribution of reinforced phase in the whole system nanofibres are carefully dispersed in a liquid substrate. Thermoplastic based composites, obtained this method, can be easily moulded what significantly extends the range of their applications. In this work the "*in situ*" synthesis as a method of polymer nanocomposites preparation as well as the effect of CNM on the properties of condensation polymers - poly(ethylene terephthalate (PET) and poly(ether-ester) block copolymer (PEE) are presented. Carbon nanomaterials consisting of amorphous carbon, carbon nanofibres and multi-walled nanotubes were synthesized using high voltage atmospheric pressure discharged plasma method (HVAPDP) and fluorinated. Polymer composites with different concentrations of modified (CNM-F) and unmodified (CNM) nanofillers were prepared and compared. For materials characterization the thermal analysis (DSC, TG), microscopic investigations (SEM), mechanical testing and electrical measurements were performed. (Presented research were financed from INTAS Research Project No. 04-80-6932)

14:20 Oral

Study of influence of size, structure and electrophysical properties of iron-containing nanoparticles depending on concentration in the polymer matrix.

Ivan P. Dotsenko[1], Gleb Y. Yurkov[1], Vitaliy G. Shevchenko[2], Sergey P. Gubin[1], Anatoliy T. Ponomarenko .[2]

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Now in the days of scientific advance it is impossible to lose an opportunity of research of nanotechnology. The systems of nanoparticles (NPs) embedded in various matrices are particularly promising. We demonstrate that it is possible to combine properties of polymers with conductivity and magnetization of metal NPs. Materials with metal NPs in polymer matrix were synthesized by decomposition of iron carbonyl in the solution-melt of polyethylene in oil. The many syntheses were held to create series of materials under equal circumstances but with different concentrations of NPs. As a result the series of samples with concentration from 1 up to 30 % (with 2-3% step) was obtained. Earlier the cavity perturbation method of measuring of complex dielectric permittivity and magnetic permeability was applied to investigate microwave electrophysical properties of nanocomposites. However, such research was held only for few samples with big step in concentration. Now the effects of concentration in polymer matrix and phase ratio of NPs on magnetic and dielectric permittivity and losses are presented. For characterization, we used TEM, XRD etc. The main major task is to determine the influence between the concentration of NPs, sizes, com-

position, and electrophysical properties. This work was supported by RFBR (04-03-32090, 04-03-32311, 04-03-32597, 05-03-32083), grant of the President MK-2733.2005.3 and the RAS program «Development of methods of synthesis of chemical substances and creation of new materials».

14:40 Oral

Powder coatings modified with silicone-containing polymer particles of hybrid structure

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Powder coatings are among the most quickly developing coating materials due to the fact that they are 100% solids (contain only solid resins, fillers and other solid additives) and therefore are environmentally friendly. They are widely used for coating of metals, in particular in manufacturing of appliances or machinery equipment, and recently also in the automotive industry. One of the major problems to be solved in the field of powder coatings is development of tough materials which in the same time would have high degree of flexibility and therefore also high impact resistance, and would retain certain other features like good adhesion to metal substrates, high surface hardness and abrasion resistance and high water resistance. One of possible approaches to reach this goal is modification of powder coatings with solid nano-particles of core-shell structure where the core is silicone elastomer of low Tg (below -50oC) and the shell is hard thermoplastic polymer of high Tg (over 100oC). In this paper the results of modification of powder coatings based on epoxy resin-carboxylic polyester resin system with hybrid particles added in the form of a powder obtained from silicone-acrylic dispersions by drying will be presented. Those results fully confirmed that introduction of small amount of hybrid particles to the powder coating masterbatch led to great increase of impact resistance, cupping resistance, abrasion resistance and elasticity of the coating while other substantial coating properties (hardness, adhesion, water resistance, gloss) remained at the same level. Examination of coating surface by SEM revealed significant differences between unmodified and modified coatings which could be interpreted by enrichment of the latter with silicone, the phenomenon reported earlier for other silicone-modified coatings.

15:00 Oral

Polymer nanocomposites containing modified smectites

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The work deals with hydrophobisation of smectic clays (aluminosilicates of layered structure) with quarternary ammonium salts in order to make them applicable for modification of polymers. The clays used in this work contained a large proportion of montmorillonite and the method of their modification (hydrophobisation) was essentially the same as described in recent patents [1, 2]. The effect of treatment of the smectic clays by various ammonium salts (both pure reagents or technical products) on the compatibility of the modified products with unsaturated polyester resins (UP) and/or epoxy resins (EP) was predicting by the sorption of monomers (styrene) or organic solvents (benzene, nonane) vapours on the surface of the modified bentonites.

It has been found that admixture of the modified clays in the amount of up to 5 wt.% to a commercial UP or EP resulted in markedly improved tensile strength (25 - 72% for UP and 27 - 78% for EP), Young modulus (48 - 77% for UP and 42 - 81% for EP), Brinell hardness (31 - 111% for UP and 25 - 97% for EP) and impact strength (without notch) (48 - 266% for UP and 34 - 180% for EP).

The composites containing modified aluminosilicates and synthetic resins have the properties typical for nanocomposites: improved stiffness, strength, optical clarity, and fine-lamellar structure of the fracture surface as observed in the electron scanning microscope.

[1]. Polish patent 178900, 2000

[2]. Polish patent 178866, 2000

The work was financed by Polish Committee of Scientific Research (KBN) grant no. *PBZ-KBN-095/T08/2003*

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony

(continuation of Mondazqs Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Parallel Session
Thursday morning, 7 September, 9:00

9:00 Invited oral

Polymer nanocomposites with layered silicates

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Polymer-layered silicate nanocomposites are a new class of materials with improved properties. For some polymer systems the use of specific compatibilizers is necessary to achieve exfoliation during melt compounding. Another way to enhance exfoliation are so-called "exfoliation promoters" (EP) [Polish Pat Appl]. Organo modified MMT (o-MMT) weakly nucleates crystallization of polypropylene (iPP) nanocomposites in static conditions while in shear very strongly. Mechanical properties improvement of polymer nanocom-

posites is usually below expectation because MMT platelets are curved when exfoliated and first they straighten before fully loaded. Melt compounding and any processing of MMT nanocomposites causes irreversible orientation of platelets. This is advantageous in film blowing where the orientation of platelets parallel to the film surface increases barrier properties. Prolongation of melt compounding of polylactide (PLA) with o-MMT led to a full exfoliation as revealed by TEM and SAXS. An increase of the dispersion MMT layers improved the thermal stability and barrier properties and HDT and elastic modulus of PLA and decreased its crystallizability. Thermooxidative and thermal stability of iPP nanocomposite with fully exfoliated MMT studied by simultaneous DSC and TGA up to 500oC permitted to identify precisely the specific mechanisms of polymer degradation. Simultaneous DSC and TGA demonstrated that alkylammonium modified clay exhibited a strong stabilizing effect on iPP in oxygen-free atmosphere. In all systems a fraction of o-MMT is resistant to exfoliation and to wetting by hydrophobic polymers. Those particles contain quartz and should be removed by thorough filtering.

9:40 Invited oral

Polymer-Metal Nanocomposites for Functional Applications

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Recently, there is much interest in hybrid materials consisting of metal nanoparticles dispersed in a dielectric matrix due to their novel functional properties offering hosts of new applications. Polymers are particularly attractive as matrix. Consequently, various approaches have been reported to incorporate metal nanoparticles into polymers. The present talk is concerned with the preparation of polymer-based nanocomposites by vapor phase co- and tandem-deposition and the resulting functional properties. The techniques involve evaporation and sputtering, respectively, of metallic and organic components and inter alia allow the preparation of composites which contain alloy clusters of well defined composition. Emphasis will be placed on soft-magnetic high frequency and optical composites, but antibacterial coatings and sensors for organic vapors will also be addressed. In particular, a novel approach to produce magnetic nanorods for potential applications in high-density data storage and other fields will be presented.

Coffee break
Thursday morning, 7 September, 10:30

Parallel Session
Thursday morning, 7 September, 11:00

11:00 Oral

Consolidation of ancient architectural materials: performances and reinforcing mechanisms in restoration

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Over last decades, ancient architectural materials, such as bricks, stones and surface finishing materials, have undergone a deep deterioration, mainly due to the combined action of pollution, rising moisture and weathering agents. This deterioration often leads to a serious loss of mechanical strength and cohesion, to such an extent that consolidation treatments must be carried out, by different kind of impregnation procedures. In this work the consolidating action of some polymeric materials is investigated by both a theoretical approach and laboratory tests on porous building materials. As a matter of fact, the consolidating action might be partially ascribed to the 'Poisson effect' exerted by the high-modulus phase on the how-modulus adhesive phases. Thus a highly porous material can be regarded, after impregnation, as a polymer-ceramic composite where pores in the high-modulus material are filled with the so-called impregnating-consolidating restoration materials. Comparison of the performance of impregnated building materials with those of not impregnated ones was carried out.

11:20 Oral

Physical properties of SiC nanoparticles and functional hybrid guest-host films

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The development of synthesis processes of semi conducting SiC nanoparticles have allowed their investigations and evidences of their versatile electronic and optical features. Particularly, based on a laser pyrolysis method, different SiC nanopowders can be obtained and systematic studies are carried out as a function of the nanoparticle sizes, surface states and the stabilised crystalline polytypes. These parameters influence also their photoluminescence marked by red emission which is also enhanced by oxidation effects and by the involved active electronic centres at the nanoparticle boundaries. In the aim of potential applications, a development of original process was dedicated to functional materials based on nanosized SiC embedded in host matrices. The more relevant ones are the hybrid architectures which combine polymers with regard to their easy preparation, low cost, optical transparency and flexibility and the nanoparticles. The organic-inorganic interfaces modify the boundary charge density which contributes to the second order optical susceptibility as well as to the refractive index changes. These physical parameters govern respectively the non-linear optics (NLO) and

electrooptical (EO) behaviour of the hybrid films. As specific application, we have realized functional hybrid materials as polymer/ SiC nanocrystals and investigated their EO properties where the interfaces polymer-nanocrystal play a key role. Optimisation of all the process allow us to obtain linear EO parameters in the order of 5-7 pm/V; i.e. in the same order as realized in the standard inorganic monocrystals.

11:40 Oral

Nanostructure of fibre forming molecular composites iPP-LCO

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Recently considerable efforts have been taken by many researches in order to obtain a new category of high-performance polymeric materials called molecular composites. Molecular composites consist of two polymeric components with dissimilar characteristic: a stiff and strong rigid rod polymer dispersed at the molecular scale in a matrix of flexible polymer. The molecular composites were prepared from iPP and liquid crystalline oligoester (LCO). The results of the nanostructure, thermal and mechanical properties investigations of liquid crystal oligoester and molecular composite in non thermal conditions are presented. The main interest is focused on the formation and changes of the nanostructure during dynamic melting and crystallisation process. The new type of molecular nanocomposite obtained from liquid crystal oligoester and polypropylene is of particular importance for material science. The nanostructure was characterised by the degree of crystallinity, the value of repeated distance (long period), crystalline lamellar thickness. These data were obtained by analysing the correlation functions. The SAXS and WAXD investigations were carried out by means synchrotron beamline of the EMBL in Hasylab of the DESY in Hamburg at wavelength of 0.15 nm.

12:00 Oral

Effect of DC glow discharge treatment on surface resistivity of thin films of polypropylene (PP) and polystyrene (PS)

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A change in the surface resistivity of a thin film of polypropylene (PP) and polystyrene (PS) of thickness 100 μm has been investigated. Using the Direct Current (DC) glow discharge the thin film of the PP and PS have been treated for various discharge powers and treatment time and the modification in the surface energy and consequently in the surface resistivity has been observed. To investigate the modification in the surface energy after DC glow discharge treatment, contact angle of two test liquids formamide and de-ionized water over the surface of PP and PS were measured. By measuring the contact angle the change in surface energy and it's two, components polar and dispersive has been measured. It has been observed that at a given power level of DC glow discharge surface energy and its polar component increases with increasing the treatment time, attains a maximum value and then becomes almost constant. With the increase in the surface energy a decrease in surface resistivities has been observed correspondingly for both the samples polypropylene and polystyrene. For the maximum power we used in the experiment i.e. 5 watt the surface resistivity of polypropylene (PP) decreased to 1.24×10^{12} Ω/cm^2 and for polystyrene (PS) decreased to 1.43×10^{9} Ω/cm^2 for the treatment time of nearly 35 seconds, which before the treatment was measured to be 4.59×10^{15} Ω/cm^2 and 2.32×10^{12} Ω/cm2 for polypropylene (PP) and polystyrene (PS) respectively.

Key words: Polypropylene (PP), polystyrene (PS), DC glow discharge treatment, surface

modification, surface resistivity.

Lunch break
Thursday afternoon, 7 September, 12:30

Parallel Session
Thursday afternoon, 7 September, 14:00

14:00 Oral

Role of nanoadditives on effectivenes of intumescent FR

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The latest world development in use and production of small particles of the nanometer size created new possibilities for the fire retardancy research.

Application of modifiers in form of so called "nano-particles" in intumescent systems improves the fire retardancy and thermal insulation effectiveness of the system. The high dispersion of particles has the influence on the decomposition of intumescent systems and on the combustion process. Therefore the structure and thermal resistance of formed carbon layer are improved.

The INF has developed a new, effective intumescent system in form of a transparent wood and wood derivates coating which contain silicon compound in "nano" scale.

Effectiveness of this intumescent system in of wood and the influence of the dispersion degree of introduced silicon compound will be presented. Parameters determined with cone calorimeter tests such as time to sustained ignition, heat release rate, mass loss rate and others, will be discussed in the paper.

Keywords: intumescent system, nano particles, fire retardant, fireproofing efficiency

14:20 Oral

Bacteriostatic textile-polymeric coat materials modified with nanoparticles

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The Institute of Textile Materials Engineering (IIMW) in co-operation with the
Institute of Material Science and Technical Mechanics (IMMT) of the Technical
University of Wrocław undertook studies aimed at the development of technology
for the preparation of appropriate nanoparticles with antibacterial properties
and conditions for their use in the production of textile-polymeric coat
materials with stable bacteriostatic properties (PBZ-KBN-095/T08/2003).
Appropriate antibacterial materials have been developed with the use of
the "sol-gel" technique by the group of IMMT. Their carriers consist of
submicro-globules, mainly SiO_2 and TiO_2. On their surface was permanently
deposited an antibacterial agent in the form of "nano-islets" of metallic
silver, Ag^o, to impart the required bio-activity to the final products. It was
of paramount importance to make these materials capable of dispersing, required
by the technological processes of coating, ensuring also a good stability of
the obtained dispersions in coating pastes as well as in the cross-linked
polymeric matrices-membranes.

14:40 Keynote lecture

Modification of polymer materials by nanoparticles, a current status and perspectives of progress

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In the lecture a survey of polymer materials modified by nanoparticles is presented.Different types of nanoparticles currently used for that purpose are mentioned.Factors determining their activity toward polymers are characterised.Perspectives of progress in the area have been predicted, taking into consideration both scientific as well as economical aspects.Organization of studies on polymer nanomaterials in Poland will be presented.

Coffee break
Thursday afternoon, 7 September, 15:30

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Mondazqs Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

15:50 Poster D1

PVC/MMT nanocomposites prepared in-situ

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PVC/MMT nanocomposites were prepared in suspension polymerization of vinyl chloride. MMT vinylated and neat were used. Both MMT - neat and vinylated - were rather well dispersed in PVC. Mechanical properties of nanocomposites were investigated and their structures were analysed by SEM and TEM methods. Comparison of mechanical properties showed slightly better elongation at break (+ 20 %) and tensile strength (+ 10 %) in case of nanocomposite with neat MMT. In case of vinylated MMT we found semi-intercalation (with the distance between MMT plates about 50 nm) and semi-exfoliation, confirmed by TEM images. Neat MMT formed regular parallel bands in PVC matrix (visible in high resolution SEM). The colours of plastified moulded pieces suggested an occuring of initial PVC degradation.

15:50 Poster D2

Synthesis, microstructure and properties of urea-urethane hybrid nanocomposites

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Urea-urethane elastomers were synthesized in a polyaddition reaction of ethylene oligo-adipate (OAE) of an average molecular weight 2000 a.u. with MDI diisocyanate. Their micro-structure and properties were studied. Dicyandiamide was used as a chain extender. In order to obtain hybrid nanocomposi-tes, OAE was modified by the reaction with methylalumoxanes (MAO) prior to use. The excess of active methyl groups of MAO was deactivated by the reactions with water, alcohols or alkylphosphates. This method allowed to introduce nano-sized aluminum-oxide based moieties into the polyurethane. The amount of nanoparticles varied from 2 to 5 % by weight. The mixture of substrates was cast into special moulds and curing process was then carried out at elevated temperature. The aim of the study was the homogeneous molecular dispersion of aluminum-oxide units in order to obtain urea-urethane nanocomposites with higher fire-resistance and improved mechanical properties. Microstructure of the nanocomposites was studied with the HRSEM. Mechanical properties were examined by standard testing procedures. The parameters meas-ured were: tensile at 100% and 300% elongation, tensile strength, elastic modulus, tear strength, elongation at break, permanent set, hardness, abrasive wear. Fire-tests were also made using Cone calorimeter.

The results of the microstructure studies show uniform incorporation of the nano-sized aluminum-oxide units in the polyurethane bulk. Such properties as Young's modulus, hardness and tensile at 100% elongation increased. A significant decrease in the heat release rate was also observed.

15:50 Poster D3

Siloxane microgel nanoparticles, grafted with polystyrene- as modifying agents for block copolymers

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Polymer nanocomposites are a new class of materials filled with inorganic nanoparticles. Compared with microcomposites (which use μm sized filler particles)- they exhibit improved properties. However the filler used should fulfill the condition of appropriate surface modification degree in order to achieve good compatibility with the polymeric matrix. One of possible ways to attain this is polymer grafting.

The goals of the presented work were twofold: synthesis of organosilicon microgel nanoparticles grafted with polystyrene and subsequent evaluation of their ability to modify block copolymer matrices.

The microgels were produced in a sol-gel process carried out in microemulsion according to method developed by Baumann et al [1]. The average diameter of nanoparticles ranged from 20 to 30 nm. After surface modification with appropriate initiator they were used as macroinitiators in controlled radical polymerization using the ATRP mechanism [2].

Spherical core-shell structures with grafting density values between 0,14 and 0,31 [nm-2] were obtained. GPC analysis of the cleaved chains revealed molecular weight ranging from 8500 to 29500 [g/mol] with average polidyspersity about 1,10.

Prepared core-shell structures after property characterization were used as modifying agents for elastoplastic block copolymers. The morphology and physical properties of the resulting systems were examined using such techniques as: SAXS, SEM, rheometry and TGA.

[1] Baumann F. et al. Macromolecules 1994, 27, 6102

[2] Pyun J. et al. Macromolecules 2003, 36, 5094

15:50 Poster D4

Formation of bimetallic oxide nanoparticles from alkoxide precursors.

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Multimetallic oxides, because of their specific properties may represent an important class of new materials. Unfortunately the high processing temperatures typically applied make them less useful for nanoscaled materials. Because of this to generated bimetalic oxide we use molecular single-source alkoxdie precursors which are suitable for formation of oxides or other materials.

During our investigation of manganese systems, we have found that heterometallic alkoxide precursors are excellent to generate oxide materials. The method allows these oxides to be formed in one step at molecular level. In this communicaion synthesis and crystal structure of alkoxide precursors - $[Mn_2Ba_2(OC_6H_4OCH_2CH_3)_8(CH_3OCH_2CH_2OH)_2]$ (**1**) will be presented. The thermogravimetric analysis (TGA) investigation with the temperature varied from 25 to 1000°C in steps of 10°C shows that decomposition of **1** is noticeable at about 400 ^{o}C in dinitrogen. The properties of the resulting oxide: $BaMnO_3$ were characterized by powder X□ray diffraction spectroscopy and scanning electron microscopy.

ACKNOWLEDGMENT We thank the State Committee for Scientific Research (Poland) for support of this research (Grant 3 T09A 083 26).

15:50 Poster D5

Synthetic strategy to access organometallic precursors for oxide-ceramic materials

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We report here new synthesis method of organometallic-aryloxides. In the first step we have obtained barium complex with 2,3-dihydro-2,2-dimethyl-benzofuran-7-ol (ddbfoH) ligand. In the next step barium aryloxide can be easily modified by organometallic species such as MMe_3 (M = Al, Ga, In) and $ZnEt_2$, giving compounds which general formulas are: $[Ba\{(\mu\text{-ddbfo})_2MMe_2\}_2]$ and $[Ba\{(\mu\text{-ddbfo})_2ZnEt\}_2]$.??? The crystal structures for these complexes were determined. The advantage of this technique is that the desired stoichiometry may be achieved on the molecular level by selective formation of the binary metallic alkoxide and the resulting molecule subsequently thermolyzed yield oxide product. The thermal behavior of obtained precursors was studied by TGA analysis over a range of 25 - 1100 ^oC under a dinitrogen atmosphere. The TGA plot shows that bimetallic precursors undergo thermal decomposition to appropriate oxide products $BaZn_2O_3$ and BaM_2O_4 (M = Al, Ga, In), which were determined by powder X-ray diffraction and SEM techniques.

??? Utko, J.; Szafert, S.; Jerzykiewicz, L.B.; Sobota, P. *Inorg.Chem.***2005**, *44*, 5194

15:50 Poster D6

Laser ablation by MAPLE at cryogenic conditions to deposition of biodegradable polymer

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Matrix Assisted Pulsed Laser Evaporation (MAPLE method) is based on a laser ablation at cryogenic conditions of target comprising the dissolved polymer in an optically absorbing solvent with a high vapor pressure, chosen especially. Application of MAPLE method is performed for ablation of a biodegradable Glycolid/ L-Lactyl copolymer. Chloroform was used as a solvent. The frozen target by liquid nitrogen was subjected to ablation using KrF excimer laser with energy density varied in the range: 0.1 to 0.3 J/cm^2. Titanium alloy Ti6Al4V, austenitic stainless steel 18-8 and polycrystalline silicon were used as substrates. Variation of surface morphology in respect to energy density is studied with application of atomic force microscopy (AFM) and environmental scanning electron microscopy (ESEM). Copolymer structure of deposited layers was subjected to examination using infrared spectroscopy (FTIR). Characteristic absorption FTIR patterns for group consisting polymer under examination are found after deposition. The applied fluencies for ablation suggest that the most uniform microstructure is achieved for fluency of 0.2 J/cm^2. The stated deviations in absorption patterns could be caused by variation in ordering as well as differentiation in the thickness of deposited layers. Micro-calorimetric examinations of crystallization temperature of copolymer are in progress.

15:50 Poster D7

Polymers doped with metal oxide nanoparticles with controlled refractive index

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Polymer nanocomposites for photonics applications, with controlled refractive index in the range 1.46-1.78 were prepared by chemical route and sol-gel process, starting from polymethylmethacrylate (PMMA), hydroxyethylmethacrylate (HEMA), polyvinyl alcohol (PVA) and metal oxides from alkoxides or inorganic salts. The properties of these materials, modified by metal oxide nanoparticles depend on the composition/metal oxide concentration, particle size and dispersion homogeneity. Ellipsometric method was used to analyse the changes in the refractive index of the nanocomposites with respect to increasing particle concentration and the spectrophotometry to determine the optical transmittance. The size and dispersity of the nanoparticles in the polymer matrix were appreciated with scanning electron microscopy (SEM) and atomic force microscopy (AFM). As is expected, the refractive index measured on the polymer thin films spincoated onto substrates rise with respect to oxide concentration for the polymers modified with TiO_2, ZrO_2 or CuO in the entire visible region and decrease with the oxide concentration when the polymers are modified with SiO_2. For the all compound the transmittance remain above 90% in the visible wavelength spectra. The particle dimensions are in the range 10 - 100 nm and were controlled through the parameters and the method of synthesis.

15:50 Poster D8

Elastomeric nanocomposites with bioactive and nanocrystalline TiO_2

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Nanocomposites consisting of polymer matrix and ceramic nanofiller are a wide group of materials with modulated properties, which

depend on filler concentration and chemical structure of the polymer matrix. New trends in biomedical applications include composites where the nanofiller improves mechanical properties and additionally enhances the biocompatibility of polymer matrix. It is possible to produce various nanocomposites based on thermoplastic polymer matrices. Poly(L-lactic acid) or poly(caprolactone) are commonly used, but they are stiff materials of limited elongation and thus not suitable for several applications. Therefore thermoplastic elastomers, as materials of high flexibility with a wide range of possibilities to tailor their structure and properties, represent attractive materials, especially for soft tissue applications. In this work we will be present an original method of bioactive nanocomposites preparation using *in-situ* polycondensation. We used ultrasound processing to prepare dispersion of nano-TiO_2 particles in one of the monomer, which is dilinoleic acid. With this original process elaborated in our laboratory, well dispersed nanoparticles within the thermoplastic elastomer matrix can be prepared. The nanocomposite effect has been gained as demonstrated by enhanced mechanical properties. At a concentration of 0.2 wt% TiO_2 the bioactivity of nanocomposites was confirmed by SEM-EDX, where calcium phosphate crystals were observed on the nanocomposite surfaces.

Acknowledgements. This work was financially supported by Polish Ministry of Education and Sciences grant nr 3T08E03268.

15:50 Poster D9

Processing of cordierite foams by direct foaming

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Open-cell ceramic foams have properties that make them attractive as catalyst supports. The "replication" process is the most widely and versatile method used for producing cellular ceramic materials. One of the drawbacks of this process is that the ceramic ligaments (struts) contain a hollow cavity possessing sharp edges due to the burnout of the polymeric template. The presence of such voids in the struts decreases the cohesion of the material in comparison to foams possessing dense struts manufactured by conventional direct foaming techniques. The aim of this work was therefore to develop cordierite-based foams by a direct foaming method and to compare the resulting properties with those obtained for foams previously developed by the replication process. The ultimate goal is to fabricate supports for depositing metal zeolites suitable for catalytic combustion of VOCs.In this work reticulated cordierite foams have been prepared by mixing of ceramic particles within a polyol followed by reaction with a MDI type isocyanate to form a polyurethane foam. Some of the experimental parameters, which were optimized, include the type and amount of surfactant, catalyst, polyol as well as the content of solids, plasticizer and blowing agent that need to be added. After curing, the polymer was burnt out under air, and the resulting ceramic foam was then sintered.Up to now, foams possessing porosities higher than 90% were obtained. Efforts are being made in order to increase density by adding coupling agents to promote a better dispersion of the inorganic filler within the polymer mix. The presence of surfactant has been found to play an important role in stabilizing the liquid-gas interface of bubbles during the expansion step, thus affecting significantly the cell characteristics of the resulting cordierite foams. Alternative ways for controlling foam density are discussed.

15:50 Poster D10

Varnises for use in electrical insulation modified with nanoparticles

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Nanotechnology enable developing of new dielectrics which show not only better mechanical, thermal and barrier properties but also display the better dielectric properties. If the size of the particle approaches that of the polymer chain length the particles stop to behave like foreign inclusions. The micron scale fillers produces significant Maxwell-Wagner polarization which is negligible in a case of nanofillers. This paper presents the results of testing of polyester (PE) and polyesterimide (PEI) varnishes modified by adding of various nanoparticles: fumed nanosilica, nanosilica and titanium dioxide obtained via sol-gel process, zinc oxide and montmorillonite (MMT) at loading 1- 3 wt %. The processing, mechanical, electrical and thermal properties have been examined for both basic pure varnishes and varnishes with nanofillers. The incorporation of nanoparticles into varnishes increased temperature of varnish degradation from 5 to 40 ^{o}C and also the bond strength by about 20% to 40%. The varnishes PE contained of fumed SiO_2, ZnO and TiO_2 showed the decrease in dielectric permittivity and maintained almost the same thermal dependence of dielectric loss factor and electrical resistivity as pure varnish. In the case of PE varnish modified with nanosilica the electric strength significantly grew at higher temperatures. The varnish PEI with fumed nanosilica exhibited much better resistance to impulse voltage and partial dischargers. Improving of dielectric properties of electroinsulating varnishes is possible by incorporation of nanoparticles. Nanofilled varnishes exhibit also the increased thermal endurance and bond strength and less water absorption.

15:50 Poster D11

Modified bentonites as thixotropic fillers

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Conditions were sought for optimal modification of smectic

(layered) aluminosilicates (LAS) with quaternary ammonium salts (QAS). The effect of structure of substituents at nitrogen atom in QAS on thixotropic properties of unsaturated polyester resin (UP) modified with LAS was studied. The results of these studies as well as the properties of thixotropic UP resins modified with bentonites treated with QAS are to be presented.

The properties of modified resins were measured at 25ºC with three types of rotary viscometers. Thixotropic properties of resins were evaluated by measuring the index of thixotropy as recommended by literature. A new alternative parameter measuring the thixotropic properties was also proposed and called the *absolutethixotropy index*. Compared with the previous methods of characterizing thixotropic properties, namely the thixotropy index, the new approach has the advantage of eliminating the arbitrary choice of shear rate and time of shear before moving to the next rate. The new index can also be related to the molecular interpretation of the phenomenon as a measure of energy of intermolecular interactions that are destroyed by shear force.

By taking into account the surface area of hysteresis loop, energy of intermolecular interactions, absolute thixotropy index (I_{at}) and classical thixotropy index (IT), the best set of composition and processing parameters were established for the structure of QAS modifying bentonites as thixotropic fillers. The best properties had the QAS with at least one benzyl substituent and an aliphatic group consisting of > 8 carbon atoms and having branched structure. The optimal content of filler in the UP compositions was ca. 2 wt.%

15:50	Poster	D12

An influence of boehmite modification on the dispersion in the polyurethane matrix

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A series of polymeric composites have been prepared using layered particulate components like nano-clays or plate-shaped, for example kaolin, mica and aluminium hydroxides. These components have been added to the polymeric matrix to obtain required heat resistance, high modulus and some others physical and mechanical properties. However, the macroscopic properties of such composites heavily depend on the dispersion of the added particles in the polymer matrix. In order to improve this dispersion, modified nanofillers are used and special processing routes are employed.
In the present study boehmite modified by exposure to lactic acid were used. Unmodified and modified boehmite particles were introduced into a polyurethane matrix by in situ polymerisation. The fillers were added to the polyurethane matrix in 0.5 and 3.5% wt. Components used for PUR nanocomposites synthesis were: polyester diol (PEA) with MW=2000, 4,4'diphenylmethane diisocyanate (MDI), mixture of chains extenders (DIOL A, DIOL B) and modified boehmite. The synthesis was performed in one-shot technique. Polyurethanes composed of PED:MDI:DIOL A: DIOL B with molar ratio 6:9:2:1 were synthesized.
The characterization of the nanocomposites has been carried out using atomic force and high resolution scanning microscopy, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). It has been found that by using boehmite modified with lactic acid, nano-composites with high degree of dispersion of the nanofiller can be obtained via mechanical stirring alone.
The analysis of the microstructure images obtained with that microscopic techniques make it possible to explain properties of fabricated nano-composites properties in terms of their relationships with microstructure characteristics.
The study was financially supported by Polish Committee for Scientific Research PBZ-KBN-095/T08/2003.

15:50	Poster	D13

Pretreatment of cellulose matrix for the generation of one-dimensional nanostructures

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Recently one-dimensional nanostructures based on different oxides (TiO2, ZnO) prepared through chemical precipitation, thermal evaporation under various cooling down procedures, sol-gel or other processes have been run up [1, 2]. The resulting structures are nanoparticles, nanorods, or nanolinters what are intriguing targets for different applications like semiconductors, photocatalysts etc. In this work a cellulose network as an intermediate framework to orientate producible nanostructures was used to create linter-like nanostructures. Wood cell wall structure has been self-assembled by nature in complex way forming tight fibrilar network and this structure was used as a matrix. The cellulose network plays an important role, nevertheless, after complete forming of nanostructures it must be removed. The surface of unbleached SW pulp fibres is generally rough and the fine cellulose fibril network hidden. Surface layer of detached fibres contains remnants from primary cell wall, compound middle lamella, and is highly folded due to the mass loss during pulping. To open the cellulose fine network treatment with supercritical CO2 was carried out. Depending on the duration and other parameters of this process various etching depths were obtained. Resulting structures were investigated with scanning electron microscope and atomic force microscope. Obtained fibre surfaces have highly porous structures with open network of cellulose fibrilar clusters. The depth of highly porous layer directly depends on the duration of the process. Treated in this way fibres are ready for the following introduction of precursors of one-dimensional nanostructures. Pretreatment with supercritical CO2 manifold enhances surface specific area of lignocellulosic matrix and facilitates higher reaction area.

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15:50 Poster D14

The influence of dispersing agents on the morphology and properties of synthesized nanoparticles of silica

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In our studies the influence of different dispersing agents on the synthesis and character of silica particles prepared by a sol-gel reaction was investigated. The silica with different surface morphology was synthesized from tetraethoxysilane (TEOS) in suspension medium - decane (an appropriate model for the elastomer). The character and structure of used dispersants strongly interacted on the shape, the particle size distrbution and morphology of silica. In comparision the reaction in the presence of dispersants was conducted "in-situ", directly, in elastomeric medium. For this purpose the mixtures of hydrogentated butadiene-acrylonitrile HNBR and ethylene-propylene rubbers containing different amount of tetraethoxysilane and dispersants were prepared. Before the vulcanization the rubber mixtures were swelled in atmosphere of carbon dioxide or were allowed to stand in atmosphere with high moisture to catalyze the sol-gel reaction. The rubber mixtures containing synthesized in decane nanoparticles of silica were also prepared.

The work was supported by State Committee for Scientific Research (3T09 B 08027)

15:50 Poster D15

New Method For The Purification Of Carbon Nanotubes and Preparation Of Polymer-Nanotube Composites

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The study and research of carbon nanotubes represents an ever growing and immensely important area, especially in the field of mechanical reinforcement. The unique mechanical properties of carbon nanotubes, namely their high strength and stiffness, could lead to their use in the creation of composite materials. This may lead to a large increase in their mechanical properties.

However, the purification of arc discharge nanotubes is a great problem due to the large amount of amorphous graphitic material simultaneously created in the discharge process. Despite the variety of different methods for nanotube purification there is still a great demand for a cost effective nanotube purification procedure. The aim of our work was to develop and optimise new method of purification and modification of MWNTs, which would provide an access to high purity and homogeneity of the final nanotube product. Our new approach is based on the modified one-step process of purification and generation of polymer (Kevlar) coated nanotube composites using Kevlar solution in the mixture of nitric and sulphuric acids. This technique has provided a single simple step for purification method for arc-discharge nanotubes and formation of a strong polymer coating. As a result new potentially very strong Kevlar-nanotube composite materials have been developed and characterised by a variety of techniques including Raman Spectroscopy and Transmission Electron Microscopy (see figure below for TEM images). We believe that new nanocomposites have a great potential as additives for the plastic reinforcement.

15:50 Poster D16

Modification of Bentonite Fillers Using Ionizing Radiation

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Nanofillers are a class of particles that are applied to polymeric composites and other materials to improve some of their properties. Manufacturing and investigation of properties of the composites recently have focused attention of many laboratories in polymer science. The main problem in the mixing process of polymers and fillers is the incompatibility of these materials. Inorganic compounds are hydrophilic, while main types of polymers are hydrophobic. For good mixing the fillers should be modified to obtain hydrophobic layer at the surface. In recent work we have concentrated our attention on using a new method to modification of classical nanofillers: layered aluminumsilicates known as montmorillonites and occurred in bentonites. Modification of the different types of bentonite using maleic anhydride and other organic anhydrides as modifying agents and ionizing radiation as a grafting method show that particles obtained in this process are good fillers for production of nanocomposites on a basis of polypropylene. Mechanism of modification of montmorillonite by the above method was tested using IR, ESR, WAXD and DSC techniques. The obtained polymeric materials were tested for mechanical and thermal properties. The features of some final materials were better than initial polypropylene. The best mechanical characteristics were obtained for polypropylene filled with modified domestic bentonite "Special".

15:50 Poster D17

Thermoplastic polyurethane elastomer reinforced with fumed silica nanoparticles: effects on thermal, mechanical and tribological properties

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Modification of the properties of a variety of polymeric materials

has become crucial for many applications because of the necessity of the replacement of more expensive metals and ceramics [1]. One way to improve certain properties of polymers consists in adding nanoparticles to create a polymer-based nanohybrid.

Thermoplastic polyurethanes find application in many fields including medical and footwear industries [2]. We have used a thermoplastic polyurethane elastomer as the polymeric matrix and fumed silica nanoparticles as the dispersed phase. Mechanical, tribological and thermal properties of the nanohybrids were studied. We find that the tensile strength, the scratch resistance and thermal stability increase along the silica content. Improvement of these properties is due to the properties of the nanosilica and interactions with the polymer matrix indicating that the fumed silica nanopowder is an effective filler for the polyurethane.

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15:50 Poster D18

Investigations on the utilization of nano zinc oxides synthesized by hydrothermal method in rubber compounds

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Recently reduction of zinc oxide level in rubber compounds has become an important issue because of concern over levels of zinc in Europe's waterways and harmful effect of soluble zinc compounds to aquatic organisms.

The aim of our work was to evaluate possibility to reduce zinc oxide amount in isoprene rubber (IR) compound by using high surface area zinc oxide obtained by hydrothermal method. In the second stage of our work possibility of utilization of zinc oxide coated with poly(oxyethylene)glycol in rubber compounds was investigated. The presence of polyglycol that is an interphase transfer catalyst improved dispersion of zinc oxide in rubber compounds and increased vulcanization rate. The application of zinc oxide coated with polyglicol enables to reduce its amount in IR compounds to 2 phr without deteriorating mechanical propertis of vulcanizates.

15:50 Poster D19

Study on photovoltaic devices based on polymer and SiC nanoparticles

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We present a study on potovoltaic devices based on polymer and SiC nanoparticles (~30nm) in the matrix of polythiophen. The photovoltaic cell structure was ITO/polymer with SiC/Al. The photophysics of such photoactive devices are based on photoinduced charge transfer from donor type semiconducting conjugated polymers to acceptor type SiC nanoparticles. In the general context of organic photovoltaics, polymeric materials have a cutting edge since they combine the photoelectrical properties of semiconductors with the large scale/ low cost technology of polymeric materials.

For determination of basic parameters these new materials, the optical investigations have been performed. The optical methods comprised: integrating sphere, double beam reflectometer, XY optical profilometer measurements in the range from 190 nm to 2500 nm. Additionally for refraction *n* and extinction *k* coefficients the ellipsometric study has been done.

15:50 Poster D20

The influence of nano - filler on microstructure homogeneity and selected properties of dental ceramic - polymer composites

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The mechanical and physical properties of polymer matrix composites can be strongly affected by the introduction of nano-sized particles. However, the main difficulty in this term is their appropriate distribution. The aim of this study is to determine the microstructure homogeneity of nano-particles containing composites intended for dental applications. Its influence on selected mechanical and physical properties was also investigated.
In the present study, the composites based on the bis - GMA and TEGDMA resins reinforced with a ceramic glass micro - filler and a nano - silica were fabricated. Volume fraction of ceramic phase was 45 - 60 %, including nano-silica. The volume fractions of the nano - filler were: 5, 10, 15, 20 %. The microstructures of composite materials were observed using high resolution scanning electron microscope. The homogeneity of nano-particles distribution was analyzed using the tessellation methods. It was found that for larger volume fractions, the nano-particles form agglomerates. Mechanical and physical properties, such as water sorption and solubility were determined and correlated with the tendency of nano-particles to agglomeration.

15:50 Poster D21

FMR study of carbon coated cobalt nanoparticles dispersed in paraffin

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Agglomerated cobalt magnetic nanoparticles coated with carbon dispersed in paraffin matrix have been prepared and investigated by FMR (ferromagnetic resonance) at room temperature. Four samples with different C/Co content, ranging from 0.175 to 1.011, dispersed at low concentration in paraffin, has been studied. Very intense and broad FMR spectra have been recorded. FMR spectrum has been analyzed by superposition of Lorentzian lines including lines at negative magnetic field. A strong dependence of the FMR signal intensity and position on concentration of magnetic nanoparticles has been observed. Various magnetic interactions affecting the observed FMR spectrum have been analyzed. It has been found that the magnetic dipole interaction between agglomerates does not play an important role.

15:50 Poster D22

Organic-Inorganic Hybrids in the Nanotechnology of Inorganic Particles

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The relatively new tendency in the nanotechnology of inorganic particles is a development of technologies based on the organic-inorganic hybrids, which allow to achieve of the inorganic mesoporous objects of macroscopic dimensions (the so-called monoliths). Calcination of the above mentioned hybrids may lead to the hierarchically ordered mesoporous inorganic materials from nanoorganisation of a single pore to the assembly of pores and finally to the macroscopic morphology. Ozin calls materials that are controllable on all of these length scales "panoscopic materials" [1].

The synthesis and the analysis of microstructures of organic-inorganic hybrids as well as inorganic structures after their calcination are the goal of this work. Two classes of hybrid organic - inorganic materials were considered. Class I is characterized by inorganic molecules embedded in polymer matrix. Class II involves materials displaying strong interactions between inorganic and organic components. Here a new molecular precursor is first synthesized and then used in a conventional sol-gel process to get the hybrid material. Precursor of the inorganic phase was a complex of the (tetra-isopropyl)orthotitanate with the methacrylic acid. The mesomorphic polymer (2-hydroxypropyl)cellulose (HPC) was used as self-organization matrix, around which there were formed "in situ" nanoparticles phase of TiO2.

As a result of our investigations we obtained:

(a)-organic-inorganic nanocomposites of HPC/TiO2, (b)-mesoporous and nanocrystalline particles diameter 200-500nm of TiO2 (anatas) as well as solid supported mesoporous and transparent thin films of TiO2, (c)-wires of nanocrystalline TiO2 (30mm x 1cm), showing high optical anisotropy.

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15:50 Poster D23

Anodic 2D and 3D Immobilization of Nano-sized Alumina Particles in a Fibrin Network

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Anodic Coagulation Casting (ACC) of fibrinogenic nano-sized (median diameter ≤ 1 µm) alumina suspensions is a novel processing technology to green form complex 2D and 3D ceramic shapes, which has not been reported so far. This technology is based on the electrically induced transformation from the soluble fibrinogen into the insoluble fibrin to immobilize ceramic particles. Contrary to the direct coagulation casting (DCC) technology, green formation does not require a pH-shift, further, as the fibrin coagulate forms on an anode it can be combined with the electrophoretic deposition (EPD) technology.

The deposition thickness ranging from a few micrometers to several millimetres and the porosity of the precipitate were controlled by the applied voltage, ranging from 1.0 V to 4.0 V and the processing-time lasting from 1 minute to 2 hours. The selection of the established green bodies via ACC-technology included two-dimensional and simple three-dimensional shapes, as well as multilayered precipitates.

The deposited ceramic was characterized using SEM and Synchrotron µ-CT, while the coagulation mechanism was studied using HPLC (high pressure liquid chromatography), SDS-gel-chromatography and ATR/FTIR.

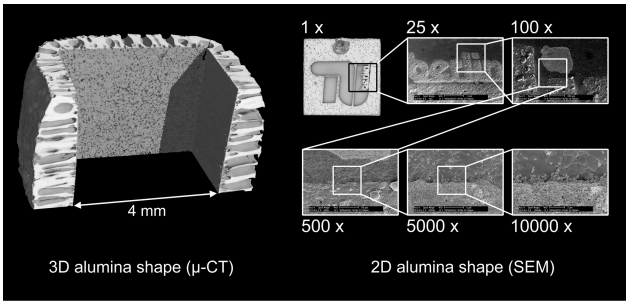

Figure:
3D (left) and 2D (right) shapes made via ACC-technology

15:50 Poster D24

Characterization of Polyazomethine thin films by optical measurements

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Polyazomethine thin films have been prepared by the chemical vapor deposition (CVD) technique via polycondensation process of *para*-phenylene diamine (PPDA) and terephthal aldehyde (TPA), with Ar as a transport agent. The Ar stream has been divided into two equal streams, one flowing over a boat containing PPDA and the other over a boat with TPA. The PPDA- and TPA source temperatures have been about fixed at 340 K and 326 K, respectively, while the substrate temperature has been kept at 298 K. Then, both streams have merged into one stream, molecules of both monomers have been mixed on their way to the substrate forming finally on it a thin film of polyazomethine. Most of the films have thickness, as estimated by means of an interference microscope, has been of the order of 200 nm. the optical and AFM measurements have been performed. The diffuse reflectance and transmittance investigations prove existence centres of high Rayleigh scattering, They have spherical structures (spherolites) and are created in process of crystallization, in PZT films, being responsible for the volume scattering of light.

15:50 Poster D25

Characterization of polyether-block-amide thermoplastic elastomer containing fumed silica nanoparticles

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Polyether block amides (PEbA) are plasticizer-free engineering thermoplastic elastomers (TPEs) [1]. These TPEs consist of linear chains of rigid polyamide blocks covalently linked to flexible polyether segments via ester groups. Commercial PEbA elastomers are mostly based on nylon 12 and poly(tetramethylene oxide)s. However, various nylons and different poly(alkylene oxide) have also been used to synthesize TPEs based on polyamide [2]. Their unique properties and easy processing allows this copolymer to achieve a wide range of applications, including: athletic shoes and sports equipment; industrial tubing and piping; drive belts; films and packaging; medical delivery catheters.

In recent years, inorganic nanoparticles filled polymer-based materials have received increasing interest due to their reinforcing effect. The use of fumed silica as filler in thermoplastics: PP, HDPE, PET, PS and PES have been documented in the literature. No data have been reported on silica nanoparticles filled PEbA as yet, what has attracted our attention. We studied the influence of nano-silica concentrations on performance of PEbA by DSC, DMA, TGA, SEM, friction testing, scratch testing, sliding wear and tensile testing. The mechanical, tribological and thermal properties were enhanced by an increased silica content due to the interaction of the amide groups and silanol groups of the silica.

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15:50 Poster D26

Applications of quantitative image analysis to the description of the morphology of polyurethane/carbon nanotubes composites

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Quantitative image analysis of polymers, plastics and composites is used to monitor and characterize a variety of processes. This paper presents the application of stereology methods to the description of morphological properties of polyurethane(PUR)/multiwall carbon nanotubes (MWCNTs) composites. Stereological analysis was performed on hard domains of the polyurethane matrix. The images of cut surface have been obtained using high resolution electron microscopy technique and atomic force microscopy. Quantitative analysis of the structure images obtained with that technique allowed us to explain the mechanism the changes of polyurethane nanocomposites properties, as well as allowed us to determine the relationships between the structure characteristics and the properties of examined materials.
Components used for PUR nanocomposites synthesis were: polyester diol (PED) with MW=2000, 4,4'diphenylmethane diisocyanate (MDI), mixture of chains extenders (DIOL A, DIOL B) and MWNTs. The filler was added to the polyurethane matrix in 0,05 and 0,1% wt. respectively to the whole weight of the polymer. Synthesis was performed in one-shot technique. Polyurethanes composed of PED:MDI:DIOL A: DIOL B with molar ratio 6:9:2:1 were synthesized.

The mechanical and thermal properties of polyurethane nanocomposites were investigated using DSC, DMA and TGA.

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15:50 Poster D27

Structure and properties of polyurethane/YAG:Tb^{3+} nano-composites with luminescence properties

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Elastic polyurethane foils with luminescent properties have attracted increasing attention, both as a subject of fundamental and applied research in the domain of electro-optical phenomena and devices. In the present study, nano-composites were prepared, by in-situ polymerization, which contained 0.05 to 0.2% weights of nano-fillers. These nano-composites were subsequently used to investigate the influence of the nano-particles content on the structure and properties of polyurethanes composites.

The following components were used for PUR synthesis: polycaprolactone diol (PCL) Mn=2000, 4,4'-dicyclohexymethane diisocyanate (H'MDI), chains extenders (DIOL A, DIOL B). The nano-filler in the form YAG containing 10% Tb^{3+} was added and samples of composites were synthesized employing pre-polymer fabrication route.

Series of nano-composites with various amounts of nano-filler were obtained which exhibited high luminescence and intensity of emissions. Microstructure of these composites was investigated via Atomic Force Microscopy in a Tapping Mode. The grain size analysis of nano-fillers was performed with HRSEM and TEM. The mechanical and electro-optical properties of the specimens have been correlated to their microstructure.

This research was financed by the Polish State Committee for Scientific Research (PBZ-KBN-095/T08/2003).

15:50 Poster D28

Structure and physical properties of TiC nano- and microparticle filled polyester and polyurethane

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Two kinds of TiC nanoparticles, different as regards methods of preparation, were applied as a filler for polymer composites. For a comparison one sample was synthesized using sol - gel technique [1] and second was prepared by crushing microcrystalline aggregates with ball grinder. Ester elastomers based polymer nanocomposites were obtained by introducing nanoparticles into polymer during its synthesis. In order to ensure a homogenous distribution in a whole system they were dispersed in a liquid substrate (ethylene glycol, EG) using ultrasonic homogenisator (Sonoplus, Bandelin) and ultra-high speed stirrer (Ultra-Turrax T25, IKA-Werke). Poly(ether-ester) elastomers (PEE) were obtained using the periodic vacuous method proceeded in two stages : transestrification of dimethyl terephthalate with EG and polycondensation [2]. Dog-bone-shaped samples for further investigations were formed by injection moulding (Boy 15, Dr BOY). For preparation of poly(ester-urethane) elastomers (PUA) based nanocomposites the reaction injection moulding (RIM) method was applied. Samples for testing were cut out from a plate annealed at 80^0C within 1h. Obtained composites contained from 0.1 to 1.0 wt% of nanofillers. The morphology of TiC/polymer nanocomposites was characterised using electron microscopy and the effect of TiC nanoparticles on thermal and mechanical properties of ester elastomers was investigated on the basis of DSC analysis and static tensile / abrasion resistance tests. The addition of TiC nanoparticles resulted in an increase of abrasion resistance of ca. 30%.

This paper and the work it concerns were partially generated in the context of the MULTIPROTECT project, funded by the EC (contract N° NMP3-CT-2005-011783, 6th Framework Programme) and the project no. PBZ-KBN 095/108/2003 financed by Polish Ministry of Science and High Education.

Reference:

1. A.Biedunkiewicz, "Crystalisation of TiC and TiN from colloidal system", *Materials Science*, 21(4) (2003) 445-452

2. T. Bodziony et al. "Ferromagnetic resonance study of Fe_3O_4 and Fe_3C magnetic nanoparticle mixture filling the PTMO-blok-PET polymer" *Rev.Adv.Mater.Sci.* 8(2004) 86-91.

15:50 Poster D29

Properties of SBR, NBR and EPDM vulcanizates reinforced by layered silicates.

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The aim of the research was to obtain rubber composites based on SBR, NBR and EPDM and montmorillonite intercalated with ammonium cations having long alkyl chains (Cloisite® 10A and 15A) and to study the influence on vulcanizates properties. To further improve dispersion of organoclay in EPDM compounds blend of EPDM and maleic anhydride grafted EPDM-M was applied. Additionally the intercalated organoclays were modified with oleic acid. The best reinforcement by the intercalated montmorillonites was obtained in NBR and EPDM/EPDM-M (85/15) compounds. Modification of the intercalated montmorillonites with oleic acid also improved their dispersion in SBR and EPDM compounds and increased level of reinforcement.

Results: Because of better dispersion of organoclays in polar rubber NBR the achieved level of reinforcement was higher than for non-polar rubbers SBR and EPDM. To improve dispersion of organoclays in non-polar rubbers SBR and EPDM and increase level of reinforcement compatibilizers should be used. The good compatibilizer for EPDM compounds is maleic anhydride grafted EPDM (less than 2% of maleic anhydride). Improvement of organoclay dispersion in SBR and EPDM compounds can also be achieved by additional modification of organoclay with oleic acid.

15:50 Poster D30

Synthesis and catalytic activity of nanosized platinium catalyst stabilized by polyvinilpirrolidone

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Nanosized colloidal particles of platinum were obtained by reduction of H_2PtCl_6 in methanol – water mixtures by refluxing. The reduction time, molecular mass and concentration of polyvinylpirrolidone (PVPD) were widely varied. The sizes of obtained platinum particles were determined by electron microscopy. It was shown that in case of PVPD with MM= $12*10^3$ very small particles of metal are forming with practically equal sizes – 1-2 nm. With increasing of PVPD MM up to $24*10^3$ together with small particles rather big mellow spheric clusters (12, 18, 30 and 42 nm) consisting of small particles appear. Fraction of such big aggregates increases with increasing of polymer MM. Decreasing of polymer concentration supports aggregation process. Significant increasing of metal particles sizes was observed in case of reduction by methanol during many hours of refluxing. High catalytic activity of obtained colloidal platinum in hydrogenization of unsaturated compounds and in hydroconversion processes of n- alkanes was shown. Possibility of remarkable decreasing of platinum content in supported catalysts by using colloidal platinum, stabilized by PVPD was demonstrated.

15:50 Poster D31

Structure and thermal properties of epoxy resins nanocomposites with flame retardants

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Nano-composites exhibit advantageous mechanical and physical properties already at small additions of modifying particles, frequently lower than 5 wt %. The present studies focused on polymeric nano-composites containing flame retardant nano-clay. The matrix used in the present study was a two-component low-molecular-weight epoxy resins. Such a matrix is used to fabricate composite structures used in aviation. As a filler, three types of modified natural montmorillonite (MMT) and various types of commonly used flame retardants were employed. The aim of the study was to develop flame retardant nano-composites containing the lowest possible amount of flame retarding fillers. To this end, a series of nanocomposites with MMT and flame retardants were fabricated.
The nano-composites produced in the present study were subjected to extensive studies of the mechanical and thermal properties. Among others, such mechanical properties were measured as elastic modulus, tensile strength and ultimate elongation. The thermal properties of the cured epoxy resin were characterized via thermogravimetric analysis. The fire reaction properties were measured using a cone calorimeter. The properties of the samples have been correlated to their microstructure, studied on images revealed on cross sections and investigated by HRSEM and AFM.
This research was financed by the Polish State Committee for Scientific Research (PBZ-KBN-095/T08/2003).

15:50 Poster D32

The effect of solvent choice on the mechanical and morphological properties of carbon nanotube-polymer composites.

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Role of the solvent in determining morphological hence mechanical properties of double wall carbon nanotubes (DWNT) polyvinylalcohol (PVA) composites were studied using differential scanning

calorimetry (DSC), thermal gravimetric analysis (TGA), differential thermal gravimetric analysis (DTGA), dynamic thermal analysis (DMTA), and tensile testing (TT). H_2O (water), DMSO (dimethylsulfoxide), and NMP (N-methylpyrrolidone) were used as solvent for solution based composite formation. Increase in crystallinity of solution based composites and reduction in crystallinity of melt processed composite was observed than their respective polymer only samples. The composites prepared with water as solvent showed maximum reinforcement indicating stress transfer and the one with DMSO exhibited intermediate reinforcement due to entrapment of solvent in matrix It was found in the recrystallization that DMSO samples were more crystalline than water and NMP samples. NMP composites showed reduction in mechanical prosperities than their respective polymer only samples suggesting that there was no stress transferred because of NMP entrapment at DWNT-PVA interface. Entrapment of NMP at PVA-CNT interface is consistent with all the data.

Symposium E

Welcome

Currently, there is high scientific interest in the development of dilute magnetic semiconductor (DMS) and other materials exhibiting ferromagnetism for spin-based sensors, light-emitters, and transistors. The magnetic behavior of such materials is due to the introduction of transition metal (TM) ions, such as Mn and Cr, into appropriate crystals. Ferromagnetic ordering in Mn-doped narrow band gap semiconductors, such as GaAs and InAs, has been studied for some time. These DMS have a relatively low Curie temperature (T_c < 200 K), which limits their potential applications. Recently, a number of research groups have reported achieving ferromagnetism at room temperature in TM-doped wide bandgap materials, such as GaN and ZnO. From then on, there is an increased interest for integration of photonic (light-emitting diodes), electronic (field-effect transistors) and magnetic (memory) devices on a single substrate. A new class of optoelectronic devices based on these materials may offer multipurpose functionality for sensing, information storage, and ultra-low-power switching elements. For the scientific and industrial communities, there is an urgent need for discussion and assessment of advances in this technical area. An international symposium with scientists providing their knowledge and insight into these areas will assist the development of device technologies involving DMS materials.

The topics will include but not be limited to:

- **Materials** - semiconductors, oxides, organic
- **Transition metals** - doping, concentration, activation
- **Synthesis** - MBE, MOCVD, PECVD
- **Ferromagnetic ordering** - Curie Temperature, carrier mediation
- **Theory** -mean field, others
- **Applications** -light emitters, sensors, transistors

Scientific Committee:

W.M. Chen (Linköping Uni), J. Cibert (LSP-Grenoble), T. Dietl (IFPAS, Warsaw), V. Etgens (INSP, Paris), G. Faini (LPN, Marcoussis), T. Foxon (Uni. Nottingham, UK), J. Furdyna (Uni. Notre Dame USA), J.M. George (UMP CNRS-Thales, Palaiseau),W. Jantsch (Uni Linz), D. Look (Uni. Colorado, USA), B. Jonker (NRL, Washington DC, USA), J. Misiewicz (Uni. Wroclaw), L. Molenkamp (Uni. Würzburg), C. Morhain (CRHEA, Valbonne), H. Ohno (Uni. Tohoku, Japan), S. Pearton(Uni.Florida, USA), P. Ruterana (SIFCOM, Caen), D. Scalbert (GES, Montpellier), Z. Wilamowski (IFPAN-Warsaw), C. Wood (ONR, Arlington, USA), J. Zavada (ARL, Durham,USA)

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Proceedings

After a regular review process (2 referees), the accepted manuscripts will be published in a special issue of **Physica Status Solidi (a)**.

Programme

Monday, 4 September

Spin Injection +ZnO
Monday morning, 4 September, 9:00
Chair: Marek Godlewski, Pierre Ruterana, Weimin M. Chen

9:00 Invited oral

Spin injection and detection in GaMnAs-based tunnel junctions : Theory and Experiments

Henri Jaffres[1], M. Elsen[1], J-M. George[1], R. Mattana[1], Albert Fert[1], Aristide Lemaître[2]

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High tunnel magnetoresistance (TMR) has been achieved using p-type ferromagnetic semiconductor GaMnAs as electrodes of tunnel junctions [1][2]. A large variation of the tunneling current was also reported depending on the orientation of GaMnAs magnetization attributed to the anisotropy of the valence band [3]. I will report on TMR and TAMR observations on various types of GaMnAs-based junctions constituted of different III-V barriers. A TMR of 170% is obtained considering an InGaAs barrier. The TAMR reaches 16% in out of plane experiment. In the framework of the 6x6 transfer matrices approach adapted to the kp theory [4], we have calculated TMR and TAMR in good agreement with our experimental data and those of Saito et al. [5]. The role of key parameters as spin splitting, Fermi level will be discussed.

[1] Tanaka and Higo..Phys. Rev. Lett. **87**, 026602 (2001) ; [2] M. Elsen et al., Phys. Rev. B **73**, 035303 (2006) ; [3] C. Ruster et al., Phys. Rev. Lett. **94**, 027203 (2005) ; [4] T. Dietl et al., Phys. Rev. B **63**, 195205 (2001) ; [5] H. Saito et al., Phys. Rev. Lett. **95**, 086604 (2005)

9:30 Oral

Ferromagnetic Fe-implanted ZnO

Kay Potzger[1], Shengqiang Zhou[1], Helfried Reuther[1], Arndt Mücklich[1], F. Eichhorn[1], Norbert Schell[1,2,3], Wolfgang Skorupa[1], Manfred Helm[1], Jürgen Fassbender[1]

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Room-temperature ferromagnetism has been induced within ZnO single crystals by implant-doping with Fe ions. For an implantation temperature of 623 K and an ion fluence of $4x10^{16}$ cm^{-2} (maximum atomic concentration of 5 %), very tiny Fe particles formed inside the host matrix are responsible for the ferromagnetic properties. These particles can be identified only by application of high resolution analysis methods like synchrotron X-ray diffraction, transmission electron microscopy or Mössbauer spectroscopy. On the other hand, Fe ions implanted at a temperature of 253 K at the same ion fluence of $4x10^{16}$ cm^{-2} are monodispersed within the host matrix. However, no magnetic coupling between these ions has been observed. This fact is related to the high damage level of the as-implanted ZnO single crystals. The damage is lowered avoiding secondary phase formation by using flash lamp annealing technology resulting in ferromagnetic properties of the Fe doped ZnO at room temperature.

9:50 Oral

Magnetic doped ZnO nanowires

David Zitoun[1], Guylhaine Clavel[2], Nicola Pinna[2], Benjamin Yuhas[3], Peidong Yang[3]

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The introduction of impurity atoms into semiconducting materials, better known as doping, is the primary method for controlling the properties of the semiconductor, such as its band gap.One such material is transition-metal doped zinc oxide ($Zn_{1-x}M_xO$). Although this system has been under experimental study for some time, the vast majority of research conducted on this material is done on bulk crystals or thin films. There are very few reports on the fabrication of one-dimensional nanostructures of $Zn_{1-x}M_xO$. While this approach has proven quite effective for the production of a multitude of nanoscale semiconductors, gas-phase syntheses have considerable limits on nanowire yield and reaction scalability.

We present the synthesis and characterization of cobalt- or manganese-doped zinc oxide ($Zn_{1-x}Co_xO$ and $Zn_{1-x}Mn_xO$) nanowires grown from a solution phase synthesis.[1] To our knowledge, this is the first reported synthesis of a one-dimensional DMS material produced in such a manner. We also note that our synthetic scheme allows for doping of different transition metals (e.g. Ni, Fe, Cu).

To determine the oxidation state of the cobalt in the wurtzite lattice, we employed electron-energy loss spectroscopy (EELS) combined with high resolution electron microscopy (HREM). The magnetic measurements are performed using SQUID and VSM magnetometer and EPR spectroscopy. Optical properties are studied on an ensemble and on single nanowire to enlighten the bandgap and dopant electronic transitions.

[1]Yuhas BD, Zitoun D, Pauzauskie P, He R, Yang P *Angew. Chem. Int. Ed.***2006**, *45*, 420

10:10 Oral

Raman studies of ZnO:Co thin films

Huijuan Zhou[1], Limei Chen[2], Vivek Malik[1,3], Christoph Knies[4], D,M. Hofmann[4], Kanwal P. Bhatti[3], Sujeet Chaudhary[3], Peter J. Klar[2], Wolfram Heimbrodt[2], Heinz Kalt[1]

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Recent theoretical predictions have shown that transition metal doped ZnO can be ferromagnetic with a Curie temperature above room temperature. However experimentally diverse magnetic properties from paramagnetic behavior to ferromagnetism over large temperature range are observed. A common question is whether the magnetic properties are intrinsic, or arise from segregation phases from transition metal clusters.

In this paper we investigate cobalt doped ZnO thin films prepared via sol-gel methods from different groups. Within the concerned doping concentration (0.5%-10%) XRD shows only the diffraction peaks from wurtzite ZnO without secondary phase, as widely reported in literature. Magnetic measurements of the corresponding samples show evidence for ferromagnetic behavior in highly doped samples (> 5%), while normal paramagnetism is found in the lowly doped films. Raman spectroscopy reveals the existence of cobalt oxides like CoO and Co_3O_4 already in intermediately doped samples. In addition, the Raman shifts of the doped samples exhibit a shift to higher energy with increasing Co doping content. Presently temperature dependent Raman measurements are under way so as to clarify the phase transition of cobalt oxides. The influence of the segregation phases on the magnetic properties of ZnO:Co thin films will be extensively studied.

Coffee break

Monday morning, 4 September, 10:30

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Characterization
Monday afternoon, 4 September, 14:00
Chair: Pierre Ruterana, Weimin M. Chen

14:00 Invited oral

Current Status and Future Perspectives of Spin-Polarized STM in Spintronics

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Spin-polarized Scanning Tunneling Microscopy (SP-STM) and Spectroscopy (SP-STS) allow the visualization of atomic-scale spin structures and the investigation of the spin-dependent local density of states spatially resolved. Spin-dependent scattering at single atomic impurities was visualized in real-space reflecting the orbital nature of the electronic states involved as well as their spin character. Ferromagnetic semiconductors like InMnAs receive a great amount of interest as the pivotal material for future spintronic devices. Recent experiments using STM suggest that the anisotropic shape of the acceptor wave function might affect the interaction of the magnetic dopants. We performed STS on Mn-doped InAs at low temperatures and found an anisotropic, cross-like shape of the Mn, which fits nicely to the Mn-acceptor wave function as calculated with the tight binding method. In contrast to the GaAs-case, Mn appears as a cross-like protrusion in the occupied density-of-states (DOS) and as a cross-like depression in the unoccupied DOS. To extend the possibility of atomic-scale spin mapping to insulating material systems we have recently succeeded in establishing magnetic exchange force microscopy (MExFM) as a reliable and reproducible technique.

14:30 Oral

Non-Collinear Instability of Ferromagnetic Mn_5Ge_3 compound

Alessandro Stroppa, Maria Peressi

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Mn_5Ge_3 thin films epitaxially grown on Ge(111) exhibit metallic conductivity and strong ferromagnetism up to about 300 K. Recent experiments suggest a non-collinear spin structure. In order to gain deep insights into the magnetic structure of this compound, we have performed fully unconstrained ab-initio pseudopotential calculations within Density Functional Theory, investigating the different magnetic states corresponding to Collinear (C) and Non-Collinear (NC) spin configurations. We focus on their relative stability under pressure and strain field. Under pressure, the C and NC configurations are degenerate, suggesting the possible occurence of *accidental magnetic degeneracy* also in Mn_5Ge_3 real samples. We found a continuos transition from a ferromagnetic C low-spin state at small volums to a NC high-spin state at higher volumes. Remarkably, the degeneracy is definitely removed under the effect of uniaxial strain: in particular, NC spin configuration is favoured under tensile uniaxial strain.

14:50 Oral

MBE growth of Mn doped $MgGeAs_2$ on GaAs (0 0 1) and (1 1 1)B

Zhen Li[1,2], Mathieu Malfait[3], Victor V. Moshchalkov[3], Gustaaf Borghs[1], Willem Van Roy[1]

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Mn doped II-IV-V_2 chalcopyrites are promising candidates for room temperature ferromagnetic semiconductors. Although several materials such as $CdGeP_2$:Mn, $ZnGeP_2$:Mn have already been reported as room temperature ferromagnetic semiconductors, most of them are bulk materials synthesized at high temperature.

Among this category, we chose $MgGeAs_2$:Mn as a new candidate for MBE growth on GaAs substrates. Before the study of Mn doping, we investigated the growth of $MgGeAs_2$ on GaAs (001) and (111)B. Stoichiometric growth conditions were established with assistance of XPS measurements. On GaAs (001), RHEED, XRD and TEM revealed phase separation with the formation of Mg_3As_2 and surface roughening. On GaAs (111)B, we obtained single crystalline films of 35nm with a smooth surface and a lattice constant of 5.66Å at an optimized growth temperature of 600^{o}C and a BEP flux ratio Mg:Ge:As = 1:3.1:800. With flux variations of ±10%, Hall measurements at room temperature showed n-type conduction (n = 2e18~3e18) in Ge rich samples and p-type conduction (p = 4e17~2e19) in Mg rich samples.

Two ways of Mn incorporation were tried on (111)B grown $MgGeAs_2$: (1) in-situ solid state reaction by annealing a Mn layer deposited on top of $MgGeAs_2$ film at 600^{o}C and (2) co-evaporation of Mn during the host material growth. Using both methods, Mn was incorporated by replacing 20% of Mg without structural change. RHEED and XRD did not reveal the existence of additional phases. A more detailed investigation by TEM is in progress. Magnetic properties are studied by VSM, SQUID, and magnetotransport measurements.

15:10 Oral

Scattering-dependence of bias-controlled magnetization-switching in ferromagnetic resonant tunneling diodes

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It is predicted from ballistic quantum transport theory that the Curie temperature (Tc) of a resonant tunneling diode (RTD) with a dilute ferromagnetic semiconductor (DFS) well should switch with increasing voltage from its equilibrium value to nearly zero in two steps. Here, we consider theoretically the effect of scattering in this system within the Buttiker probe picture. An RTD can operate in three regimes: coherent, sequential, or, no resonance - in order of increasing scattering strength. In the coherent regime the broadening of the resonance due to scattering is much lesser than the 'coherent broadening' - that due to the barriers as shown in Fig. 1. In the sequential regime, the broadening due to scattering is greater than the coherent broadening, while in the third regime it is large enough to destroy the resonance altogether. Fig. 2 and 3 show the corresponding Tc-V and I-V plots, respectively. The two steps in the Tc-V get smoothed out as scattering increases - being sharp in the coherent regime, rounded but discernible in the second and non-existent in the third. The relative robustness of the second transition is shown to arise from the asymmetry in the barriers at non-zero bias. The decrease in the equilibrium Tc with scattering follows from the broadening of the DOS shown in Fig. 4 that moves more of it outside the range of filled states, leading to a decrease of the spin susceptibility. Mapping the scattering to a bulk mobility, we show that the magnetization switching effect should be observable for typical scattering rates.

Materials/characterization

Monday afternoon, 4 September, 15:50

Chair: Hiroshi Katayama-Yoshida, Xavier Marie

15:50 Keynote lecture

Incoherent and coherent spin manipulation in ferromagnet - dilute magnetic semiconductor hybrids

Simon Halm[1], Patric Hohage[1], Frank Seifert[1], Tilmar Kümmell[1], Ellen Schuster[2], Werner Keune[2], Matthias Sperl[3], Joachim Puls[4], Fritz Henneberger[4], Gerd Bacher[1]

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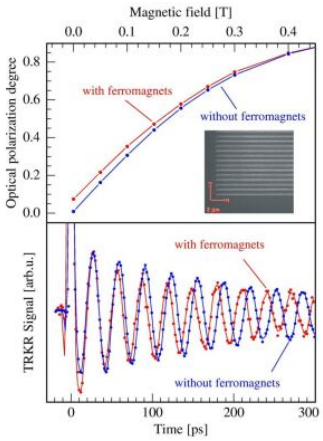

Hybrid systems of a semiconductor (SC) and nanostructured ferromagnets (FMs) have attracted great interest, since they allow a local control of the spin property of the SC charge carriers. Several routes are being followed to obtain a spin polarization in such hybrids, including spin injection, ferromagnetic proximity polarization or the usage of local magnetic fields. We present a scheme that employs the magnetic fringe field from nanoscale FMs with a well defined magnetization to locally manipulate the spin states in a ZnCdMnSe/ZnSe dilute magnetic semiconductor quantum well (DMS QW) both incoherently and coherently.

We demonstrate a spin analogon to the "classical" electric gate: instead of electric fields which control the local charge density, magnetic fields arising from Fe/Tb nanomagnets with remanent out-of-plane magnetization are used to locally accumulate spins of a defined orientation in a SC. In an optimized hybrid structure we obtain a spatially confined, remanent spin polarization of 12 % at 4 K. The spin polarization can be switched via magnetization of the FMs and can be observed up to a sample temperature of 80 K. Highly spatially resolved photoluminescence measurements allow a local scan of the spin polarization on a sub-micrometer scale.

By using time resolved Kerr rotation measurements, we are able to demonstrate the impact of the magnetic fringe field on the coherent magnetization dynamics in the DMS QW: Fringe fields arising from nanostructured Co FMs on top of the SC effectively modify the total magnetic field, which results in a local variation of the Mn spin precession frequency. We demonstrate that even tiny fringe fields alter the coherent spin dynamics in the SC noticeably, which implies that this technique can be used as a sensitive magnetometer for local magnetic fields.

16:20 Oral

Formation of epitaxial MnSb and MnBi layers on GaMnAs

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GaMnAs remains to be a candidate material for future spintronics.

For some time it was believed that the Curie temperature of this system would belimited toaround 110K forfundamental (but unknown) reasons. Eventually it became clear that point defects may have a detrimental influence on the magnetic properties, and that the density of defects can be reduced by different annealing treatments. Thus, several groups have been able to produce GaMnAs with ferromagnetic properties above 160K. Most importantly, it now seems that the limiting mechanisms are more of practical than fundamental nature, and it is believed that RT ferromagnetism is within reach. Since the Curie temperature is proportional to the density of charge carriers and to the density of magnetic ions, an obvious strategy would be to maximize these quantities. Unfortunately, to raise the Mn content in GaMnAs the growth temperature must be lowered, and this in turn promotes generation of point defects. Therefore, this approach is by no means trivial. An alternative way to achieve new magnetic properties is by including single or multiple layers of MnAs, MnSb, or MnBi in GaMnAs. In the present work we demonstrate that by applying post growth annealing under As capping we are able to obtain continuous epitaxial films of MnAs on GaMnAs, and by As/Sb or Bi exchange reactions also MnSb and MnBi films can be grown. The latter are particularly interesting, both being half metals with Curie temperatures far above room temperature.

16:40 Invited oral

Electron spin relaxation in charge-tunable InAs/GaAs quantum dots

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The hyperfine interaction between electron and nuclei in InAs/GaAs quantum dot is supposed to produce a partial relaxation of electron spin in the nanosecond timescale. As a result, this should limit to30% the average circular polarization of positive trions X^+ (consisting of a single electron associated with two holes). To address this issue, we have studied the optical orientation of X^+ in charge-tunable quantum dots. Actually, we observe that under intra-dot
circularly-polarized excitation the X^+ photoluminescence of individual quantum dots can be
polarized above 70%. We show that this effect is due to the dynamic polarization of nuclei in zero magnetic field produced by the hyperfine interaction itself. Indeed, analyzing the fine structure analysis of several X^+ lines reveals a splitting (or Overhauser shift) of the X^+ components by □12μeV which is clearly large enough to screen the nuclear field fluctuations
($g_e \mu_B \Delta_B$ □ 2 μeV) responsible for the spin relaxation. This effect is confirmed by
using a σ^+/σ^- 50kHz-alternated circular polarization provided by a photo-elastic
modulator. Under such excitation conditions, the X^+ polarization falls down to 30% as expected in the case of zero nuclear field, but can be progressively restored to 70% by applying a longitudinal magnetic field of 150mT which similarly screens the nuclear field fluctuations. Coming back to the steady-state excitation, we show in addition that this magnetic-like nuclear field can be as well screened by an external longitudinal magnetic field. This is evidenced by a dip in the X^+ polarization for a field of 70mT. Dynamic nuclear polarization and hyperfine-induced spin relaxation have to be treated self-consistently to reproduce the field dependence of X^+ polarization

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20
Chair: Marek Godlewski, Pierre Ruterana, Weimin M. Chen

Tuesday, 5 September

Theory

Tuesday morning, 5 September, 9:00
Chair: Philippe Schieffer, Tomasz Dietl

9:00 Invited oral

Theory of Ferromagnetic Semiconductors

Hiroshi Katayama-Yoshida[1], Kazunori Sato[1], Tetsuya Fukushima[1], Masayuki Toyoda[1], Hidetoshi Kizaki[1], Peter H. Dederichs[2]

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By using the KKR-CPA method within the local-density approximation, the electronic structure of II-VI and III-V-based DMS is calculated. It is found that the range of the exchange interaction in II-VI and III-N, being dominated by Zener's double exchange mechanism, is very short ranged due to the exponential decay of the impurity wave function in the gap. On the other hand, in (Ga,Mn)As and (Ga,Mn)Sb, where Zener's p-d exchange mechanism dominates, the interaction range is weaker but long ranged, because the extended valence hole states mediate the ferromagnetic interaction. Curie temperatures (Tc's) of DMSs are calculated by using the mean-field approximation (MFA), the random-phase approximation, and the, in principle exact, Monte Carlo method. It is found that the Tc values of (Ga, Mn)N and (Ga,Cr)N are very low since, due to the short-ranged interaction, percolation of the ferromagnetic coupling is difficult to achieve for small concentrations. It is found that the MFA strongly overestimates the Curie temperatures for low concentrations due to shortrangeness of interactions in (Zn,Cr)S, (Zn,Cr)Se and (Zn,Cr)Te. The Curie temperatures of (Zn,Cr)Te calculated by Monte Carlo simulation agree very well with recent experimental values. We show that 2-dimensional (2D) spinodal decomposition under layer-by-layer crystal growth condition leads to characteristic quasi-one-dimensional nano-structures (1D Konbu-Phase) in DMS. We design a new fabrication process in the bottom-up nanotechnology to realize the Tera-bit-density nano-magnets by controlling the self-organized two-dimensional spinodal decomposition. We show that the growth position, shape and density of quasi-one-dimensional nano-magnets in the DMS can be controlled by the nano-scale seeding and the vapor pressure of the doped magnetic-impurities under

the thermal non-equilibrium crystal-growth condition in MBE, MOVPE or MOCVD.

9:30 Keynote lecture

Theory for vacancy or non magnetic impurity induced "d^0" ferromagnetism.

Georges Bouzerar[1], Timothy Ziman[2]

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We present a theory for vacancy or non magnetic impurity induced ferromagnetism. The model depends only on very few free parameters. The theoretical approach is based on a correlated model for anions (oxygen orbitals) with on-site potentials on the neighbouring oxygen sites of the defect (vacancy or non magnetic impurity). First, for a fixed value of the correlation parameter it is shown that beyond a critical value of the on-site potential strength a magnetic moment appears on the oxygen sites neighbouring the non magnetic defect. By using techniques (Green's function approach for the exchange couplings and Self Consistent-Local RPA) which allow us to treat the randomness exactly, we are able to calculate the magnetic couplings between the total induced moment around the vacancies/non magnetic impurity, the Curie temperature and the dynamical properties (magnetic excitation spectrum) as a function of the strength of the disorder, doping, density of defects and strength of the correlation parameter. For physically reasonable parameters, our theory predicts Curie temperature well above room temperature for relatively small concentration of defects (few percent).Some of our results are discussed in relation to questions of stability and reproducibility raised in some recent experiments. Finally, we predict ferromagnetism with high Curie temperature in a new class of materials as $Zr_{1-x}K_xO_2$, $Hf_{1-x}Na_xO_2$ and $Ti_{1-x}K_xO_2$ for example. Some of our predictions were recently confirmed by first principle calculations.

10:00 Keynote lecture

Ab-initio modeling of spintronic materials

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Half-metallic ferromagnets with high Curie temperatures are important ingredients for spintronics applications. In this respect, diluted magnetic semiconductors and other stoichiometric ferromagnetic compounds have received enormous attention in theoretical and experimental research for the last few years. As the physics of these systems is extremely complicated, a theoretical understanding in the microscopic scale is an absolute necessity. Here, we present ab-initio calculations on diluted magnetic semiconductors and other half-metallic ferromagnets. Structural, electronic and magnetic properties of several III-V and II-VI semiconductors will be presented with the emphasis on several types of defects present in these systems. We will also present results on the calculations of Curie temperatures using Monte-Carlo simulations with effective Heisenberg Hamiltonian within the framework of ab-initio methods. Volume effects on the exchange interaction parameters and hence Curie temperatures will be presented too.

Coffee break

Tuesday morning, 5 September, 10:30

GaN-I

Tuesday morning, 5 September, 11:00

Chair: Ahsan Nazmul, Daniel R. Gamelin

11:00 Invited oral

Growth, Fe incorporation, and properties of GaN:Fe and (Ga,Fe)N:Mg

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It has become clear over the recent couple of years that a progress in the development of functional diluted ferromagnetic semiconductors requires the determination of growth phase diagrams for particular hosts and for particular magnetic dopants, including a quantitative information on the substitutional vs. interstitial incorporation of the magnetic ions as well as on spinodal decomposition and on precipitation of different crystallographic phases [1]. In this talk, I will review our recent work on the growth [2] and characterization [3] of the GaN:Fe and (Ga,Fe)N:Mg materials systems with special attention at the incorporation of the transition metal ions into the hexagonal GaN matrix. All our GaN:Fe and (Ga,Fe)N:Mg samples have been grown by means of metalorganic chemical vapor deposition on c-sapphire substrates and thoroughly characterized via high-resolution x-ray diffraction, transmission electron microscopy [4], secondary-ion mass spectroscopy, photoluminescence, spectroscopic ellipsometry [5], Hall-effect, electron-spin resonance [6], SQUID magnetometry [7] and extended X-ray absorption fine structure [8]. These studies provide information on the solubility limit and on the charge state of Fe in GaN substitutional sites for various growth conditions, and elucidate the relation between the character of Fe incorporation and magnetic, optical and transport properties of this model materials system.

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[2] A. Bonanni et al., Phys. Stat. Sol. (a), in print
[3] H.Przybylinska et al., Mat. Sci. and Eng. B 126 (2006) 222
[4] T.Li et al., Appl. Phys. Lett. 86, 241911 (2005)
[5] K.Schmidegg et al., J. Cryst. Growth 275, 1763 (2005)
[6] A.Wolos et al., Phys. Rev. B 69, 115210 (2004)
[7] M.Sawicki et al., Phys. Rev. B 70, 245325 (2004)
[8] F.d'Acapito et al., Phys. Rev. B 73, 035314 (2006)

11:30 Oral

The growth of Cr-doped GaN by MOVPE for spintronics

Yong Suk Cho[1], Nicoleta Kaluza[1], Nicolas Thilosen[1], Vitalyi Guzenko[1], Thomas Schaepers[1], Hilde Hardtdegen[1], Uwe Breuer[2], Mohammed Reza Ghadimi[3], Marian Fecioru-Morariu[3], Bernd Beschoten[3], Hans Lueth[1]

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Transition metal doped GaN based diluted magnetic semiconductors are very attractive candidate for future "spintronics" devices based on carrier-induced ferromagnetism. Theoretical calculations predicted that Cr-doped GaN was found to have the most stable ferromagnetic state in transition metal doped GaN. Up to now, Cr-doped GaN layers have been grown only by molecular beam epitaxy. Although metal organic vapor phase epitaxy (MOVPE) is a mature technique regarding the layer quality, no growth of Cr-doped GaN layers by MOVPE has been reported, yet. In this study we investigate the growth of Cr-doped GaN layers by MOVPE and their characterization. Conventional Ga and N precursors were used while bis(cyclopentadienyl)chromium (Cp_2Cr) was employed as the Cr precursor. The influence of precursors supply, carrier gas type and growth temperature on Cr incorporation efficiency in the GaN layers will be discussed. Secondary ion mass spectrometry (SIMS) was used to verify the incorporated Cr fraction in the layers. A linear dependence between mole fraction of Cp_2Cr in gas phase and incorporated Cr in solid phase was found. So far Cr-doped GaN layers with Cr concentrations in solid phase up to 2×10^{19} at/cm^{-3} were grown. Scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD) and photoluminescence (PL) measurements were performed to investigate the surface morphology, structural and optical properties of these layers. Superconducting quantum interference device (SQUID) zero field heating measurements showed a remanent magnetization even above room temperature for the Cr-doped GaN layers.

11:50 Invited oral

Electron Spin Dynamics in Dilute Nitride Semiconductors at Room Temperature

Laurent Lombez[1], Delphine Lagarde[1], Xavier Marie[1], Thierry Amand[1], Vladimir Kalevich[2], Eugeniyus Ivchenko[2], Andrey Shiryaev[2], Anton Egorov[2]

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We report optical studies in undoped GaAsN epilayers and InGaAsN quantum wells, which show that a strong electron spin polarization can persist at room temperature. Introducing less than 1% of nitrogen in the binary (GaAs) or ternary (InGaAs) alloy increases the spin relaxation time at T=300 K by a factor greater than 20 in *as-grown* material.

The spin properties have been investigated by cw and time-resolved photoluminescence (PL) experiments. The PL circular polarization degree can reach values up to 35-40% and this giant value persists within 2 ns, which exceeds the photoelectron lifetime by more than an order of magnitude [1-3]. Moreover, the application of a transverse magnetic field (~8 kG) or the change of photoexcitation polarization from circular to linear both result in a decrease of the PL decay time by a factor of 2.

We explain all these effects by a *spin-dependent recombination* process of free conduction electrons on deep paramagnetic centers. Since the capture of a free electron in the same spin state as the one of a localized electron is not efficient [4], the dynamic spin polarization of paramagnetic centers arises. This results in an increase of the average spin polarization of free electrons. The free-electron density is then entirely controlled by the spin relaxation in the conduction band. As the same spin relaxation drives the difference of spin-up and spin-down free electrons their giant spin polarization is constant in time.

[1] A.Y. Egorov et al., JAP 98, 13539 (2005)

[2] V.K. Kalevich et al., JETP Lett. 82, 455 (2005)

[3] L. Lombez et al., APL 87, 252115 (2005)

[4] C. Weisbuch et al., Solid State Com. 14, 141 (1974)

Lunch break

Tuesday afternoon, 5 September, 12:30

GaN-II

Tuesday afternoon, 5 September, 14:00

Chair: Henri Jaffres, Roland M. Wiesendanger

14:00 Keynote lecture

Comparative study of Mn and Fe incorporation into GaN by metalorganic chemical vapor deposition

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Wide bandgap diluted magnetic semiconductors (DMS) have been of interest recently due to theoretical predictions of room temperature ferromagnetism in these materials. However, the mechanism of the observed ferromagnetism of the nitrde-based DMS is still controversial, and may originate from a carrier-mediated, defect-related or nanoscale clustering mechanism. In this work, we present a comparative study of the incorporation of various transition metals and their effect on the optical, structural, and magnetic properties of GaN. Metal-organic chemical vapor deposition (MOCVD) has been em-

ployed to produce epitaxial films of varying thickness and manganese and iron doping using bis-cyclopentyldienyl(magnanese,iron) as the transition metal sources. High-resolution X-ray diffraction reveals no secondary phases under optimized growth conditions. Magnetic hysteresis is observed at room temperature in both GaMnN and GaFeN, though the strength of the magnetic ordering is roughly an order of magnitude weaker in the Fe-alloyed samples. Increasing Mn concentrations significantly affect long-range lattice ordering, and the observation of local vibrational modes (LVMs) supports the formation of nitrogen vacancies, even under optimized MOCVD growth conditions. Such vacancies form shallow donor complexes and thus contribute to self-compensation. A disorder-induced mode at 300 cm^{-1} and a LVM due to vacancies at 669 cm^{-1} were revealed by Raman spectroscopy. The iron-doped samples also show the disorder-induced mode at 300 cm^{-1}, but the vacancy-related mode is not observed which is attributed to Fermi level and defect formation energy considerations. These results will be compared with additional optical studies and discussed in relation to the prevailing theories of the origin of ferromagnetism in these materials.

14:30 Oral

Giant Zeeman effect for Mn in wide gap semiconductors: (Ga,Mn)N and (Zn,Mn)O

Wojciech Pacuski[1,2], David Ferrand[2], Piotr Kossacki[1], Joël Cibert3, Jan A. Gaj[1], Andrzej Golnik[1], Stephan Marcet[2], Ekaterina Chikoidze[4], Yves Dumont[4], Eirini Sarigiannidou[2], Henri Mariette[2]

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We present a magneto-optical study of the coupling between Manganese spin and band carriers in wide bandgap diluted magnetic semiconductors (DMS). We use reflectivity, transmission and photoluminescence. We analyze similarities and differences between the giant Zeeman effect in (Ga,Mn)N and in (Zn,Mn)O.

ZnO and GaN are wide bandgap semiconductors with the wurtzite structure, with a weak spin-orbit coupling and a strong electron-hole exchange interaction within the excitons. This combination was an origin of controversies in the determination of basic material parameters. It results also in a complex behavior of the giant Zeeman effect of A and B excitons in the presence of magnetic impurities. It was shown for (Zn,Co)O [1], that not only the transition energies, but also the oscillator strengths are strongly affected by spin-carrier coupling.

We studied (Ga,Mn)N epilayers (grown by MBE), in which most of the Mn ions are in the d^4 electronic configuration (Mn^{3+}) with spin S=2 [2]. In the wurtzite structure, spin-orbit coupling in Mn^{3+} leads to a significant spin anisotropy, with the c-axis as a hard axis. We analyzed the circular polarization causes by the giant Zeeman effect, and we find a ferromagnetic p-d exchange. It is the same sign as in the case of Cr^{2+} in II-VI DMS's - another example of the d^4 electronic configuration.

In ZnO, as in other II-VI's, Mn ions are in Mn^{2+} state with spin 5/2 and zero orbital momentum. Then the anisotropy is negligible. Thus, the temperature and magnetic-field dependence of the giant Zeeman effect in (Zn,Mn)O follows the usual Brillouin function. Assuming the "normal ordering" for the valence band in (Zn,Mn)O samples grown by MOCVD, the p-d exchange is found to be antiferromagnetic for Mn^{2+}, as for Co^{2+} [1].

[1] W.Pacuski et al., Phys. Rev. B 73, 035214 (2006)
[2] S.Marcet et al., cond-mat/0604025 (2006)

14:50 Invited oral

Magneto-optical spectroscopy of spin injection and spin relaxation in ZnMnSe/ZnCdSe and GaMnN/InGaN spin light-emitting structures

Irina A. Buyanova[1], Weimin M. Chen[1], Yasuo Oka[2], Cammy R. Abernathy[3], Stephen J. Pearton[3]

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All-semiconductor spintronics has attracted increasing attention due to its promise to combine new spin enabling functionality with well-established electronic and optical properties that have widely been explored in the present microelectronics and optoelectronics. Among many challenges, efficient spin injection and reliable spin detection are among the key elements required for the success of future semiconductor spintronic devices. In this talk we shall review our recent results from in-depth investigations of physical mechanisms which govern spin injection from spin aligners based on diluted magnetic semiconductors (DMS) and also spin depolarization within non-magnetic spin detectors, by employing cw and time-resolved magneto-optical spectroscopy in combination with tunable laser excitation. Two types of spin-injection structures based on II-VIs (e.g. ZnMnSe/Zn(Cd)Se) and III-Vs (e.g. GaMnN/Ga(In)N) were studied as model cases. We shall show that in ZnMnSe/Zn(Cd)Se two physical mechanisms are responsible for optical spin injection, i.e. (i) commonly believed tunneling of individual carriers or excitons and (ii) e*nergy transfer* via localized excitons and spatially separated localized electron-hole pairs (LEHP) located within DMS. Unexpectedly, the latter mechanism is in fact found to dominate spin injections. We shall also show that spin depolarization in the studied structures is largely determined by efficient spin relaxation within non-magnetic spin detectors, which is an important factor limiting efficiency of spin detection. Detailed physical mechanisms leading to efficient spin depolarization will be discussed.

Coffee break

Tuesday afternoon, 5 September, 15:30

ZnO/Mechanisms

Tuesday afternoon, 5 September, 15:50
Chair: Grzegorz Karczewski, Yoon Shon

15:50 Oral

Manipulating High-T_C Ferromagnetism in Doped ZnO

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The theoretical prediction and experimental observation of ferromagnetism above room temperature in oxide diluted magnetic semiconductors has stimulated intense efforts to understand the origins of the magnetic ordering [1-5]. To test the importance of charge carriers, we have used targeted p- and n-type chemical perturbations to activate and control the high-T_C ferromagnetism in polycrystalline and epitaxial Mn^{2+}:ZnO and Co^{2+}:ZnO films. In the case of Mn^{2+}:ZnO, p-type chemical perturbations resulted in robust ferromagnetism (T_C > 350 K, M_S ~ 1.5 μ_B/Mn^{2+}), whereas n-type chemical perturbations yielded only paramagnetism at all temperatures. For Co^{2+}:ZnO, the exact opposite correlation was observed. These results provide experimental evidence for the importance of bound carriers in the magnetic ordering of these materials.

[1] T. Dietl, H. Ohno, F. Matsukura, J. Cibert and D. Ferrand, Science 287, 1019 (2000).

[2] K. Sato and H. Katayama-Yoshida, Semicond. Sci. Technol. 17, 367 (2002).

[3] J.M.D. Coey, M. Venkatesan and C.B. Fitzgerald, Nature Materials 4, 173 (2005).

[4] D.A. Schwartz and D.R. Gamelin, Adv. Mater. 16, 2115 (2004).

[5] K.R. Kittilstved, N.S. Norberg and D.R. Gamelin, Phys. Rev. Lett. 94, 147209 (2005).

16:10 Oral

ZnO:Mn DMS - growth and characterization

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As a wide bang gap material, with large exciton binding energy, ZnO has been paid attention as a material for realizing transparent DMS's. When Mn is incorporated in ZnO, a large controversy is found between magnetic properties: room temperature ferromagnetism [1] or at low temperature[2], even attributed to extrinsic phase[3].We report the correlation between structural, magnetic, optical and electrical properties of a series of thin films of $Zn_{1-x}Mn_xO$. layers have been grown by MOCVD technique. Mn content varies x=0.00 -0.44 range. Veggard's law behavior for cation-cation distance has been found, justifying the achievement of Zn1-xMnxO. EPR measurements prove the substitution incorporation of Mn^{2+} in zinc site. SQUID and EPR measurements reveal an antiferromagnetic coupling for ZnMnO layers with an effective exchange constant of J1/kB = -15 K [4]. Raman spectroscopy results show Mn^{2+} related band and alloy effect well pronounced for ZnO matrix related phonon lines.

1. P. Sharma et al., Nature Materials, 2 (2003) 673

2. S. W. Jung,S.-J. An, and Gyu-Chul Yi , C. U. Jung , Sung-Ik Lee et all Appl.

Phys. Lett. 81 4561 (2002)

3. Darshan et al., Nature Materials, 3(2004) 709

4. E. Chikoidze, J. von Bardeleben, Y. Dumont et al. J. Appl. Phys. 97 D 316 (2005)

16:30 Oral

Formation of precipitates in Mn doped ZnO layers deposited by magnetron sputtering

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Transition-metal-doped ZnO is attracting the attention of researchers as a promising diluted magnetic semiconductor (DMS) material for its use in spintronics. Based on the prediction of Dietl et al., considerable effort has been focused on achieving reliable ZnO-based DMS with a Curie temperature above room temperature by doping with transition metals, especially Mnand Co.However, doping ZnO layers with these transition metals atoms may lead to the formation of various interphase precipitates, which may have magnetic properties. One of the easiest and low cost deposition method for introducing high doses of nitrogen may be deposition by rf sputtering. However, the deposited layers may be of poorer crystalline quality when doped with transition metals, and the existence of structural imperfections can impede the clarification of experimentally observed ferromagnetism in DMS materials. In this work, our aim is to develop a better understanding of the observed magnetic behaviour by carrying out a detailed microstructural analysis of Mn-doped ZnO thin films. In samples which show a ferromagnetic effect, we also observed numerous precipitates. We will report on their relationship with the deposition conditions, as well as their connection to the measured magnetic properties.

16:50 Invited oral

Ferromagnetic semiconductors, Tunnelling Anisotropic Magnetoresistance & Coulomb Blockade Anisotropic Magnetoresistance

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We firstly review some of the recent developments which firmly es-

tablish GaMnAs as a well understood carrier mediated ferromagnetic semiconductor. We then introduce and discuss the tunnelling anisotropic magnetoresistance and Coulomb blockade anisotropic magnetoresistance. These effects have recently been discovered in GaMnAs heterostructures and nanostructures. These effect should be present in other magnetic systems with strong spin-orbit coupling and may well prove to be of importance in a wide range of materials. In normal tunnelling magnetoresistance, spin conserving tunnelling results in low/high resistance states for parallel/anti-parallel magnetisations of two ferromagnetic layers. Very recently it has been demonstrated experimentally and theoretically that a large tunnelling anisotropic magnetoresistance, which arises from the dependence of the tunnelling density of states on the orientation of the magnetisation with respect to the crystallographic axes, is present in ferromagnetic tunnelling structures. This leads to strong magnetoresistance in vertical devices in which tunnelling occurs between a single (Ga,Mn)As layer and a non-magnetic layer. It has also been discovered that the tunnelling anisotropic magnetoresistance effects of ~100,000% can be achieved in tunnelling structures with two FS contacts. We show that TAMR is present in a range of vertical tunnelling structures. Recently, in lateral nanoconstrictions, we demonstrated ~400% MR effects which we ascribed to TAMR. Very recently we have observe low-field hysteretic magnetoresistance in a (Ga,Mn)As single electron transistors which can exceed three orders of magnitude. Experimental data are interpreted in terms of electrochemical shifts associated with magnetization rotations. This Coulomb blockade anisotropic magnetoresistance is distinct from previously observed anisotropic magnetoresistance effects as it occurs when the anisotropy in a band structure derived parameter is comparable to an independent scale, the single electron charging energy.

Wednesday, 6 September

Coffee break
Wednesday morning, 6 September, 10:30

-
2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

III-V and IV I
Wednesday afternoon, 6 September, 14:00
Chair: Dmitri R. Yakovlev, Irina A. Buyanova

14:00 Invited oral

Ferromagnetic III-V semiconductor heterostructures with Mn delta doping

Ahsan Nazmul, Masaaki Tanaka

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With the development of epitaxial growth technology such as molecular beam epitaxy (MBE), a variety of semiconductor heterostructures are now grown with atomically controlled layer thicknesses and abrupt doping profiles. Such growth technique has made it possible to design magnetic semiconductor heterostructures, in which the wavefunction of carriers can be controlled in artificially designed potentials [1,2]. We found that in appropriately designed p-type AlGaAs/GaAs heterostructures with Mn delta-doping, the increase of the Mn dopant concentration and maximizing the overlap of the Mn local spins with holes led to significant enhancement of TC up to 172-250 K [2,3]. In this paper, we review our study on the magnetotransport properties and Curie temperature of ferromagnetic III-V semiconductor heterostructures (Mn delta-doped GaAs/p-AlGaAs heterostructures).
It was recently revealed that (Ga,Mn)As epilayers have a cubic magnetic anisotropy with the easy axis of <100> at low temperature [4] and showed a change to the uniaxial anisotropy with the easy axis of [110] with increasing temperature [5]. We present our recent results on the giant planer Hall effect (PHE) and magnetic anisotropy of Mn δ-doped GaAs/p-AlGaAs heterostructures [6]. The sample structure is (from the surface to the substrate) GaAs(2nm) / Be-doped Al0.8Ga0.2As(15nm) / GaAs(2nm) / Mn delta-doped(0.75ML) / GaAs(20nm) / Al0.8Ga0.2As(15nm) / GaAs(200nm) / GaAs(001) substrate. We performed systematic investigations of PHE, including its temperature dependence and angular dependence. The heterostructures show distinct in-plane uniaxial anisotropy along [110].
[1] A. M. Nazmul et al., Appl. Phys. Lett. 80, 3120 (2002). [2] A. M. Nazmul et al., Phys. Rev. B 67, 241308 (2003). [3] A. M. Nazmul et al., Phys. Rev. Lett. 95, 017201 (2005). [4] H. X. Tang et al., Phys. Rev. Lett. 90, 107201 (2003). [5] U. Welp et al. Phys. Rev. Lett. 90, 167206 (2003). [6] H. T. Lin et al., submitted.

14:30 Oral

Study of the initial stages of Mn growth on Ge(001) substrates

Sion Olive-Mendez[1], Lisa Michez[1], Alain Ranguis[1], Vinh Le Thanh[1], François A. Davitaya[1], Jacques Derrien[1], Thibaut Devillers[2], Matthieu Jamet[2], Andre Barski[2]

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In recent years, there has been a growing interest in synthesizing di-

luted magnetic semiconductrs (DMS), obtained by substituting 3*d* transition metal ions into nonmagnetic semiconductors. Among the various systems, which have been largely studied such as (Ga,Mn)As or II-VI DMS, Mn-doped Ge is of particular interest since it offers a direct route for integrating magnetism with existing silicon technology [1]. A result of particular interest is the recent observation of a new high-temperature ferromagnetic phase, consisting of nano-columns of ~4 nm in diameter and 10 nm in spacing [2]. To complete this work, we have combined STM with TEM to investigate the initial stages of Mn deposition on Ge(001) surfaces. The Ge deposition was carried out at two substrate temperatures: at 80 °C, a temperature which is usually used for growing MnGe DMS, and at 250 °C, temperature at which intermetallic precipitates have tendency to be formed. For Mn deposition at 80 °C, two *distinct* kinds of Mn islands are observed even for Mn coverage smaller than a monolayer. Big islands have an average size of ~4 nm and small islands have size of 1-2 nm. When the Mn coverage increases, coarsening between small islands with newly deposited adatoms occurs, giving rise to the formation of mono-sized islands. Interestingly, these islands have an average size of 4 nm and are separated by a spacing of 10 nm. High-resolution TEM analyses indicate that those islands are epitaxial, defect free and perfectly coherent with the Ge substrate. For a Mn deposition carried out at 250 °C, only one kind of islands is observed and they appear to be pinned at the surface steps. Nanoscale chemical analysis of the island composition by means of energy dispersive X-ray spectrometry and magnetic characterizations of samples are now in process.

[1] Y.D. Park et al., Science 295 (2002) 651;

[2] M. Jamet et al., French patent n° 06/01149 (2006); Accepted in Nature Materials 2006.

14:50 Oral

Use of ion implantation in the realization of a group-IV Mn_xGe_{1-x} diluted magnetic semiconductor

Adriano Verna[1], Luca Ottaviano[1], Maurizio Passacantando[1], Franco D Orazio[1], Franco Lucari[1], Roberto Gunnella[2], Giuliana Impellizzeri[3], Francesco Priolo[3]

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The use of high temperature Mn^+ ion implantation onto Ge (100) substrates in view of the fabrication of a group-IV diluted magnetic semiconductor is explored. Different implant conditions have been tested and a large number of different experimental techniques (TEM, EXAFS, EELS, XRD) has been used to investigate the effective dilution of Mn species. Use of relatively low temperatures (240°C) and a Mn^+ dose of $2 \cdot 10^{16}$ ions/cm^3 reveals to be effective in diluting about 60% of the overall Mn atoms, that occupy substitutional Ge lattice sites. Pure dilution is observed in the subsurface (up to ~35 nm) Ge implanted layer with peak Mn concentration of ~7 at.%. This result has never been achieved in MBE-grown MnGe alloys. In the deeper region of the implanted layer Mn atoms precipitate in amorphous Mn-rich extrinsic clusters (size range 3-15 nm), coexisting with the surrounding diluted Mn_xGe_{1-x} alloy. Upon annealing at 400°C the clusters evolve in two different ways: smaller ones tend to dissolve, releasing their Mn content to the surrounding host matrix in diluted form; on the contrary, bigger clusters tend to acquire Mn atoms from the surrounding matrix and to crystallize in Mn_5Ge_3 phase. Consistently, MOKE measurements, performed to analyze the magnetic response of the samples, show magnetic signals associated with: i) diluted MnGe semiconductive matrix, ii) amorphous Mn-rich precipitates, iii) Mn_5Ge_3 nanocrystals. In the as-implanted sample, the weak superparamagnetic contribution of amorphous clusters can be easily isolated, while Mn_xGe_{1-x} diluted semiconductor manifests magnetic hysteresis up to 130 K.

15:10 Oral

Negative giant longitudinal magnetoresistance in NiMnSb/InSb: An interface effect

Spiros Gardelis[1,2], John Androulakis[2], Zacharias Viskadourakis[2], Evie L. Papadopoulou[2], Sanjay Rai[3], Gyanendra S. Lodha[3], Sindhunil B. Roy[3], John Giapintzakis[2,4]

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We report on the electrical and magneto-transport properties of the ohmic contact between polycrystalline NiMnSb thin films grown using pulsed laser deposition (PLD) and n-type degenerate InSb (100) substrates. An unusual negative giant magnetoresistance (n-GMR) effect is found when the external magnetic field is parallel to the in-plane current direction. A similar effect is also observed when Ni films are deposited on InSb substrates. On the other hand, no n-GMR effect is displayed when the deposited film is nonmagnetic. Grazing-incidence x-ray reflectometry (XRR) shows the formation of a low-density NiMnSb layer at the interface. The presence of such a layer coincides with the appearance of the n-GMR. We argue that the n-GMR effect is due to magnetic precipitates formed at the interface during the growth of the magnetic films. We propose that these precipitates align their magnetic moments in the direction of the external magnetic field and thus, the spin dependent scattering of the electrons is reduced. The effect of these precipitates on the magnetoresistance depends on the thermal processing of the system.

This work was supported by the EU contract FENIKS: G5RD-CT-2001-00535.

Coffee break

Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

III-V and IV II

Thursday morning, 7 September, 9:00
Chair: Alberta Bonanni, Bryan L. Gallagher

9:00 Invited oral

Interfaces between Fe and III-V semiconductors

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Generally, the interdiffusion processes between the ferromagnetic metals and the semiconductors take place during the interfaces formation and semiconductor atoms are released into the metallic layers and/or on the films surface. Using X and UV photoelectron spectroscopy as well as high energy electron and photoelectron diffraction we have studied and compared the growth of iron films at room temperature on GaAs(001)-(2x4) and $Al_{0.52}In_{0.48}As$(001)-(2x4) surfaces. Our results suggest that the same reaction mechanisms are implied during the interface formation for the two systems. There is an interdiffusion between the Fe and the semiconductors, and the interfaces are formed for coverage higher than 0.8 nm Fe. Our quantitative measurements demonstrate that a Fe solid solution is formed with 20-30 % of foreign atoms in the vicinity of the interfaces (~ 5 atomic planes). For the ultrathin films (0.5-2 nm) the two systems show a dominant in plane uniaxial magnetic anisotropy (with an easy axis along the [110] substrate direction) which is due to the anisotropic chemical bonding at the Fe/semiconductor (001) interface. Despite the reaction processes, the Fe/GaAs interface is rather sharp and the magnetic moment of Fe atoms at the interface is close to that of the bulk.

9:30 Keynote lecture

Structural, Optical, Magnetic Properties, and Anomalous Hall Effect of InMnP:Zn Epilayer

Yoon Shon[1], Eun Kyu Kim[2], Sejoon Lee[1], Tae Won Kang[1], Yongmin Kim[3]

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The p-type InP:Zn epilayers were prepared by MOCVD and subsequently doped with Mn by heat treatment after evaporation of Mn on top of InP:Zn epilayers using MBE. The MnO_2 of XRD peaks with the Mn concentration ranged from 0.019 to 0.290 % coincides with the $P_{4/2}$/mmm structure of MnO_2 proved by the electron diffraction analysis of TEM without showing any other precipitates. The analysis of PL showed that optical broad transitions related to Mn appeared at 1.233 eV(Ev+0.187 eV), 1.247 eV(Ev+0.173 eV), and 1.261 eV(Ev+0.159 eV) based on band gap energy of 1.42eV at 0K. The samples with the Mn concentrations of 0.290 and 0.062 % revealed that both in the calculated diffraction pattern and experimental pattern of TEM, {010} and {030} spots are missing, indicating the FCC lattice was still maintained after the Mn doping. The forbidden, regularly spaced spots suggest presence of superlattice (a = 11.738 Å), arising from the possible ordering of Mn atoms in the cubic structure of InP. The regularly spaced spots of ordered Mn produce the anomalous Hall effect (AHE) showing the characteristics of diluted magnetic semiconductor, which is caused by hole-mediated ferromagnetism due to the increase of hole concentration in tetrahedrally coordinated semiconductor. It is found that a ferromagnetic semiconductor at 150 K demonstrated by the analysis of TEM and AHE can be successfully formed in diluted magnetic semiconductor based on InMnP:Zn epilayers additionally co-doped with Zn.

10:00 Keynote lecture

Electronic structure and density of states of dilute and precipitated MnGe alloys probed via Scanninng Tunneling Microscopy/Spectroscopy measurements

Luca Ottaviano[1], Pietro Parisse[1], Maurizio Passacantando[1], Silvia Picozzi[1], Adriano Verna[1], Giuliana Impellizzeri[2], Francesco Priolo[2]

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Effective dilution of Mn impurities constitutes the main issue in the fabrication of diluted magnetic semiconductors. However, also the study of the extrinsic phases, that Mn impurities form with host atoms, deserves interest for future application in nanomagnetics. Samples for our analysis has been prepared through high temperature (240-300°C) implantation of Mn^+ ions onto Ge (100) substrates at 100 keV and subsequent 400°C annealing. TEM and EELS measurements reveal the coexistence (in a 130 nm thick implanted layer) of Mn dilution into the crystalline Ge matrix and of MnGe precipitates embedded in it. The precipitates with smaller size (3-10 nm) are principally amorphous, whereas the greater ones (8-15 nm) are mainly in the Mn_5Ge_3 crystalline phase. The samples have been progressively sputtered with Ar ions, in order to explore with surface probes different depths of the implanted layer. STM measurements show the presence of nanometer sized mounds that protrude few nm out a flat surface. These clusters are clearly identified as the MnGe precipitates (harder to etch than the surrounding Mn-diluted Ge matrix). Current Image Tunneling Spectroscopy (CITS) maps evidence significantly higher current values in correspondence with the protruding MnGe clusters, in particular for those of greater dimensions, well distinguished from the much less conductive Mn-diluted Ge matrix. Averaged I-V measurements show that the smaller MnGe precipitates (identifiable as the amorphous ones) are semiconductive (band gap of 0.45±0.05 eV), whereas the bigger ones (identifiable as the Mn_5Ge_3 nanocrystals) are metallic This is the first direct investigation of the electronic structure of the two types of clusters. The Mn-diluted Ge matrix has a band gap of 0.60±0.05 eV (close to intrinsic Ge). The experimental findings on the electronic structures

are also compared with ab-initio calculations.

Coffee break

Thursday morning, 7 September, 10:30

Oxides +

Thursday morning, 7 September, 11:00

Chair: Luca Ottaviano, Olivier Krebs

11:00 Oral

Ferromagnetic properties of Co-doped CeO_2

Boris Vodungbo[1,2], Yunlin Zheng[1], Dominique Demaille[1], Franck Vidal[1,2], Victor H. Etgens[1], Dante H. Mosca[3]

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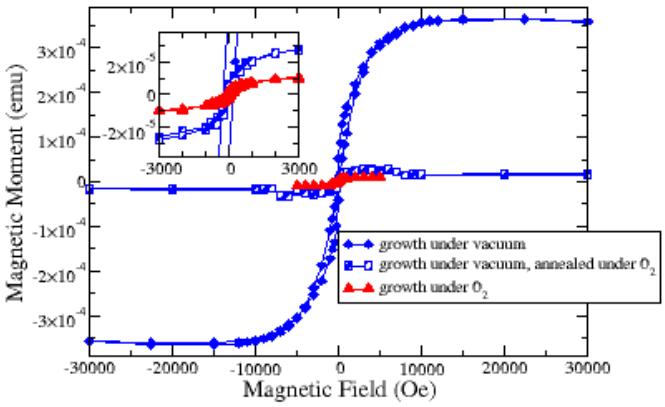
Crystalline $Ce_{1-x}Co_xO_2$ epilayers have been grown by pulsed laser deposition on $SrTiO_3$(001) substrates. It turns out that the epilayers are ferromagnetic at room temperature with the magnitude of the magnetic moment depending on the growth parameters. Films grown under vacuum (P=10^{-6} mbar) exhibit higher magnetic moment than films grown under oxygen pressure (P=5.10^{-2} mbar). Moreover, when films grown under vacuum are subsequently annealed in an oxygen atmosphere, their magnetic moments is strongly reduced. This behaviour strongly suggests that the ferromagnetism is mediated by defects. Epilayers have been studied using x-ray diffraction (XRD) and high-resolution transmission microscopy (HRTEM). All the films are (001) oriented and diplay a 45 ° in-plane rotation from $SrTiO_3$. HRTEM micrographs has shown a very good crystal quality with no visible inclusions such as Co precipitates. Preliminary electrical measurements show that the films are highly resistive. All these results points CeO_2 as a promising material for applications in spintronics.



11:20 Oral

Time-resolved ODMR investigations of II-VI based DMS heterostructures

Vitalii Y. Ivanov[1], Marek Godlewski[1,2], Dmitri R. Yakovlev[3], Sergij M. Ryabchenko[4], Andreas Waag[5], Grzegorz Karczewski[1]

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Determination of spin relaxation times in DMS nanostructures with 2D electron gas is one of the crucial aims of magneto-optical investigations. In this communication we demonstrate power of a new experimental method developed by us. Time-resolved ODMR is applied to determine spin-related relaxation times in a range of II-VI DMS heterostructures. We investigated ZnMnSe/ZnBeSe and CdMnTe/CdMgTe heterostructures with magnetic quantum wells and with a low density of 2D electron gas. Influence of a microwave radiation (60 GHz) on excitonic transitions in above-mentioned heterostructures was studied. Setting conditions of magnetic resonance of Mn^{2+} ions we could suppress magnetization of structures studied. Also a nonresonant (by moving magnetic field out of the resonance) magnetization quenching was observed imposed by a heating of spin subsystem caused by interaction with microwave heated free electrons. These two effects could be separated in a time-resolved study. We could thus directly evaluate their importance and measure characteristic relaxation times for spin-lattice and spin-2D carriers interactions.

11:40 Invited oral

Dopants and Carriers in Oxide Diluted Magnetic Semiconductor Nanocrystals and Thin Films

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Diluted magnetic semiconductors (DMSs) are pivotal architectural elements in many proposed spintronics devices. In particular, wide band gap DMSs are being intensely investigated for their potential use in high-temperature spintronics technologies. This talk will summarize our group's recent findings related to: (a) Direct chemical synthesis and magneto-optical spectroscopy of DMS nanostructures (b) Chemical manipulation of room-temperature spin ordering in oxide DMSs (c) Charge transfer electronic structures of oxide DMSs (d) Spin generation and manipulation in collidal DMS nanostructures. Apart from the technological advantages of reliable, controllable, and even switchable high-Tc ferromagnetic semiconductors, this research is motivated by the new fundamental insights it provides into the microscopic origins of ferromagnetism in this class

of materials.

Lunch break
Thursday afternoon, 7 September, 12:30

II-VI
Thursday afternoon, 7 September, 14:00
Chair: Marek Godlewski, Pierre Ruterana, Weimin M. Chen

14:00 Invited oral

Dynamics of spin interactions in DMS heterostructures

Dmitri R. Yakovlev

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Magnetic, optical and transport properties of heterostructures based on diluted magnetic semiconductors (DMS) are determined by coupled systems of magnetic ions, lattice (the phonon system) and free carriers. The system of free carriers (its concentration, temperature and spin polarization) is of great importance for static characteristics and dynamical response of DMS materials as it modifies strongly efficiency of energy- and spin transfer between the systems of DMS heterostructures.

This talk will give an overview of our recent experiments on II-VI DMS quantum wells with a type-I band alignment (ZnMnSe/ZnBeSe and CdMnTe/CdMgTe) and with a type-II band alignment (ZnMnSe/BeMnTe) [1-6].

1. A.V.Scherbakov *et al.*, Appl. Phys. Lett. 86, 162104 (2005).

2. M. Kneip *et al.*, Phys. Rev. B 73, 035306 (2006).

3. M. Kneip *et al.*, Phys. Rev. B 73, 045305 (2006).

4. M. Kneip *et al.*, Appl. Phys. Lett. 88, 152105 (2006).

5. M. Kneip *et al.*, Appl. Phys. Lett. 88, 212105 (2006).

6. A. V. Akimov *et al.*, Phys. Rev. B 73, 165328 (2006).

14:30 Invited oral

MBE Growth and Optical Study of Magnetic CdMnTe Quantum Dots

Grzegorz Karczewski

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In bulk semiconductors and in semiconductor quantum wells the carrier spin relaxation time is short - in the order of tens picoseconds. Since in quantum dots (QDs) the carriers are confined in all three dimensions the scattering processes are strongly suppressed resulting in spin-relaxation times of the order of nanoseconds. This makes the QDs particularly good suited for gaining the ability to control and manipulate spin states in semiconductor structures.

We report recent results of spectroscopy of magnetic CdMnTe QDs. The investigated structures have been grown by MBE. We focused our study on QDs containing only few Mn ions. The insight into properties of these structures was obtained by performing a high spatial resolution PL measurement in a magnetic field. The spectra are fully circular polarized already at 2T. In order to obtain precise information about the size and the number of Mn ions inside a particular dot we compare the evolution of a corresponding PL-line in an external magnetic field to the theoretical simulations in terms of the muffin tin model. In particular, we present magnetic field dependence of two lines, of which one is coming from low energy part of the spectrum and the other from the high energy part. We found that there are from 5 to 25 Mn ions coupled to excitons in our QDs depending on the size of the dot. We also estimated the QDs size distribution in our ensemble. Due to particular stability of the exciton magnetic polarons in CdMnTe QDs, where the localization of electrons and holes is comparable to the magnetic exchange interaction, the optically induced spin alignment persists to temperatures as high as 160 K.

Concluding Rump Session
Thursday afternoon, 7 September, 15:00

Coffee break
Thursday afternoon, 7 September, 15:30

Joint Session Symposia E, F & K, Room 219
Thursday afternoon, 7 September, 15:50

15:50 Invited oral

Ferromagnetic oxide semiconductors: using off-stoichiometry to tune low-dimension magnetism and consequently the iron valency

Niels Keller, Yves Dumont, Elena Popova, Michel Tessier, Marcel Guyot

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Today's research in diluted magnetic semiconductors (DMS) is concentrated on large band gap semi-conductors with the scope to obtain carrier mediated high temperature ferromagnetism by substitution of appropriate transition metal ions into the host matrix. This study investigates the possibility of alternative approach, such as the creation a of DMS starting from a ferromagnetic oxide. The control of stoichiometry during growth of thin oxide film will be used as a tool to modify the physical properties of the oxide such as magnetism, valence state and consequently transport properties. In particular, ferrimagnetic iron oxides like garnets or illmenites show already high intrinsic Curie temperatures (T_C > 400 K) and are suitable candidates for this case study. Among the different systems, the Yttrium Iron Garnet (YIG) will be presented in detail. Pulsed laser deposition of Yttrium Iron Garnet thin films allows to explore a new part of its phase diagram, e.g. controlled stabilization of iron and yttrium vacancies within the oxygen sub-lattice, and to tune magnetism by off-stoichiometry. Magnetization and Curie temperatures are measured

by polar magnetic circular dichroism (MCD). A significant increase of the Curie temperature (+10%) indicates changes of the super-exchange coupling through the variation of the Fe-O-Fe distance. Simultaneously, an important increase of the magnetization (up to 120%) is observed. The temperature dependent MCD measurements demonstrate that the increase of magnetization is due to a preferential occupation of the iron vacancies on the octahedral sites. Iron valence presumably changes from Fe^{3+} to Fe^{4+} accompanied by the presence of iron and yttrium vacancies. Its consequences to the carrier doping in application to spintronics will be discussed.

16:35 Invited oral

Origin of ferromagnetism and phase separations in diluted magnetic semiconductors

Tomasz Dietl

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A considerable effort has been devoted to understand the origin of ferromagnetism, often persisting up to above room temperature, in a number of semiconductors doped with transition metals. In the talk, I will argue that ferromagnetic DMS can be divided into three categories. The first consists of (Ga,Mn)As and related compounds. Here, theory built on Zener's model of carrier-mediated ferromagnetism and the Kohn-Luttinger *kp* theory of semiconductors describes thermodynamic, micromagnetic, optical, and transport properties. To the second group belong compounds, in which the proximity of the localisation boundary and/or a competition between long-range ferromagnetic and short-range antiferromagnetic interactions leads to an electronic nanoscale phase separation that results in characteristics similar to colossal magnetoresistance oxides. Finally, in a number of compounds a chemical nanoscale phase separation is observed, reminiscent of spinodal decomposition. Mechanisms accounting for this effect in particular materials will be discussed.

The work is supported by NANOSPIN E.C. project (FP6-2002-IST-015728), by Humboldt Foundation, and carried out in collaboration with M. Sawicki, K. Osuch, H. Przybylińska, M. Kiecana, and A. Lipińska in Warsaw, as well as with groups of H. Ohno in Sendai, S. Kuroda in Tsukuba, K. Trohidou in Athens, J. Cibert in Grenoble, J. Jaroszyński in Tallahassee, and A. Bonanni in Linz.

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20
Chair: Marek Godlewski, Pierre Ruterana, Weimin M. Chen

17:20 Poster 1

Investigation of the Semiconducting ScN and its Alloy ScMnN Grown by Radio Frequency Molecular Beam Epitaxy

Hamad A. Albrithen[1,2], Haiqiang Yang[2], Arthur R. Smith[2], Daniel Gall[3]

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ScN films have been grown on MgO(001) substrates using rf MBE. Both reflection high energy electron diffraction and x-ray diffraction confirm the (001) orientation of the films. Scanning tunneling microscopy shows only 1×1 surface reconstruction. Optical absorption results indicate a direct transition at 2.15 eV and a sub-transition with an indirect behavior. Scanning tunneling spectroscopy of ScN surfaces confirms the existence of the indirect transition with E_g of 0.9 ± 0.1 eV. The ScN(001) films on MgO(001) have been used as substrates to grow ScMnN alloys. The incorporation of the Mn into ScN has been studied as a function of the growth temperature. Structural measurements indicate the face-centered tetragonal rocksalt-type crystal structure with Sc and Mn cations and N anions. In addition to the solute incorporation into the lattice, atomic force microscopy images show that the surface of the alloy grown at $T \leq 518\ ^{o}C$ contains dot-like features, indicating surface accumulation. The areal dot density is found to decrease as the growth temperature increases, whereas the Mn incorporation increases at 518 ^{o}C. Moreover, growth at 612 ^{o}C leads to increased desorption rate, resulting in less Mn incorporation, suggesting nearly optimized growth at T = 518 ^{o}C. The alloy is found to have lattice parameters which depends on the Mn/(Mn+Sc) bulk ratio; the lattice constants follow Vegard's law depending on the Mn bulk fraction and the lattice constants of ScN and theta-MnN

17:20 Poster 2

UV beam treatments for optical and magnetic materials

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In general, high power UV beams, or lasers are used often as activating sources in metal organic chemical vapor deposition (MOCVD). In other cases, they have been applied to anneal electronic materials such as silicon and high-k dielectric film. Hatano et al. used a pulsed KrF excimer laser (λ=248 nm) to improve both device performance and uniformity, by changing the microstructures of silicon films, where this pulse laser crystallization is an efficient technology for obtaining poly-Si TFTs[1]. Fang et al. reported that the low temperature annealing step improved leakage current densities, using UV laser (λ=222 nm)[2]. These results strongly imply that UV irradi-

ation process can change the microstructure of a deposited film, and be effective in the low temperature surface treatment. In this work, a photo-assisted crystallization (PAC) method will be used to crystallize the phase change optical recording layer such as AgInSbTe and GeSbTe, using Xe-type UV-lamps. Furthermore, equatomic FePt or CoPt alloys have been attractive as materials for perpendicular magnetic recording media due to their advanced perpendicular magnetic properties, which comes from structural change from disordered to ordered $L1_0$ structure. Multilayered FePt or CoPt thin films will be annealed using the UV beam treatment to change their structural ordering with UV beam power and irradiated time.

17:20 Poster 4

Photoelectron spectroscopy of CVD grown thin films of $V(TCNE)_x$ - organic-based room temperature magnet

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$V(TCNE)_x$, is especially interesting since it features an ordering temperature above room temperature (T_C~400K) [1]. We have prepared and characterized thin films of $V(TCNE)_x$ in ultra-high vacuum using a novel film growth technique based on *in-situ* chemical vapor deposition of tetracyanoethylene and *bis*-benzene vanadium. The *in-situ* preparation method enabled, for the first time, experimental analysis of the electronic structure [2]. N(1*s*) and C(1*s*) XPS spectra clearly indicate the predominant presence of $TCNE^-$. The charge state of the vanadium ion was assigned as V^{2+}, using the XPS V(2p) peak positions. Upon comparing the valence band spectra with those of two model compounds, condensed TCNE and Rb-intercalated TCNE [3], strong modifications are observed in the TCNE-related states in $V(TCNE)_x$ that point to a covalently bonded network as opposed to an ionic charge transfer complex. The electronic, chemical structure and the magnetic properties of $V(TCNE)_x$ are studied using synchrotron radiation techniques such as photoelectron spectroscopy (PES), resonant photoelectron spectroscopy (RPES), near edge X-ray absorption fine structure spectroscopy (NEXAFS), and X-ray magnetic circular dichroism (XMCD).

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17:20 Poster 5

Periodic boundary conditions in micromagnetic modeling

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Elongated ferromagnetic nanostructures attract much attention due to the recent progress in fabrication of good quality nanowires / nanotubes. These structures offer possible interesting applications as future device components. For instance, using them one could build cylindrical transistors [1], or fast spintronic spin-injection devices [2]. Other structures focusing attention recently are periodic ferromagnetic networks, like the one discussed in Ref. [3]. When simulating all these cases, however, periodic boundary conditions (PBC) are helpful, if they are included in the micromagnetic modeling program. Although one can find commercial simulation packages where PBC is built-in [4, 5], so far it was not present in popular no-cost systems like OOMMF [6] or "magpar" [7].

In our work we concentrated on the OOMMF simulation package. It has been extended by adding a special module, which allows including PBC in exchange and demagnetization interactions. Especially the second case was difficult because of the long-range character of dipole-dipole interactions. We had for instance to control carefully the errors of the demagnetization computations.

In spite of our extensions, the software runs at same speed as without them. Only the initialization step can take longer compared to no-PBC case. We present the structure and schematic idea of our PBC implementation. We also present the results of comparative tests between our PBC simulations and available analytical solutions for the infinite rod.

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17:20 Poster 7

Electronic structure and chemical bonding of Cr_2AlC and Cr_2AlN

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Cr_2AlC is a recently developed layered ternary carbide. In this work, we have investigated the electronic structure of nanolaminate layered ternary carbide and nitride Cr_2AlX (X=C and N) for three magnetic configurations, antiferromagnetic (AFM), ferromagnetic (FM) and paramagnetic (PM) by means of *ab-initio* total energy spin-polarized calculations. We have employed an all-electron full-potential augmented plane-wave plus local orbitals (APW+*lo*) method in the local spin density approximation (LSDA). We have calculated the equilibrium lattice parameters, bulk moduli, band structures, total and partial densities of states (DOS) and charge densities. The total densities of states are calculated using the relaxed structures at the equilibrium volumes. Our calculated equilibrium volume and c/a values are in good agreement with experiments. Based on a comparison of the DOS for all three magnetic configurations we have identified the FM configuration to be metastable. The charge density distribution suggests that the chemical bonding between Cr and C in Cr_2AlC is very similar to the one between Cr and N in Cr_2AlN.

17:20 Poster 8

Structural characterization of GaN and $Ga_{1-x}Mn_x$N layers obtained by Sandwich Sublimation Method

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Sandwich Sublimation Method allows to obtain layers of gallium nitride and gallium nitride doped with manganese. This method is cheap and fast. It doesn't need high pressure nor hazardous conditions. Experimental temperature is within the range 1100^{O}C to 1200oC. Layers were grown on GaN thin films obtained by MOCVD. Reactions were carried out in a tubular quartz reactor with induction heating in a stream of ammonia. Obtained layers of GaN and $Ga_{1-x}Mn_x$N were then characterized by various techniques.

17:20 Poster 9

Optical, Structural, and magnetic properties of p-type GaN implanted with Fe^+ (5 and 10 at %)

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GaN and ZnO are good examples of a diluted magnetic semiconductor (DMS) that is classified as a wide band gap material has attracted much attention. P-type GaN:Zn epilayers were prepared by metalorganic chemical vapor deposition and subsequently implanted with Fe^+ of 5 and 10 at %. In the case of the Fe concentration of 5%, the magnetic properties of all epilayers show paramagnetic behavior. These results agree with those that the triple axis diffraction (TAD) of X-ray diffractometry (XRD) do not produce the ferromagnetic GaFeN domain and the Fe-related PL transitions happen very weakly, and also atomic force microscopy (AFM) and magnetic force microscopy (MFM) images do not reveal clear, symmetric ferromagnetic domains. However, the systematic enhancement of ferromagnetic hysteresis loops takes place with an increase in the Fe concentration (5→10 at%) and in the annealing temperature from 700 to 850 ^{O}C. The trends of magnetic properties coincide with those of results that the full width at half maximum of triple axis diffraction for GaN (0002) including the appearance of ferromagnetic GaFeN domain increases and the Fe-related photoluminescence transitions enhance, and also the sizes of symmetric ferromagnetic domains in AFM & MFM increases systematically

17:20 Poster 10

Photoluminescence and Hall studies of GaN:Fe and (Ga,Fe)N:Mg layers

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GaN:Fe and (Ga,Fe)N:Mg are promising candidates as ferromagnetic diluted magnetic semiconductors (DMS) for spintronics applications [1]. To gain insight into the ordering mechanisms of this novel DMS, temperature dependent photoluminescence (PL) and Hall measurements are essential.

All samples were fabricated by metallorganic chemical vapor deposition (MOCVD) on (0001) sapphire substrates. Several series of samples were grown with iron and magnesium incorporated both homogeneously and as delta-layers. High-resolution transmission electron microscopy (TEM) and X-ray diffraction (XRD) confirmed the device-quality of the films. The PL spectra of GaN:Fe at 6 K exhibit an excitonic region between 3.3eV and 3.5eV dominated by a optical transition whose intensity scales with the Fe content. By evaluating the activation energies from temperature dependent PL, we attribute this feature to a bound exciton. The activation energies and results from secondary ion mass spectroscopy (SIMS) of different GaN:Fe samples point most likely to oxygen as the binding site for the excitons. The O-concentration, due to the high affinity of oxygen to iron, increases with increasing magnetic ions content. Hall measurements confirm the GaN:Fe layers to be n-type. The carrier concentrations increase with increasing Fe flux and lie between 10^{16} and 10^{17}cm^-3 at 300 K. Their temperature dependence suggests O as donor as well.

Depending on sequence and thickness of the single layers and the dopants' concentrations in the delta-codoped (Ga,Fe)N:Mg samples, we measure strong excitonic features in the near band gap region, a shallow donor-to-acceptor transition at 3,25 eV and a yellow lumin-

escence band which centers around 2,23 eV.
Furthermore, we found that by delta-codoping with Mg we achieve an optimized concentration of about 10^20 acceptors/cm^-3.
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17:20 Poster 11

First-principles calculations of magnetic properties of $Zn_{1-x}Mn_xO$ alloys

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There is currently widespread interest in the development of novel diluted magnetic semiconductors (DMS) having ferromagnetic properties at room temperature because of their technological impacts for spintronic devices. We report here on first-principles total-energy calculations of the structural, electronic, and magnetic properties of $Zn_{1-x}Mn_xO$ alloys in 32-atom wurtzite supercell using the hybrid full-potential augmented plane-wave plus local orbitals (APW+*lo*) method. We have used the local-spin density approximation (LSDA) for the exchange and correlation potential. The ground state properties, equilibrium lattice constants, bulk moduli, charge densities, band structures, and densities of states are determined. Our calculations show that the hybridization between *d*-states of magnetic impurities and the host valence bands plays an important role to mediate ferromagnetic interactions between ions. By increasing the Mn content, the alloys exhibited increases in both the c-axis lattice constant and fundamental band gap energy.

17:20 Poster 12

Method of Manganese co-doping of LT ZnO films

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Lately various methods of doping of ZnO films by Mn have been investigated. In the presented work we used Atomic Layer Deposition to grow ZnO at low temperature (LT) and we studied procedures of doping of these films with Mn ions during the growth process. LT growth conditions were selected to minimize Mn diffusion and formation of various Mn-related oxides. Mn is know to be a fast diffuser in ZnO, which results often in highly inhomogeneous doping, formation of foreign phases and of Mn precipitates. We demonstrate that most of these effects can be avoided by an appropriate selection of growth conditions and by post-growth annealing. The ALD grown LT ZnMnO films show fairly homogeneous in-plane and in-depth Mn content, as we conclude from SIMS, TEM, EPMA and X-ray investigations. These films do not show responses due to inclusions of foreign phases of Mn_xO_y in magnetic investigations.

17:20 Poster 13

Role of defects on the magnetic interactions in Mn-doped ZnO

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The magnetic properties of Mn-doped ZnO were studied by means of the Korringa-Kohn-Rostoker-Coherent-Potential-Approximation (KKR-CPA) method, in the framework of density functional theory. It is found that ZnO doped with 5% Mn has a very low Curie temperature (~2K). The role of defects, such as vacancies, interstitials, or N substitution, has been studied. Zn interstitials lead to a strong antiferromagnetic coupling between the magnetic cations, while Zn vacancies or oxygen substitution by nitrogen can stabilize the ferromagnetic state. This trend is explained by the increase in the number of holes mediating ferromagnetism. But, due to the short-ranged character of the exchange interactions, the calculated Curie temperatures by Monte Carlo simulations do not exceed 50K for any of the doping situations.

17:20 Poster 14

Ferromagnetism in ZnO:Mn thin films deposited by PEMOCVD

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ZnO is one of the most perspective materials for spintronics and for applications as diluted magnetic semiconductor being doped by transition metals such as Mn or Co. We have deposited ZnO thin films doped by different Mn amount (1, 5, 10, 15 wt% in precursors mixture) by plasma enhanced metalorganic chemical vapor deposition (PEMOCVD). Sapphire (0001) was used as substrate and films thickness was up to 300 nm. Structure analysis was realized by XRD, and no Mn separate phase has been found. Only peaks, peculi-

ar to ZnO were observed. However all samples had the textured orientation along c-axis, the films crystallinity was found to deteriorate with Mn content increasing. Also the ZnO lattice parameters were changed, thus we assumed that Mn has incorporated into ZnO lattice. By means of XRF analysis we determined the real content of Mn in films what was in correlation with precursor's mixture composition. Morphology of ZnO:Mn films was investigated by AFM and the surface roughness was observed monotonically to decrease with Mn content increasing. Magnetic properties were investigated by SQUID and the ferromagnetic behavior was observed at low temperatures. Such magnetic behavior was found to increase with Mn content increasing. The most reliable hysteresis loop was observed for ZnO with 7 wt. % of Mn. The results obtained are discussed.

17:20 Poster 16

Optical and magneto-optical properties of ZnMnO and ZnMnFeO single crystals and thin films

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At present the existing or absence of room temperature ferromagnetism in zinc oxide-based diluted magnetic semiconductors is main point for discussion and an increased interest to these materials. It was found that their magnetic properties depend on content of magnetic ions and the growth parameters. In this work single crystals of $Zn_{1-x}Mn_xO$ with $0<x<0.1$ were grown by chemical vapour transport technique. Thin films of $Zn_{1-x}Mn_xO$ (x=0; 0.06; 0.1) and $Zn_{1-x-y}Mn_xFe_yO$ (x=0.05; y=0.07) were deposited by pulsed laser ablation and rf magnetron sputtering methods on quartz and sapphire substrates. Optical absorption, magnetoabsorption and Faraday rotation spectra were measured at temperature range of (5-400) K in magnetic field up to 25 T. In the absorption spectra new structure has been found which can be attributed to the charge-transfer transitions. The observed peculiarities in magneto-optical spectra in $Zn_{1-x}Mn_xO$ single crystals and thin films are explained in framework of strong spin exchange interaction between d-electrons of Mn^{2+} and s,p - band carriers. The revealed negative sign of the Faraday rotation angle, typical temperature and magnetic field dependences suggest of paramagnetic state of $Zn_{1-x}Mn_xO$ single crystals and thin films. On the other hand, $Zn_{1-x-y}Mn_xFe_yO$ film samples exhibit ferromagnetic-like behavior at room temperature. Origin of this ferromagnetic ordering is discussed. Application of the studied materials for spintronic purposes is considered. The work was supported partially by grants No. M/128-2004 and No.D3/152-2005 from Ministry of Education and Science of Ukraine.

17:20 Poster 17

Influence of Annealing Condition on Exchange Bias of IrMn/GaMnAs Heterostructures

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The exchange biasing properties of MnO/GaMnAs have been investigated by Eid *et al.* in detail by varying annealing condition. Moreover, a promising antiferromagnetic material IrMn has been identified due to its high exchange bias energy (Jk), high blocking temperature (Tb), and low critical thickness (~70 Å). The present work is a comparative study of exchange biasing of GaMnAs/IrMn heterostructures. $Ga_{0.95}Mn_{0.05}As$ (30 nm) epilayers were grown by MBE system on semi-insulator GaAs (001) substrates. Then, the samples were transferred to another ultra high vacuum chamber to deposit the overgrown layers IrMn (10 nm)/Ta (5 nm) by dc magnetron sputtering system. In order to set up good exchange bias and decrease structural defects, the samples were annealed at temperature range from 150 to 200^{o}C in a 1300 Oe field for 30 minutes. For as-prepared sample the magnetization curves show symmetric with respect to zero field, indicating absence of exchange bias. Moreover, the exchange bias effect was enhanced and demonstrated for GaMnAs/IrMn heterostructures after anneal procedure. Furthermore, the sample shows atomically flat with a root mean square (rms) roughness ~0.3 nm by atomic force microscopy (AFM). In spite of these new works, the origin of exchange bias with thermal treatment is not yet fully understood. Auger electron spectroscopy (AES) and Secondary Ion Mass Spectrometer (SIMS) analysis were carried out in order to study the extent of GaMnAs/IrMn interdiffusion during the thermal treatment. Furthermore, more magnetic characterization and detailed microstructures were performed and discussed by SQUID magnetometer and HRTEM analysis, respectively.

17:20 Poster 18

Tunneling Anisotropic Magnetoresistance effect in p^+-(Ga,Mn)As/n^+-GaAs Esaki diode structure.

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We have fabricated devices to investigate electrical spin injection from a ferromagnetic (Ga,Mn)As layer into a non-magnetic GaAs layer in the lateral transport geometry. The Esaki diode structure p^+-(Ga,Mn)As/n^+-GaAs is used to circumvent the problem of fast spin relaxation of holes injected from a *p*-type ferromagnet into GaAs. Under a reverse bias applied to such a structure spin-polarized *elec-*

trons, with longer spin relaxation times than holes, tunnel from (Ga,Mn)As valence band through the depletion layer into GaAs conduction band [1]. Here we report on magnetotransport investigations of the Esaki diode in both in-plane and perpendicular external magnetic field. The sample was rotated in the in-plane field to investigate the dependence of the resulting magnetoresistance on the angle between applied in-plane field and directions of crystallographic axes of the (Ga,Mn)As layer. Obtained results resemble the Tunneling Anisotropic Magnetoresistance (TAMR) effect discovered recently in a normal-metal-insulator-ferromagnetic-semiconductor tunneling device [2].

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17:20 Poster 20

Magnetotransport properties of ultrathin GaMnAs layers

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Ferromagnetic GaMnAs is currently being used to investigate prototype spintronics devices. The progress in material fabrication in recent years led to a remarkable increase of Curie temperature (Tc) and improved conductivity of GaMnAs. This made it possible, e.g. to observe the Coulomb blockade anisotropic magnetoresistance effects in ferromagnetic GaMnAs yielding an interesting material system combining nanoelectronic and spintronic properties. Because of large carrier density, typically in the range of 10^{20} to 10^{21} cm^{-3} the depletion of GaMnAs via the field effect is only possible in nanoscale structures. It is therefore desirable to investigate the ferromagnetism of GaMnAs towards ultrasmall thicknesses. It was already reported that the thickness limit for ferromagnetism in single GaMnAs layers is close to 5 nm. So far it is unclear which factors cause the ferromagnetism in very thin GaMnAs layers to vanish. To address this problem we have investigated single GaMnAs layers with thicknesses between 2 and 5 nm. Samples were grown by MBE on GaAs(100) substrates. Magnetic properties of the samples were studied by SQUID magnetometry and transport experiments employing standard Hall effect measurements. Magnetisation and transport measurements revealed that the lower limit for the FM phase transition in a single $Ga_{0.95}Mn_{0.05}As$ layer is close to 3 nm. Samples with thicknesses between 3 and 5 nm exhibited normal behaviour both in transport and in magnetisation measurements. This required however the insertion of suitable buffer layers which needed to be deposited prior to GaMnAs growth and a suitable post-growth annealing treatment.

17:20 Poster 21

Studies of Mn incorporation in GaAs close to the equilibrium doping limit

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Mn disorder effects play an important role in ferromagnetism of III-Mn-V's. In ferromagnetic GaMnAs, a structurally metastable material, Manganese is introduced with compositions above 1 at.%, a much higher level than the equilibrium doping limit. Consequently, an atomic ordering of Mn atoms in the lattice (of short range order type) is expected to exist (even before any Mn-enriched precipitates occur). It is not clear to what extent it is present in ferromagnetic layers [Ref. 1]. To study Mn-Mn short range order we intended to prepare Ga(1-x)Mn(x)As crystals with Mn composition only slightly above the equilibrium doping limit. Using annealing techniques, an atomic order could, in principle, be controlled and studied.

Bulk Ga(1-x)Mn(x)As crystals with Mn composition up to 0.1 at.% were grown using equilibrium technique (Czochralski). Samples were characterized by magnetization measurements (SQUID) and by Hall effect. GaMnAs samples with Mn composition up to 0.1 at.% were paramagnetic, Mn composition was evaluated fitting Brillouin function. Sample with 0.1 at.% Mn was studied by EXAFS technique (at HASYLAB, Hamburg) to evaluate local environment of Mn ions in the lattice.

GaMnAs samples were subjected to an annealing at various temperatures (700-1200 deg C) during 0.5 to 100 hours. Such thermal treatment was expected to change properties of GaMnAs if Mn composition exceeded an equilibrium value. Hole concentration was used as a most sensitive probe of changes caused by annealing. For GaMnAs with 0.1 at.% of Mn no changes of electrical properties were observed at any annealing temperature. This indicates that Mn composition of 0.1 at.% is still below an equilibrium doping limit.

Such a conclusion is confirmed by EXAFS studies, which revealed a well-resolved structure up to 4-th coordination shell, which could be fitted assuming solely substitutional Mn(Ga) incorporation.

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17:20 Poster 22

Ferromagnetic Fe-implanted 6H-SiC: new results towards Diluted Magnetic Semiconductor

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In 2000, Dietl et al predicted that hole-mediated ferromagnetism with high Tc can be obtained in wide band gap semiconductors with low spin-orbit coupling. SiC is such a semiconductor and Tc up to 270 K has been observed in Fe-implanted 6H-SiC. SiC is now recognized as a good candidate for diluted magnetic semiconductors (DMS) applications, but the origin of the ferromagnetic contributions in implanted SiC is still to be determined. Recently, we have shown that interesting ferromagnetic behaviour up to room temperature has been detected in 6H-SiC multi-implanted with $^{57}Fe^{+}$ and $^{56}Fe^{+}$ ions at 380°C and subsequently annealed at 1000°C.
A microstructural study of such ferromagnetic Fe-implanted 6H-SiC samples with Mössbauer spectroscopy and X-ray techniques has shown that strongly disordered Fe-containing regions might be responsible of the observed ferromagnetic behaviour. In this work, we propose a continuation of this study by extending the annealing temperature range from 600°C up to 1400°C. Investigation of the intimate structure on these entities and Fe location, new microstructural results and systematic characterization of their magnetic properties with Polarized Neutrons Reflectivity and magnetometry with superconducting quantum interference device and of their transport properties will be given, with the aim to contribute in deciding whether this system could really be a DMS or not.

17:20 Poster 23

High Curie temperature ferromagnetism in self-organized $Ge_{1-x}Mn_x$ nano-columns

Thibaut Devillers[1], Matthieu Jamet[1], Andre Barski[1], Romain Dujardin[1], Valier Poydenot[1], Pascale Bayle Guillemaud[1], Johan Rothman[4], Edith Bellet-Amalric[1], Alain Marty[1], Joël Cibert[3], R. Mattana[2], Serge Tatarenko[4]

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Room temperature ferromagnetic semiconductors, compatible with the silicon technology is one of the major challenges in the spintronic research. In this work, we show that, grown by molecular beam epitaxy (MBE), Mn-doped Ge, with a mean manganese content of 6%, exhibits ferromagnetic ordering over 400K. Structural and chemical analyses performed using Transmission Electron Microscopy (TEM) and Energy Electron Loss Spectroscopy (EELS) revealed strong non-homogeneities of Mn distribution in our MBE grown GeMn samples. Growth process induces the segregation of Mn and formation of Mn rich nanocolumns surrounded by a nearly pure Ge matrix. The average diameter of nano-columns is 3 nm and the average distance between nanocolumns is about 10 nm. This indicates that the concentration of manganese in nanocolumns is about 30% which is an unknown phase of the GeMn into the binary phase diagram of GeMn. The magnetic properties of our GeMn films grown on Ge (001) were investigated with a superconducting quantum interference device (SQUID). Magnetization measurements revealed a Curie temperature higher than 400K. The zero-field cooled (ZFC) and field cooled (FC) SQUID investigations exclude the presence in our GeMn films of the GeMn clusters and of the known stables phases of GeMn: Ge_3Mn_5 or Ge_8Mn_{11}. Magneto-transport measurements evidence a pronounced anomalous Hall effect and confirm: (i) p-type character of our GeMn layers, and (ii) pronounced holes spin-polarization.

17:20 Poster 24

Anomalous Hall effect in Mn-implanted p-type Si

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Si1-xMnx alloy has been regarded as the most compatible material of DMSs to semiconductor industry of today. The Curie temperature (T_C) reported so far is around 75 K, even higher than room temperature. Si1-xMnx samples were fabricated by implanting Mn+ ions into p-type silicon (100) substrates with an energy of 150 keV at the doses of (5~10) $x10^{15}/cm^2$ to produce average volume concentrations of 1~3 at% in the top 200 nm of Si substrates. In order to activate the Mn ion into Si lattice site and remove the damages caused during ion implantation process, post annealing were carried out at temperatures ranging from 700 to 900°C for 30 sec under Ar atmosphere. In particular, B^+ ions were first implanted at energy of 25 keV, the corresponding projected range calculated by TRIM simulation program shows B^+ atoms was located at interior side than Mn^+ atoms. Due to conductivity enhancement by co-doping B atoms, the current can be confined and accurately flows into the Si1-xMnx layer while the magnetotransport measurement The field dependence of the Hall resistance (R_{Hall}) in Si1-xMnx sample were performed by Van der Pauw method at temperatures ranging from 10 K to room temperature. Strong anomalous Hall effect clearly reveal that the present sample is ferromagnetic for temperature below 30 K. Moreover, we have observed the specific variation in magnetic and electrical behaviors while the $Si_{1-x}Mn_x$ samples were under different post annealing condition. The further magnetic characterization and detailed microstructure were also performed by SQUID magnetometer and TEM analysis, respectively. The local composition distribution of $Si_{1-x}Mnx$ was precisely characterized by an energy dispersive x-ray spectrometer. The implication of the effect of the additional B+ implantation and the corresponding magnetotransport properties will also be discussed.

17:20 Poster 25

Magnetic properties of Si-Ge whiskers

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Si-Ge solid solution whiskers are used as sensors of various physical parameters (temperature, pressure, strain) operating in wide range of temperatures including cryogenic ones. In present paper magnetic properties of Si-Ge<B> whiskers were studied in magnetic fields (0,3-4,0) kQe in temperature range 4,2-300K. In particular magnetization and magnetic susceptibility of the whiskers were investigated. The results obtained in temperature range 77-300K show two peculiarities: 1) substantial paramagnetic contribution in whisker magnetization and magnetic susceptibility, respectively; 2) nonlinear dependency of magnetization on magnetic field. These facts indicate in existing in the whiskers ferromagnetic ordering. Really observing of magnetization hysteresis at 4,2 K and substantial growth of magnetization (in several orders of magnitude) at T< 7 K confirm the above supposition. Possible reasons of the effect observed are discussed.

17:20 Poster 26

Magneto-optical and Raman spectroscopes of SnO2 doped by Co

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Diluted magnetic semiconductors exhibit many interesting properties such as magnetic, magneto-optical, and magneto-electronic ones. In this work the RT ferromagnetism is observed in chemically synthesized samples of Sn1-xCoxO2 compound (x=0.07 and 0.1). the deposited films were annealed in 02 atmosphere at T=450 oC. Magnetic properties of the samples were measured by MO Kerr effect system. The absorption spectra a(hw) of SnO2:Co films were recorded on Fourier-transform spectrometer. Raman-scattering experiments were made at 300 K and in the magnetic fields up to 5 kOe. A interband absorbtion edge is revealed at ~3.4-3.6 eV. The a(hw) increases with doping Co in-gap. The absorption spectra show addtional shoulders at ~3.9 and 4.8 eV. The peaks in the Raman spectra for Sn1-xCoxO2 films at ~634, ~775 and ~477 cm-1 resemble the vibration modes of microcrystal SnO2. Additional Raman peak at ~545 cm-1 may be related to the Co2+ ions vibrations or polaron formation including O-vacancy presence. The peak positions of main Raman lines did not show any measurable change in external magnetic field, but the intensities of the additional peaks at ~545 cm-1 enhances whereas the applied magnetic field H increases. Our measurements of the absorption and the Raman spectra show that the vacancies tend to form a near-neighbour complex with Co atoms suggesting a spin-polaron magnetism.

17:20 Poster 27

Co-doped TiO_2 films grown on Al_2O_3 (0001) by laser ablation

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Of the various groups of semiconductors, the dilute magnetic oxide semiconductors appear to be one of the most promising routes to high Curie temperature ferromagnetic semiconductors. Indeed, both the anatase and rutile structure-types of TiO_2 doped with Co have been found to be ferromagnetic at room temperature [1, 2].

In this paper we report on Co-doped TiO_2 thin films that were prepared by Pulsed Laser Deposition (PLD) on sapphire substrates. Ablation was carried out in argon atmosphere at total pressure between 7 and 50 Pa, for different substrate temperatures up to 400 ºC, by using polycrystalline targets of TiO_2 rutile with a few mol% of Co_2O_3. Some of the samples were prepared by adding a small amount of hydrogen to the gas phase during growth.

The effect of varying the laser fluence, hydrogen and argon flux, total pressure and growth temperature on the structure and physical properties of the films was systematically studied. Characterisation of the as-deposited films was performed using various structural, compositional, optical and magnetic characterisation methods.

1. Y. Matsumoto, M. Murakami, T. Shono, T. Hasegawa, T. Fukumura, K. Kawasaki, P. Ahmet, T. Chikyow, S.Y. Koshihara, H. Koinuma, Science 291 (2001) 854.

2. Y. Matsumoto, R. Takahashi, M. Murakami, T. Koida, X.-J. Fan, T. Hasegawa, T. Fukumura, M. Kawasaki, S.-Y. Koshihara, H. Koinuma, Jpn. J. Appl. Phys. 40 (2001) L1204.

17:20 Poster 28

Photonic crystals based on titania and Co-doped titania

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There has been a considerable interest in the investigation of photonic crystals (PCs) due to the unique possibilities that they offer in having control over photons. From the point of view of materials science, PCs are dielectric composites with a highly periodic structure in which the refractive index varies on the scale of visible light wavelength. The key to making PCs is the requirement to synthesize ordered dielectric lattices with a high refractive index contrast n_1/n_2, where n_1 and n_2 refer to the high and low refractive indices in the

structure. For this reason, PCs made of ordered spherical voids in titania are of great interest, because TiO_2 possesses both high refractive index (2.5-2.9, depending on the crystalline phase) and low absorption at visible wavelengths. Moreover, since Co-doped titania is a well-known dilute magnetic semiconductor exhibiting ferromagnetism above room temperature, this material is a very promising candidate for tunable PCs.

In the present work we report the preparation of PC films based on titania and Co-doped titania. Colloidal crystal composed of latex monodisperse microspheres were used as templates; interstitial voids between the spheres are infiltrated with alkoxide precursors of TiO_2 or Co:TiO_2; after gelation the initial spheres are selectively removed by sintering. Structural and optical properties of both undoped and Co-doped titania PCs are discussed.

The work was supported by the Russian Foundation for Basic Research (grants nos. 05-03-32778 and 04-03-39010) and the Program for Fundamental Research of Russian Academy of Sciences.

17:20 Poster 29

Ferromagnetism of $In_{1-X}Mn_XSe$ and behaviour of excess Mn in InSe crystal

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Static magnetic susceptibility (MS), electron spin resonance (ESR) and magnetic force microscopy of annealed crystals InSe doped 5%at. Mn in the temperature range 2.4-300 K were studied.The XRD investigation of samples phase composition has shown, that the hexagonal phase InSe (P63/mmc) with reduced lattice constant a=4.0026Å;c=16.634Å is basic one. The lowering of lattice periods testifies to creation of solid solution $In_{1-X}Mn_XSe$ which fills more than 90 % at crystal volume. Besides it samples contain second (cubic) phase MnSe (Fm3m,a=5.456Å) which is antiferromagnetic one with Neel temperature 135K.The magnetization dependences on magnetic field in the temperature range 2.4-271 K were measured and family of magnetic hysteresis loops was obtained testifying to ferromagnetic state of $In_{1-X}Mn_XSe$ solid solution. Paramagnetic constant background of magnetic susceptibility originates of atypical Van-Vleck $Mn^{3+}(d^4)$ ions which are in a state of nonmagnetic singlet in crystal field. Very narrow (2 Oe) ESR line with g=1.999 appeared at temperatures lower 23K which is apparently due to electrons localized on the level of space quantization.The occurrence of magnetic field gradient for a series of 35x35μm^2 areas in the crystal plane of InSe<Mn> was established. The change of vibrating frequency of silicon probe covered with ferromagnetic coating of cobalt was used. It was revealed that dominating area of a plane has no magnetic features. Separate inserts (1-7μm) with magnetic properties distinguished from dominating material and localized on imperfections were discovered. Taking into account magnetic measurements, RFA and XRD data they can be identified as inclusions of antiferromagnetic phases MnSe and Mn.

17:20 Poster 30

Investigations of dilute magnetic material HgTe:Cr

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The doping of semiconductors by d-elements impurities enables to operate with their characteristics, in particular with their magnetic and kinetic properties. The behaviour, differences of action of Cr impurity and its effect on properties of HgTe are shown. The nature of defect states is discussed. Magnetic susceptibility (MS), specific resistivity, Hall coefficient R, thermoelectromotive force α and electron spin resonance (ESR) of HgTe doped by Cr in the region 4.2÷350 K in magnetic fields up to 7 kOe were investigated. Electron concentration was $(6\div25)\cdot10^{16}$cm^{-3} at 4.2 K. General concentration of chromium doping impurity was in limits of $2\div50\cdot10^{18}$ cm^{-3}. The investigation results of R(T) in HgTe:Cr show a presence of a peculiarity at T~25 K. A magnitude of effect increase with Cr concentration increase. It may be explained by existance of resonance acceptor Cr-level near a bottom of conduction band. Thermoelectromotive force of HgTe:Cr had an extraordinary temperature dependence - a saturation at T>150 K. This may be explained by action of two competition mechanisms: increase of α~T, lnT and decrease of $\alpha\sim n^{-2/3}$, (α_0-lnn), where n~exp(-ε/kT). Temperature dependence of MS of HgTe:Cr show the presence of additional paramagnetism, which does not depend from temperature, grow with increase of Cr concentration and can be considered as paramagnetism Pauli of ions Cr^{2+}. At high concentration the Cr impurity leads to occurrence of orientational paramagnetism, which increase with temperature decrease. The exchange interaction between the magnetic centres is absent and the mechanism of single introduction of impurity atoms in a lattice is observed. Absence of ESR signal which is obliged to chromium testifies that almost all Cr is in nonmagnetic state. The Cr ion has a configuration $Cr^{2+}(d^4)$, which leads to Van-Vlek paramagnetism independent from temperature.

17:20 Poster 31

Nanoscale formations in $Hg_{1-x}Mn_xTe_{1-y}Se(S)_y$ crystals

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The investigation of effective magnetic moments of Mn atoms and paramagnetic Curie temperatures, which characterize the fettle of magnetic subsystem (formed by Mn atoms) and can be defined from $1/\chi_{Mn}$ = f(T), before and after thermal treatment, allow to judge about changes, which occur in the crystals of marganseinclusive solid solutions on the basis of $A^{II}B^{VI}$ in the process of thermal treat-

ment.

The process of thermal treatment of semimagnetic solid solutions on the basis of mercury chalkogenide samples in the vapour of chalcogen leads to formation new or to change the sizes of clusters (nanoscale formation Mn-Se-Mn-Se, Mn-Te-Mn-Te, Mn-Te-Mn-S) which exist in the crystal. Increasing or decreasing the claster sizes determines the conditions of thermodynamic equilibrium in the crystals (that is accordance of cluster size to x composition). It leads to change of the effective magnetic moments of Mn atoms and of the paramagnetic Curie temperatures, which characterize the fettle of magnetic subsystem (which formed by Mn atoms). The increasing of thermal treatment continuance leads to improvement the homogeneity of cluster consistence in solid solution. The change of paramagnetism in investigated crystals afterward their thermal treatment in the Hg vapour occurs by such reasons: the change of cluster size, the change of Hg account in the junctions and interjunctions, the change of the charge carriers concentration and increasing of non-interactive between themselves Mn atoms.

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium F

Under construction

Welcome

The Symposium will cover all aspects of basic and applied research on wide-bandgap II-VI compounds and their alloys, including diluted magnetic semiconductors, i.e. -- on such materials as ZnO, ZnMnO, ZnSe, ZnSeS, CdZnTe, and CdMnTe.
Emphasis will be given to new technological and application concepts concerning both bulk crystals and various quantum structures with reduced dimensionality, ranging from two to zero.
We presume that the presented results of the application-oriented materials research on the wide-bandgap II-VI compounds will be focused on:
- light emitting structures,
- infrared and visible light detectors,
- X- and gamma radiation detectors,
- materials for spintronics.

The Symposium will be devoted mainly to the materials research in the three groups of topics:
(1) Oxide crystals (e.g. ZnO, ZnMnO):
- growth of the substrate crystals,
- p-type doping,
- homo- and hetero-structures and their applications mainly as optoelectronic sources of light.
(2) Tellurium- and selenium-based wide-bandgap semiconductor compounds, like CdTe, CdZnTe, ZnTe, CdMnTe, ZnMnTe, ZnSe, and ZnCdSe. Potential applications as:
- substrates for infra-red detectors,
- semiinsulating platelets for X and gamma radiation detectors,
- Faraday optical isolators for telecommunications based on lasers and optical fibres,
- materials for spintronics,
- materials for light sources.
Special place belongs to the applications of the ultrafast magnetooptical phenomena,
(3) Epitaxial structures based on oxides, tellurides and selenides:
- growth,
- characterization,
- possible applications, mainly in optoelectronics.

Certain topics, concerning all the materials, will be discussed. These topics are:
- growth techniques,
- defect structures,
- defect control,
- doping procedures,
- optical properties,
- degradation

Organisers

Scientific-Organising Committee:

Prof. Andrzej Mycielski (Chairman), Institute of Physics PAS, Warsaw, Poland
Prof. Marek Godlewski, Institute of Physics PAS, Warsaw, Poland
Prof. Jacek Kossut, Institute of Physics PAS, Warsaw, Poland
Prof. Robert Triboulet, CNRS/LPSC
Prof. Andreas Waag, Institute of Semiconductor Technology, Braunschweig Technical University

Proceedings

The proceedings of the conference will be published as a regular issue of ***physica status solidi (b)*** . All manuscripts will be reviewed according to the standards of ***physica status solidi*** by two referees.

Programme

Monday, 4 September

Opening Ceremony
2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Session Chairman
Monday afternoon, 4 September, 14:00
Chair: Jacek Kossut

14:00	Invited oral

Preparation and properties of ZnO based heterogeneous devices

Hadis Morkoc, Vitaly Avrutin, Umit Ozgur, Yahya I. Alivov, Natalia Izyumskaya

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ZnO is a wide band gap semiconductor with potential applications to optical emitters due to its very high emission intensity and large exciton binding energy (~60 meV) which persists even at room temperature. In addition, unlike GaN, bulk ZnO substrates with very high quality are available. The bottleneck, however, has been the unambiguous attainment of p-type conductivity. In the interim period, heterojunctions of ZnO with other semiconductors with p- type conductivity are used to demonstrate the potential of this material. In this presentation, growth by MBE, and electrical and optical properties of ZnO/GaN and ZnO/SiC heterojunctions inclusive of band discontinuities will be discussed. In addition, preparation and optical properties of ZnO nanowires will be presented.

14:45 Invited oral

Homoepitaxy of ZnO : from the substrate to doping

Bruno K. Meyer

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In order to realize controlled p-type doping in ZnO it is absolutely necessary to control and understand the role of point and extended defects in the epitaxial films. This starts with the choice of the substrate since for sapphire and GaN on sapphire substrates there is a severe contamination by diffusion of the group III elements in the ZnO films. However, also the choice of a ZnO substrate matters. In the first part of the talk we address to the properties of the ZnO substrates in terms of crystallinity, surface morphology and preparation, polarity etc. Secondly, we report on the homoepitaxial growth and demonstrate the influence of the II/VI ratio on the electrical and optical properties of the films. In the last part the doping with Li, Na and P from the evaporation of solid precursors will be presented and discussed.

Coffee break

Monday afternoon, 4 September, 15:30

Session Chairman

Monday afternoon, 4 September, 15:50

Chair: Hadis Morkoc

15:50 Oral

Effect of the Annealing Atmosphere on the Quality of ZnO Crystal Surface

Krzysztof Grasza[1,2], Elżbieta Łusakowska[1], Paweł Skupiński[1], Krzysztof Kopalko[1], Jadwiga Bak-Misiuk[1], Andrzej Mycielski[1]

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Zinc oxide crystals were grown by CVT contactless technique and thermally annealed in the air, nitrogen or oxygen atmospheres, or zinc or arsenic vapours. Wide ranges of annealing times and temperatures were applied. The variety of morphology and quality of the crystal surfece was investigated by AFM and XRD. It was found, that thermal annealing in the air, nitrogen, oxygen and zinc resulted in similar evolution of the crystal surface, although there are observed differences in the rate of the changes. The annealing in zinc vapour resulted in smooth morphology and stabilization of colour of the crystal, which was dependent on the pressure of the zinc vapour. The obtained roughness (RMS) was typically lower than 1 nm. The annealing in arsenic vapour leads to degradation of the crystal surface. The improvement of the surface was not observed in case of thermal annealing of the rough ZnO surfaces obtained by ALE homoepitaxy on the smooth CVT as-grown ZnO surfaces.

This work was supported by Ministry of Education and Science under grant No. 3 T0A 051 28.

16:10 Oral

Growth of ZnO thin films by metalorganic chemical vapor deposition for optoelectronic and spintronic applications

William E. Fenwick, Tahir Zaidi, Vincent Woods, Matthew H. Kane, Ian T. Ferguson

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Zinc oxide (ZnO), with its wide bandgap (~3.37eV) and high exciton binding energy (~60meV), is a promising material for use in optoelectronic and spintronic devices. ZnO growth by metal organic chemical vapor deposition (MOCVD) is a useful technique in because of its flexibility and scalability to larger commercial systems, provided it can consistently yield high-quality undoped, n-type, and p-type ZnO. This work presents results obtained from an investigation into factors affecting MOCVD growth of ZnO and the effects of transition metal incorporation on structural, optical, and magnetic properties of ZnO. DEZn and O_2 were used as the precursors in a vertical injection, rotating disk MOCVD reactor. Trends in materials properties were correlated to changes in growth parameters in order to build an understanding of the kinetics of ZnO growth by MOCVD. Total volume flow and injection velocity were found to significantly effect the growth kinetics and crystal quality. Undoped ZnO thin films have been produced that show both strong luminescence and good crystal quality as measured by X-ray diffraction and Raman spectroscopy. PL spectra show a strong luminescence peak around 3.28eV, suggesting that the dominant emission peak is due to an LO phonon replica of the band-to-band emission. XRD θ-2θ scans show a linewidth of 180 arcsec. Raman spectra showed the E_2(high) mode at 437cm^{-1} while the 2nd-order phonon mode near 332cm^{-1} was of very low intensity. Initial results will also be presented on p-type doping and TM-doping of ZnO thin films by MOCVD.

16:30 Oral

Investigations of ZnO substrates after high temperature annealing

Christian Neumann, Swen Graubner, Bruno K. Meyer

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In order to promote growth of ZnO films on ZnO substrates, defects introduced by the surface polishing procedure have to be removed. We investigate the influence of a high temperature annealing in O_2-atmosphere on the structural porperties of the ZnO substrates by atomic force microscopy. Only at temperatures above 1100°C atomic steps (terasses) are seen, the remaining defects can be assigned to dislocations in a density between 10^4 to 10^5 cm^{-2}. Interestingly the electrical properties also change from high resistive to n-type con-

duction, which make the substrates - apart from the homoepitaxial growth on a perfect template - suitable for top-to-bottom contacts.

16:50 Oral

Photoconductivity of Ga doped ZnO Film grown by Reactive Plasma Deposition Method

Seiichi Kishimoto, Takahiro Yamada, Aki Miyake, Hisao Makino, Tetsuya Yamamoto

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Zinc oxide has been extensively studied as the versatile semiconductor materials for the practical applications.One of the interesting application of ZnO is the ultra-violet light (UV) sensors. In this study, we report the photoconductivity of Ga doped ZnO (GZO) films.

The polycrystalline GZO films were prepared on glass substrates by reactive plasma deposition. From out-of-plane XRD measurement, we have reported that these GZO films showed a very high level of orientation along the c-axis [1]. For the photoconductivity measurement, thermally evaporated aluminum or indium electrodes on the GZO film in a coplanar configuration were prepared. The contact study was also performed under various conditions forming the electrode.

For a GZO film with a film thickness of 186 nm prepared under the condition that Ga content was 4 wt% and O_2-gas flow rate was 20 sccm, we find the resistivity of 7.81×10^{-4} cm, the carrier concentration of 3.6×10^{20} cm^{-3}, and the Hall mobility of 21.6 cm^2/Vs by Hall effect measurement. For GZO films grown under the same condition, almost temperature independent carrier concentrations indicate the formation of impurity band. The GZO film exhibited the photoconductivity at the wavelength, ranging widely less than 380 nm. In other words, the GZO film can use to detect UV-A, UV-B, and UV-C bands. The photoconductivity properties of GZO films are discussed with structural, electrical, and optical properties. These results indicate the possibility that the unique UV sensor with the high optical transmission in the visible region.

[1] T. Yamamoto et al. Superlattices and Microstructures 38 (2005) 369.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Session Chairman

Tuesday morning, 5 September, 9:00
Chair: Bruno K. Meyer

9:00 Invited oral

Manipulating the spin states of a single magnetic atom in a II-VI quantum dot.

Henri J. Mariette[1], Lucien Besombes[1], Yoan Léger[1], Laurent Maingault[1], Catherine Bougerol[1], Joël Cibert[2], David Ferrand[1]

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Precise control of electronic spins in semiconductors should lead to development of novel electronic systems based on the carriers' spin degree of freedom. Magnetic semiconductor quantum dots (QDs), where excitons can interact strongly with the magnetic atoms, hold particular promise as building blocks for such spin-based systems. This requires the ability to detect and manipulate individual spins. We will show in this presentation how we can optically probe the magnetic state of a single Mn atom embedded in an individual QD.

In the case of a quantum dot incorporating a single magnetic atom (spin S) and a single confined exciton, the exchange interaction between the exciton and the magnetic atom acts as an effective magnetic field, so that the atom's spin levels are split even in the absence of any applied magnetic field [1,2]. A set of (2S + 1) discrete emission lines can be resolved in magneto-optic micro-spectroscopy experiments, providing a direct view of the atom's spin states at the instant when the exciton annihilates.

The influence of the number of confined carriers on the spin splitting will be then considered by investigating both the biexciton and trion (X^-) transitions in the same Mn-doped QD. The injection of the second electron-hole pair cancels the exchange interaction with the Mn ion and the spin degeneracy is almost restored. Bias controlled single carrier charging allows us to tune the presence of excess carriers in the dots. The fine structure of charged excitons coupled with a single Mn spin differs strongly from the exciton-Mn one [3]. This can be attributed to the absence of electron-hole exchange interaction in the case of charged exciton.

[1] L. Besombes et al. Phys. Rev. Lett. 93, 207403 (2004)
[2] Y. Léger et al. Phys. Rev. Lett. 95, 047403 (2005)
[3] Y. Léger et al. to be published in PRL (2006)

9:45 Invited oral

CdSe quantum dots and their application in surface emitters and micropillars: A comparison to analogous nitride structures

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The growth of CdSe quantum dots (QDs) embedded in a ZnMgSSe matrix will be discussed. Laser diodes with CdSe/ZnSSe quantum

dot stacks show a superior stability compared to conventional LDs with a quantum well as optically active region. For low power applications CdSe QDs as active region can be a real alternative to other semiconductor materials for the green spectral region. Approaches to reach a single photon emitter at room temperature will be presented. The monolithic growth of a vertical cavity surface emitting laser (VCSEL) requires a reflectivity well above 99%. This can be realized by stacks of distributed Bragg reflectors (DBR). For the low index DBR a short period superlattice ZnCdSSe/MgSe is used. The stop-band can be tuned over the whole visible spectral region. Optically pumped lasing of such a monolithic VCSEL has been obtained. Using a focused ion beam (FIB) for nano-structuring, air gap micropillars can be realized with a very high aspect ratio. The dependence of the optical modes on pillar diameter will be presented including theoretical modelling. First results on CdSe QDs in micropillars show the Purcell effect (enhancement of emission) when such a QD comes in resonance with the cavity mode.

InGaN quantum dots can be easily realized on open surfaces but are highly unstable when overgrown by GaN. Using a novel growth procedure the problems can be overcome and evidence for single quantum dot emission was obtained. Such InGaN quantum dots show a high T-stability up to 150 K which make them, in connection with their low density of mid 10^8 cm^{-2}, a good candidate for single photon emitters. The approach developed for II-VI has been used for nitrides as well to obtain high reflectivity DBRs for VCSELs. One problem here is the cracking due to a strong thermal mismatch of the materials (AlN-GaN). Nevertheless, first microcavities containing InGaN quantum wells could be realized showing the characteristic optical modes.

Coffee break

Tuesday morning, 5 September, 10:30

Session Chairman

Tuesday morning, 5 September, 11:00

Chair: Henri J. Mariette

11:00 Oral

Nonexponential photoionization of the DX related centers in indium doped $Cd_{1-x}Mn_xTe$. On the relationship between the stretched exponential and two-exponential relaxation models.

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The kinetics of phototransients due to the presence of defects in semiconductors is commonly analyzed in the terms of the multiexponential fitting or by means of the stretched exponential function.The motivation of this paper is to explain the relationship between both models.For this purpose the kinetics of phototransients due to DX centers in indium doped $Cd_{0.93}Mn_{0.07}Te$ crystals grown by the Bridgman method were studied.Photoconductivity transients, measured at 77K for different photon fluxes with fixed photon energy,were analyzed. The obtained data can be well fitted with the stretched exponential function.On the other hand, it appears that the transients are also well fitted by a superposition of two weighted exponents with the short and long time constants b1and b2, respectively.To explain the result we propose the probabilistic representation of the relaxation function $\Phi(t)=\langle e^{-\beta t}\rangle$, given by a weighted average of the exponential decay with respect to the distribution of the random relaxation rate β,representing the intrinsic nonexponential behavior of the entire system.As it is well-known from probability theory, such an average can be expressed either as a sum of weighted exponentials $\Sigma p_i e^{-b_i t}$,if β takes positive discrete values b_i with probabilities p_i,or as the integral $\int e^{-bt}\rho(b)db$,if β takes values from $[0,\infty)$ with probability $\rho(b)db$.This "dual" representation of the relaxation function explains a relationship between the stretched exponential pattern and the two-exponential fitting.In the studied case the probabilities p_1,p_2 of taking two different time constans b_1,b_2 attributed to the two-step photoionization of the DX centers, are shown to be in agreement with the long-tailed stable distribution of the effective relaxation rate corresponding to the stretched exponential relaxation response.

11:20 Oral

Dephasing Of Free Carriers And Excitons In Bulk CdTe

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CdTe is widely used for fabrication of solar cells, X-ray and gamma-ray detectors. All these applications are based on the manipulation with incoherent carriers (i.e., carriers that do not have any definite phase relationship among themselves). Recently, however, also the coherent regime in semiconductors attracts a lot of attention due to the envisioned applications in the fields of quantum computing and spintronics. Here we report on the measurements of the carrier dephasing (i.e., loss of orbital coherence) using a self-diffraction technique of the ultrafast laser spectroscopy. The samples were thin platelets prepared from undoped bulk CdTe. We concentrated on the measurements in two samples that differ only in the preparation procedure - one was mechanically polished and the other was polished and also chemically etched. The etching leads to removal of several micrometers of CdTe from the polished surface and, consequently, to considerably lower concentration of preparation induced dislocations. The thickness of both samples was about 1 μm. We observed that the increased concentration of defects leads to a pronounced acceleration of carrier dephasing. The characteristic time constants describing the decay of the measured signal in the etched sample at 10 K are 1.3 ps for excitons and 0.34 ps for free carriers while these time constants are shorter than the time resolution of our experiment (0.08 ps) in the polished sample. The dephasing time of free carriers in the etched sample is a non-monotonous function of the lattice temperature, and above 100 K it is dominated by an interaction with optical phonons. We also observed that the dephasing of spin coherence is considerably slower than that of orbital coherence - the characteristic decay times are 60 ps for excitons and 150 ps for electrons

in the etched sample at 10 K. This shows that the spin coherence is rather appealing for applications where long coherence times are essential.

11:40 Oral

Sub-µm-mesa waveguides with strong light confinement for urtrafast intersubband optical switches based on wide-gap II-VI quantum wells

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Short-wavelength intersubband traditions (ISBT) in semiconductors quantum wells (QWs) with ultra fast carrier relaxation (~1ps) has attracted much attention, especially by aiming at potential applications for all-optical signal processing for high-bit-rate (above 160 Gbit/s) optical fiber communication system at λ~1.55 µm. Recently, sub-ps all-optical modulation at optical communication wavelength have been demonstrated in ISBT-waveguide devices based on several material systems, such as InGaAs/AlAsSb[1], GaN/AlGaN[2], (CdS/ZnSe)/BeTe[3].

In this presentation, we discuss the fabrication and characterization of ISBT-waveguide devices for telecom wavelength applications in wide-gap II-VI semiconductor based (CdS/ZnSe)/BeTe multiple quantum wells (MQWs).

The waveguide has a separate confinement heterostructure (SCH) structure which is consisted by ZnMgBeSe cladding layer (CL, n=2.35), a ZnBeSe optical confinement layer (OCL, n=2.45), (CdS/ZnSe)/BeTe MQW (n=2.54) active layer. The active layer has 40 period of QWs which is designed such that 15-ML(mono layer)-thick BeTe barriers with ZnSe/CdS/ZnSe (1/~2/1 ML) well layers exhibit intersubband (ISB) absorption at a wavelength of around 1.55 µm. For the fabrication of sub-µm mesa patterns, we employed reactive ion etching in an inductive-coupled-plasma using Ar and BCl_3 gases.

For the switching characteristics, we estimated ISB absorption saturation and recovery time which correspond to extinction ratio and gate time window for the ISBT switching device. A 10 dB extinction ratio is achieved by pump pulse energy of 7pJ (1565 nm). As the gate open time, the modulation band width of 0.36 (FMHW) ps is estimated. These results indicate that optical modulation up to 1 Tbit/s is possible in present II-VI-based ISBT waveguide.

[1] S. Sekiguchi., et al, OFC 2005 [2] N.Iizuka., et al, Opt. Express, 13 (2005), 3835 [3] R.Akimoto., et al, APL. 87 (2005), 181104

12:00 Oral

Deposition and properties of CdTe nanowires prepared by template replication

Ionut Marius Enculescu[1], Marian Sima[1], Monica Enculescu[1], Mihaela Enache[1], Lucian Ion[2], Stefan Antohe[2], Reinhard Neumann[3]

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CdTe nanowires were prepared by electrochemical deposition in nanoporous polycarbonate membranes. The method offers some very important degrees of freedom, i.e. the possibility to choose the desired density of nanopores by selecting the appropriate heavy ion irradiation fluence and the desired size and shape of the nanopores by using the appropriate etching conditions. Thus one can employ templates with the desired characteristics: high pore density for measurements of optical, structural or morphological characteristics of the nanowires and low pore density membranes for transport characterization.

The present report deals with the influence of the preparation conditions on the properties of CdTe nanowires. Thus the influence of parameters such as deposition potential or temperature on morphological or structural properties were investigated. Moreover, differences between wires deposited at constant potential and in a pulsed regime were emphasized.

Transport properties were also investigated, special attention being paid to metal semiconductor contacts properties at nanoscale.

Lunch break

Tuesday afternoon, 5 September, 12:30

Session Chairman

Tuesday afternoon, 5 September, 14:00

Chair: Paul Siffert

14:00 Invited oral

Material properties limiting the use of cadmium zinc telluride X- and gamma detectors

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Cadmium zinc telluride (CZT) is one of the most promising materials for the production of large-volume gamma-ray spectrometers and imaging arrays operable at room temperature. The performance of

CZT devices, the global capacity for growth of detector-grade crystals, and the size of the commercial market have progressed steadily over the past 5-10 years. Because of deficiencies in the quality of the material, commercial high-resolution CZT spectrometers are still limited to relatively small dimensions (< 2-3 cm^3), which makes them inefficient at detecting high photon energies (> 1 MeV) and somewhat ineffective for weak radiation signals except in proximity to the source. The detectors are very attractive for a much broader range of spectroscopic and imaging applications; however, increases in their efficiency are needed without sacrificing the ability to spectrally resolve gamma energies. Achieving the goal of low-cost efficient CZT detectors requires progress in the following areas: better uniformity of detector response, growth of large uniform single crystals, and improved device fabrication procedures. Despite the current material constraints, several types of electron-transport-only detectors have been developed: pixel, coplanar-grid, cross-strip, drift-strip, orthogonal coplanar strip, and virtual Frisch-grid, some of which are now addressing important applications. This talk summarizes the material factors limiting performance of CZT detectors and provides new insight into the critical role of small-scale defects (i.e., tellurium-rich inclusions) on the energy resolution and efficiency of detectors. Conclusive data demonstrating the relationship between Te precipitates (size, concentration, and spatial distribution) and the performance of CZT detectors are presented, together with a model of charge trapping for electrons transiting through areas populated with Te secondary phases.

14:45 Invited oral

Materials requirements in the group - CdTe, CdZnTe and CdMnTe - and recent advances for X-ray and gamma-ray applications.

Arnold Burger[1], Andrzej Mycielski[2], Michael Groza[1], Helen Jackson[1], Yunlong Cui[1], Andrzej J. Szadkowski[2], Barbara Witkowska[2], Wojciech Kaliszek[2]

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Although various other classes of materials including layered compounds and III-V compounds have also been considered and investigated, for more than four decades, CdTe-based crystals continue to be one of the leading materials for X-ray and gamma-ray spectroscopy applications at room temperature aimed at medical, space and national security applications. One of the major advantages of the CdTe, CdZnTe and CdMnTe group is the easiness to expand to higher energy gap ternaries without severe deterioration of the intrinsic transport properties. Even if quite related, large variations in the purity of starting materials, the yield of single crystal growth process, the uniformity of Zn or Mn and compensating dopants, and the formation of tellurium precipitates exist in this group, thus providing materials science research opportunities that are yet to be explored. Recent results and future challenges will be presented and discussed.

Work in Warsaw was partially funded by the Polish Ministry of Science and Higher Education through grant 3 T08A 046 30.

Coffee break

Tuesday afternoon, 5 September, 15:30

Session Chairman

Tuesday afternoon, 5 September, 15:50

Chair: Detlef Hommel

15:50 Oral

Self-compensation processes in CdTe<In> single crystals

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CdTe crystals doping by shallow donors are widely used to produce material for gamma- and X-ray detectors. This is possible due to self-compensation (SC) phenomena, which ensure high resistivity of obtained crystals. SC is well studied in CdTe<In> crystals. But all information concerns room temperature properties, even if the samples were treated by annealing. In this work in situ high temperature PD equilibrium and SC phenomena in CdTe crystals, doped by different quantities of In (10^{17}-10^{20} at/cm^3), were studied. High temperature Hall effect measurements under well defined Cd/Te pressure up to 1200 K were performed.

At low In content in the samples controlled conductivity was observed. Heating to T>1000 K under small Cd vapor pressure (P_{Cd}) afterwards resulted in electron density [e^-] decreasing in 1-1.5 orders of magnitude. The electron density became the same as in undoped CdTe. The samples with high In concentration revealed stable and high [e^-], which didn't depend on P_{Cd}. On the base of obtained results models of point defect concentration on temperature, component pressure and dopant activity were built. Some thermodynamic constants of In introducing into the CdTe lattice were defined also. SC models for temperature dependencies at different P_{Cd} were calculated.

16:10 Oral

Optical Studies of Wide Band Gap III-Nitride Semiconductor Quantum Wells and Superlattices

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A detailed optical study of several sets of AlN/GaN superlattice (SL) as well as AlGaN/GaN multiple quantum well (MQW) structures has been reported. In this study all materials were grown by MOCVD, as opposed to most previous studies where MBE was em-

ployed. Doublet excitonic features are observed in the PL spectra. They are explained in terms of discrete well width variations by one c-lattice parameter, i.e. two GaN monolayers. The residual photoluminescence (PL) line width in MQW structures is varying from about 10 meV for 15 Å QWs to > 15 meV for 45 Å QWs, also comparable to the best MBE structures reported. While the recombination process in undoped samples is excitonic, in MQWs doped with Si above about 5×1018 cm-3 free electrons (a 2DEG) are dominant, and the PL process is a free electrons-localized hole transition at low temperatures. The hole localization prevails up to very high n-doping, as was previously observed in bulk GaN. The hole localization is demonstrated via several experiments, including results on PL transient decay times and LO phonon coupling. Near surface band bending, due mainly to dopant depletion in doped structures or interaction with surface states in case of higher Al content in barriers, influence the distribution of electron filling among the QWs, making a detailed modeling of the spectral shape somewhat ambiguous. It is found that AlN barriers promote a strong room temperature PL signal from the QWs, as opposed to the case with AlGaN barriers.

16:30 Oral

Characterization of Growth Defects in GaN Layers withX-ray Microbeam

Rozaliya I. Barabash[1,2], C Roder[3], G E. Ice[1], Sven Einfeldt[3], S. Figge[3], Detlef Hommel[3], R. F. Davis[4]

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The spatially resolved distribution of strain, misfit and threading dislocations, and crystallographic orientation in uncoalesced GaN layers grown on Si(111) by maskless cantilever epitaxy or by pendeo epitaxy on SiC was studied by white beam Laue x-ray microdiffraction, scanning electron and orientation imaging microscopy. Tilt boundaries formed at the column/wing interface depending on the growth conditions. A depth dependent deviatoric strain gradient is found in the GaN. Density of misfit dislocations as well as their arrangement within different dislocation arrays was quantified. Two different kinds of tilt (parallel and perpendicular to the stripe direction) manifested themselves by mutually orthogonal displacement of the (0006)GaN Laue spot relative to the Si (444) Laue spot. Origin of tilts is discussed with respect to the miscut of the Si(111) surface and misfit dislocations formed at the interface.

16:50 Oral

Two-dimensional dopant profiling of InGaN/GaN light emitting diodes by electron hologarphy

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Electron holography in the TEM(trasmission electron microscopy) measure the two-dimensional voltage maps. We investigated the dopant profile of InGaN/GaN light emitting diode samples. The samples were grown by MOCVD(Metal Organic Chemical Vapor Deposition) and doped Mg, Si. We are preparing for electron holography measurement using FIB (focused ion milling) with low kV milling for which samples was removed ion damage layers. We compare electron holographic measurement to secondary-ion mass spectrometric data and discuss the results.

References

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2. Armin T. Tilke, Andreas Lenk, Uwe Muhle, Cajetan Wagner, Claus Dahl, and Hannes Lichte, IEEE TRANSCATIONS ON ELECTRON DEVICES, Vol. 52, No. 6, 1067 (2005).

3. A. E. Thesen, B.G. Frost, D.C. Joy, J. Vac. Sci. Technol. B20(6), 3063 (2002).

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

Session Chairman
Wednesday afternoon, 6 September, 14:00
Chair: Marek Godlewski

14:00 Oral

Metal-Organic Vapor Phase Epitaxy Growth and Crystallographic Study of Vanadium-doped ZnSe

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Recently, as a new functional device material, diluted magnetic semiconductors (DMS) have attracted special interest from many researchers. However, Curie temperature for most of DMS is lower than room temperature so that it has been desired to get DMS with

Curie temperature higher than room temperature for practical applications. K. Sato *et al*. reported that Vanadium(V)-doped ZnSe is expected theoretically as DMS inducing ferromagnetism above room temperature without carrier doping.[1)]

V-doped ZnSe film was grown on (100)GaAs substrate by MOVPE method in an atmospheric pressure under a hydrogen flow. Dimethyl Se and dimethyl Zn were used as the source materials. Vanadocene ($V(C_5H_5)_2$) was used as a dopant source for vanadium. The detailed experimental condition is presented in the previouns paper.[2)] The local structure and the crystallinity of sample was investigated by using XAFS and XRD.

On the research of the lattice distortion of V-doped ZnSe crystal three models were constructed as following cases; vanadium atoms (1) cohere and have the metal structure, (2) are replaced with the Zn-site, (3) are replaced with the Se-site. Fig.1 shows the relations between notation radial distribution function and magnitude of Fourier transformation $F(r)$ obtained by XAFS spectra for experimental and calculation results. Theoretical spectra calculated for the Se-site case comparatively agrees with the experimental one. This result indicates that vanadium atoms are replaced in the Se-site and/or exist in interstitial sites.

1) K.Sato *et al*. : Jpn. J. Appl. Phys., 40(2001) 651.

2) M.Tahashi *et al*. : Materials Transactions, 46(2005) 1908.

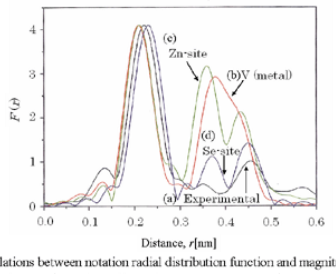

Fig.1 Relations between notation radial distribution function and magnitude of Fourier transformation $F(r)$ obtained by XAFS spectra for the experimental result (a)vanadium doped ZnSe sample and calculation results that vanadium atoms exist in (b) metal structure, (c) Zn-site and (d) Se-site in ZnSe crystal.

14:20 Oral

Two color spectroscopy of ZnSe:Cr

Vitalii Y. Ivanov[1,3], Marek Godlewski[1,2], Tom Gregorkiewicz[3]

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Chromim doped ZnSe is very promising material for optoelectronics (tunable lasing in Mid Infrared) and spintronics (high temperature ferromagnetism) applications.

In this communications two-color spectroscopy (pulse-to-probe) experiments are discussed. We show that two strong Cr^{2+} intra-shell emissions, the first at 930-1300 nm (with life time τ=1.8 ms) and the second at about 2200-2800 nm, can be efficiently pumped upon the $Cr^{2+} \rightarrow Cr^{+}$ photoionization transition. The intra-shell emission can be enchanced upon additional illumination with Free Electron Laser (FEL) with photon energy tuned at 0.08-0.14 eV range, as we demonstrate for the 930-1300 nm photoluminescence, for which we first present arguments for the Cr^{2+}-centers relation. Enhancement of th intra-shell emissions we relate to ionization of shallow ZnSe acceptors, followed by capture of free holes by Cr^{+} ions. Hole capture by Cr^{+} centers proceeds via excited intra-shell states of Cr^{2+} ions, and thus is followed by two easily detectable Cr^{2+} intra-shell emissions, opening a simple method for detection of THz radiation for photon energies above 80 meV.

14:40 Oral

ZnO/ZnMgO multiple quantum wells on sapphire: MBE growth, structural and lasing properties

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ZnO/ZnMgO heterostructures are promising for applications in ultraviolet light-emitting devices. Recently, we developed a specific procedure for the fabrication of ZnO/ZnMgO single quantum wells with abrupt interfaces and Mg mole fractions as high as x = 0.40 by radical-source MBE. These structures show very narrow luminescence linewidths and distinct excitonic emission up to room temperature (RT). We extended this approach to the growth of ZnO/ZnMgO multiple quantum wells (MQW) on *a*-plane sapphire and demonstrate RT lasing under optical pumping. To compensate for the lattice mismatch with the substrate, the growth is initiated with a thin ZnMgO nucleation layer followed by a 600 nm thick ZnMgO buffer layer. The five ZnO/ZnMgO MQWs periods are grown combining low-temperature growth of ZnMgO and an annealing step at each interface. TEM data reveal the high quality of the interfaces and uniformity of the layers composing the heterostructure. The thickness of the well layers agrees well with a value calculated from RHEED oscillations ruling out intermixing phenomena. The two ZnMgO buffer layers with significantly different defect densities are clearly distinguished. The substrate/ZnMgO interface region has a much higher dislocation and stacking fault density and dislocation annihilation rate. Above this region, there are threading dislocations with mainly edge character visible. Their density decreases only gradually with layer thickness. Nevertheless, the structures demonstrate strong excitonic emission and lasing under optical pumping with a threshold of 150 kW/cm^2 at RT. The lasing mechanism differs from the formation of an electron-hole plasma and exciton-exciton scattering, typically observed for ZnO epilayers and ZnO/ZnMgO MQWs. The small energy shift of 64 meV at RT between spontaneous and stimulated emission bands indicates that localized states are responsible for the laser action.

Coffee break

Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Session Chairman
Thursday morning, 7 September, 9:00
Chair: Tomasz Wojtowicz

9:00 Invited oral

Novel semiconductor nanorod/nanowire architectures.

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In this lecture we will present the structural and optical properties of ZnO nanorods grown by chemical vapor deposition (CVD) and wet-chemical syntheses. The CVD-growth is catalyzed by gold islands, pre-patterned on sapphire substrates by use of the nanosphere lithography (NSL) technique, resulting in laterally ordered ZnO nanowires of diameters less than 100 nm and a length of up to several micrometers. By modifying the NSL mask using annealing or chemical treatment, the holes between adjacent nanospheres can be reduced, which results in smaller, well-separated catalytic islands on the substrate. Tilting and turning the sample holder during catalyst evaporation produces many different structures. Rods grown by the wet chemical approach from zinc organics or zinc salts as precursors are much thinner with diameters less than 10 nm and aspect ratios up to 10. We show that the use of long-chain amines induces one-dimensional growth. Currently, we are carrying out doping experiments on both CVD and wet-chemical synthetic routes, to influence the conductivity, magnetism or luminescence of ZnO. We are going to present first results on these doped ZnO nanorods, which have potential applications as light emitting devices, sensors, bio-labels or piezoelectric devices. All produced rods are characterized structurally by electron microscopy (SEM, TEM, HRTEM) and optically by absorbance and photo-luminescence spectroscopy. Furthermore, we show results obtained by scanning near-field optical microscopy of the ZnO nanowires.

9:45 Invited oral

Cathodoluminescence study of wide band gap ZnO nanorod heterostructures.

Bernard Piechal[1], Jinkyoung Yoo[2], Abdel-Hamid El-Shaer[3], Augustine C. Mofor[3], Gyu-Chul Yi[2], Andrey Bakin[3], Andreas Waag[3], Fabrice Donatini[1], Dang Le Si[1]

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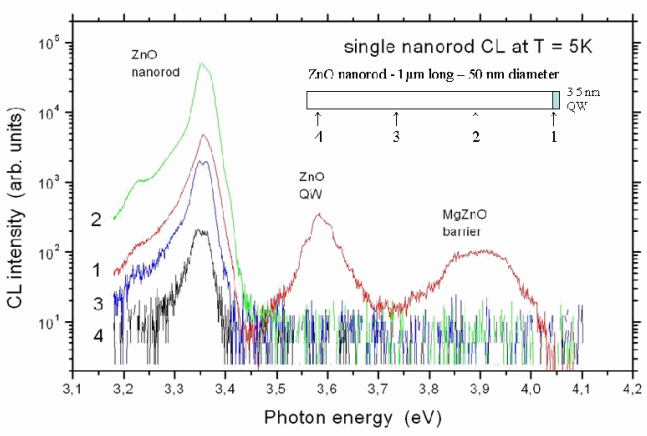
The considerable interest in nanorod structures stems from novel physics and applications opened up by their 1D character. In the case of wide band gap semiconductors, such as ZnO with record exciton binding energy of 60 meV, also high radiative quantum efficiency could be expected, which makes ZnO nanorods particularly attractive as emitters in the UV spectral range.
In this presentation we report a cathodoluminescence (CL) investigation of ZnO-based nanorods grown by MOCVD and by MBE combined with vapour phase transport. Samples are ZnO/ZnMgO quantum wells grown on top of ZnO nanorods with lengths of 1-5 µm and diameters of 50-500 nm. CL of single nanorods has been measured to probe structural and compositional properties along the nanorod axis. CL results at T = 5K and room temperature will be discussed.

This work is supported by the European Union projects HPRN-CT-2002-00298 "Photon Mediated Phenomena in Semiconductor Nanostructures", STREP FP6-016924 "Nanophotonic and Nanoelectronic Devices from Oxide Semiconductors (NANDOS)", and the Associated International Laboratory France-Korea "Center for Photonics and Nanostructures (CPN)".



CL at T = 5 K of a single ZnO nanorod at different locations 1 - 4 along the nanorod axis.

Coffee break
Thursday morning, 7 September, 10:30

Session Chairman

Thursday morning, 7 September, 11:00
Chair: Michael Giersig

11:00 Invited oral

ZnO nanostructures, thin films and devices

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ZnO based semiconductor devices are potentially interesting for optoelectronics in the UV and blue spectral range, transparent electronics, and magnetoelectronics operating at room temperature. An additional aspect of ZnO is the possibility to fabricate ZnO nanopillars by self-organisation. Due to the small footprint of nanopillars on the substrate, virtually any substrate can be used, while defect density remains small. ZnO thin films, ZnMgO-ZnO heterostructures and ZnO nanostructures were fabricated by molecular beam epitaxy (MBE), vapour phase transport (VPT) and aqeuous chemical growth approach (ACG). The possibility to employ several fabrication technologies is of special importance for realization of unique device structures. MBE was implemented for ZnO-based layers and heterostructures as well as ZnMgO-ZnO quantum wells growth. A novel advanced VPT approach of ZnO layers and nanopillars growth on sapphire, SiC, ZnO epitaxial layers, and even plastic and glass is developed. Low-cost and low temperature ACG fabrication of ZnO nanopillars on silicon, plastic, glass is also realized. The nanopillars arrays were patterned and also selective growth approach is demonstrated showing further step forward to ZnO-nanopillar-based device fabrication. Nanopillar fabrication technique is combined with MBE technology: MBE ZnMgO-ZnO quantum well structures were embedded into ZnO nanopillars presenting significant progress towards nano-LEDs realization.

11:45 Oral

Electrical characterization of ZnO nanorods

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There have been intensive studies in the field of nanosized materials in order to improve the performance of electronic devices. ZnO seems to be the ideal candidate for possible applications in micro-, opto- and magnetoelectronics. It is not only a wide band gap (3.37 eV) semiconductor with a large exciton binding energy of 60 meV, it furthermore has the property of self-organised growth resulting for instance in well aligned nanorods building regular block-structures.

Most of the research on ZnO nanostructures was focused on the deposition techniques using processes like metal-organic vapor phase epitaxy (MOVPE), vapor phase transport (VPT), molecular beam epitaxy (MBE), pulsed laser deposition (PLD) or aqueous chemical growth (ACG). However, there have been only few reports on the electrical characteristics of single ZnO nanostructures.

Here we report on our findings concerning the electrical property measurements of ZnO nanorods synthesized by VPT. The rods are single-crystalline and of high quality as TEM pictures reveal. They grow in vertically aligned pillar arrays and reach widths of 70 nm to 1 µm and up to 15 µm in length. Current-voltage characteristics were measured for single as well as for bundles of nanorods. E-beam lithography has been used to make electrical contact to single nanorods in various geometries. The specific resistance of a nanorod was determined to be about 8×10^{-2} Ωcm. Assuming an electron mobility of 100 $cm^2V^{-1}s^{-1}$ this leads to a carrier concentration of 7.8×10^{17} cm^{-3}. Additionally, we compare the results with the ones obtained for ZnO nanorods grown by ACG.

12:05 Oral

Lattice Dynamics of Wurtzite ZnO: an Experimental and Ab-initio Study

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Zinc oxide is a wurtzite semiconductor with a band gap of 3.4 eV that has stimulated renewed interest in its properties for optoelectronic applications in the ultraviolet energy range. To this aim, knowledge of the thermal properties and heat transport of ZnO is essential. The latter are mostly dominated by the lattice dynamics, which can be investigated by measurements and theoretical calculations of the phonon dispersion relations along high symmetry directions in the Brillouin zone. Unfortunately, until now little information is available in this respect, obtained mainly from Raman and infrared spectroscopy. The phonon dispersion of ZnO has 12 branches from which only some quasi-acoustic phonons have been reported in the literature [1].

We present here the phonon dispersion relations of ZnO obtained by inelastic neutron scattering measurements. The results are compared with ab-initio lattice dynamical calculations that have shown an excellent agreement with empirical data obtained under pressure and temperature. They also account for the observed dependence on isotopic mass [2]. The calculations reproduce very well the experimental dispersion along all investigated symmetry directions. The main deviations occur for the lower E_2 modes, which require an increased accuracy in the calculated interatomic forces, and for the longitudinal optic modes.

[1] A. W. Hewat, Solid State Commun. 8, 187 (1970); K. Thoma et al., Solid State Commun. 15, 1111 (1974).

[2] J. Serrano et al., Phys. Rev. Lett. 90, 055510 (2003); Phys. Status Solidi B 235, 260 (2003); Phys. Rev. B 69, 094306 (2004).

Lunch break

Thursday afternoon, 7 September, 12:30

Session Chairman

Thursday afternoon, 7 September, 14:00
Chair: Dang Le Si

14:00 Oral

Optical properties of p-type ZnO:(N, As, Sb)

Ewa Przezdziecka[1], Eliana Kaminska[2], Iwona Pasternak[2], Elzbieta Dynowska[1], Witold Daniel Dobrowolski[1], Rafał Jakieła[1], Adam Barcz[1,2], Lukasz Klopotowski[1], Krzysztof P. Korona[4], Jacek Kossut[3]

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Zinc oxide, due to its specific is an important semiconductor material which already has many diverse applications. Undoped ZnO is usually n-type which is associated with presence of native point defects and/or residual hydrogen impurities. Fabrication of ZnO with p-type conductivity represented considerable challenge for many years. Recently, however, several groups achieved p-type ZnO by doping with group V elements. It has been shown that nitrogen has a simple substitutional character, while As and Sb form more complicated acceptor complexes [1].
Here we report on fabrication of p-ZnO layers doped with N and/or As and Sb acceptors by thermal oxidation of Zn based compounds (i. e., ZnTe, Zn3N2, Zn3Sb2). Optical study showed meaningful differences between samples doped with different acceptors.
Photoluminescence spectrum in the excitonic region shows three peaks in our samples: the peak located at 3.367 eV which we attribute to ionized-donor bound exciton, the peak at 3.361eV - to neutral donor bound exciton, and the peak located at 3.355 eV - to exciton bound to a neutral acceptor. We also observe additional peaks in ~3.3 eV region in some samples. The PL peak located at 3.311 eV is observed in samples doped only with N. It is worth to notice that the emission at this energy is absent in samples doped only with As. On the other hand, in ZnO:As and ZnO:Sb samples we observe emissions at 3.322 eV and 3.316eV, respectively. Therefore, we conclude that the 3.322 eV, 3.316 and 3.311 eV lines derive from excitons bound to the As, Sb and N acceptors, respectively. In nitrogen doped sample we observe also a peak, whose origin is not clear yet, located at 3.329 eV. Part of the research was supported by a Subsidy 8/2003 and grant 1P03B08430.

[1] S. Limpijumnong, S. B. Zhang, Su-Huai Wei, C. H. Park, Phys. Rev. Lett. 92, 155504 (2004).

14:20 Oral

Fabrication of ZnO Nanorod-based Single Quantum Well Structures

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In the past decade, much effort has been made in fabricating semiconductor nanostructures, including nanorods on different material bases, including ZnO. Due to its wide band gap (3.37 eV) alongside many fascinating properties of this material, ZnO is a potential candidate for light-emitting devices in the blue and ultra-violet (UV) region of the electromagnetic spectrum. While much success has been reported on the fabrication of ZnO nanostructures, the fabrication of device structures from these nanostructures has rather proven to be challenging. With the persistent difficulties in incorporating acceptors in ZnO, quantum well structures will provide a dependable alternative for achieving lasing on ZnO basis. With their anticipated high crystalline quality, densely packed nanostructures would provide a possibility for high device density.

We have fabricated ZnO anorod-based single quantum well heterostructures in a two-step process. Nanorods were first grown at 800° C and 20 mbar reactor pressure using a vapour transport system [1]. Then ZnO/Zn $Mg_{1-x}O$ heterostructures were grown on the nanorods using a modified Varian Gen II molecular beam epitaxy system. The nanorods grow preferentially along the *c*-axis of ZnO and exhibit, besides peculiar optical properties, a very narrow rocking curve full width at half maximum of 0.23°. Quantum confinement was clearly observed within the ZnO *wells* of different widths (1-5 nm). These quantum wells show relatively narrow photoluminescence peaks of full width at half maximum less than 40 meV.

Details of the fabrication and characterisation processes shall be presented.

Rerence:

[1] A. C. Mofor, A. S. Bakin, A. Elshaer. D. Fuhrmann, F. Bertram, A. Hangleiter, J. Christen and A. Waag: phys. stat. sol. (c) **3**, No. 4, p. 1046 (2006)

14:40 Oral

Controlled Precipitation of Monodisperse ZnO Nanocrystals via Acid-Catalyzed Estrification of Zinc Acetate

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The synthesis of ZnO nanocrystals has been the subject of active re-

search due to existing and further potential applications. Although numerous chemical strategies have been reported, the preparation of monodisperse crystals in large volumes with homogeneous properties still remains a challenge. In this work, we describe a chemical precipitation process to obtain ZnO nanoparticles of high crystal perfection through acid-catalyzed esterification of zinc acetate dihydrate with 1-pentanol. Particle formation was promoted by an esterification catalyst, p-toluene sulfonic acid monohydrate (p-TSA), which yields nearly monodisperse ZnO nanoparticles at short growth times. Crystalline powders were prepared on the gram scale and characterized by X-ray diffractometry (XRD), scanning electron microscopy, high-resolution transmission electron microscopy (HRTEM), and photoluminesence (PL) spectroscopy. The crystal size can be tuned in the diameter range of 20-80 nm by changing the p-TSA concentration and the reaction time. The presence of this acid catalyst does not cause structural differences to be seen by HRTEM and XRD. All room temperature PL spectra exhibit a strong and sharp UV emission band at ~385 nm and a weak and very broad green-yellow visible emission centered at ~550-560 nm. For the samples precipitated in presence of p-TSA, the UV emission is enhanced by a factor of 4, which can be correlated with the improvement of crystal perfection.

Coffee break
Thursday afternoon, 7 September, 15:30

Joint session Symposia E, F & K, Room 219
Thursday afternoon, 7 September, 15:50

15:50 Invited oral

Ferromagnetic oxide semiconductors: using off-stoichiometry to tune low-dimension magnetism and consequently the iron valency

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Today's research in diluted magnetic semiconductors (DMS) is concentrated on large band gap semi-conductors with the scope to obtain carrier mediated high temperature ferromagnetism by substitution of appropriate transition metal ions into the host matrix. This study investigates the possibility of alternative approach, such as the creation a of DMS starting from a ferromagnetic oxide. The control of stoichiometry during growth of thin oxide film will be used as a tool to modify the physical properties of the oxide such as magnetism, valence state and consequently transport properties. In particular, ferrimagnetic iron oxides like garnets or illmenites show already high intrinsic Curie temperatures (T_C > 400 K) and are suitable candidates for this case study. Among the different systems, the Yttrium Iron Garnet (YIG) will be presented in detail. Pulsed laser deposition of Yttrium Iron Garnet thin films allows to explore a new part of its phase diagram, e.g. controlled stabilization of iron and yttrium vacancies within the oxygen sub-lattice, and to tune magnetism by off-stoichiometry. Magnetization and Curie temperatures are measured by polar magnetic circular dichroism (MCD). A significant increase of the Curie temperature (+10%) indicates changes of the super-exchange coupling through the variation of the Fe-O-Fe distance. Simultaneously, an important increase of the magnetization (up to 120%) is observed. The temperature dependent MCD measurements demonstrate that the increase of magnetization is due to a preferential occupation of the iron vacancies on the octahedral sites. Iron valence presumably changes from Fe^{3+} to Fe^{4+} accompanied by the presence of iron and yttrium vacancies. Its consequences to the carrier doping in application to spintronics will be discussed.

16:35 Invited oral

Origin of ferromagnetism and phase separations in diluted magnetic semiconductors

Tomasz Dietl

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A considerable effort has been devoted to understand the origin of ferromagnetism, often persisting up to above room temperature, in a number of semiconductors doped with transition metals. In the talk, I will argue that ferromagnetic DMS can be divided into three categories. The first consists of (Ga,Mn)As and related compounds. Here, theory built on Zener's model of carrier-mediated ferromagnetism and the Kohn-Luttinger *kp* theory of semiconductors describes thermodynamic, micromagnetic, optical, and transport properties. To the second group belong compounds, in which the proximity of the localisation boundary and/or a competition between long-range ferromagnetic and short-range antiferromagnetic interactions leads to an electronic nanoscale phase separation that results in characteristics similar to colossal magnetoresistance oxides. Finally, in a number of compounds a chemical nanoscale phase separation is observed, reminiscent of spinodal decomposition. Mechanisms accounting for this effect in particular materials will be discussed.

The work is supported by NANOSPIN E.C. project (FP6-2002-IST-015728), by Humboldt Foundation, and carried out in collaboration with M. Sawicki, K. Osuch, H. Przybylińska, M. Kiecana, and A. Lipińska in Warsaw, as well as with groups of H. Ohno in Sendai, S. Kuroda in Tsukuba, K. Trohidou in Athens, J. Cibert in Grenoble, J. Jaroszyński in Tallahassee, and A. Bonanni in Linz.

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster F-1

First-principles calculations of the optical band-gap properties of $Mg_{1-x}Zn_xO$ alloys

Nadia Abbouni[2], Lilia Beldi[2], Bachir Bouhafs[1,2], Abderrahmane Kadri[4], Pierre Ruterana[3]

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$Mg_{1-x}Zn_xO$ alloys became of great interest recently, since in principle a wurtzite-rocksalt combination spans a wider range of optical band gaps than either a rocksalt-rocksalt or a wurtzite-wurtzite alloy. For example, alloys of ZnO (E_g=3.4 eV) with MgO (E_g=7.7 eV) could span a range from blue to deep UV, which is of interest for optical laser and light-emitting diode applications.

We report here on first-principles total-energy calculations of the structural and electronic properties of ZnO, MgO and ordered $Mg_{1-x}Zn_xO$ alloys in various crystal structures (CuAu-I and Luzonite) using the hybrid full-potential augmented plane-wave plus local orbitals (APW+*lo*) method. We have used the local-density approximation (LDA) for the exchange and correlation potential. The ground state properties, equilibrium lattice constants, bulk moduli, charge densities, band structures, and densities of states are determined.

17:20 Poster F-2

Theoretical studies of $ZnS_{1-x}O_x$ alloy band structures

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We report the structural and electronic properties of ordered $ZnS_{1-x}O_x$ alloys calculated in various structures (CuAu-I, Cu_3Au, Luzonite and Famatinite) using a first-principles total-energy calculations based on the hybrid full-potential augmented plane-wave plus local orbitals (APW+*lo*) method. We have used the local-density approximation (LDA) for the exchange and correlation potential. The calculated gaps range from 0.97 for O-rich to 1.55 eV for S-rich $ZnS_{1-x}O_x$ systems. We predict that ordering affects significantly the electronic band structures. The alloys in the CuAu-I structure are found to have direct smaller band gap of 0.49 eV. The origin of the reduction in the band gaps is discussed, as well as the effects of increasing Oxygen in $ZnS_{1-x}O_x$ alloys. These alloys whose constituents are rather size mismatched, a strong reduction of the band gap is found. Beside the presence of lattice mismatch, the Zn 3*d* electrons effects are found to play an important role in the determination of the electronic structure. Their effect on the gap, and also on the k dispersion in the valence bands is significant. We have identified the microscopic origin of the main features in the band structures and found that not only ordering but also Zn 3*d* electrons effects are responsible of the reduction of the band gap in these alloy systems.

17:20 Poster F-3

Luminescence of CdMnTe Crystals in Magnetic Field

Romuald BRAZIS[1], Laima Barauskaite[1], Marek Godlewski[2,3], Vitaly Ivanov[2]

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The work is aimed at feasibility study of magnetically tunable light sources based on $Cd_{1-x}Mn_xTe$ crystals. Strong luminescence from the cleaved surfaces of crystals with the Mn mole fraction of x=0.2 and 0.3 has been observed experimentally under cw laser excitation at liquid helium temperatures, and the radiation spectrum evolution in magnetic fields up to 7 Tesla has been analyzed. The evolution has been found essentially different in the normal (Faraday) and transverse (Voigt) geometry revealing implications related to the non-zero incidence and emission angle. The implementation is discussed in term of self-organized magnetic nano-structures and exciton-polariton condensation at the cleaved crystal surface.

17:20 Poster F-4

Phonon- and Electron-Related Far-Infrared Absorption in CdTe and ZnTe Crystals

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Constants characterising the efficiency of far-infrared (FIR) photon coupling to anharmonic lattice vibrations are elucidated from existing experimental data in CdTe and ZnTe crystals. Two-phonon difference processes are concluded to play leading role in the FIR absorption in the range of 1-4 THz. Absorption modulation in electric field acting on the conduction-band electrons is considered using original Monte Carlo modeling data. If the lattice is cooled down well below the liquid nitrogen temperature the absorption is predicted to change its sign (i.e., convert to stimulated emission) under condition of inverted phonon band filling produced by electrons accelerated in electric field.

The work is partially supported by the Lithuanian State Science and Studies Foundation project V-06085

17:20 Poster F-5

Excitons in ZnO/$Zn_{1-x}Mn_xO$ quantum wells

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wurtzite structure ZnO has particularly large coefficient of piezoelectric and spontaneous polarization. ZnO-based quantum structures grown in 0001 direction, are expected to reveal effects caused by the presence of built-in electric field, leading a redshift of exciton emission, reduction of exciton binding energy and oscillation strength[1].We studied electron hole Coulomb interaction in $ZnO/Zn_{0.82}Mn_{0.18}O$ QWs. Exciton energies and probabilities of their radiative decay are calculated for from 2-7 nm well width. Built-in electric field is included by direct diagonalization method.Built-in electric field is assumed to be 0.9MV/cm. For 2-3nm well width electron hole interaction is very high and binding energy is increased as much as 4-5 times with respect to the bulk ZnO; Electric field effect is not significant and electron hole wave functions separation is not pronounced probability of radiative decay is close to 1.Starting from 4nm well width ground state exciton energy and decay probability decrease sharply, while binding energy of excitons of excited states and their decay probability increase, because wave functions of electrons and holes of higher levels are shifted to the area of higher potential toward the centre of QW, increasing their overlap. That menace that radiation of high energetic excitons in $ZnO/Zn_{1-x}Mn_xO$ are expected to be intensive.

1. C. Morhain, T. Bretagnon,P. Lefebvre, et al, Phys. Rev B **72**, 241305R (2005)

17:20 Poster F-6

Peculiarities of the microhardness in $Cd_{1-x}Mn_xTe$

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The $Cd_{1-x}Mn_xTe$ ($0 \le x \le 0.55$) single crystals were grown by the vertical Bridgmen method. The Vickers microhardness measurements on the grown crystals showed a solid solution hardening effect with two hardness maximums at $x \approx 0.15$ and $x \approx 0.40$. In this paper we report on investigations concerning possible phase separation, ordering effect and structural distortion. To understand this complicated variation of the microhardness, we carried out electronic band structure calculations (under both normal and strained conditions) using supercell psedopotential method, with a set of particular atomic arrangements. We demonstrate a close correlation between the calculated shear modulus and experimental microhardness. Summarizing the calculations we conclude that the first maximum is connected with distortion phenomena. The second hardness maximum we explain by a non-random distribution of the cations and some nanoclastering of cations which could play the role of "nano" phase separation.

17:20 Poster F-7

The Effect of Treatment on CdTe, $Cd_{1-x}Mn_xTe$, $Cd_{1-x}Zn_xTe$ Surface Stoichiometry

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It is known that stoihiometry of the CdTe, $Cd_{1-x}Mn_xTe$, $Cd_{1-x}Zn_xTe$ surface, as a rule is broken, moreover the relation between components depends on the treatment method, and also on etchant's composition. In the present paper using thermodynamics calculations, potentiometric analysis and Auger-spectroscopy measurements we studied the surface composition and the formation mechanism of after treatment oxides films in such solutions. The treatment with bromine methanol solution gives an enrichment of surface layer by Te and TeO_2. The relation of reduced intensities of the Cd/Te component was of 0.5 for CdTe; 0.4 for $Cd_{1-x}Mn_xTe$ and 0.3 for $Cd_{1-x}Zn_xTe$. The most perfect surface is obtained with the use of chemico-mechanical polishing with modified alkaline colloid solution SiO_2 containing H_2O_2. The presence of fine dispersed modified colloid silica (2÷20 nm) in etchant mixture, which acts as an abrasive and forms complexes, affects favorably the surface quality. The speed of chemico-mechanical polishing depends on alkali and H_2O_2 concentration and is within the limits of 0.1 ÷ 0.7 mm/min. Such treatment gives the surface with the minimum damaged layer of 13÷26 nm. The relation of reduced intensities of the Cd/Te component for studied materials surfaces was of 0.8-1.1.

17:20 Poster F-8

Phase transitions in ZnTe doped with Mg and O

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Self-assembling of isoelectronic impurities in GaAs doped with Al and N, AlN doped with Ga and As, Ge doped with C and Sn and ZnS doped with Mg and O was predicted and described in [1]. The equality conditions of the free energies of the random alloys and alloys with impurity tetrahedral clusters were represented. Such self-assembled cluster ordered state is a new phase state of the alloys. However, a phase transition between the cluster ordered and homogeneous alloys is not described yet. The cluster order - disorder phase transition in ZnTe doped with Mg and O is described.

The self-assembled cluster ordered state is characterized by the cluster order parameter that is a part of oxygen impurity atoms placed in 1O4Mg clusters. The ultra dilute limit for the oxygen impurity from $y = 1' 10^{-8}$ to $1' 10^{-3}$ was chosen. The enthalpy is a continuous function of temperature and heat capacity undergoes the fi-

nite discontinuity at an occurrence of 1O4Mg clusters. Such temperature dependences of the enthalpy and heat capacity correspond to the second-order phase transition. The critical temperature of Mg(x)Zn(1-x)O(y)Te(1-y) with x = 1.52′ 10^{-4} and y = 1′ 10^{-5} is equal to 786 K. The free energies of this alloy at the completely cluster ordered and random states are equal to each other at temperature of 773 K. At this temperature the thermodynamically stable state corresponds to the alloy in which one-half of oxygen impurities is placed in 1O4Mg clusters. At the completely cluster ordered state this alloy becomes stable at temperature of 747 K. The occurrence of the complete cluster order at this temperature is also a result of the second-order phase transition.

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17:20 Poster F-9

Structure and optical properties of screen-printed CdS-CdSe films

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CdS_xSe_{1-x} films at present time are widely applied in production of photoelectrical devices for visible and near-IR regions. The vacuum sputtering commonly used for fabrication of such films are relatively expensive and therefore are inexpedient for preparation of large area photosensitive layers. The screen-printing method can be viewed as an alternative to sputtering and is capable to find a wide application in technology of large area solar cells, photoresistors, etc. In this method a paste containing material to be deposited is pressed through the screen onto the substrate and then subjected to heat treatment to achieve the required photoresponce.

In this work the structural and optical properties of CdS_xSe_{1-x} films produced by screen-printing method were investigated. The initial pastes were produced on the base of fine-dispersed CdS, CdSe and dopant-containing powders, $CdCl_2$ flux powder and binder. The as-screen-printed films were dried and then annealed at 500 - 600 °C. The study of XRD, surface morphology, composition and its in-area and in-depth distribution, and also distribution of intensity of different photoluminescence bands by the film area testify that CdS-CdSe films can be transformed into solid solutions CdS_xSe_{1-x} after annealing. It was shown that the film properties were strongly sensitive to the CdS/CdSe powder ration, type of binder and heat treatment regimes (temperature and duration).

17:20 Poster F-10

Growth and properties of $Zn_{1-x-y}Be_xMn_ySe$ Crystals

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$Zn_{1-x-y}Be_xMn_ySe$ is promising for spintronics as a spin filter layer with possible applications in memory technology. This work deals with photoluminescence (PL), Raman and EPR studies of bulk $Zn_{1-x-y}Be_xMn_ySe$ grown by Bridgman method. The contents of Be and Mn were varied from 0.0 to 0.2. PL spectra were measured in the temperature range from 30K to RT. EPR studies were made with EPR spectrometer working at X-band. PL spectra at low temperatures of $Zn_{1-x-y}Be_xMn_ySe$ with Be content 5% and Mn content not higher than 20% consist of exciton line, shallow donor-acceptor emission and yellow luminescence band positioned at the energy about 2.05 eV associated with Mn ions. For larger concentration of beryllium only yellow emission is observed at temperatures from 35K to room temperature. The photoluminescence-excitation spectra, when the emission is detected at 2.05 eV, consist of four clearly resolved components interpreted as due to transitions from the ground $^6A_1(^6S)$ state of Mn to different excited states associated with crystal field splitting. From PL measurements the slight increasing of the excitonic energy gap with increasing of Mn content was observed in the investigated range of composition, in contrast to ZnMnSe.

The room temperature Raman spectra consist of ZnSe-like, MnSe-like and BeSe-like transverse and longitudinal modes. Because of the phonon energies in ZnSe and MnSe do not differ so much, the respective Raman lines are overlapped. EPR measurements show that Mn content in $Zn_{1-x-y}Be_xMn_ySe$ samples influences the amplitude of the EPR signal, the lineshape and the line position, directly related to the g-factor value. Applying the DPPH as a standard, the Mn content per sample weight unit was estimated.

17:20 Poster F-11

High-temperature electrical properties of CdTe<Pb> crystals under Te saturation

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CdTe is a very promising material for photorefractive devices, such

as optical memory, holographic real time cameras, optical communication. Usually CdTe, doped by Ge or V, are used for these purposes. CdTe single crystals, grown in Chernivtsi University, ensure the largest gain factors known till now in all semiconductor photorefractives without applied electric field. Similar properties - conductivity of p-type and high resistivity - reveal CdTe<Pb> crystals and they can find an application in non-linear optics devices. But such material was investigated at low temperatures only. It is important to study the CdTe properties at high temperatures, where the point defect structure of semiconductor is formed.

The single crystals were grown by Bridgman technique. Pb concentration in the melt was 5×10^{18}-5×10^{19} at/cm^3. Hall effect measurements at 600-1100 K under well defined Te vapor pressure were performed. At 300 K all samples were of p-type conductivity with 40-70 cm^2/Vs hole mobility. The dominant charge carriers were controlled by the E_V+0.43 eV level. Under Cd saturation the samples gained n-type conductivity with lower electron concentration comparing to undoped CdTe. Measurements under Te vapour pressure demonstrated hole conductivity up to 600 K. After 900 K the conductivity became electronic. During heating-cooling cycles the p-n transition temperature shifts to higher values. The obtained results are explained using Kröger's quasichemical defect formation theory.

17:20 Poster F-12

Sb-treatment effect of GaAs substrate on CdTe growth by MOVPE

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CdTe is an attractive matrial, because of its wide application field, for example, the X-ray detector, the solar cell, the diluted magnetic semiconductor and so on. It is necessary to obtain high-quality materials in order to realize such devices. We reported the Sb-treatment of GaAs substrate is effective to obtaining high-quality $Cd_{1-X}Mn_XTe$ (CMT) film on (100)GaAs by metal-organic vapor phase epitaxy (MOVPE).[1] We will disscuss the phenomena at the interface of the GaAs substrate and the CdTe layer concerning to the Sb-treatment and the relation between the Sb-treatment conditions and the quality of CdTe grown on it.

A CdTe film was grown on a (100)GaAs substrate by MOVPE method under atmospheric pressure. Dimethyl cadmium (DMCd), diethyl telluride (DETe) and triethyl antimony (TESb) were used as the source gases for cadmium, tellurium,and antimony, respectively. H_2 was used as the carrier gas. The substrate was heated on the graphite susceptor induced by RF irradiation. Prior to the growth of CdTe, the GaAs substrate was heated in H_2 introducing TESb for 15 min(Sb-treatment). The TESb supply rate was varied from 0 to 3 μmol/min and the temperature of the GaAs substrate was varied from 500 to 650^{o}C, to elucidate the effect of TESb treatment. After the TESb treatment, the CdTe film was grown for 2 hours. The typical growth conditions of CdTe are presented in the previous paper[1].

The CdTe film grown on (100)GaAs substrate treated with TESb of 3μmol/min was high-quality, featureless and free from cracks. The X-ray diffraction shown in Fig.1 suggests that the formation of Sb layer and/or GaSb layer at the interface is important to obtain featureless CdTe film on GaAs substrate.

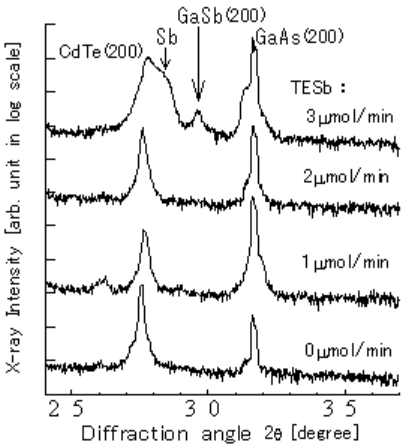

Fig.1. X-ray diffraction of CdTe
CdTe was grown at 520°C on Sb-treated GaAs(100) substrate.
GaAs substrate was treated in use of TESb at 650°C for 15minutes.

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17:20 Poster F-15

Electrical characterization of He^+ irradiated n-ZnO

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Due to its direct wide bandgap of 3.37eV, Zinc oxide (ZnO) has become the focus of many studies. Devices such as detectors, lasers and diodes operating in the ultra-violet (UV) and blue regions of the spectrum have been reported. It has previously been reported that the carrier removal rate in ZnO by MeV protons is almost two orders of magnitude lower than that in GaN. To investigate if this is due to a material property of the ZnO, or perhaps a case of radiation enhanced annealing, He^+ irradiation were performed at room temperature as well as at low temperatures. ZnO Schottky barrier diodes (SBD's) were fabricated with 20/80/40/80 nm Ti/Al/Pt/Au ohmic contacts on the O face and circular 0.5 mm in diameter 500 nm thick Ru Schottky contacts in the Zn face. The carrier concentration of the ZnO prior to irradiation was approximately 5×10^{16} cm^{-3}. These diodes were irradiated with 1.8 MeV He^+ with fluences ranging from 1×10^{12} cm^{-2} to 5×10^{13} cm^{-2}. Deep level transient spectroscopy (DLTS) was used to study the defects introduced. These measurements revealed that this implantation introduced a defect with an energy level at 0.54 eV below the conduction band. The introduction rate of this defect for 1.8 MeV He ions was calculated to be 220 cm^{-1}. This appears to be the same defect that is introduced during proton implantation of ZnO.

17:20 Poster F-16

Evaluation of deep-impurity governed photoelectrical properties in differently doped CdTe

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Fast response and high sensitivity in the near-IR region makes II-VI compounds the promising semiconductor materials for telecommunication, adaptive optics, and real time holographic devices. Defect engineering efforts are ongoing for selection of an appropriate deep midgap dopant, optimization of its density, electrical activity, and to achieve technological reproducibility.

We investigated nonequilibrium carrier generation, transport, and recombination processes in differently doped CdTe crystals. Doping by Ge, Pb, Sb, Se, Si, or Sn was reached by Bridgman-growth, whereas vanadium doped CdTe ingots were grown by the vertical Bridgman-Stockbarger method. The samples were excited by 23 ps laser pulses at 1064 nm wavelength to ensure nonequilibrium carriers generation from deep levels. Time-resolved four-wave mixing technique enabled us to study peculiarities of carrier generation, trapping, and transport in light-created space charge (SC) field, and to determine the photoelectric parameters such as photo-generated carrier lifetime, diffusion coefficient. We measured the SC-field dependent diffusion coefficient values at various excitation energies and determined the sign of the photogenerated excess carriers. The experiments revealed that in samples doped with V, Sb, Si, or Ge, the majority carriers are electrons, while in Pb, Se, Sb, Ge (cut from the different place of an ingot), or Sn-doped crystals - the holes. Very strong feed-back effect of the internal electric SC field to subnanosecond free carrier dynamics was observed in the crystals doped with Ge, Pb, Sb, or Sn. The crystals doped with Ge, Sb, or Sn revealed longer than 40 ns carrier lifetimes together with long living SC field component, what makes them very promising photorefractive media for real time holographic devices.

17:20 Poster F-17

Laser control of magnetization of bulk (Zn,Mn)Te

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There is an intense search for techniques of magnetization control in MBE-grown semimagnetic semiconductors ((Zn,Mn)Se, (Cd,Mn)Te) by means other than magnetic field. We report the way for the control of the magnetization of bulk (Zn,Mn)Te. We demonstrate that by tuning the intensity of laser illumination at a constant magnetic field, the magnetization of the (Zn,Mn)Te crystals could be decreased by 60%.

(Zn,Mn)Te crystals were grown by the high pressure Bridgman method. The Zeeman effect was used to find out the sample magnetization (M). In zero magnetic field the increase of the excitation intensity (P) from 10 to 200 mW practically does not change the peak energy of the exciton, donor-acceptor pair (DAP), and intra-Mn PL emission, but strongly increases the intensity of these bands. At P > 200 mW, the exciton band shows a red shift which reaches 5 meV at P=1.2 W. These findings indicate that there is a direct energy transfer from the photocarriers to the band states and Mn ions subsystem. We have found that at a given field a relatively small increase of the excitation intensity causes a large blue-shift of the exciton line indicating a strong suppression of the Zeeman splitting, i.e. the magnetization decreases. For example, at B=6 T the ratio M(600mW)/M(10mW)=0.4. Comparing the free exciton shifts from the PL and reflectance spectra as a function of magnetic field, we found that in the (Zn,Mn)Te crystals the Mn ion system is being heated under even smallest laser excitation density. The Mn-spin temperature was deduced from the Zeeman splitting, and found to be 5.06 K at P=10 mW while the lattice temperature was kept at 1.7 K. Moreover, for a given laser excitation the heating of Mn ions system causes a strong increase in the intra-Mn and the integrated DAP PL intensities with increasing magnetic field.

17:20 Poster F-18

Low temperature growth of ZnO thin film by metalorganic chemical vapor deposition

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Zinc oxide is a II-VI compound semiconductors with a wide direct bandgap of 3.37 eV, a large exciton binding energy of 60 meV, and a hexagonal wurtzite structure. Recently, the interest of short-wavelength optoelectronics and transparent conductive oxide has stirred up the research on ZnO thin films. ZnO thin films also have display applications on polymer and glass substrates due to the high transmittance, low resistivity, and relatively low growth temperature.

In this study the ZnO films of ~ 0.5 μm thickness were grown at relatively low growth-temperature (< 300 °C) by metalorganic chemical vapor deposition (MOCVD), compared to the previous MOCVD results. The used substrates are Si (111) and sapphire substrates. While the growth temperature below 300 °C is required to utilize glass and polyimide substrates, ZnO films grown at low temperature showed the deteriorated crystallinity. To promote the ZnO film growth at low temperature, the additional Ar gas was injected through by-pass line except carrier. The XRD analysis revealed that the c-axis orientation of ZnO films increases up to 240 °C, regard-

less of substrate types. The ZnO film grown at 240 °C showed a strong (002) diffraction peak, a near-bandedge emission at 3.29 eV, and a reduced yellow level emission. This indicates that the ZnO films at low temperature show fairly good characteristics.

17:20 Poster F-19

Study on Defect States Using Deep Level Transient Spectroscopy of ZnO Grown by Pulsed Laser Deposition

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Wide-bandgap semiconductors such as GaN, ZnO and 6H-SiC have been widely investigated for their applications to ultraviolet (UV) light emitters and high-power and high-temperature electronics due to their wide-bandgap nature and high breakdown electric fields. Since the first commercial device was realized using GaN, many GaN-based devices have been developed. Recently, however an attention to ZnO is growing rapidly because of its advantages over GaN including availability in bulk and much higher exciton binding energy (60 meV) compared with GaN (24 meV) which guarantees a stability of exictonic emission mechanisms above room temperature.

In this study, the defects in ZnO layers grown by pulsed laser deposition (PLD) were investigated by using capacitance-voltage (C-V) measurements and deep level transient spectroscopy (DLTS) which is the most widely used technique for characterization of defects in semiconductors. The ZnO was grown on a sapphire wafer at room temperature. For the DLTS measurement, Pt was deposited on the surface of the ZnO layer for a Schottky contact and Ti/Au was deposited over the large area of the sample to form an Ohmic contact. On the prepared sample the DLTS measurements were performed at temperature range of 20 K~700 K.

17:20 Poster F-20

Modification of the photoluminescence characteristics of CdZnTe/ZnTe QWs by CdTe monolayer film insertion

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Heterostructures containing submonolayers of narrow band gap semiconductor embedded in wide band gap one are attractive objects. Light-emitting diodes and lasers with fraction-monolayer active region based on II-VI compounds demonstrated better characteristics in comparison with corresponding quantum well (QW) devices. The influence of single CdTe monolayer film insertion on the luminescence and structural characteristics of CdZnTe/ZnTe QW was investigated by the photoluminescence (PL) and high resolution X-ray diffraction (HRXRD) methods. The structures studied were grown by MBE on 3^0 off - (100) GaAs substrate and contained 8nm thick $Cd_{0.4}Zn_{0.6}Te$ QW with or without CdTe monolayer film embedded in the middle of QW. Low temperature PL investigations showed that insertion of CdTe layer resulted in two times narrowing of the QW PLband and one order of value increase of the QW PL intensity. Temperature dependencies of the QW PLspectra and time-resolved PL investigations indicated that these changes are caused by the increase of shallow localized state density and the decrease of deep one. Smearing of interference fringes in the HRXRD profiles proved the absence of flat interface between CdTe and CdZnTe layers. The latter testifies to that shallow localized states in CdZnTe QW are due to the CdTe insertion.

17:20 Poster F-21

Magnetic properties of Fe doped SiC crystals

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Recently, ferromagnetic semiconductors have attracted much interest due to potential applications in spin-based information-processing technologies. SiC is a good candidate for spintronic investigations because of its wide bandgap (3.0 eV for 6H polytype) and due to potential technological applications. Recently, magnetic properties of Fe- implanted SiC were investigated [1]. In the present studies SiC crystals were grown by Physical Vapor Transport method. We used Fe enriched source material to dope crystals. The growth temperature was equal to 2150^0C. DC magnetization measurements performed at low temperatures and low magnetic fields revealed ferromagnetic type of magnetic ordering. In the low magnetic field magnetization data the hysteresis loops are present. The structural investigation using powder XRD were performed. The presence of nonmagnetic FeSi precipitates ([2]) was observed. We will present the systematic magnetic measurements including AC susceptibility and DC magnetization up to 9T in the range of low and high temperatures as well as structural studies using powder XRD. The chemical analysis using SIMS will be performed. The origin of ferromagnetic behaviour will be discussed.

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17:20 Poster F-22

Photoinduced changes of photoconductivity and exciton luminescence in ZnO crystals

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Zinc oxide has many device applications in electronics, so the study of processes responsible for its parameter instability is an actual task. In this work, the influence of light irradiation on ZnO characteristics was studied. Nominally undoped ZnO single crystals with ρ = 10-30 Ω·cm were investigated. Photoconductivity and exciton luminescence (ExL) spectra were measured at 300 and 77K before and after sample irradiation with focused light of xenon lamp at 300-700K. After the irradiation, considerable reduction of photosensitivity and the change of ExL spectrum shape were found to occur. Simultaneously, the decrease of dark conductivity was observed. The initial characteristics were restored with time in dark. Observed effects were shown to result from rearrangement of highly conductive layer on ZnO crystal surface. The origin of this layer and the mechanism of its rearrangement were recently published [1].

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17:20 Poster F-23

Photoluminescence of ZnO films studied by femptosecond sapphire:Ti laser

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ZnO films were deposited by PEMOCVD, magnetron sputtering (MS) and reactive thermal evaporation on Al_2O_3 (0001), SiO_2/Si (100) and SiN_x/Si. Photoluminescence (PL) of as-grown and annealed ZnO films were studied at room temperature in range 350-800 nm by femptosecond sapphire:Ti laser (37 mW, 170 fs, 76 MHz). Annealing of ZnO films was carried out in air at 600-1000^oC during 2-9 h.

As-grown ZnO films deposited by PEMOCVD on SiN_x/Si were revealed the most intense near band edge (NBE) photoluminesense peaked at 3.27 eV. ZnO films deposited on SiO_2/Si (100) and Al_2O_3 (0001) were characterized both NBE and broad deep-level luminescence. Annealing of samples results in significant decreasing of NBE PL. At the same time the deep-level luminescence at 2.39 eV sufficiently increases. It was revealed the influence of ZnO texture on PL intensity of films deposited by MS. The most intense PL was observed in samples with c-axis situated in plane of Al_2O_3 substrate. Annealing the samples also results in suppression of NBE PL.

Obtained results can be explained by oxygen diffusion through film-substrate interface during processes of growth and annealing, increasing of stresses in films at annealing as a result of large misfit between ZnO and Al_2O_3 substrates in periods of crystal lattice and thermal expansion coefficients.

17:20 Poster F-24

In-doped transparent and conducting cubic Magnesium Zinc Oxide thin films grown by Pulse Laser Deposition

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In-doped n-type semi-conducting Magnesium Zinc Oxide (MZO) thin films have been fabricated on MgO (100) and $LaAlO_3$ (LAO) (100) substrates under low oxygen ambient pressure by Pulse Laser Deposition (PLD). X-ray diffraction and TEM studies reveal the presence of pure phase cubic MZO (c-MZO). High quality cube-on-cube epitaxy is obtained in films grown on MgO. For films deposit on LAO, they exhibit heteroepitaxial relationship of $(100)_{c\text{-}MZO}||(100)_{LAO}$ and $(011)_{c\text{-}MZO}||(010)_{LAO}$. All as-prepared c-MZO thin films show good optical transmittance over the whole visible spectrum. Samples with In dopant concentration above 8 at. % yield an electrical resistivity of less than 10^{-2}Ωcm and can be used as Transparent Conducing Oxide (TCO) thin films.

17:20 Poster F-25

ZnMgO epilayers grown by chemical vapor deposition

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ZnMgO heterostructures grown by MOCVD require very high substrate temperatures (>900 °C) which make p-type doping by N or P rather difficult if not impossible. It was therefore our aim to explore the growth of ZnMgO epilayers using CVD at much lower substrate temperatures. The substrate temperature was between 600 and 650 °C, metallic precursors (Zn, Mg) were used with NO_2 as oxygen precursor. The properties of the films were investigated by low temperature photoluminescence, the Mg content was determined by SIMS and EDX. We find a linear dependence of the band edge recombination on the magnesium content. The maximum Mg content in our samples was 28 ± 2 %.

17:20 Poster F-26

Optical characterization of type II $Zn_{0.97}Mn_{0.03}Se/ZnSe_{0.92}Te_{0.08}$ multiple-quantum-well structures

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Recently some spin injectors and spin aligners, consisting of II-VI compound diluted magnetic semiconductor heterostructures as well as III-V compound semiconductor heterostructures, have been fabricated. Spintronics has attracted a good deal of attention owing to its potential applications in quantum computation, as well as its interesting physical properties of its building materials. In this paper, we present the interesting optical properties of ZnMnSe/ZnSeTe multiple-quantum-well (MQW) structures using photoluminescence (PL) and photoreflectance (PR) measurements. We prepared two sets of $Zn_{1-x}Mn_xSe/ZnSe_{1-y}Te_y$ (x=0.03,y=0.08) MQW samples grown on GaAs substrate by molecular beam epitaxy (MBE). For the samples A, B and C (D, E and F), the thickness of the ZnSeTe (ZnMnSe) layers is fixed at 20nm and the thickness of ZnMnSe (ZnSeTe) layers is varied among 5, 3 and 1 nm, respectively. PL data reveal that the band alignment of the ZnMnSe/ZnSeTe system is type II. Comparing with the theoretical calculation based on the Schrodinger equation, the valence band offset can be estimated. From the power-dependent PL spectra, it is observed that the peak position of PL spectra shows a blueshift under different excitation power. This effect can be interpreted in terms of the band-bending and band-filling effects due to the spatially photoexcited carries in a type-II aligment. We also found that the PL spectra exhibit a large in-plane polarization with the polarization degree up to 50%. The polarization does not depend on the excitation intensity as well as temperature, which excludes the possibility of extrinsic mechanisms related to the in-plane anisotropy. Photoreflectance has been widely used to investigate the intersubband transitions. In this study, for PR spectra the optical features corresponding to the ground and excited state transitions are found and discussed.

17:20 Poster F-27

The role of radiation defects in HgCdTe epitaxial growth

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X-ray diffraction methods as well as transmission electron microscopy were used to study the controlled doping and structural homogeneity of $Cd_xHg_{1-x}Te$ epitaxial layers. The investigated layers were obtained by the evaporation-condensation-diffusion (ECD) method in the process of isothermal growth. Two types of substrates for $Cd_xHg_{1-x}Te$ ECD growth were used: (110) and (111) CdTe monocrystals with As ion implanted surface layer at dose $<5\times10^{15}$ cm^2 and 100 keV energy. Structural changes in damaged areas of CdTe crystals that arise at the ion beam implantation as well the influence of radiation defects on the dopant diffusion process and quality of obtained layers are analyzed.

17:20 Poster F-28

Strain Relaxation Effect on the Properties of Ultra Thin ZnO Film on Sapphire (0001) Substrates by Pulsed Laser Deposition

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In ZnO/sapphire (0001) heteroepitaxy, the properties of the ZnO thin films are ultimately affected by the biaxial strain induced by the lattice mismatch. In this work the strain relaxation process in the initial growth stage of ZnO/sapphire (001) by PLD and its effect on the film properties have been investigated. Detailed structural evolution of ZnO films was carried out using XRD and TEM. At low temperature and high O2 pressure, the strain is released at the very early stage of growth by roughening the film surface and forming 3D islands. TEM analysis showed a columnar growth mode, and the existence of two types of in-plane rotation domains, ZnO [10-10]//sapphire [10-10] and ZnO[11-20]// sapphire [10-10]. Interestingly, the films grown at high temperature and low O2 pressure exhibit (0002) rocking curves dominated by a sharp peak with FWHM of 0.02 degree, and a single in-plane orientation ZnO[11-20]// sapphire [10-10]. TEM investigation revealed also highly crystalline layers which contain only basal stacking faults. This improved crystalline quality at optimized growth condition is induced by a much slower strain relaxation, so that the ZnO epitaxial layer is aligned to the lattice of the underlying sapphire substrate.

17:20 Poster F-29

High temperature electrical conductivity in ZnSe:In and in CdSe:In under selenium vapor pressure

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There are differences in In-doping mechanisms of II-VI compounds. Doping with In-dopant of some compounds gives rise to large concentrations of donors, but often the electron concentration is a small fraction of the donor concentration as a result of self-compensation

with formation of complex. In this report high temperature electrical conductivity (HTEC) isotherms and isobars of ZnSe:In and of CdSe:In are compared to construct the high temperature defect equilibrium (HTDE) models of these systems as we made earlier for II-VI systems doped with Al and Ga [1,2]. When HTEC isotherms and isobars of ZnSe:In and of CdSe:In, measured under metal component vapour pressure give both n-type conductivity then differences appear in the measurements under the selenium vapor pressure. Measured at the last conditions ZnSe:In HTEC isotherms are characterized by the conductivity type conversion but no change of HTEC type is observed on CdSe:In isotherms. Under these conditions the activation energy for ZnSe:In isobars is ΔE = 1.3 - 1.6 eV and for CdSe:In is ΔE = 1.3eV. The onefold ionized substitutional In at metal component place is proposed to be compensated by native defects in ZnS:In and in CdSe:In at high selenium component vapour pressure. The native defect for compensation may be onefold ionized zinc vacancy for ZnSe:In and twofold ionized zinc vacancy for CdSe:In. Defect associations occur at lower temperatures. HTDE models under selenium component vapor pressure conditions are presented and compared.

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17:20 Poster F-30

Local structure around Mn in Mn containing ZnO nano crystals

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Diluted magnetic semiconductors, either in form of thin films or in form of nanostructures have been of great interest in the last years due to the combined magnetic properties from the magnetic elements and the transport properties from the carriers of the host. Especially ZnO is experiencing a renaissance with the incorporation of Mn because of the recent predictions from theory. We have investigated the electronic structure of the ground state Mn^{2+} in ZnO nanocrystals as well as in $Zn(OH)_2$ by electron paramagnetic resonance (EPR) measurements. For a better assignment to the atomic structure we have complemented the EPR measurements with X-ray absorption spectroscopy (XAS) on Mn and Zn on samples with different particle size and different Mn concentration. Both types of experiments show that Mn substitutes Zn in ZnO as well as in $Zn(OH)_2$.

17:20 Poster F-31

Growth and characterization study of multidimensional hierarchical ZnO nanostructures

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We report on the fabrication as well as on the optical and electrical characterization of ZnO hierarchical structure. ZnO hierarchical structure fabricated with sequence of ZnO films/ZnO rods/ZnO buffer/c-plane sapphire substrate by continuous controlling growth condition in metal organic chemical vapor deposition (MOCVD) system. A growth temperature used in this experiment was not exceed the temperature of 300 °C, which is very low compared to the previous results on the ZnO films or rods grown by MOCVD. There were no seams or cracks in the interfacial region between ZnO films and end of ZnO nanorods. Transmission electron microscopy (TEM) characterization showed that ZnO nanorods in hierarchical structure had the single crystal hexagonal wurtzite structure with <0002> growth direction. Growth mechanism of transition from two-dimensional (2D) growth to one-dimensional growth or from 1D growth to 2D growth analyzed in atomic scaled via high resolution TEM (HRTEM). Due to the film layers located at end of nanorods, the optical and electrical property of ZnO nanorods could be observed directly without any metal contact which demands complicated process. Only near band edge (NBE) emissions with very weak deep level emission are observed around 3.28 eV. This indicates that ZnO hierarchical structure grown by MOCVD possess a good optical quality and less interior defects.

17:20 Poster F-32

Hydrogen passivation of electrically active defects in CdTe created by the bromine-methanol etching

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We report the change of the defect structure in CdTe crystal surface layer after etching in 2% Br/Me solution. The resistance of the etched samples is lowered by more than one order of magnitude and their low-temperature PL excitonic lines are blurred. The explanation of these processes is the forming of the Cd depleted surface layer and increasing of the Cd vacancies concentration which are known to be shallow acceptors. As a consequence crystalline quality of this layer is worsened and surface leakage current increases considerably. We studied the possibility of improving the chemically treated sample properties and electrically active centers passivation

by means of hydrogenization. CdTe:Cl single crystals grown by modified PVT are investigated. H diffusion into previously etched CdTe samples was performed by their exposure to H+ plasma created by glow discharge during 1 hour at 300 K and 0.025 Torr. Analysis of the temperature dependences of dark current and photocurrent testifies to partial restoring of the surface electrical properties for hydrogenized samples. It is seen from their resistivity increasing almost to those of cleaved samples and photosensitivity rise. We believe that these effects are caused by the hydrogen passivation of shallow acceptors centers; their ionization determines the dark conductivity at low temperatures. This assumption is confirmed by the intensity decreasing of PL bands in the edge region after hydrogenization. The improvement of surface quality is indicated by the change in the excitonic region of PL spectra. Bound exciton lines become much more prominent. Weak line of free exciton is also seen in hydrogenated as well as in cleaved samples spectra whereas it is not visible in the case of the chemically etched crystal.

17:20 Poster F-33

Photoelectric Properties of ZnO Thin Films

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Electron-beam deposition and sol-gel methods were used to obtain ZnO:X thin films doped with different impurities (X=Al, Ga and Li). Influence of various concentrations of impurities on electric, photoelectric and spectral properties of zinc oxide films has been investigated. The developed technique has allowed to obtain both transparent high conductive and dielectric ZnO films doped by different impurities. Measurements of dark and photoconductivity were carried out in a wide frequency range (0-10^{10} Hz). The value of resistivity depends on impurity and is changed from 10^{-4} up to 10^{6} Wcm. Photoelectric property studies have shown that with Li doping it is possible to achieve a significant increase of the photoconductivity. The current-voltage characteristics, current-optical power sensitivity and kinetics of rise and decay times of slow and fast components of the photoresponse were studied. It was found that the dark current and photocurrent in Li doped ZnO films have different conductivity mechanisms: hopping mechanism or charge transfer in a Hubbard-model impurity band for dark conductivity current, and drift mechanism of charge transfer in the conduction band for photoconductivity current.

The investigations have shown that ZnO film is perspective material for using as active layer in solid-state UV photodetectors. We studied photoelectric properties of the developed photosensitive field-effect transistor (currents ratio, charge carriers mobility, ampere-watt sensitivity in UV diapason, NEP sensitivity, and photocurrent kinetics). The open and close current ratio was 10^{6} and the field-effect mobility was ~10 cm^2/Vsec.

17:20 Poster F-34

Influence of hydrogen on optical spectra of hydrogenated CdTe

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The presence of oxygen impurity in semiconducting materials affects the electrical properties of crystals and significantly limits their application. To remove oxygen impurity, ultra-pure hydrogen is used while growing Te-containing crystals such as CdTe, CdZnTe, and ZnTe. The hydrogenation of CdTe crystals is a technological process that purifies the basic material from oxygen, mainly cadmium and tellurium oxide compounds incorporated in CdTe crystalline lattice. In the present work we analyses the deformations induced by hydrogen and oxygen atoms in CdTe crystals looking at their influence on the near fundamental band (NFB), middle infrared (MIR) and far infrared (FIR) reflectivity spectra as well as on cathodoluminescence (CL) spectra. Comparison of the hydrogenated CdTe phonon structure profiles confirms the presence of hydrogen atoms bounded inside the lattice.The possible localization of hydrogen and oxygen ions within the tetrahedron coordinated lattice is discussed in the framework of a model that shows a good agreement with recent NFB, MIR and FIR experiments carried out on hydrogenated CdTe crystals. Measured reflection spectra in the wavelength range 190-1400 nm (NFB) indicate the appearance in CdTe(H_M) and CdTe(H_L) of additional maxima at 966 nm related to the electron transitions from level about 0.2 eV above the valence band. The CL spectra confirmed existence of this electron level. We present a possible H_2 alignment similar to the single H model i.e., over the face (at about 0.38 Å). For this model the angle from the central atom to the H atoms is equal to 64° which is also close to the bonding angle of CdH_2. The propose model was verified by TEM images.

17:20 Poster F-35

Non-stationary photoconductivity processes in CdTe single crystals grown by modified PVT

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We performed the complex investigations of the non-stationary photoconductivity processes for undoped and Cl-doped (N_{Cl}=10^{17}-5·10^{19} cm^{-3} in the charge) CdTe single crystals grown by modified PVT. For the first time the CdTe photoconductivity kinetics measurements in the temperature region as wide as T=10-325 K were made. Numerical analysis of the decaying parts of photoconductivity signals revealed that the experimental curves are well fitted with a sum of two exponents with relaxation times τ_1 and τ_2 at lower temperatures. When the temperature increases the contribution of the second component reduces and at RT becomes negligible compared with the rapid one. Concentration dependences of the relaxation constants were also studied. Low-resistivity undoped CdTe is photosensitive in a narrow temperature range (30-60 K). τ_1 values of moderately and highly doped samples decrease from more than 40 μsec at 10 K to about 3-4 μsec at 100-150 K and remain practically constant with further temperature increasing. The τ_1(T) dependence of low-doped crystals (N_{Cl} = 10^{17} cm^{-3}) has a complex character: rapid falling (from 30 μsec at 10 K to 2 μsec at 70 K) followed by raising to the initial value in the vicinity of 150 K and then decreases again to less than 10 μsec but more slowly. The observed peculiarities are explained by the rebuilding of the CdTe defect structure as a result of doping with Cl.

17:20 Poster F-36

Optical and magnetooptical properties of the p-type ZnMnO.

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Zinc oxide is an II-VI compound semiconductor with a wide direct band gap of 3.37 eV at room temperature. Undoped ZnO is usually n-type, which is associated with native point defects and/or residual hydrogen. On the other hand, Mn doped p-type ZnO is predicted to be ferromagnetic with the Curie temperature reaching 300K [1].
ZnMnO layers were fabricated by thermal oxidation of nitrogen doped ZnMnTe films grown by MBE on GaAs and ZnTe substrates. In samples grown on GaAs, As is accumulated in the ZnO region as revealed by the SIMS analysis. Arsenic most probably, diffused from the GaAs substrate during the oxidation process. As a consequence samples were doped with both As and N acceptors. The Hall measurements demonstrated p-type conductivity with the high hole concentration. The XRD on ZnMnO samples present wurtzite polycrystalline character with metallic Te inclusion.
In photoluminescence spectrum from p-ZnMnO sample we observe an intensive peak located at 3.357eV, probably related to exciton bound to a neutral acceptor A0X or possible to an exciton bound to a neutral donor D0X is resolved.
Magnetooptical studies were performed in the Faraday configuration. PL peak dependence on the magnetic field shows an energy shift and a characteristic saturation. Magnetization measurements show a paramagnetic behavior and allow to evaluate the molar faction of Mn to be x=0.003, 0.0045 and 0.006. We fit the PL peak position vs. magnetic field using the Brillouin function and estimate the exchange constant No(α-β)=0.1eV. This extremely small value in II-VI DMS is discussed.
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17:20 Poster F-37

Temperature influence on the electroless Pt contact behaviour on CdTe and nuclear detection quality

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Contact deposition on CdTe is not a simple process, the nature of the chosen deposition method as well as the surface treatment are of great importance. However, contacts can decrease the quality of a good material. Electroless contacts on CdTe allow us to avoid the polarisation problem, knowing that even with good material, the detection spectrum can be of different quality and will depend on the deposition process. Furthermore, Electoless Pt contact deposition process on the CdTe is dependant on many parameters. This time, the time deposition, the dilution and the pH of the solution were fixed at 4 min., dilution 5, and pH=1.8 respectively while the varied one was the temperature. Analysis of Pt thickness and Cd lack was studied by RBS and SIMS and were correlated with the detection quality. The defect concentration and energy were studied by PICTS, TSC, TEES. In addition resistivity and gamma-ray detection spectra were showed as function of the solution temperature. A discussion is exposed about the role and quality of a possible presence of the TeO2 layer at the interface contact-semiconductor.

17:20 Poster F-38

The structure of nucleation Zn(Al)O layers for transparent metal oxide application

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For efficient detectors in a wide range of wavelengths, high transparency, and good electrical conductivity can only be achieved by creating electron degeneracy in a wide band gap semiconducting oxide. To this end, transparent conducting oxides such as Sn doped In2O3 (ITO), SnO2 (NESA) and ZnO are widely studied. In particular, ZnO films are usually doped with Si, Ga, Al, and so on. Among these, the Al-doped ZnO (AZO) transparent conducting films have high transmittance in the visible region and low resistivity. Besides, AZO has also several advantages such as non-toxicity, low cost and high stability against hydrogen plasma. Because of these many advantages, this material is suitable for the fabrication of solar cells, flat panel display electrodes, surface acoustic devices, optical waveguides, gas sensors, and micro-machined actuators. In this work, the single crystalline transparent conducting Al-doped ZnO films grown on sapphire(0001) by Rf-magnetron sputtering of various AZO targets are studied. We report on the structure of the thin Al:ZnO films and crystalline quality versus Al content, layer thickness as well as their behaviour during post deposition annealing.

17:20 Poster F-39

Post-annealing effect upon phosphorus-doped ZnTe homoepitaxial layers grown by MOVPE

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ZnTe is a promising material for pure green LED. Phosphorus (P) is considered to be a suitable species for *p*-type dopant in ZnTe, since the pure green LED is obtainable by a thermal diffusion of Al into P-doped *p*-type ZnTe. In our previous study, we have grown P-doped ZnTe layers by MOVPE using tris-dimethylaminophosphorus (TDMAP). Although the layer with a high carrier concentration of 1.3×10^{18} cm^{-3} can be obtained, it shows only donor-accepter pair emission (DAP) in the low temperature photoluminescence (PL) spectrum. In order to improve the P-doped ZnTe layer, we have investigated the post-annealing treatment (PAT), which is expected as one of the approaches for overcoming this problem.

The samples for PAT were as-grown P-doped ZnTe layers prepared by vertical MOVPE at atmospheric pressure on Ga-doped (100) ZnTe substrates using TDMAP as a dopant source. They were annealed in N_2 flow at a low temperature of 420 ºC for 2 h. In order to investigate the effect of PAT upon the P-doped ZnTe layers, the PL spectra of the layers were compared before and after PAT. The PL properties of the layers are drastically influenced by PAT. In each spectrum of the annealed layers, DAP vanishes and instead free-to-bound transition emission and broadened acceptor-related excitonic emission (I_a) appear. These changes indicate the reduction of the compensation and an activation of the P atoms, which may be a result of the decrease in the number of the defect related donor and/or the displacement of P atom into regular lattice site of Te by PAT. The broadened I_a shift towards lower energy side with increasing dopant transport rate, indicating the carrier concentration is enhanced by PAT.

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17:20 Poster F-40

Investigation of ZnTe thin films grown by Pulsed Laser Deposition method

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Significant progress in the nanoelectronic can be achieved at usage of efficient and low-cost technologies that allow the growth of thin films with good crystalline structure of semiconductor multicompound materials. Among varieties technologies that are used for growth of ZnTe films the Pulsed Laser Deposition (PLD) method is a highly efficient and flexible thin-film growth method. The energetic nature of the ablated species enhances the PLD process, potentially enabling deposition of high quality films on substrates at significantly reduced temperature. This paper is devoted to optimization of PLD growth condition of ZnTe films on various substrates and subsequent investigation of relevant parameters of growth process, structural, morphological and optical properties of grown films. Study the effect of growth parameters on the structural quality and properties of grown films were carried out. X-ray diffraction measurements showed that the ZnTe films, which have been deposited at optimal substrate temperatures, were characterized by a (111) preferred orientation with large average grain size. The surface morphology as function of the substrate temperature was investigated. We found that morphological properties of ZnTe films show a strong dependence on the growth temperature. The effects of subsequent deposition conditions on the optical properties of pure ZnTe thin films were studied. The optical transmission and reflectance in the energy range 1,5-5,5 eV for films grown at various substrate temperatures were measured. We calculated the variation in the absorption coefficient with the photon energy from the transmittance spectrum for samples grown at various substrate temperatures. Obtained data were analyzed and value of the absorption coefficient, for allowed direct transitions, was determined as a function of photon energy.

17:20 Poster F-41

Comparative study of II-VI based diluted magnetic semiconductors in form of bulk crystals, thin films and nanocrystals

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The giant Faraday effect is one of the remarkable properties of diluted magnetic semiconductors type of $II_{1-x}Mn_xVI$. Namely this effect has provided fertile ground for different optoelectronic applications including magnetic field sensors. In this work, the focus is on comparative magneto-optical study of CdMnTe, ZnMnTe, CdMnS in form of single crystals, thin films, nanocrystals and their application in magnetic field sensing. Single crystals were grown by the Bridgman method. Pulsed laser deposition technique has been applied to prepare thin films of CdMnTe. Growth regimes have been found for which physical characteristics of the thin films are similar to those for bulk material. Pulsed laser deposition technique was also applied for fabrication of CdMnTe and CdMnS nanocrystals embedded in SiO2 dielectric matrix. In Faraday rotation spectrum of CdMnTe film typical S-shape dispersion can be seen. The obtained results correlate with the data of the giant Faraday effect in the single crystals. Spectral dependence of Faraday rotation for nanocrystals of CdMnTe grown by laser deposition technique was found to be qualitatively similar to that of the related thin films. However, it was found additional enhancement of the Verdet constant in case of nanocrystal material almost for one order. The variant of one-way sensor scheme was used for designing of magneto-optical magnetic field sensor. Advantage of using thin films and nanocrystals in magnetic field sensing is low cost of manufacturing of such materials. This work was supported in part by grants No.M/128-2004 and No. D3/152-2005 from Ministry of Education and Science of Ukraine.

17:20 Poster F-42

Anisotropic optical properties of II-VI superlattices

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Band-edge optical properties of superlattices (SLs) can be discussed by modelling the superlattice as an effective anisotropic medium in which quasifree carriers propagate and interact. In the low barrier limit the electron and the hole motion in the confinement direction is determined by the SL potential and is replaced by an effective-mass motion with the appropriate masses obtained from the miniband dispersion. Here we present a calculational scheme which starts with the Kronig-Penney model to obtain SL miniband parameters, in particular, anisotropic effective masses for electrons and holes, and band gaps. Having these parameters, we can apply the real density matrix approach (RDMA) [1] adapted to the case of SLs [2,3], taking into account both the Coulomb interaction between the electron and the hole, and coherence between the electron-hole pairs and the radiation field. The RDMA gives the SL effective anisotropic dielectric tensor and thus enable to compute the SL optical functions. The method is applied to a $ZnSe/Zn_{1-x}Cd_xSe$ SL where we have computed bandoffsets, bandwidths and effective masses for the first 6 minibands (electrons, heavy- and light-holes). Then we have determined the SL dielectric tensor and computed the optical functions (absorption and reflectivity) for the case of oblique angle of incidence and for p- and s-polarisation. We also have computed ellipsometric parameters ψ, Δ and Stokes parameters. Fair agreement with available experimental data [4] has been obtained.

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17:20 Poster F-43

Structure properties of bulk ZnO crystals

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The intensive study of physical properties and growth process of ZnO crystals is caused by its unique electrical and optical properties. Zinc oxide crystals can be considered as an alternative to gallium nitride for use in optoelectronic devices. In the present work ZnO crystals were grown by Chemical Vapour Transport (CVT). During growth, partial pressure of zinc vapour was varied. The high-resolution diffraction study was performed in triple-axis mode using an X'Pert MPD diffractometer. The studied crystal had natural (110) and (001) external faces. Lattice parameters were determined from 110 symmetric and 112 asymmetric reflections. The changes of the colour observed in the crystal suggest that the Zn:O ratio varies along the crystal. It is necessary to admit, that the change of colour was not related to the change of crystallographic orientation. The parts of the crystal exhibiting different colours are found to have distinct *c* lattice-parameter values , whereas the *a* parameter is virtually the same in all parts of the crystal. The determined lattice parameters are different then the standard JCPDS. These deviations from standard parameters can be explained by the different oxygen concentration at different crystal parts. In the present work, the structural characteristics of bulk zinc oxide crystals, and their dependences on CVT growth process were done in details. The work was supported by the Ministry of Education and Science of Poland under the grant Nr.3T08A05128.

17:20 Poster F-44

Gallium and Arsenic Co-doped ZnO Thin Films Deposited by Pulsed Laser Deposition

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The transparent conductive oxides as ZnO have been widely studied. As a promising wide band gap semiconductor, ZnO thin films with varied dopant is important in fabricating the photonic devices to meet the various needs. In this study, Gallium and arsenic co-doped ZnO thin films were deposited at different temperatures (100-600^{o}C) on sapphire (001) and Si (100) by using pulsed laser deposition technique. X-ray diffractmeter (XRD), atomic force microscope (AFM), spectrophotometer, spectrometer were used to characterize the structural and optical properties the thin films. Hall measurements were also carried out to identify the electrical properties of the thin films. XRD results indicated that the co-doped ZnO thin films showed preferred (002) orientation. The transmittances of the thin films were nearly 100%. The band gap energies of the thin films were determined by a linear fit of the transmittance spectra. The photoluminescence measurements indicated that the thin films were very high optical qualities. The luminescence due to defect was highly quenched, only the near band edge emissions were observed. Hall measurements indicated that the co-doped thin films were very conductive and with high hall mobility.

17:20 Poster F-45

ZnO Thin Films on Sapphire (0001) Substrates Annealed in Oxygen

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Pulsed laser deposition (PLD) is a very efficient technique for the fabrication of the thin films, especially for the fabrication of metal oxide thin films at low temperature. In PLD system, the KrF excimer laser (l = 248 nm) is used to ablate ZnO target. During ablation the target is melted because of the irradiation of the high-energy pulsed laser. The melted zinc oxide is then evaporated and the plasma is produced during the process. High-quality zinc oxide thin films are deposited on the sapphire (0001) substrates at room temperature. Rapid thermal annealing process is followed to process the thin films at different temperatures (100, 200, 300, 400, 500, and 600^{o}C, respectively) in oxygen or in air for 10 minutes. X-ray diffraction (XRD), optical transmission, Raman spectroscopy, photoluminescence (PL), and atomic force microscopy (AFM) are used to characterize the thin films. It is found that the thin films annealed at higher temperatures are preferred (002) orientation, but the thin films annealed at low temperatures are amorphous nature. The band gap energies are determined from the transmission data by spectrophotometer. PL results reveal that the thin films annealed in air have two emissions. One is the near band emission; the other is the famous green-yellow emission in ZnO thin films. The green-yellow emissions are depressed when the thin films are annealed in oxygen.

17:20 Poster F-46

ZnO:Mn:Cu nanowires prepared by template method

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In the last few years, a large number of scientific publications dealt with the study of zinc oxide nanowires. The reason for this intensive research of zinc oxide nanostructures is given by the wide field of possible applications in the domains of optoelectronics and spintronics. On the other side, by ZnO doping with transitional metals one can obtain a room temperature diluted magnetic semiconductor with high Curie temperature; this property making ZnO a good candidate as a material for spintronic applications above room temperature. The present work has as main objective preparation of ZnO nanowires doped with Cu and Mn ions. By doping ZnO with manganese the possibility of obtaining diluted magnetic semiconductor nanowires will be opened; co doping with copper ions will facilitate the control of charge carriers responsible with the exchange interaction contributing in this way to the increase of the Curie temperature and luminescence yield for the studied structures. The properties of doped and undoped ZnO semiconductor are sensitive to the preparations conditions. We prepared by electrochemical deposition one-dimensional nanostructures having uniform diameters using polycarbonate membranes as templates. Nanowires morphology, composition and structure were characterized by scanning electron microscopy, energy dispersive X ray analysis and X ray diffraction, respectively.

17:20 Poster F-47

Photoluminescence from ZnO films prepared by wire explosion technique

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ZnO films deposited on glass, quartz and Al on silicon mono-crystal Si (100) substrates by using the wire explosion technique were investigated by X-ray diffraction (XRD), UV-VIS spectroscopy, scanning electron (SEM) and atomic force microscopy (AFM) measurements. X-ray diffraction measurements have shown that ZnO films are mainly composed of (100), (002), (101) orientation crystallites.

The post-deposition thermal treatment at 600 ^{o}C temperature in air have shown that the composite of Zn/ZnO film was fully oxidized to ZnO film. XRD spectra of the film deposited in oxygen atmosphere at room temperature presents high intensity dominating peak at 2h=36,32^{o} corresponding to the (101) ZnO diffraction peak. The small fraction of the film (7 %) correspond to (002) peak intensity at 2h=34,42^{o}. This result indicates the good crystal quality of the film and hexagonal wurtzite-type structure deposited by zinc wire explosion. The SEM analysis shows that ZnO films presented different morphologies from fractal network to porous films depending on deposition conditions. AFM analysis revealed the grain size range from 50 nm to 500 nm. The nanoneedles up to 300 nm length were found as typical structures in the film. The optical properties of the deposited ZnO nanostructures were investigated by Scanning Near Field Optical Microscopy and UV-VIS spectroscopy and revealed at room temperature a green photoluminescence band. The optical absorbtion spectra shows the bands at 374, 373, 371 nm corresponding to deposition conditions.

17:20 Poster F-48

Determination by Raman scattering of free charge-carrier concentration in p- type CdTe

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In this work we have measured the room temperature Raman spectra of the LO-phonon-plasmon modes of oriented CdTe samples prepared from commercially available p-type CdTe single crystals grown by vertical Bridgman process. The aim was to determine the free carrier concentration and to compare the results with other methods, namely photoluminescence (PL) and Hall effect. In the former, the energy difference between the free-to-bound and donor-acceptor transitions is used. In this paper we discuss the details of measurements and compare the results obtained by the three experimental techniques. We show that the three processes can yield similar results. While the Hall method can only be used in samples of low resistivity and requires adequate ohmic contacts and the PL method requires sample cooling, the Raman method is simpler and quicker: sample preparation is easier and experiments can be done at room temperature.

17:20 Poster F-49

Photo-EPR studies of charge tunneling processes in $Cd_xZn_{(1-x)}Se$:Fe,Cr ($0 \le x \le 0.3$) crystals

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Mechanisms by which localized excitations move through a crystal are of great interest in various fields of solid state physics. Such kind of transfer takes place in, e.g., laser, cathodoluminescent and electroluminescent materials.

In this communication we analyze tunneling processes of photo - generated electrons and holes between impurities in $Cd_xZn_{(1-x)}Se$:Fe,Cr (x = 0, 0.1, 0.2, 0.3) bulk crystals. To study migration of electrons among donor, iron, chromium, and acceptor after switching off external illumination, changes of electron paramagnetic resonance (EPR) signals of the Fe^{3+} and Cr^{1+} ions were detected. Measured decay times of the EPR signals at liquid helium temperature were long ($\tau_d \sim 10^3$ s). However, decay of the Fe^{3+} signal was a bit faster than decay of the Cr^{1+} signal. Increase of temperature caused decrease of the measured decay times for both impurities. Observed temperature dependence of τ_d has so called activation character. Energy value determined from such dependence correlates well with thermal ionization energy of donors present in the CdZnSe:Fe,Cr system. Results are compared with previous data of Zakrzewski and Godlewski obtained for ZnS:Cu,Fe,Cr.

17:20 Poster F-50

SIMS study of Al thermal diffusion in ZnTe

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ZnTe is a promising material for a variety of optoelectronic devices such as pure green LED because of its direct band gap of 2.26 eV at room temperature. Recently, Al thermal diffusion technique is attracted attention as a potential method to fabricate *pn*-junction ZnTe LED at low cost. In order to fabricate LEDs with better performance, the Al concentration in the diffusion layer has to be optimized [1]. However, the diffusion properties of Al in ZnTe has been scarcely reported except for a paper which reported the diffusion coefficient of Al estimated from the diffusion depth measured by scanning electron microscope [2]. In this study, we have evaluated the depth profile of Al in the diffusion layer by secondary ion mass spectroscopy (SIMS), and analyzed the diffusion property exactly.

SIMS measurements were performed on Al-diffused ZnTe from the backside of the diffusion layer to evaluate the Al depth profile ex-

actly. According to the Fick's second law of diffusion, the diffusion profile must obey an erfc function. However, the SIMS depth profile deviates from an erfc curve in the Al concentration range below $10^{18}cm^{-3}$. We employed the Boltzmann-Matano analysis to evaluate the concentration dependent diffusion coefficient of Al in ZnTe, and found that the diffusion coefficient of Al decreases at the Al concentration range below $10^{18}cm^{-3}$. This result indicates that a rapid decrease in Al concentration is occur at the diffusion front, yielding the steep *pn*-junction leading the better performance.

This study was supported by Industrial Technology Research Grant Program in 2005 from NEDO of Japan.

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17:20 Poster F-51

The effect of deformation phenomena on optical and photoelectric processes in cadmium telluride single crystals

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We studied the changing of the CdTe single crystalline samples properties caused by purposive introducing of fresh dislocations by means of uniaxial compression under constant load as well as by indentation. The dislocation density (DD) increased by more than two order of magnitude after the deformation. The temperature dependences of dark conductivity in the region of 100-300 K correspond to the activation energy of E_i = 82 meV both for initial and deformed samples but the carriers concentrations increase with DD rising. The RT carriers lifetimes determined from the decaying parts of transient photoconductivity signals decreased in 2-5 times after the deformations. The shallow level E_t = 11 meV participates in the relaxation processes of the initial samples. A recombination through deep centers dominates in deformed crystals indicating that there the overcompensation took place. The analisys of the photoconductivity kinetics revealed the increase of the surface recombination centers concentration and persistent photoconductivity in the last case. The values of the transmission cut-off energy point out the optical band gap narrowing up to 8% in deformed samples. The optical absorption edges become more broadened and their temperature shifts become a little smaller with the increasing of the DD. The last facts are explained by the disordering processes and deformational goffering of energy gaps.

17:20 Poster F-52

Localized electronic states of oxygen impurities in II-VI compounds.

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With the aim of tailoring the properties of efficient interband solar cells we
investigate localized impurity states in different II-VI wide gap semiconductors.
As the host material we chose binary, ternary and quaternary compounds of the
family (Zn,Cd)(Se,Te). The impurity band is formed by isoelectronic oxygen states replacing VI-group atom. For technological applications the impurity band should be positioned within the fundamental band gap. We investigate the role of the local environment around the impurity by changing the composition of the oxygen neighboring shells. The atomic scale effects of the electronic structure and in particular the localization are studied with the ab initio pseudopotential approach and the Wannier function analysis [1,2].

[1] www.pwscf.org
[2] www.wannier.org

17:20 Poster F-53

Electrical contacts to semi-insulating (Cd,Mn)Te:V

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The high-resistivity (Cd,Mn)Te is believed to be suitable to successfully replace the commonly used (Cd,Zn)Te system as a material for manufacturing large-area X- and γ-ray detectors.

The purpose of our study was to elaborate a method of preparing high quality (Cd,Mn)Te crystal plates as well as a technique of producing good electrical contacts to that material.

(Cd,Mn)Te was grown using the Bridgman method. The crystals were doped with vanadium to the level of 10^{16} cm^{-3} . The crystals are twinned in the (111) plane, but by slicing the crystal parallel to the twinning plane we obtained monocrystalline plates of large area (e.g. 30 × 30 mm^2), which is essential for application purposes. Proper annealing of those plates in cadmium vapours allowed us to reduce the number of cadmium vacancies forming during the growth process. Due to the vanadium dopant acting as a compensating centre we obtained a semi-insulating material. In order to obtain good contacts to the (Cd,Mn)Te plates we used the method proposed initially for CdTe by D. Rioux [1]. The ZnTe:Sb layers (~1 μm thick) were grown on the epi-ready (Cd,Mn)Te:V plates by the MBE technique. The grown layers were p□type and formed a good electrical contact to the crystal plates. Finally - a standard tech-nique was used to cover the ZnTe layer by a metal layer. The contacts formed on both the (100) and (111) surfaces of (Cd,Mn)Te were studied. We believe that the contacts to the (111) surface are important for applications.
In the paper we describe techniques of preparing electrical contacts and results of their characterization.

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17:20 Poster F-54

Structural properties of ZnO polymorphs

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The first principles calculations by using the plane-wave pseudopotential method in the scheme of density functional theory were performed for polymorphs of ZnO: wurzite-, zinc-blende-, rocksalt-structures, and yet undiscovered CsCl-structure. The elastic behaviour and the stability of the structures are investigated and compared to experimental data, where available.

17:20 Poster F-55

The Effect of Formation Conditions of Nucleation Center on CdTe, CdMnTe and CdZnTe Single Crystals Structure

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The structure of CdTe, CdMnTe, CdZnTe single crystals obtained from the melt at different formation conditions of nucleation center is investigated. For growing of large single crystal by Bridgman method form of the bottom of an ampoule, where crystals growth begins, is very important. For crystals less than 20 mm in diameter the best single crystals reproducibility is observed if ampoules with the system of neckings is used, for crystals of 20-30 mm in diameter - at the use of ampoules with a cone bottom. For crystals of 30-60 mm in diameter the best results gives the use of flat bottom with light conductor or heat conductor. Interesting results were obtained at the use of ampoules with a flat bottom, light conductor on the center and with double walls. A gap between walls was filled by the CdTe melt. This melt crystallized earlier, than the melt which in the internal ampoule. The released heat of crystallization heated the internal walls of the ampoule and aligned crystallization front. The temperature conditions of this process were studied theoretically.

17:20 Poster F-56

Influence of underlying layers on the growth of 1-D MgZnO nanorods

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ZnO is a II-VI oxide semiconductor with a wide band-gap (3.37 eV) and a large exciton binding energy. ZnO-based one-dimensional materials have received increasing attention over the past few years due to their potential applications. Recently, doping of ZnO nanostructures has become the focus of intensive research because their kinds of doping elements can efficiently adjust their electrical, optical, and magnetic properties, which are important for practical applications. By alloying Mg in ZnO, the band gap can be modulated from 3.3 to 7.7 eV by alloying it with different concentration of MgO. In this study, we investigated the characterization of the MgZnO nanorods fabricated by thermal evaporation method on underlying layers such as ZnO template, GaN, and MgZnO template. MgZnO nanorods were fabricated by a two-step growth technique. In the first step of low temperature, Zn seed metals with low melting temperature formed the droplet, and then MgZnO ternary nanorods were grown by injecting oxygen and evaporating Mg atoms in high temperature process of the second step. The vertical growth of the MgZnO nanorods with large-area distribution and uniformity was successfully performed on various underlying layers. We characterized the optical and crystal properties of the vertically grown 1-D MgZnO nanorods using SEM, XRD, Raman, PL, and TEM.

17:20 Poster F-57

The effect of oxygen content on the electrical characteristics of ZnO films

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Zinc Oxide (ZnO) is currently considered as a promising material for very bright ultraviolet and blue optical devices, like light emitting diode (LED) and laser diode (LD), because of its.interesting characteristics, such as a large direct band gap of 3.37 eV at a room temperature and a large exciton binding energy of 60 meV which is 2.4 times of GaN. However, most of the deposited ZnO films tend to show many point defects, such as Zn interstitials and oxygen vacancies. These oxygen vacancies are responsible for natural n-type of ZnO. Thus, it is of great significance to understand the properties of ZnO films with oxygen contents. This paper reports the effects of oxygen content in ZnO films on the electrical properties. To control oxygen concentration in ZnO films, several kinds of post thermal annealing have been performed in N2 and air at different temperatures of 600 to 800 oC, where oxygen concentrations in ZnO films were measured by Auger electron spectroscopy. The electrical characteristics were measured by Hall measurement in the van der Pauw configuration and transmission line method (TLM). As results, the electron concentration and resistibity can be controlled from low 10^16/cm^3 to mid 10^18/cm3 and from 10^-2 to several tens ohm-cm, respectively. In special, we will discuss^ quantitative relation between oxygen content and carrier concentration.

17:20	Poster	F-58

Growth of Zinc Oxide Crystals and Their Scintillation Properties

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ZnO is a wide band-gap semiconductor (Eg = 3.37 eV) with a high exciton binding of 60 meV. It has great potential for application in optoelectronics, piezoelectricity, and UV-blue LED. Since exciton luminescence of ZnO shows very short decay time of < 1 ns, it is attractive as ultra-fast scintillator material. However intensity of exciton luminescence of ZnO for device application is relatively low because of following reasons:

1. Self-absorption of exciton luminescence
2. Damage of surface that results luminescence in the green-yellow spectral region.

In this study, comparison of scintillation properties of ZnO with different surface quality was performed. Moreover, growth and effect of donors on red-shift of the luminescence wavelength was examined in details in order to decrease self-absorption.

ZnO crystals were grown by hydrothermal method from Pt container and liquid phase epitaxy (LPE) with LiCl flux. Photoluminescence spectra at room temperature were measured with Xe lamp excitation and S900 spectrometer (Edinburgh Instruments). For decay time measurement, fs laser CPA2001 (Clark-MXR) was used as an excitation source. (260 nm, 160fs).

The as grown ZnO epitaxial layers with untreated surface show highest emission intensity from the band edge, and defect-related luminescence was low. It was also confirmed that exciton luminescence of ZnO can be increased by improving after-growth machining of the crystals. Exciton luminescence of In^{3+}-doped ZnO was shift to longer wavelength range (398 nm). In:ZnO crystals demonstrated ultra-short decay of two components (40 ps and 650ps).

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium H

Welcome

Most of applicable properties of contemporary materials follow from structural transformations whose dynamics governed by atomistic mechanisms must be under control within advanced technologies. This general goal cannot be achieved on one hand without elucidating the nature of nano-structural (atomistic) phenomena both experimentally and by means of modeling or simulations covering the problems of structural defect thermodynamics and physics of atomic migration in condensed phases, and on the other hand, without elaborating links between nano- and meso-scale approaches to modelling the processes.

As clearly follows from many meetings and discussions, advanced methods and concepts of condensed-matter physics definitely enter the domain of contemporary materials science. This is in great extent due to the current fast development of hardware and software computational facilities.
The main idea of the organized event is to gather physicists, materials engineers and other scientists carrying out interdisciplinary research in one symposium dedicated to the multiscale modelling of time-evolving phenomena in materials. Although such problems have recently been taken up in contributions scattered over various conferences/sessions, organization of a specially dedicated symposium with the programme definitely focused on materials dynamics will stimulate synthesizing insight into the subject matter and will give rise to the development of new material technologies well-grounded in materials physics.
The symposium will focus on two basic aspects of investigations:
(i) current development of theoretical and model approaches to structural kinetics (links between quantum electron theories of solids and non-equilibrium thermodynamics);
(ii) computer simulations as an effective tool for studying atomistic mechanism of structural kinetics (Monte Carlo and Molecular Dynamics: suitability, efficiency, limitations, results).

In both cases reports on mustiscale-character investigations, as well as communications of results of pure nano- or mesoscale modelling are welcome.

Scientific Committee:

Christian Abromeit (Hahn-Meitner-Institut, Berlin), John Agren (Royal Institute of Technology, Stockholm), Robert W. Cahn (Department of Materials Science & Metallurgy, University of Cambridge), Jiři Čermák (Institute of Physics of Materials, Czech Academy of Sciences, Brno), Marek Danielewski (AGH University of Science and Technology, Krakow), Didier de Fontaine (University of California, Berkeley), Andriy Gusak (Cherkassy State University), Georges Martin (CEA Saclay), Carlo Massobrio (IPCMS, Strasbourg), Yuri Mishin (George Mason University, Fairfax), E.J. Mittemeijer (Max Planck Institute for Metals Research, Stuttgart), Tetsuo Mohri (Hokkaido University, Sapporo), Wolfgang Pfeiler (Vienna University), Veronique Pierron-Bohnes (IPCMS, Strasbourg), Maciej Pietrzyk (AGH University of Science and Technology, Krakow)

Organisers

- **Rafal Kozubski**, Institute of Physics, Jagellonian University, Krakow, Poland
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- **Pawel Zieba**, Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Krakow, Poland

Proceedings

The manuscripts submitted to this symposium will be reviewed and published in **"Solid State Phenomena".**

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Programme

Monday, 4 September

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Simulation techniques for bridging length and time scales
Monday afternoon, 4 September, 14:00
Chair: Tetsuo Mohri, Tomasz Wejrzanowski, Veronique Pierron-Bohnes

14:00	Invited oral

Bridging different length and time scales in diffusion problems using variable length lattice models

Irina V. Belova, Graeme E. Murch

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Many diffusion problems span widely different length and time

scales. In this presentation we show how lattice-based random walks of virtual particles can be used to address a variety of complex phenomenologically conceived diffusion problems. Examples include the determination of grain boundary depth profiles in the presence of grain boundary slabs, the determination of the effective diffusivity (and thermal conductivity) in the presence of various shapes of grains with and without solute segregation, the determination of time-dependent concentration profiles of solute that segregates at metal-ceramic oxide interfaces and the effective electrical conductivity of composite electrolytes. In some examples we also show how the finite element method can also be used. Comparison is made of the results of these two methods where possible.

14:30 Oral

Electro-Mechano-Chemistry; Transport Problem in Three Time Scales

Marek Danielewski[2], Maciej Pietrzyk[1], Bartłomiej Wierzba[1]

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The mass transport in the presence of stress, electrical, mechanical and chemical potential gradients in multicomponent solid solution is analyzed. We simulate the deformation field during the diffusion caused by the gradients of the chemical potential of all elements. The method is based on the Darken concept, the calorimetric equation of state and Gauss equation. We effectively couple the mass conservation (continuity equations) with the energy and momentum conservation laws. The diffusion fluxes of the components are given by the Nernst-Planck formulae and take into account the electrochemical and mechanical potentials.

We are presenting the numerical results for Cu-Fe-Ni alloy. The simulations show that the model is compatible with experimental results, and can be effectively used for modeling the energy, momentum and mass transport problems in compressible multicomponent solid solutions. The electro-mechano-chemical transport is a multi-scale problem. The transport is governed by light velocity ($\sim 10^{8}$ ms^{-1}), strain field characterized by "sound velocity" in the medium ($\sim 10^{4}$ ms^{-1}) and slow diffusion process characterized by self-velocity of diffusion ($\sim 10^{-2}$ ms^{-1}). The theoretical and numerical problems and methods of solution are presented.

14:45 Oral

Concurrent multiscale Kinetic Monte Carlo-continuum models for the evolution of solids via diffusion

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An atomistic Kinetic Monte Carlo (KMC) model for diffusion is found to exhibit classical behaviour in the limit of small gradients of slope (for surface diffusion) and concentration (for lattice diffusion). Equivalent continuum models are derived in this limit and the two systems are shown to be compatible. A mixed KMC-continuum model is proposed which allows discrete atomistic simulations to be embedded within a smooth continuum at points where the gradients are large. A blending region is introduced between the two model descriptions, which allows mass to be transferred between them in a consistent manner. This allows the model to simultaneuously work across multiple length and time scales. The implementation of this multiscale modelling approach is illustrated with a number of simulations, including the decay of a rough surface.

15:00 Oral

Comparison of the strain distribution obtained from multi scale and conventional approaches

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Multi scale modelling of material response under various loading conditions is a commonly used method of solving practical problems. One of the approaches is multi scale technique based on combination of cellular automata (CA) and finite element (FE) method - CAFE method. Creation of rheological model in the micro scale, capable of predicting discrete and discontinuous processes, is the main advantage of this approach.

This work is part of an investigation connected with application of the developed CAFE model to prediction of strain localization in various industrial processes. Model capabilities to predict the development of micro shear and shear bands in different scales are crucial during metal forming operations, when localized deformation limits ductility and may lead to fracture. On the other hand, materials formability may be extended when the development of shear bands is precisely controlled during entire deformation.

Comparison of the strain fields predicted by FE and CAFE models is the general objective of the project. Extrusion process, which involves strong strain localization, was selected as an example. This selection was motivated by experimental observations showing highly localized shear band zone with flow discontinuity that occurs in the deformation area. Shear zone influences the final product microstructure and may lead to coarse grain layer close to the surface. Modelling of the shape of this zone and prediction of strains will create a possibility of computer aided design of extrusion process and optimization of technological parameters. Conventional FE models give the results, which do not agree well with experimental observations. The developed CAFE model predicts larger values of strains and narrower strain localization zone, which is closer to the experimental observations. Advantages and disadvantages of the CAFE model are discussed as well.

Coffee break
Monday afternoon, 4 September, 15:30

Phase Transformations

Monday afternoon, 4 September, 15:50
Chair: Hualong Li, Paweł Dłużewski, Stefan Müller

15:50 Invited oral

Atomistic Monte Carlo simulations of phase transformations under irradiation

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We present some recent applications of Monte Carlo simulations to the study of Radiation Induced Segregation (RIS) and Precipitation (RIS) kinetics in metallic alloys. These phenomena are experimentally well known and can have an important impact on nuclear industry. At the atomic scale, they result from the permanent fluxes of point defects sustained by irradiation towards sinks such as grain boundaries, dislocations, etc. The coupling with fluxes of solute atoms can lead to local segregation and precipitation processes.

RIS and RIP behaviours can be predicted by Kinetic Monte Carlo simulations based on an atomic model of diffusion under irradiation which takes into account the creation and migration of vacancies and interstitials, their mutual recombination and their annihilation at sinks. In model binary alloys, KMC simulations have been used to show how the kinetic pathway and the microstructural evolution are controlled by the details of diffusion properties, i.e. by the various migration barriers and the way they depend on the local atomic configurations. We especially focus on the case of dilute iron-copper alloys. In this system, the KMC parameters (effective interactions and migration barriers) have been computed by ab initio calculations, in the framework of the Density Functional Theory.

16:20 Invited oral

Multi-lattice kinetic Monte Carlo simulation of interface controlled solid-state transformations

Cornelis Bos, Ferdinand Sommer, Eric J. Mittemeijer

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To overcome timescale limitations associated with other atomistic simulation methods a kinetic Monte Carlo method has been developed for the simulation of interface controlled solid-state transformations. In the simulation method the atoms can take place on sites from (at least) two intertwining crystal lattices. To enable the atoms to also take positions between the ideal lattice sites, a collection of randomly placed sites can be included. These 'random sites' allow for irregularities in the atomic structure of transformation interfaces. The atoms move by independent, thermally activated jumps. The activation energy for the atomic jumps can be determined for every jump separately based on the arrangement of the neighbouring atoms. The simulation method has been used to study the interface mobility in the austenite to ferrite transformation in iron for different interface orientations. Simulation results indicate that the excess volume associated with the interface plays a key role for the activation enthalpy for the interface mobility. The rate controlling process is the rearrangement of free space at the interface by series of (unfavourable) jumps by different atoms to create a path from the parent to the product phase.

16:50 Invited oral

First principles calculation of ordering transition

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First-principles calculations have been rapidly developing in various fields of materials science. So far, most of the works have been centred around the calculation of static equilibrium such as ground state stability and phase equilibria. The authors' group also attempted a series of first-principles calculations on binary phase equilibria for Fe-based alloys by combining FLAPW electronic structure calculations with Cluster Variation Method of statistical mechanics. The calculations for Fe-Pd and Fe-Pt were quit successful to reproduce the order-disorder transition temperatures with quite a high accuracy. Also, for Fe-Ni system, we found a stable ordered phase which is missing in the conventional phase diagram. Furthermore, the calculation of the Coefficient of Thermal Expansion of Fe-Ni at Invar concentration reproduced the experimental results quite well. Recently, our attention has been focused on the extension of the first-principles calculations to transformation dynamics. For this, we further incorporated Phase field Method and attempted to calculate the evolution process of anti phase boundary associated with disorder-L10 transitions. We reproduced the essential morphology of the anti phase boundaries including triple point junctions. Moreover, the spatial scale and crystallographic orientations are uniquely fixed in the calculated microstructure. However, one serious problem is the time scaling. Coarse graining operation adopted in our calculations is not applicable to fix the time scale. The extension of the coarse graining operation to time scale is attempted based on the Path Probability Method.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Ordering Processes

Tuesday morning, 5 September, 9:00
Chair: Alain Barbu, Carlo Massobrio, Robert Sot

9:00 Oral

Molecular Dynamics Simulation of Swift-Ion-Induced Phase Transformations in a Model NiAl Alloy

Christian Abromeit[1], Nikolai P. Lazarev[2], Rolf Gotthardt[3], Robin E. Schäublin[4]

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The track evolution of ions with energies Ei > ~ 100 MeV results in various phase transformations in a martensite phase as e.g. crystal-liquid-glass-crystaltransitions, martensite transformations or compositional order-disorder transformations. They occur at length scales of ~10 nm from track centre and at time scale of up to 1 ns. In order to get more insight in these phenomena we perform molecular dynamics simulations of phase transformations in a model NiAl alloy. The advantage of the Ni-Al system is that the embedded atom potentials are known. We start with the study of the thermodynamics and kinetics of this model alloy. We observe a wide hysteresis on the temperature dependence of the free energy at cooling and heating of initially B2 ordered alloy with composition ranges from 60 to 65 at. % of Ni. A strong influence of cylindrical and spherical extended defects on the character of martensite phase transformations in this alloy is observed. In order to distinguish the structural changes during the phase transformations we use a new approach for the determination of local order parameter of the alloy undergoing structural rearrangements. This method is based on combination of Voronoy tessellations and the common-neighbor analysis. Also, the basis of an analysis of local chemical order changes is discussed. Such method has been applied for local order-disorder transformations modelled by MC simulations.
The development of new phases around the swift ion track in NiAl alloy is simulated. The central part of the track appears to be highly disordered and has the tendency to transform into amorphous state. The outer part shows the formation of heterogeneously distributed austenite B2 ordered regions.

9:30 Oral

Multiscale simulation of ordering processes in GaInN and GaAlN

Katri Laaksonen, Maria G. Ganchenkova, Risto M. Nieminen

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The III-V semiconductors with nitrogen as the fifth-group element have unique properties that make them very attractive for short-wavelength light emitters and high-power/high-temperature electronics applications. In order to provide the adequate performance of electronic devices, these materials must have reliable electronic properties. It is well known, however, that the electronic properties of multicomponent semiconductors are very sensitive to the uniformity of their chemical composition. The inhomogeneities can affect device performance via changes of the band gap energy and localized variations of polarization field.
There exists experimental evidence that III-V alloys are often unstable against phase separation, spatial fluctuations in the second component concentration, or partial ordering. Compositional inhomogeneity (clustering or phase separation) has been experimentally observed, in particular, in InGaN. The microscopic reasons for ordering in InGaN and the ordered structure patterns have been already extensively studied in the literature. Unfortunately, the majority of the theoretical investigations are based on analytical models, which makes the reliability of such predictions uncertain.
In this work we study the minor component ordering in wurtzite $Ga_{1-x}In_xN$ and $Ga_{1-x}Al_xN$ alloys by the multiscale approach that combines the accurate total-energy density functional calculations and lattice kinetic Monte-Carlo simulation. According to our results In in GaN forms [0001] aligned pairs or chains and, at higher In concentrations, zigzag chains in c-direction, while Al forms a random alloy with the matrix material. The increase of In concentration above 25-30% is shown to lead to the phase separation and formation of InN regions. The effect of the minor component (In,Al) ordering pattern on the band gap of the ternary Ga(In,Al)N alloy is also discussed.

9:45 Oral

Effect of Fe addition on ordering kinetics in $Ni_3Al_{1-x}Fe_x$. Monte Carlo simulation

Rafal Kozubski, Ewa Partyka

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Experimental studies of the effect of Fe alloying on the long-range ordering kinetics in $Ni_3Al_{1-x}Fe_x$ revealed a decrease of the activation energy for "order-order" relaxations with an increase of Fe concentration. For low Fe concentrations the activation energy was close to the one measured for Fe^* tracer duffusion in Ni_3Al.

The effect has now been modeled in atomistic scale by means of Monte Carlo simulations of a quasi-binary $L1_2$-ordered system $A_3B_{1-x}C_x$ with an Ising-type Hamiltonian. "Order-order" kinetics were simulated within Glauber dynamics implemented with vacancy mechanism of atomic jumps. The reality of $Ni_3Al_{1-x}Fe_x$ has been simulated by evaluating the atomic interaction energies in the way that the stability of $L1_2$ superstructure gradually decreases when the C concentration x increaes. The multi-time scale character of the relaxations known from the case of Ni_3Al was again observed. Detailed analysis of atomic jump statistics revealed: (i) a dominance of the C-atom jumps in the creation/elimination of antisite defects; (ii) an increased vacancy availability in the 1st co-ordination shell of the C-atoms with respect to the A- and B-atoms. The results elucidate the role of Fe alloying in the kinetics of chemical ordering in Ni_3Al.

Coffee break
Tuesday morning, 5 September, 10:30

Precipitate Formation

Tuesday morning, 5 September, 11:00

Chair: Christian Abromeit, Robert Filipek, Simon P. Gill

11:00 Invited oral

Ab-initio statistical physics of precipitate evolution in metal alloys

Stefan Müller

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Quenching a solid solution of a binary metal alloy into the two-phase region of the phase diagram leads to the formation of characteristic precipitate microstructures. Of special importance are coherent precipitates that have no dislocations between the precipitate and the matrix. It will be demonstrated how the size. shape, temperature-dependence and time evolution of the precipitates' distribution can be studied by combining density functional theory calculation with the so-called Mixed-Space Cluster Expansion method (MSCE) and Kinetic-Monte-Carlo simulations. Thereby, the MSCE Hamiltonian allows for a detailed analysis of interface and strain energies and their influence on the precipitate's shape. The focus will be on Al-based lightweight materials, namely Al-Zn, Al-Cu, and Al-Li. For the latter, the precipitates form an intermetallic compound itself, namely the well-known $L1_2$ structure which is not stable in the bulk phase diagram of Al-Li.

11:30 Invited oral

Cluster dynamics modelling of materials : advantages and limitations.

Alain Barbu

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In material science, cluster dynamics (CD) is based on kinetic equations describing the formation and evolution of clusters of solute atoms or point defects such as vacancies or self interstitial atoms (SIA). Whatever the efficiency of algorithms, CD that can be seen as a meso-scale modelling, cannot be replaced by Lattice Kinetic Monte Carlo method to address long time aging in many cases. Indeed, it is a very efficient method in term of computational cost. This efficiency is due to a drawback coming from the basic hypothesis of uniform distributions of clusters (a gaz of clusters): the real system is replaced by an effective medium in which all processes occur continuously in time and space. The spatial correlation between clusters is consequently not considered explicitly.

After a short introduction describing the basis of the model and the numerical schemes used to solve the set of ordinary differential equations describing the evolution of the number density of clusters of every size and type, we will give several examples of applications in the field of precipitation and irradiation. The limitations of the model and the conditions of utilisation will be discussed. Unsurprisingly, it will be shown that this method goes in a very satisfactory way when the objects are distributed homogeneously as for annealing of microstructure produced under irradiation or for homogeneous nucleation of precipitate. Conversely, it will be shown that the source term describing the primary damage under irradiation, by nature heterogeneous in space and time, is difficult to introduce especially when displacement cascades are produced.

12:00 Oral

Early stages of island nucleation and growth on Ag/Ag(100)

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During deposition, the morphology of the initial layer will crucially influence further layer growth.

We introduce a suitable kinetic Monte Carlo model for island nucleation and growth on Ag/Ag(100) substrate, leading to the formation of non-equilibrium structures consisting of near square islands as observed on STM images. We simulate the homoepitaxial deposition of Ag adatoms on the Ag(100) substrate at a temperature of 200 K for values of the incident flux of particles ranging between 0.01 ML/s and 1.00 ML/s.

We characterize the nucleation process by studying the dependence of the mean island density (per unit site) during growth and the island size distribution on the incident flux of particles.

Interesting non trivial behavior of the time dependence of the mean island density arises from the interplay between island formation due to deposition of adatoms and difusion and coalescence of existing islands.

The island density also shows an interesting dependence on the flux of deposition leading to two different regimes, since for higher fluxes the system does not have time to agregate.

12:15 Oral

An Atomistic Approach to the Effect of Cr on the Rate of Void Formation and Agglomeration in bcc Fe

Byeong-Joo Lee

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For an elaborate control of microstructures and materials properties, it is desired to understand the materials behavior from more fundamental level, e.g. the atomic level. Atomistic simulations (Molecular dynamics or Monte Carlo) can be a useful tool to analyze and predict phase transformations, defects, structural evolutions and mechanical behaviors of hard materials. The atomistic approaches use results of first-principles or Calphad thermodynamic calculations as well as experimental information for determination of interatomic potential parameters. Most of the properties covered by the atomistic approaches are those that cannot be investigated using the first-principles or Calphad thermodynamic calculations. In this sense, the atomistic approaches can be regarded as to link the first-principles

and Calphad thermodynamic calculation techniques in a multi-scale framework.

Void swelling is one of the typical degradation phenomena in irradiated steels. It is known that the length of transient regime (void formation stage) gives a decisive effect on the overall rate of swelling in ferritic (bcc) steels. However, because of the highly limited experimental environment (neutron irradiation), the effect of alloying elements on the rate of void formation or agglomeration is not clearly known. In the present work, the effect of Cr, the typical alloying element in ferritic nuclear steels, was investigated by using atomistic approaches (molecular dynamics and Monte Carlo simulation) based on a recently developed semi-empirical interatomic potential (the second nearest-neighbor modified embedded atom method potential) for the Fe-Cr binary alloy system. It was found that the Cr atoms segregate and even form clusters on the void surfaces, and give a significant effect on the rate of void formation or agglomeration. Details of simulation methods and results will be presented as well as the reliability of the interatomic potential used.

Lunch break

Tuesday afternoon, 5 September, 12:30

Simulation of Defect Processes

Tuesday afternoon, 5 September, 14:00

Chair: Dariusz Chrobak, Irina V. Belova, Jiri K. Bursik

14:00 Invited oral

Diffusion-limited Processes Treated With Accelerated Molecular Dynamics

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We treat diffusion-limited processes using Accelerated Molecular Dynamics. On-the-fly kinetic Monte Carlo is combined with the Dimer
Method to find the saddlepoints exiting a valley, based on energetics from the Embedded Atom Method. With this technique, we treat two cases
involving diffusion-limited processes in situations with very low symmetry. First, we calculate the tracer diffusivities in ordered intermetallics as a function of composition and temperature. We investigate both a strongly ordered case (Ni3Al) and a less-strongly ordered case (Cu3Au). In the latter, we investigate especially the diffusivity near the order-disorder transition. Second, we treat the motion of an anti-phase boundary (APB) in the same intermetallics. We
demonstrate how an APB can be moved perpendicular to itself perpendicular to itself via vacancy motion. We conclude that the on-the-fly kMC is useful over most of the range. We have had to introduce some modifications to overcome the "blind spots" at certain
temperatures and compositions where the time boost would otherwise not be sufficiently significant.

The author acknowledges support from NSF.

14:30 Oral

Interdiffusion of two $L1_0$ phases without order decrease : experiments and molecular dynamics simulations

Christine Goyhenex[1], Romaric V. Montsouka[1], Mirosław Kozłowski[2], Veronique Pierron-Bohnes[1]

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The $L1_0$ ordered MPt(0 0 1) thin films (M = Fe or Co) are very interesting for perpendicular recording due to their magnetic anisotropy and magneto-optical behaviors. Nevertheless, in this type of recording, the problem remains, since the strong magnetic anisotropy involves a high coercive field, difficult to overcome with a miniaturized writing head. $(Fe\text{-}Ni)_{50}Pt_{50}$ thin films consist in a good compromise to decrease the coercivity and the Curie temperature keeping large enough remanent magnetization and magnetic anisotropy.

Epitaxial $L1_0$-ordered NiPt(0 0 1) / FePt(0 0 1) bi-layers were co-deposited on MgO(1 0 0) substrates by MBE. The $L1_0$ order parameter is high with the concentration modulation along the growth direction. Some $FeNiPt_2$(0 0 1) thin films were obtained by interdiffusion of the bi-layers. The $L1_0$ order parameter is conserved after interdiffusion, which can be explained by different mechanisms:

- a second neighbor jump,
- a six jump cycle mechanism,
- a double vacancy diffusion,
- a triple defect mechanism.

14:45 Oral

Stability and mobility of interstitial clusters in alpha-iron: ab initio and empirical potential calculations

Mihai-Cosmin Marinica, François Willaime

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Interstitial-type defects formed by the clustering of self-interstitials produced under irradiation have rather peculiar properties in alpha-iron. Very small interstitial clusters are formed of <110> dumbbells, whereas larger clusters have either a <111> or a <100> orientation. This contrasts with other BCC metals where they are predominantly <111>. The competition between these different orientations raises the question of their relative stabilities as function of size and temperature, and of the transformation mechanism from <111> to <100> orientations observed experimentally. Their mobilities are also a key issue in the interpretation of experiments. We have addressed these questions by combining first principles and empirical potential approaches.

We have determined the stability and mobility of small self-interstitial defects by ab-initio calculations performed on cells containing up to 250 atoms using the SIESTA code. We then used these results to fit and validate a new semi-empirical potential for iron. This potential allows to access dynamical properties and larger sizes. (i) Using the activation-relaxation technique (ART) we performed on small clusters a systematic search of configurations and migration/rotation mechanisms. (ii) Using lattice-dynamics we studied the vibrational properties of small clusters: low frequency modes have been evidenced in <111>-type defects ; the associated large vibrational entropy is shown to have a significant effect on the relative stabilities at finite temperature. (iii) In connection with experiments we have investigated the properties of loops with up to 1000 defects: the relative stabilities as function of Burgers vector, their migration energies, and the loop-loop and loop-surface interactions. We discuss the dependence on the potential of these results.

15:00 Oral

The stability of vacancy clusters in fcc crystals

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The present paper deals with the research of vacancy clusters stability in fcc metals Ni, Cu, Al by the method of molecular dynamics. The calculated block of an ideal crystal contained 27000 atoms in the model. Periodical boundaries were applied on its block. Interatomic interactions were described by pair central Morse potentials. Vacancy clusters were constructed by the removal of corresponding atoms from the calculated block. The following types of clusters were studied: pores, vacancy discs, stacking fault tetrahedrons, vacancy "tubes", which were constructed by the removal of the part of closely-packed atomic row, accidentally distributed single vacancies. The number of vacancies in every case varied from several units to several hundreds. The annealings of crystal block at the temperature from 0.5Tm to Tm during 100-500 ps with further cooling to the temperature of about 0 K were made to determine clusters stability. Diffusion coefficient and potential energy of the calculated block were calculated after cooling. Studied was the structure of crystal block by the visualizators of a distribution of the potential energy and atomic displacements. Vacancy clusters having tetrahedron form were more stable and energetically profitable. The sides of the tetrahedron were oriented along the planes <111> and looked like stacking fault defects, sides - as partial dislocations oriented along the directions <110>. All the types of the considered vacancy clusters tended to take a tetrahedron form. Similar stacking fault tetrahedra were stable up to the melting temperature. Pores were less energetically profitable than flat clusters. Diffusion at the presence of pores in the crystal took place mainly along the internal surface by so-called "rolling stone" mechanism. It was found that the bigger the size of a vacancy disk or a pore, the less mobilie it was. Consequently, the activation energy of cluster-migration-mediated diffusion was high.

15:15 Oral

Kinetics of point defects in hcp-Zr from first principles

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Zirconium based materials, which are widely used in the nuclear industry, have a particularly challenging microstructural evolution under irradiation because of their hexagonal close packed structure. Which are the configurations adopted by self-interstitial atoms (SIAs) among the non less than eight possible ones is not clear, and whether point defects exhibit or not diffusion anisotropy is an open question. We have investigated the structural and migration properties of vacancies and SIAs by combining calculations based on an empirical potential (EP) and on first principles (FP) methods. The latter are performed with the Density Functional Theory SIESTA code on cells with up to 385 atoms. The extensive study of the convergence as function of size and shape of the supercell of the SIA formation energies confirms the conclusion of previous DFT works according to which four SIA configurations are nearly degenerate in energy in Zr. We have in addition evidenced a new family of low-energy configurations, both within EP and FP calculations. These new configurations are shown to play a particularly important role: they are stabilized at high temperature by low-frequency vibrational modes and they are intermediate configurations for the migration mechanism between other configurations. We used the ART method (Activation Relaxation Technique) in order to search for other configurations and to determine in a systematic way the various possible migration pathways. The energy landscape which emerges from the FP calculation of the SIA migration barriers is compatible with internal friction experiments. The migration barrier of the vacancy within the basal plane is shown to be lowered by electronic effects, which yield a significant diffusion anisotropy (~0.15 eV) within FP calculations.

Coffee break

Tuesday afternoon, 5 September, 15:30

Microstructure, Layers and Thin Films

Tuesday afternoon, 5 September, 15:50

Chair: Cornelis Bos, Frederic Soisson, Pawel S. Zieba

15:50 Oral

Multiscale modeling of the interphase layer and its applications in mechanics of materials

Sergey A. Lurie, Natalia P. Tuchkova

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Continuum model of the interphase layer is developed as a model of media with kept dislocations. The basis of approach is the theory of

defects [Int J of Solids & Structures, 2005,v43, N1. 91-111], which includes classification of continuous media with defects, definition of different levels defects, investigation of defects generation and healing mechanisms. The multiscale interphase layer model is developed using variational kinematic principle [Int J Comp Mater Scs N3-4, 529-539]. The cohesion and adhesion interactions are modeled as the length scale effects. The model has been successfully applied to prediction/modeling of cohesion type interactions and both cohesion and adhesion superficial interactions[Int. J Comp Mater Scs; A., 2005, V36, N2. 145-152]. Mathematical model of the Barenblatt's cohesion field in fracture mechanics is given; existence of local cohesion fields near a crack tip has received the formal substantiation, new physical cohesion parameter is found as a fracture factors [In book: Analysis and Simulation of Multifield Problems, Springer, 2003; V12, 101-110]. For modelling of the mechanical properties of composites we use the different scale levels: the nano- and micro-mechanical scale, the scale of substructure (cell with inclusion, mono-layer) and the macro-scale (filled composites and layered composites as whole). The homogenization procedure [Comp Math And Math Phys.2006, V46, N7, 1318-1337] allows to predict the effective properties linking the different levels of modelling. Concept of the quantum-mechanical description of materials is formulated which allows to link characteristics of materials with parameters of potentials. Two new potentials are offered, which describe the cohesion interactions and Van der Waals interactions.

16:05 Oral

Electrochemical impedance spectroscopy simulations of multilayer systems: from nano- to mesoscale films.

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A model of electrochemical impedance spectroscopy (EIS) responses of multilayer (multiphase and multicomponent) systems is presented. EIS spectra are simulated by a numerical solution of the Nernst-Planck-Poisson problem. The method of lines with a finite difference spatial discretization is applied. The continuity and field equations are integrated by means of the Rosenbrock solver in Mathcad 12. The model can be applied to a system under phase transition in which the planar symmetry is retained. Depending on the thickness of segregated layers, different characteristics of impedance spectra are expected. From a single bulk arcs for nanoscale films to well defined bulk and diffusional arcs for thick films. A moderate behavior for mesoscale films is shown.The model may be applied for in-situ, non-destructive monitoring of transient processes from nano- to mesoscale.

16:20 Oral

Monte Carlo Simulation of Texture and Microstructure Transformation During Annealing Process of Steel

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Controlling texture and microstructure evolution during annealing process is very important for advanced steel. Theories to explain annealing process are complicated and always case dependent. Our recently developed simulation based Monte Carlo method offers an effective tool for studying annealing process and can be used to verify the arbitrarily defined theories that govern annealing process. The computer model is able to take Orientation Image Microscope (OIM) measurements as an input. The abundant information contained in OIM measurement allows the computer model to incorporate many structural characteristics of polycrystalline materials such as, texture, grain boundary, grain shape and size, phase composition, chemical composition, stored elastic energy, and residual stress. The outputs include various texture functions, grain boundary and grain size statistics that can be verified by experimental results. Graphical representation allows us to perform virtual experiments to monitor each simulation step of the structural transaction. An example of applying this simulation to Si steel is given.

16:35 Oral

3-D phase field simulations of grain growth in polycrystalline films

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Grain size, grain size distribution and grain orientation in polycrystalline films strongly influence their strength, electronic properties and durability. The rate of failure due to electromigration is, for instance, strongly correlated with grain size and grain size distribution.

Once the grain size is larger than the thickness of the film, grains become columnar with their grain boundaries parallel to the plane of the film. As a result, polycrystalline films show many features that are typical for 2-dimensional grain growth. However, surface energy, which is extremely important in thin films, introduces 3-dimensional aspects in the grain growth behaviour. Grooves are formed at the triple lines where grain boundaries meet the surface to balance surface energies and grain boundary energy. It has experimentally been observed that these grooves pin grain boundaries and can stop normal grain growth. Furthermore, the surface energy of the grains may depend on their orientation. The favourably oriented grains often have a high driving force for growth and break free from the pinning force of the grooves. This mechanism may provide the necessary additional driving force for abnormal grain growth (= secondary recrystallization).

A phase field model for grain growth that takes account of the sur-

face energy of the grains is presented and simulation results are discussed.

16:50 Oral

FE and MD simulation of InGaN QD formation induced by stress field of threading dislocations

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The stress induced diffusion process of In-Ga segregation in $In_xGa_{1-x}N$ layer deposited on GaN is simulated step by step by using a 3D nonlinear FEM. From the thermodynamical point of view this process is governed by the driving force induced by the gradient of residual stresses operating in an anisotropic nonlinear elastic structure [1]. The source of stresses we consider the set of threading dislocations examined in the plane view HRTEM investigation of GaN layer deposited on sapphire [2].

The chemical segregation obtained by FEM is used next in MD calculations based on the Stillinger-Weber potential. Since the size of the clusters is large, the cell must contain tens of thousand of atoms and only empirical potential methods are suitable to handle such large systems. The choice of the Stillinger-Weber potential is justified by the numerous results obtained with this potential for the analysis of extended defects in GaN [3]. A new parameterization has been defined by fitting to the crystallographic parameters, bulk modulus and elastic constants of InN.

Reference

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17:05 Oral

Why and how does hydrogen in silicon carbide thinfilms degrade the Ta barrier? - Large-scale ab initio molecular dynamics simulations of Ta growth

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Silicon carbide (SiC) films are often used to seal the highly porous ultra low-k polymers prior to the deposition of a Tantalum (Ta) diffusion barrier layer. However, hydrogen can not be eliminated because SiC films are fabricated by PECVD processes, which sometimes causes Ta layers to become an ineffective diffusion barrier against copper (Cu) diffusion. Through large scale ab initio molecular dynamic simulations, we show that such degradation is due to hydrogen atoms diffusing into the Ta layers and preventing Ta from forming crystalline dense films. These loose amorphous layers allow Cu atoms to diffuse into crevices. However, further simulations show that nitrogen addition to SiC thin films will strongly enhance the barrier effects of the Ta layers even in the presence of a high percentage of hydrogen. We predict that SixCyNz thin film is an excellent candidate to replace SiC.

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break

Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break

Wednesday afternoon, 6 September, 12:30

Simulation of Nanostructures

Wednesday afternoon, 6 September, 14:00
Chair: Marek Muzyk, Murray S. Daw, Sergey A. Lurie

14:00 Invited oral

Understanding materials at the atomic scale: first-principles molecular dynamics simulations of nanostructures and disordered systems

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Precise knowledge of the atomic structure is a crucial prerequisite to gather reliable information on the link between microscopic and macroscopic properties of a material. We have resorted to molecular dynamics based on an accurate description of the total energy and forces, directly derived from a density functional expression of the relevant interactions (first-principles molecular dynamics). A collection of results will be presented on nanostructures (isolated clusters) and disordered systems (liquid and glasses), for which no clearcut information in direct space is experimentally available. Two issues will be addressed in detail. First, the determination of a threshold of dynamical instability for silicon-doped heterofullerenes. We have

analyzed the mechanism leading to fragmentation of these clusters with increasing temperature. As a second issue, we shall focus on the structural properties of network-forming liquid and glasses characterized by intermediate range order. We were able to associate specific structural units to peculiar behaviors observed in the partial structure factors for low values of the reciprocal vector. These low values correspond to correlations establishing on distances well beyond nearest neighbors. From the methodological point of view, features common to the two classes of systems will be underlined, thereby highlighting the predictive power of first-principles molecular dynamics.

14:30 Oral

Interface shape change and shift kinetics on the nanoscale

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It is known form Fick's phenomenological laws that in a diffusion couple the displacement of a plane with constant composition (or an abrupt interface) is proportional to $t^{1/2}$. However, we have shown first from computer simulations that this rule can be violated on the nanoscale either in completely [1] or restricted miscible systems [2]. This is strongly related to the discrete character of the system on the nanoscale and to the highly neglected fact in the literature that the diffusion coefficients or mobilities depend on the composition. As we have shown recently, these studies may help to understand the atomistic meaning of the kinetic transfer coefficient in solid state reactions. [3]

Computer simulations also have shown that on the nanoscale, for strongly composition-dependent diffusion coefficients, diffuse interfaces can sharpen rather broaden in completely miscible binary systems during annealing [4]. This sharpening is surprising, because the direction of diffusion is always opposite to the direction of the composition gradient. This phenomenon could provide a useful tool for the improvement of interfaces and offer a way to fabricate of e.g. better X-ray or neutron mirrors, microelectronic devices or GMR multilayers.

These phenomena predicted by computer simulations have been later proved experimentally as well [5-6]. This shows the efficiency and importance of the computer modeling in planning and fabrication of new devices.

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14:45 Oral

Single layer colloidal films on nanopatterned substrates

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Single layer colloidal films are of great scientific and technological interest in fields like heterogeneous catalysts, photonic crystals, and quantum dots.

Physical properties of these films are quite sensitive to the morphology of the film. The latter can be significantly modified by introducing geometrical restrictions, during the irreversible adsorption of particles on the substrate, which we take for the sake of simplicity to be a regular array of square regions of size α and a distance β apart from each other.

To this end, we perform an extensive Monte Carlo study of the irreversible deposition of colloids on substrates consisting of 1000×1000 square-cells, which can be experimentally built through lithographic methods.

For the jamming state, where no more colloidal particles can be adsorbed, we study the dependence of the radial distribution function of the distance between colloids on the above two parameters.

The sequence of peaks appearing in the radial distribution function is closely related to the distance between cells while the dispersion of each peak is related to the size of the cells.

Therefore, proper control of the α and β parameters can have as a result an order or disordered colloidal film.

To summarize our findings, we construct a diagram where we categorize the jamming state into four different cases according to the number of colloids that can be adsorbed per cell (single-particle or multiparticle) and the type of interaction between colloids adsorbed in neighboring cells (non-interacting or interacting).

Our study shows the relevance of geometrical constraints to obtain different patterned colloidal monolayer films with potential for practical applications.

15:00 Oral

Multiscale modelling of mass and charge transport in electrochemical and biological systems

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The Nernst-Planck-Poisson equations (NPP) are used to describe transport of ions in electrochemical and biological membranes. The nature of electrochemical processes involve critical phenomena near the boundary of the membrane (at distances of the Debye length)

while the thickness of the membrane can vary up to milimeters. Different range of lengths from nano- to microscales implies difficulties in numerical simulations. Effective solutions of the time-dependent (transient-state) and time-independent (steady-state) NPP problem are presented for one dimension geometry.

The time-dependent form of the Nernst-Planck-Poisson equations can be used both for transient and steady-state calculations. Steady-state analysis is obtained by starting from an initial profiles, and letting the numerical system evolve until a stationary solution is reached. However it is not always obvious if the state, which has been achieved, is really the steady-state. We have devised a method for verification whether time-dependent NPP system reached its steady-state. This approach requires the solving a steady-state NPP problem with Dirichlet type boundary conditions.
Moreover the steady-state problem is interesting by its own. We will also presnt solution for Neumann like boundary conditions, which with the NPP set of equations allows to predict steady-state boundary values of the concentrations and electrical potential in the membrane.

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster H-1

Point defects and their influence on thermoactivated disordering process of Ni3Al intermetallic

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The influence of point defects on thermoactivated disordering of Ni3Al intermetallic was studied. Computer simulation was made by the method of molecular dynamics. It was found that the temperature of the beginning of diffusion process causing the appearance of disordering areas decreased. In this connection, the extent of decreasing was directly connected with the level of local stresses which caused the presence of point defects in the crystal. It concerned, first of all, a proper interstitial atom and interstitial atoms of Frenckel pairs. The temperature of the beginning of superstructure diffusion reconstruction corresponded to 50-100 K for Ni3Al intermetallide. Then, it followed a big temperature interval - about 600 K. No considerable diffusion transformations were observed. If the crystal structure had only vacancies, diffusion was noticed only at about 400 K. Vacancies began to definitely contribute to a disordering process at higher temperature. The contributions of every type of point defects to a disordering process increased while melting point was approached. It is worse to note that dynamical Frenckel pairs played an active part in a disordering process in the case of an ideal crystal. Frenckel pairs had short lifetime at the first stages and recombined rapidly. Then, a process of their aggregation was observed. The complexes in a form of dislocation dipoles and loops were formed at the aggregation of interstitial atoms. The complexes displaced collectively at thermal activation. They formed cooperative displacements of a whole group of atoms including the presence of rotary modes. The effect of unification of point defect groups and their collective participation in a diffusion process of disordering can be characterized on the basis of self-organization concept of the system in a dynamics of thermal activation process.

17:20 Poster H-2

Atomic and meso-scale simulation of the diffusion features of point defects.

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This work is devoted to the development of Moleculas Statics model of the diffusion features of point defects in bcc metals. In particular, the proposed model allows to calculate activation volumes which describe the influence of pressure on the diffusion processes in solids. Our model realizes a new approach that makes it possible to self-consistently determine atomic structure near vacancy and constants C characterizing the displacement of atoms in an elastic matrix around computational cell. We also take into account that the energies of a perfect system and a system with a defect depend in different ways on the outer pressure. This modifies the values of the migration and formation volumes. The effect is usually neglected in calculation although it may comprise a considerable part of the activation volume. Therefore, we take into account that the time an atom needs to jump to the vacancy covers a few oscillations in the lattice site and thus, we carry out the relaxation of only those atoms which are located in a distance less then four lattice parameters from a defect. Such a distance was chosen on the basis of the results obtained from Molecular Dynamics. The formation and migration energies and volumes were calculated for the vacancies and the interstitials in various bcc metals.

17:20 Poster H-3

Simulation of interstitial atom diffusion in fcc metals with point defects

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This work is devoted to simulation of interstitial atom diffusion in fcc metals with point defects. these defects are intacted with each other that gives rise to the formation of a complex defect, which can alter the surrounding atom configuration and consequently the local magnitude of the activation barrier for the jump of interstitial atom as for instance carbon or hydrogen. This problem is investigated by a combination of two simulation methods: molecular static method and Monte Carlo method. In the present study we used the molecular static method to model the activation barrier set for different configurations interstitial atom - vacancy. Knowing the activation barriers, it is possible to calculate the jump rates. Using these jump rates and basing on the Monte Carlo method, it is possible to model the interstitial atom migration. The entry parameter is only a potential function for the systems Me-Me, Me-C and Me-H. In the present study the variation of the interstitial atom diffusion coeffcient with temperature is computed. In particular, the calculations were related to the systems nickel-carbon and nickel-hydrogen. The simulation shows that the interference between vacancy and interstitial atom affects the acceleration of interstitial atom diffusion in the range of the investigated temperatures.

17:20 Poster H-4

Monte Carlo and Molecular Dynamics Simulation of Ordering and its Effect on Elastic Parameters in Ni-base Alloys

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Ordering occurs and is of practical importance in many groups of structural materials. Namely, ordering in Ni-based superalloys is the process controlling the development of the characteristic two-phase microstructure and subsequently the mechanical properties. The aim of this paper is to study the effect of ordering processes on elastic parameters of model multicomponent Ni-base alloys. System ordering at elevated temperatures was simulated using a Monte Carlo approach on face centred cubic lattice with phenomenological pair potentials and Kawasaki dynamics. Selected atomic configurations with various degree of order were subject to calculation of elastic parameters using molecular dynamics approach with fluctuation formula. For these calculations, Materials Explorer software was used with modified embedded atom method interatomic potentials. Changes of elastic parameters due to ordering in face centred cubic lattice are discussed.

17:20 Poster H-5

Analysis of the time-dependence of the segregation of oxygen at metal-ceramic oxide interfaces using lattice Monte Carlo and finite element methods.

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The presence of atomic oxygen at internal metal-ceramic oxide interfaces significantly affects the physical properties of the interfaces which in turn affects the bulk properties of the material. We address this problem for the case of a constant source of oxygen at the surface and periodic and stochastic arrangements of ceramic oxide particles (MgO) embedded in a metal matrix (Ag). We simulate the time-dependence of the oxygen concentration depth profiles into the material using a newly developed lattice Monte Carlo method and a previous finite element method, the latter making use of an extended commercial code. There is very good agreement between the results of these numerical methods.

17:20 Poster H-7

Materials hardness estimation by simulation of the indentation process

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In the present study Finite Element Method (FEM) has been applied for modelling of indentation process in polycrystalline metals. Two different model of the material, with linear and non-linear hardening, has been assumed in calculations. The indentation process has been quantitatively described by a load-depth curves. The hardness values were obtained from the unloading part of such curves, which are responsible for plastic deformation of the material during indentation process. In addition, the geometry changes of the indent after deformation has been analyzed.

The results show that simulation techniques, used in the present study, are useful in estimation of materials hardness. It has been also found that specific combination of materials properties such as hardening exponent, hardening coefficient, tangent modulus and yield stress may result in sink-in or pile-up phenomenon. This processes may influence the indent geometry and hardness measurements consequently.

17:20 Poster H-8

Stability of hollow nanospheres: a Molecular Dynamics study

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Recently, a method of fabricating hollow nanospheres of cobalt selenide and cobalt sulphide has been announced (Y. Yin et al. Science Vol. 304, p711 (2004). We have investigated the stability and mechanism of collapse of hollow nanospheres by the method of Molecular Dynamics. First of all, we have analyzed single metallic systems exemplified by Pd. We found that for small hollow nanospheres up to about 10 000 atoms, the spheres collapsed quickly but not by vacancy-assisted mechanisms but by a mechanism involving Shockley

partial dislocations. For larger hollow nanospheres, too much energy is required for that mechanism and the most likely mechanism for sphere collapse, now far slower, involves the vacancy mechanism.

17:20 Poster H-9

Thermal properties of $L1_0$-TiAl from first principle calculations

Marek Muzyk[1], Robert Sot[1,2], Krzysztof J. Kurzydlowski[1]

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First principle phonon calculation have been used to study $L1_0$-structure TiAl intermetallic compound. The lattice constant and bulk modulus as function of temperatures were computed with use of the ab initio pseudopotential and direct method. The computations requires optimization of the structure, calculation of the Hellmann-Feynman forces, and construction of the dynamical matrix. A large number of crystalline systems optimized under different pressures have been studied in terms of the phonon density of states, and thermodynamic functions.

17:20 Poster H-10

First principles study of Al(100) twisted interfaces

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The first principles density functional calculations have been performed for a number of Al(100) twisted interfaces with the purpose to investigate the most stable atomic structures. Orientation dependence of interface energy normalized to bulk is established and discussed. The obtained results can be used to model grain growth in molecular dynamics studies.

17:20 Poster H-11

Predicting site occupation of Fe in B2 NiAl from ab initio computations

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This work reports the lattice site preference of Fe in B2 NiAl. Our computations show that, at T = 0 K, Fe always preferes the Ni sublattice in B2 NiAl, with such a preference being quite strong in Al-rich NiAl, but relatively weak in Ni-rich and stoichiometric NiAl. To predict the site occupation of Fe in B2 NiAl at finite temperature, a statistical-mechanical Wagner-Schottky model was used, with the formation enthalpies of point defects being determined from our present first-principles computations. We also report on the effect of the substitution of Fe on the elastic properties of the Ni(Al,Fe) and (Ni,Fe)Al.

17:20 Poster H-12

Interface dynamics of melt instabilities on semiconductor surfaces

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The processes that take place in the pulsed laser radiation of semiconductors are essentially non-equilibrium, which causes appearance of new physical phenomena. The nonuniform temperature fields define the peculiarities of melting processes and semiconductors surface relief in the zones of light flux action. At uniform excitation of semiconductors by laser radiation with prethreshold power the locally melted region are formed on irradiated surfaces. It is induced by thermo diffusive instability of distribution of uniformly generated EHP. Therewith the forms of locally melted region give rise to a great variety of interesting patterns. The realization of instability during the melt processes on the interface within a wide interval of wave numbers and the essential instability of the whole process of shaping and next to the reproduction of self similar forms on the interface in smaller scales can by described within the framework of the problem of Laplacian growth. The mathematical model to the surface dynamics, when the instability of the front of melt arises along a chosen wave vector, is proposed. The results of computer simulation of interface dynamics of solitary melted region on the base of this mathematical model fit the experimental data and indicate the fractal character of their formation.

17:20 Poster H-13

Grain Boundary Migration in Nanocrystalline Iron

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The migration of grain boundaries in polycrystalline materials is driven by a reduction of the internal energy. This can be achieved either by decreasing the surface area of grain boundaries or by reducing their specific energy (energy per surface unit). Diffusion controlled reduction of the interface curvature as well as the rotation of

grains could be considered, respectively. However, due to the kinetic barriers for rotation of the grains in polycrystalls, this process is not observed in coarse grained materials. However, in nanomaterials it could play an important role in the process of grain growth.

In this study a series of 3D models for curved grain boundaries in pure iron have been built. Each model consisted of a spherical grain, with an initial size of about 9 nm, surrounded by a large single-crystal. Different orientations have been assigned to the grain and the matrix in order to obtain interfaces with misorientation angles in the range of 5-45 degrees in steps of 5 degrees. The molecular dynamics with EAM potential have been performed for investigation of temporal changes in grain boundary migration and rotation. The relation between grain size, grain boundary misorientation has been revealed. It was also found that the density of the material decreases with a reduction of grain boundary area.

The effect of a triple junction on the interface motion has been also studied by introducing a bi-crystal matrix instead of a single-crystal matrix.

The results are discussed in terms of grain growth investigations in nanometals.

17:20 Poster H-14

The research of thermoactivated changes of the structure of antiphase boundaries in Cu3Au and Ni3Al alloys

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It is known that physical and physics-mechanical properties of ordered alloys and intermetallides are connected with the peculiarities of a thin structure of antiphase boundaries (APB). The changes of atomic order near APB <110>{100} and ½<110>{111} in L12 superstructure on the examples of Cu3Au and Ni3Al alloys were studied in the paper. The definite type of APB were introduced in the calculated block of three-dimensional crystal containing 2×105 atoms. Periodical boundary conditions were applied outside the block. Interactions between different pairs of atoms were given by Morse potential. The definite configuration of vacancies was introduced in the system accidentally. Their distribution over the sites was regulated by the condition of keeping of stoichiometric composition A3B. Then the atoms were allowed to occupy vacant sites. Activation of atomic jumps in the dependence on temperature was simulated on the basis of the Monte-Carlo method. The change of the energy of APB formation and local distribution of energy over the planes related to the boundary were observed upon reaching the definite dynamical equilibrium by the crystal. Observed was the distribution of atoms of alloy component, the change of long and short order over the planes which were parallel to a boundary. The extent of the area with transition order with respect to a boundary plane increased with the growth of temperature. Faceting of APB relief took place at the expense of the precipitation of point displacement defects. Main contribution in deformation strengthening is made at the expense of structural reconstruction and changing of order near antiphase boundaries at relatively high temperatures. In this connection, the energy of APB formation decreases with the growth of temperature and washing of a crystal structure near APB is accelerated with the growth of vacancy concentration.

17:20 Poster H-15

Multiscale Plastic Deformation Near Fatigue Crack From Diffraction

Rozaliya I. Barabash[1,2], Yunan Sun[2], G E. Ice[1], Peter Liaw[2], Hahn Choo[1,2], David Brown[2]

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The multiscale plastic deformation in the vicinity of the crack tip was studied with neutron and x-ray microbeam diffraction. The results help to understand the overload effect, which induced large plastic deformation causing hierarchical dislocation arrangement around the crack tip. From neutron diffraction measurement anisotropic line broadening was observed in front of the crack tip. Furthermore, Laue patterns obtained from microbeam diffraction at different locations near the crack, provide a better spatial resolution and show alternating regions with high and low dislocation density. Orientation dependence on (*hkl*) of both strains and line broadening was observed indicating non random dislocation arrangement. Dislocation density and arrangement was evaluated from the line width and profile behavior for several crystallographic families. Lattice strains were analyzed by GSAS. Comparison of the results obtained with GSAS and line profile analysis allowed to understand the change of hierarchical dislocation density distribution and strain in different plastic zones. Overall, the dislocation density was found to decrease with the distance from the crack tip.

17:20 Poster H-16

Statistical model of grain growth in polycrystalline nanomaterials

Thomas B. Tengen[3], Tomasz Wejrzanowski[1,2], Radosław Iwankiewicz[3], Krzysztof J. Kurzydlowski[1,2]

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Nanomaterials, due to their fine grain size, exhibit enhanced mechanical properties. However, their low stability at relatively low temperatures might limit their future applications.

In the present work, a statistical model has been proposed in order to study grain growth processes in nanomaterials. The Hillert's approach has been extended by incorporating two mechanisms of grain growth for an individual grain: grain boundary migration (diffusion based - continuous) and grain rotation (discontinuous). The influence of the grain size distribution on the grain growth process has been studied.

It has been shown that higher rate of rotations results in aberration from the parabolic law of grain growth. Such a deviation has also been observed experimentally, especially in grain growth in nano-materials. The results reveal that growth rate increases with higher dispersion of the fine grains and the rotation mechanism can initiate growth even with low dispersion. This causes a steady increase in the coefficient of variation which, after some time interval, decays to homogeneity. It is also found that the varying misorientation angle affects the mobility constant.

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium I

Welcome

The aim of this Symposium will be to gather specialists and interested researchers in the field of phase diagrams, phase transformations, computational thermodynamics, modeling and properties of materials. Invited speakers will address topics such as phase diagrams opportunities and modeling, low temperature phase stabilities, magnetic refrigeration, phase separation and atomic ordering in semi-conductors, and other materials developments. Leading specialists in this general area will discuss development of models aiming at calculation of thermodynamic properties for various phases in binary and multicomponent systems. Contributions related to the measurements of phase properties and stability are invited. Special attention will be given to phase transitions in the liquid and solid state, and their applications in various industrial and technological processes. Application of diffusionless and diffusional processes will be discussed from the point of view of recent metallic materials studies and development, such as amorphous materials, ordered structures, and metastable structures. Contributions are also invited in the area of theoretical calculations, especially in the field of first-principles calculations and their experimental verification through measurements of thermochemical and phase equilibrium data. The application of thermodynamic data and phase diagrams in the design of industrial processes are also of interest to this symposium. Among the currently invited speakers are Profs. Karl Gschneidner, David Laughlin, Subhash Mahajan, and .L.L. Rokhlin. The Symposium will be dedicated to the Jubileum of Professor Thaddeus Massalski of Carnegie Mellon University, who will receive the 2006 Czochralski Prize, and will present the introductory lecture.

The topics of interest include, but are not limited to:
- Calculation and determination of phase equilibria
- Phase transitions and metastable phases in the solid and liquid state
- Practical application of phase diagrams

The manuscripts should be delivered at the beginning of the Symposium.

Scientific Committee:

Livio Battezzati (University of Torino, Italy), Yves Bienvenu (Ecole des Mines de Paris, France), Arkadii V. Dobromyslov (Ural Branch RAS, Ekaterinburg, Russia), Cezary Guminski (Warsaw Univesity, Poland), Karl A. Gschneidner, Jr. (Ames Laboratory, USA), Kiyohito Ishida (Tohoku University, Japan), David E. Laughlin (Carnegie Mellon University, USA), Subhash Mahajan (Arizona State University, USA), Uichiro Mizutani (Toyota Research Institute, Nagoya, Japan), Tetsuo Mohri (Hokkaido University, Japan). Henryk Morawiec (Silesian University, Katowice, Poland), Krzysztof Parliński (Institute of Nuclear Physics PAS, Kraków, Poland), Lazar L. Rokhlin (Baikov Institute RAS, Moscow, Russia), Rainer Schmid-Fetzer (Technical University Clausthal, Germany), Vladimir Sima (Charles Univeristy Prague, Czech Republic), Wojciech Suski (Institute Low Temp. Physics PAS Wrocław, Poland) Tamara Velikanova (I.N. Frantsevich Institute for Problems of Materials Science, Kiev, Ukraine), Leszek Zabdyr (Institute Metallurgy and Materials PAS Kraków, Poland)

Organisers

- **Prof. J. Dutkiewicz**, IMIM, Polish Academy of Sciences, Krakow, Poland
- **Prof. Z. Moser**, IMIM, Polish Academy of Sciences, Krakow, Poland
- **Prof. Thaddeus Massalski**, Carnegie Mellon University, Pittsburgh, USA
- **Lidia Lityńska**, IMIM, Polish Academy of Sciences, Krakow, Poland (Symposium Secretariat)

Proceedings

It is intended to publish some of the contributions to this Symposium in the Polish Journal **"Archives of Metallurgy and Materials"** covered by the Institute of Scientific Information USA.

Programme

Monday, 4 September

Opening Ceremony

2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Experimental Thermodynamics and Phase Equilibria I
Monday afternoon, 4 September, 14:00
Chair: Tadeusz B. Massalski

14:00	Invited oral

The Theory of Phase Formation in Rare Earth Metal Systems

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The seventeen elements which make up the rare earth family consist of two non-4*f*-electron members, Sc and Y, and the fifteen 4*f* electron containing lanthanide elements. In general the physical properties, which do not directly involve the 4*f* electrons, vary in a smooth and regular manner, especially for the lanthanide group. These include: the metallic radii, electronegativities, melting points, and crystal structure sequence of the metallic elements. By utilizing this sytematic behavior, we have learned a great deal about solid solution

formation and the crystal chemistry of intermetallic compounds of, not only the rare earth elements, but also the other elements in the periodic table. Furthermore, the application of systematics has demonstrated 4*f* electron hybridization with the valence electrons in metallic and non-metallic systems, and also lead to a method for predicting heats (free energies) of formation of rare earth compounds.

14:30 Oral

Thermodynamic modelling in the ZrO2-La2O3-Al2O3 and ZrO2-La2O3-Y2O3 systems

Olga B. Fabrichnaya, Chong Wang, Matsvei Zinkevich, Fritz Aldinger

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The thermodynamic parameters for the ZrO2-La2O3-Al2O3 system are assessed using phase equilibrium data. The thermodynamic description of the ZrO2-La2O3-Y2O3 system is derived from binary descriptions. The compound energy formalism and two-sublattice ionic liquid model [01Hil] are applied to describe phases in these systems. Thermodynamic parameters for the ZrO2-La2O3 system are assessed taking into account triangulation in the ternary system of ZrO2-La2O3-Al2O3. Phase equilibria between solid phases in the ternary system ZrO2-La2O3-Al2O3 constrain thermodynamic parameters in the binary system ZrO2-La2O3. Ternary interaction parameter is assessed for the liquid phase using experimental data on liquidus surface of [05Lak]. Isothermal sections at 1523, 1923 K, liquidus surface and several vertical sections are calculated and compared with experimental data of [05Lak]. It should be pointed out that the general character of liquidus surface is reproduced in calculations. However, the calculated primary crystallisation field of pyrochlore phase is wider than obtained in experimental study of [05Lak]. This results in several inconsistencies between calculations and experimental data. New experimental determination of thermodynamic properties of pyrochlore is necessary to resolve the present inconsistencies.

The isothermal sections and liquidus surface are calculated for the ZrO2-La2O3-Y2O3 system. Experimental data are very scarce for this system. The calculated diagrams are useful for planning new experiments in this system.

[01Hil] M. Hillert: J. Alloys Comp. 320 (2001) 161-176.

[05Lak] S. Lakiza, L.M. Lopato: J. Eur. Ceram. Soc. 25 (2005) 1373-1380.

14:50 Oral

Calorimetric studies of the enthalpies of formation of $NiTi_2$, NiTi and Ni_3Ti

Zbigniew Moser[1], Władysław Gąsior[1], Krzysztof Rzyman[2], Adam Dębski[1]

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Solution calorimetry operated with liquid Al bath has been used for determination of enthalpies of formation of intermetallic compounds of the Ni-Ti system needed for extensive studies on quaternary system Al-Fe-Ni-Ti within COST 535 program on advanced aluminates. At first, there were obtained enthalpies of solution of Ni, Ti and Fe in liquid Al amounting respectively: -150 ±0.4 kJ/g.atom for Ni, -128.8 ±0.7 kJ/g.atom for Ti and-120.0 ±0.6 kJ/g.atom for Fe. Using these values of enthalpies of solution, the following values of enthalpies of formation were obtained: -25.3 ± 1.7 kJ./mol of atoms for $NiTi_2$, -31.1 ± kJ/mol of atoms for NiTi and -43.8 ± 1.6 kJ/mol of atoms for Ni_3 Ti, respectively. The resulting values are in good agreement with literature data, both experimental and from *ab initio* calculations.

15:10 Oral

Phase Diagram for the Tl_2Te - In_2Te_3 System Determined by Thermal Analysis and Concentration Cell EMF Measurements

Zbigniew Sztuba, Katarzyna Wiglusz, Ewa Zaleska, Igor Mucha, Wieslaw Gawel

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From the data obtained in TA and electrochemical studies, the phase diagram for the system thallium(I) telluride - indium(III) telluride was constructed. It follows from the diagram that in the system:

- a compound is formed of component molar ratio Tl_2Te : In_2Te_3 = 5:6 to which a formula $Tl_{10}In_{12}Te_{23}$ may be ascribed.
- another compound is formed of component molar ratio Tl_2Te : In_2Te_3 = 4:3 to which a formula $Tl_8In_6Te_{13}$ may be ascribed;
- the compound $Tl_8In_6Te_{13}$ forms terminal solid solution (γ) on the $Tl_{10}In_{12}Te_{23}$ matrix 5.5 mol. % wide i. e. from 49.0 mol. % In_2Te_3 to 54.5 mol. % In_2Te_3;

thallium(I) telluride forms terminal solid solution (α) in a peritectic reaction at 723.2 K with the compound $Tl_8In_6Te_{13}$ within the range 0 - 30 mol. % In_2Te_3;

- the compound $Tl_{10}In_{12}Te_{23}$ and In_2Te_3 form eutectic of composition 84.5 mol. % In_2Te_3 and melting point 922.8 K, and a terminal solid solution (β) 5 mol. % wide.

Coffee break

Monday afternoon, 4 September, 15:30

Experimental Thermodynamics and Phase Equilibria II

Monday afternoon, 4 September, 15:50
Chair: Karl A. Gschneidner Jr

15:50 Oral

Titanium-boride eutectic materials: Phase diagrams and properties

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Titanium boride TiB has taken a position of promising phase for development of titanium-matrix composites with advanced strength properties. As a scientific background, we investigated alloy constitutions and properties for ternary Ti-Al-B and Ti-B-*X*, quaternary Ti-Al-B-*X* (where *X* was Si, Ge, Sn, Zr, V or Nb, and the Al content was 10 at.%). As-cast and annealed samples arc nelted were studied by optic microscopy, SEM/EPMA, XRD, DTA, Vickers hardness up to 800°C and other tests.

A specific titanium-boride eutectic (Ti) + (TiB) structure was found to be almost unaffected by alloying additions, comparing to the binary Ti-B system. Study of phase equilibria in the Ti-rich portions showed that *p*-elements (Al, Si, Ge and Sn) dissolve fully in matrix; *d*-elements (Zr, V and Nb) partition comparably between metal matrix and boride phases.

The *p*-elements increase hardness of titanium-boride eutectic alloys in the whole temperature range under study, and the *d*-metals increase it up to 400-500°C (at higher temperatures the hardness degraded). The *p*-elements increase the onset temperature of sharp softening to about 600-650, and the *d*-metals decrease it to 400-500°C.

The boride based on TiB was shown to be quite attractive reinforcement for titanium-matrix composites. The obtained results open up possibilities to adapt alloying to possible applications.

16:10 Oral

The application of thermodynamic calculations for the semi-solid processing design

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Semi-solid metal processing is now a commercially successful manufacturing route producing lots of near net-shape parts. One member of such processing technologies is thixoforming which is based on the special behaviour of alloys with non-dendritic microstructure in the partially liquid state. The semi-solid processing design requires the use of faithful values of thermodynamic material properties. Thermodynamic modelling is a potential tool for predicting alloy compositions suitable for thixoforming. Thixoformable alloys must have a wide melting range with some additional feature. Namely, the slope of the curve of the liquid fraction versus the temperature should be low at the liquid fraction value of 40%. The data from thermodynamic calculations might be also applied in the industrial thixoforming processes design. It is very helpful to find different thermal properties of alloys in order to simulate precisely the temperature distribution inside the formed materials. The thermodynamic calculations allow one to find the heat transfer coefficient, the specific heat value and the latent heat value. The purpose of the present paper is to provide examples of the numerical modelling of the thermodynamic properties for commercial, industrial Al alloys. All the calculations in the present work are performed using JMatPro software. The determination of the phase composition for multi-component alloys is originally based on the Gibbs energy minimisation. Furthermore, the conditions of the non-equilibrium solidification are determined using the Scheil-Gulliver equation. An additional advantage of the software used is the possibility of calculation of the formation conditions for metastable phases which have also an influence on the mechanical properties of the alloys.

16:50 Oral

The determination of solubility limit of indium impurity in PbTe<In> thin films on Si substrates

Alexander M. Samoylov, Yuri V. Synorov, Alexander M. Khoviv, Igor S. Surovtsev

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Under unfluence of the presence of III A group metals the electrical parameters of PbTe and its solid solutions can change significantly. However, some fundamental problems of the formation, saturation, and influence of quasi-local impurity levels in In-doped PbTe have not been resolved yet.

The main purposes of this study are to discuss the experimental results, which have been received during the examination of the chemical quantitative composition and the real crystal structure of PbTe<In>/Si and PbTe<In>/SiO_2/Si heterostructures, and to evaluate the solubility of In atoms in lead telluride thin films at different temperatures.

In this work the method of fabrication of PbTe<In> heterostructures offers the direct HWE one-stage synthesis, in which the doping and the condensation process proceeds simultaneously. On the basis of SEM observation, XRD, and EPMA experimental results the boundary of indium's limited solubility region in lead telluride matrix has been drawn. From the point of view of Gibb's compositional triangle of Pb - In - Te ternary system, one can make a conclusion that the analysis of the PbTe - InTe pseudobinary cross section only, as it have been done earlier, does not explain, in general, the phenomena of indium's solubility in PbTe crystal structure. For fundamental understanding of the formation of In solid solutions it is necessary to considerthe PbTe - In_4Te_3 and PbTe - In_2Te_3 polythermal cross-sections also.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Properties Relations to Phase Diagrams

Tuesday morning, 5 September, 9:00
Chair: Subhash Mahajan

9:00 Invited oral

The Regularities in the Mg-rich Parts of the Phase Diagrams, Phase Transformations and Mechanical Properties of Magnesium Alloys with Separate Rare Earth Metals

Lazar L. Rokhlin

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Some of the rare earth metals (RE) are known for a long time as favorable additives in Mg alloys for improvement of their strength. There is significant difference between effects of separate rare earth metals on strength of magnesium connected with regular change of the respective phase diagrams. Regularities of the phase diagrams of separate rare earth metals with any other metal were shown in many works. However, there is a specific feature of Mg systems. It consists of existence of solubility of RE in solid Mg which changes regularly with increasing the RE atomic number. This regularity is caused by favorable differences between atomic radii of separate RE and Mg. The higher solubility corresponds to less difference between atomic radii of RE and Mg. Nevertheless, solubility of RE of the yttrium subgroup in solid Mg is significantly more than that of RE of the cerium subgroup suggesting different electron interaction between Mg and RE of different subgroups when Mg solid solutions are formed. Decrease of the RE solubility in solid Mg with lowering temperature creates a possibility of Mg supersaturated solid solution decomposition accompanied by strengthening effect. Behavior of Mg alloys with RE of different subgroups during this process are significantly different. Solubility of RE in solid Mg and Mg supersaturated solid solution decomposition are responsible for high strength properties of Mg alloys with RE. With increasing solubility of RE in solid Mg the strength properties increase, but up to the first elements of yttrium subgroup (Gd, Tb). Although the separate RE are used as alloying elements in Mg alloys for a long time there are possibilities to develop new alloys with better strength. This can be reached by alloying Mg with RE which were not used so far and by addition of RE of yttrium and cerium subgroups together in certain ratios.

9:30 Oral

Continuous casting and processing of copper alloys containing tin : an illustration of the applicability of phase stability rules and data

Yves C. Bienvenu[1], Fayçal Sadi[1], Roland Bailly[2], Frederic Bacou[2]

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Copper alloys with up to 9 wt% Sn are used in the automotive industry and in electrical/electronic applications for their electrical, mechanical and tribological properties in the form of hot and cold rolled thin strip.
An other application in Europe is the "Nordic Gold" monetary alloy, used in 10,20 and 50 eurocents coins. It is a quaternary Cu, 5Zn, 5Al, 1Sn (all wt%) alloy whose merit is first not to contain any nickel and second to combine specific physical properties, a gold like aspect and a reasonable corrosion resistance. The composition was also selected in view of its poor processability to limit counterfeiting. Companies involved in the late 90's in the production of the large quantities of Nordic Gold met indeed with great difficulties to produce it economically, that is starting with vertical semi continuous casting.
The presentation will first address the continuous casting of tin containing alloys. A succession of peritectic transformations involving fragile intermetallics and an unusually large solidification temperature interval in the Cu-Sn phase diagram explains the microsegregation related difficulties. Differential thermal analysis of solidification and EPMA microanalysis of solidified products led to a modified Scheil type modelling of microsegregation ending with the formation of interdendritic tin rich phases. The large tin contents of the last liquid and the stresses on the first solidified skin leads in turn to the "inverse" macro-segregation of tin and eventually to the blocking of the vertical continuous casting which inclines the industry to select the horizontal continuous casting of thin (2 cm) slabs associated with a lower productivity and material yield.
The presentation will then deal with the processing steps following casting during which tin (and to a lesser the other solutes) creates a risk of "burning" during reheating and a risk of crack initiation on fragile intermetallic phases during hot rolling. This is especially true for the monetary Nordic Gold alloys owing to a composition and to an electron per atom ratio which places it at the limit of alpha phase stability especially when microsegregation is active. Ternary phase diagram information and a laboratory study guides processing optimisation.

9:50 Oral

Phase and structural transformations in Ti-Ta alloys in wide region of compositions

A. Dobromyslov[1], Galina V. Dolgikh[1], Jan M. Dutkiewicz[2], Tamara L. Trenogina[1]

1. *Russian Academy of Sciences, Ural Division, Institute of Metal Physics, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation* **2.** *Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland*

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The effect of alloy composition on the phase transformation and the microstructure of Ti-Ta alloys were investigated in detail in the present study. Alloys of eight different compositions in range of 1 to 40 at. pct Ta were quenched from β-region in ice water. The phase state and the resulting microstructures were investigated using light metallography, transmission electron microscopy, and X-ray diffraction.

It was established that with increase in tantalum content, the martensitic structure changes from hexagonal α′-phase to orthorhombic α″-phase. The martensitic α″-phase was found only in 10 and 30 at. pct Ta. The concentration dependence of the lattice parameters of the α″-phase, the temperatures of the direct martensitic transformation are given. Omega-phase precipitation were not found in quenched alloys. The alloys of 35 - 40 at. pct Ta consist of metastable β-phase upon quenching only. It was established that stress-induced $\{110\}_\beta$ and $\{332\}_\beta$ twinning appeared in metastable β-phase.

10:10 Oral

Transition Phenomena in the Diffusion Soldering / Brazing

Waldemar Wołczyński

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A model for dissolution, solidification and first solid / solid transformation is proposed. The model explains mechanism of above processes that precedes the final stage of homogenisation required by the technology. Transformation of the liquid filler metal into its liquid solution is described as a phenomenon necessary for the formation of sub-layers within the interconnection. Transition from the initial stable solidification into the final metastable solidification is explained. The connection between period of solidification and first solid / solid transformation is considered. The birth of coupled phase on the surface of dominant phase of the joint is observed. It allows the explanation of the sequence of phases appearance to be delivered. The thermodynamic justification for the mentioned sequence is shown, additionally.

Coffee break

Tuesday morning, 5 September, 10:30

Amorphous Phases and Nanomaterials

Tuesday morning, 5 September, 11:00

Chair: Lazar L. Rokhlin

11:00 Invited oral

Nano in the Materials Curriculum: Past, Present and Future

David E. Laughlin

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The current fad in advertising one's Department to potential engineering students is to claim to be at the forefront of *nano*-engineering, *nano*-technology or *nano*-materials. This talk will review some of the aspects of *nano*-materials in the teaching of Materials Science and/or Metallurgy. Since the symposium is in honor of my long time colleague and friend Professor Massalski, who will receive the 2006 Czochralski Prize, I will focus on *nano*-materials and phase equilibria. How we included *nano* in our 20th century curricula will be reviewed and how we now include it in the 21st century curricula will be presented and discussed. Some thoughts about *nano* and the future also will be presented. I will close with some thoughts on the danger of designing a curriculum around fads.

11:30 Keynote lecture

Phase transitions in binary alloys nanoparticles and nanowires

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The phase diagram of nanosystems is known to be a function of their size and shape. The variation with temperature depends on the surface tensions involved in the phase transitions. When looking at the nucleation process in nanoparticles, it turns out that it is necessary to take into account the fact that the reservoir of matter is limited. In a nanosystem, the total amount of one of the chemical components may be too small for the synthesis of the critical nucleus. This gives rise to three possibilities: phase separation, prohibition of decomposition, formation of metastable phases. A new effects arise for phase transformations in binary and multicomponent nanosize systems with change of composition - finite depletion effect. It is shown theoretically that the usual concept of phase diagram has to be reformulated when dealing with multicomponent nanosystems. The liquidus and solidus lines are shifted due to size effects. Moreover, it turns out that it is required to differentiate the solidus and liquidus curves and equilibrium curves after the first order phase transition. In this work, we study how the phase transitions of binary nanoalloys are treated in the case of nanoparticles and nanowires.

11:50 Oral

Search for New Metallic Glass Compositions in NiZrTi Base Alloys Near Multicomponent Eutectic Positions

Jan M. Dutkiewicz, Jerzy Morgiel, Lidia Lityńska-Dobrzyńska, Wojciech Maziarz, Tomasz Czeppe, Magdalena Parra Carillo

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In a new metallic glasses near muticomponent eutectic compositions even a small composition changes have a significant effect on the T_g and T_x temperatures and therefore on the glass forming ability. In the present study the alloy of composition Ni25Cu25Zr25Ti25 (1 in at%) was melt spun amorphous. The position of the eutectic was identified as Ni29.2Cu29.9Zr17Ti24.9 (2) and the melt spun ribbon shows a better glass forming ability than the alloy (1) as manifested by DT and T_g/T_l increase. The alloy (2) was modified by manganese addition as Ti25Zr20Ni22,5Cu22,5Mn10 (3). In this alloy the eutectic was identified at Ti21Zr14Ni27Cu27Mn7 (4) and as melt spun has shown slightly better T_g/T_l=0.63 as compared to 0.62 in the alloy (3). A second copper rich eutectic was also identified in this alloy near Ti16Zr14Ni24Cu39Mn7 (5). Nickel rich alloy of the com-

position Ni60Nb15Zr15Ti10 shows the eutectic composition at Ni48,5Nb21,5-Ti16.5Zr13,5 (6). The alloy (6) has been modified by silicon addition with the eutectic composition containing Ni50.3Nb14.4-Ti17.3Zr15.1Si2.8 (7). Glass forming ability was studied in NiCuZrTiAlSi alloys. The composition of the eutectic was found near Ni45.5Cu10.5Zr20Ti15-Al3.5Si5.5 (8). Present studies indicate that eutectic 4-6 component alloys lead to the increase of the T_g/T_l and DT coefficients in the melt spun amorphous ribbons.

12:10 Oral

Stability of second phase particles during processing by hydrostatic extrusion

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Phase stability and transformations are well recognized for conventional materials of the grain in the micro-meters range. Nowadays, these phenomena attract new attention in the context of nanocrystalline materials in which the large fraction of atoms are located in interfaces area leading to the change of phase stability. In addition, such materials may be produced by the severe plastic deformation (SPD) which results in a large amount of defects such as dislocations being accumulated in nano-metals. It has been already shown that SPD strongly affects stability of carbides in Fe alloys. One should expect that it also affects the stability of particles in other multiphase materials, e.g. age-hardenable aluminium alloys in which the crucial factor influencing the mechanical strength is the presence of second phase particles.

The aim of the present work is to describe the stability of second phase particles during SPD processing and their effect on the process of grain refinement. Two age-hardened aluminium alloys (2017 and 7475) were subjected to HE process. The microstructure of the alloys before and after HE processing was observed using a transmission electron microscopy and described in terms of size and shape of grains and particles. During this processing the grain size was reduced below 100 nm. At the same time, the precipitates underwent the transformation from the plate-like shape into spheroids while inclusions were profoundly dispersed. The mechanisms of these changes have been proposed. Their influence on the process of grain refinement and high-angle grain boundary formation has been discussed.

Lunch break

Tuesday afternoon, 5 September, 12:30

Interfacial Reactions-Thin Films and Applications

Tuesday afternoon, 5 September, 14:00

Chair: David E. Laughlin

14:00 Invited oral

Non-Random Distribution of Atomic Species on Mixed III-V and Group III Nitride Layers

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We will demonstrate that atomic species in mixed III-V and group III nitride layers, differing in their covalent tetrahedral radii, are not distributed at random on their respective sub-lattices. Two types of deviations from randomness are observed: phase separation and atomic ordering. In both systems, phase separation is two-dimensional in nature, evolves while the layer is growing and occurs along soft directions lying in the growth plane. This microstructural feature develops to reduce the strain energy of the system.

Mixed III-V layers exhibit *CuPt* type ordering where lattice periodicity along two of the four <111> directions is doubled. Ordering is caused by the (2 x 4) surface reconstruction induced sub-surface stresses which bias the occupation of sites by species differing in their tetrahedral radii. On the other hand, 1:1 atomic ordering in mixed group III nitride layers having the wurtzitic structure appears to be driven by the preference of atomic species, differing in their tetrahedral, for different sites on facets.

Influence of the above microstructural features on electronic and optical properties will also be discussed.

14:30 Oral

Interfacial reactions in the Sn/Te couples

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Te and Sn are the primary elements for thermoelectric materials and solders, respectively. However, there are no previous Sn/Te interfacial reaction studies which are important for the development of thermoelectric devices. A piece of 7 mm×5 mm× 2mm pure Te was encapsulated together with 2 grams of pure Sn in a quartz tube. The sample capsules were placed in a furnace at 250^{o}C and 300^{o}C for various lengths of reaction time. Only one reaction phase, SnTe, was formed. The reaction rates were very fast, and the reaction products were with very unusual cruciform patterns. The reaction layer was 800 μm thick when reacted at 300^{o}C for 30 minutes. The reaction layer was porous, but with uniform thickness, very straight and tidy boundaries through the entire couples. At the corner of the Te, the reaction products were with cruciform pattern. The special kind of reaction morphologies are found only in the Fe/Zn, Ti/Al, Ni/Ga and Nb/Al/Ni systems. It is assumed that the porous SnTe layers grew outward rapidly, cracked at the corners, and thus very unusual cruciform patterns were formed.

14:50 Oral

Magnetisation dynamic in quasi-one dimensional Ising magnet $[(CH_3)_3NH]CoCl_3x2H_2O$

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The quasi-one-dimensional compound [(CH3)3NH]CoCl3*2H2O (CoTAC) represents a canted antiferromagnet with four sublattices and Neel temperature TN=4,113 K. The CoTAC crystal has an orthorhombic symmetry with the space group Pnma. COTAC's magnetic structure consists from ferromagnetically coupled antiferromagnetic chains. A nonzero net magnetic moment in c direction is the key feature of COTAC.Relaxation processes in magnetization was measured at temperatures 2- 0.5 K along a-axis in magnetic fields up to 2 T. It was shown that relaxation processes of magnetization in this compound are motivated by domain structure and its motion. The time constants τ for CoTAC compound strongly depends on temperature and at low temperature is comparable with experiment time. For the explaining of effect of dynamic hysteresis the simple mechanism was proposed associated with a thermally activated crossing of an energy barrier whose height is reduced by the applied field. The main characteristics of magnetization dynamic process such as domain energy barriers, relaxation times, intrinsic magnetic field of domain and its volume were estimated.

15:10 Oral

Study of domain evolution in $(Pb(Mg_{1/3}Nb_{2/3})O_3)_{1-x}(PbTiO_3)_x$ single crystal by temperature-dependent piezoresponse force microscopy

Kin Sun Wong[1], Xin Zhao[1], Ji Yan Dai[1], Xiangyong Zhao[2], Haosu Luo[2]

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PMN-xPT is relaxor ferroelectrics with great potential in the application of novel devices. At the morphotropic phase boundary with x~28%-36% of PT, PMN-xPT exhibits complex crystal structure and phase transformation, which are not fully understood yet. The ferroelectric domain evolution study may reveal the mechanism of phase transformations induced by temperature or electrical field. In this work, we report the study of domain evolution of PMN-30~31%PT single crystal by means of temperature-dependent piezoresponse force microscopy (PFM) and electrical measurement. Observation of poled sample revealed that stripe-shaped domains are developed from an unstable single domain structure (generated from poling) through the aggregation and self alignment of polar nanosized regions (PNRs). Temperature-dependent PFM of the poled sample revealed a domain evolution process. During temperature increase, the stripe-shaped domains become larger through the growth and aggregation of PNRs, and the phase transformation from rhombohedral to tetragonal phase is progressing according to polarization rotation. When temperature is higher than T_m, the domain contrast becomes very weak and finally disappears. After heating and cooling cycle, the PNRs and microdomains with random arrangement can be observed in the PFM image.

Coffee break
Tuesday afternoon, 5 September, 15:30

Parallel Session
Tuesday afternoon, 5 September, 15:50

15:50 Oral

Cu diffusion in Sn/Cu couples by electromigration

Chao-hong Wang, Sinn-wen Chen

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The study investigates the electromigration effects upon the Sn/Cu interfacial reaction. A Sn/Cu/Sn/Cu/Sn couple was prepared by the casting method. The couple was reacted at temperatures ranging from 150 to 190°C with 5000A/cm^2 current density. No massive Cu_6Sn_5 phase formation is found in the couples reacted at 160°C for 120 hours. However, after current stressing at 180°C for 120 hours, at the interfaces with electrons flowing from Cu to Sn, large irregular Cu_6Sn_5 phase was formed and penetrated into the Sn matrix. However, at the interfaces with the opposite electron flow direction, two continuous reaction layers, Cu_6Sn_5 and Cu_3Sn, were formed. At the Cu/Sn interfaces, atoms are driven into the Sn phase by the electron current flow. Since grain boundaries are faster paths for Cu diffusion, large amounts of Cu atoms cram into the Sn region with the electron current flow through grain boundaries, reacted with Sn and large Cu_6Sn_5 compounds were formed. Sn grains grow with reaction time as well. When the sizes of Sn grains are large, only limited grain boundary paths are available. Instead of the grain boundary diffusion, the Cu atoms diffuse along the free surfaces, and the reaction Cu_6Sn_5 compounds are found at the surfaces of the couples. It is found that Cu dissolution in the molten Sn is significant when the casting method is used for sample preparation, and fine Cu_6Sn_5 phases precipitate after quenching. The Cu_6Sn_5 precipitates are an important source for the Cu supply at the interfaces with electrons flowing from Sn to Cu. The Cu atoms of the precipitates move toward the interfaces under current stressing, and the growth of the reaction layers are thus faster in the initial stages at the interfaces where electrons are from the Sn side to Cu side.

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session
2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

Parallel Session
All Symposia exept Symposium I
Wednesday afternoon, 6 September, 14:00

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony
(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Posters

Monday, 4 September

Poster Session 1
Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster I-1

Structural stability of zirconia ceramic in water and acid solutions

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Yttria-tetragonal zirconia is used as biomaterial for dental applications e.g. inlays, crowns, bridges. Since Y-TZP may undergo phase transformation, which can results in micro-cracking, structural stability in oral environment is of great importance.

In the present work, hydrothermal stability of low-porous yttria doped zirconia (Y-TZP) was investigated in water and acid solutions. Ceramics with fractional density of tetragonal phase from 90% to around 100%, were produced by biscuit-sintering dry-pressed pellets at different temperatures in an ambient air atmosphere.

The flexural strength, phase composition (XRD) and density were measured for samples before and after corrosion test. The influence of phase transformation on a microstructure was determined using scanning electron microscope.

The results showed that specimens sintered at higher temperature (1450^{o}-1500^{o}C) to a fractional density above 95% of the theoretical exhibit low tetragonal to monoclinic phase transformation upon autoclaving in water at 200^{o}C for 24 hours. The volume expansion due to phase transformation created micro cracks, which reduced flexural strength. The effect of environment pH on phase transformation is also discussed.

17:20 Poster I-2

Radio-crystallographic study of titanium sesquioxide at high temperature and in vacuum

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High-temperature X-ray diffractometry is used to study Ti2O3 rhombohedral oxide formed by reduction of titanium dioxide, under vacuum and in the presence of graphite or metallic titanium. The sesquioxide is characterised by a homogeneous field of very low oxygen pressure, both boundary phases appearing independently from one another between 1073K and 1988K. The c/a = 2.660 value for Ti2-xO3 oxidized form of the equivalent hexagonal cell reaches c/a = 2.735 for Ti2O3 reduced form, which does not undergo quenching (c/a = 2.640 at 293K). ABSTRACT:

17:20 Poster I-3

Diffusion brazing fundamentals and applications contemporary and ancient

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Diffusion brazing, as the name implies is a brazing procss which lasts long enough so that the liquid brazing alloy interacts (with dissolution/solidification/interdiffusion) with the solid materials to be joined and eventually solidifies under isothermal conditions. Copper-Silver alloys are well known is this context and a paper by T.B. Massalski and co-workers illustrates how equilibrium and kinetic information can be combined to understand the kinetics of the diffusion brazing process in this simple binary system. This presentation starts with a review of a selection of some theories of diffusion brazing and continues with two applications, one ancient (again with the copper-silver system) and one contemporary with nickel based brazing alloys. The first application deals with ancient counterfeiting. A. Deraisme and co-workers recently figured out which techniques were used to make forgeries of coins in Roman Gaul in the third century. To understand how craftsmen of the time made the coins, the supposed technique was recreated in modern laboratory by heat-

ing copper cylinders that had been coated with thin silver layers at different temperatures and for varying lengths of time. Four minutes and 950°C led to the composition and structure closed to that of the counterfeit coins. The interpretation of EMPA composition profiles of counterfeit coins and recreations relies mostly on the work by Massalski et al. The contemporary application is that of nickel based brazes. A classical application is the repair of nickel base superalloys components of aero gas turbines and a not so classical one is the joining of ferritic stainless steel Fe20Cr5Al (wt%) by nickel chrmium silicium commercial brazing alloys in the form of powders. Laboratory studies include differential thermal analysis and diffusion experiments combined with EMPA (microanalysis) of the diffusion couples and actual diffusion brazed joints.

17:20 Poster I- 4

Potential possibilities of DSC/QMS coupling for investigation of thermodynamic properties

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Over last decades numerous techniques of experimental thermodynamics in dependence on different aspects of phase diagram science and equilibria computation and prediction were applied. Some of them regard thermochemical measurements and thermodynamic equilibria measurements and they are able to estimate feature of phase diagrams or thermodynamic properties. Others involve methods enabling to characterise phases present in a system from the point of view of their structural feature, composition and phase abundance and also their morphology and physical chemical properties.

Both groups yield data which serve as input information for calculation of thermodynamic properties as well as of phase equilibria for various technically important systems.

Very typical technique is Knudsen effusion method. This method utilizes effusion of the gas being in equilibrium with sample (solid or liquid phase) through a small orifice.

This work presents data from Knudsen cell mass spectrometry coupled with differential scanning calorimetry. Instrument STA 409 CD / QMS 403/5 SKIMMER from firma Netzsch [1] applied for investigation of nickel based alloys as well as newly developed steels illustrates modern way how to collect source data leading to estimation of activities for particular components very trigly and effectively. Some data are compared with older ones obtained using home made equipment coupled with a monopole mass spectrometer [2]. Final data can be subsequently used for determination of thermodynamic properties using thermodynamic models involved in CALPHAD method [3].

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17:20 Poster I-5

Controlled precipitation of $CaCO_3$ sub-micro crystals of well-defined structure in a multiphase system

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In this paper we present the new method that allows obtaining of super fine calcium carbonate particles in a (CO_2 - $Ca(OH)_2$ - H_2O) system. A new rotating disc reactor was employed as a processing unit to run this multiphase process. The device enables to control inter- and intraface mass and energy transfer as well as the macro- and micromixing effects in the reacting system.

The system can be controlled by the rate of discs revolutions speed and mixing conditions in the system, reagents contact area or gas flow rate. The influence of different parameters on the reaction kinetics and precipitated powders properties were investigated.

The average particles diameter (hydrodynamic diameter) and the particles size distribution were determined with the Dynamic Light Scattering method. Crystals and agglomerates morphology was examined with the Scanning Electron Microscopy (SEM). SEM studies allowed also for particle and agglomerate size distribution measurements.

The testing with X-ray diffraction analysis confirmed that we have obtained a pure calcite, which is the most thermodynamically stable polymorph of $CaCO_3$.

Our investigations results of produced powders properties confirm effectiveness of the applied technique for the controlled precipitation of superfine calcite crystals with well-defined and controlled size, shape and structure.

17:20 Poster I-6

Bendging test of an SMA bar and its seismic applications

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This paper conducted several bending tests of a shape memory alloy bar, which includes the single and double bending test. The SMA bar, whose state is Austenite of NiTi in room temperature, is circular cross-section and has the length of 157 mm and the diameter of 25.4 mm. In the bending behaviour, the SMA bar shows different tensile and compressive strain with the same stroke, which means the Young's modulus for the tensile and the compressive behaviour are

different. To explain this phenomenon, the compressive test result of the bar is compared with that of the tensile test result. The bending test's results can be applicable for seismic restrainers for bridges. Previous application of SMA bars of seismic restrainers used the tensile behaviour of the bar. However, the application has several drawbacks. This bending application of seismic restrainers can overcome the problems of the previous study. In this paper, the bending behaviour of the SMA bar is analysed such as the stiffnesses of the loading and the unloading path and the damping ratio of the hysteretic curves. The parameters in the test are the loading velocity and the top displacement of the bar. For the tests of the single and double bending, the two special devices are manufactured to realize the end conditions of the bar which are the fixed-free condition for the single bending and the fixed-fixed for the double bending. The Austenite SMA bar can provide recentering force for the superstructure of bridges and some additional damping and restrict the movement of the superstructure during an earthquake.

17:20 Poster I-7

Phase stability and structural relaxation in $Fe_{86-x}Nb_xB_{14}$ amorphous alloys

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It is known that magnetic and mechanical properties of amorphous alloys based on iron can be controlled by applying a suitable thermal annealing leading to the so-called structural relaxation and nanocrystallization. Recently, we have reported that for the $Fe_{86-x}Nb_xB_{14}$ ($2 \le x \le 8$) amorphous alloys the structural relaxation - i.e. annealing out of free volume and formation of small iron clusters [1] - plays the main role in the process of optimization of soft magnetic properties. It makes possible to obtain a good soft magnetic material free of brittleness - the main disadvantage of nanomagnets. The aim of this paper is to study the influence of Nb content, structural changes and phase stability of amorphous structure on different physical properties. The following measurements were carried out: i) heat of transition amorphous-crystalline state (DSC, 300-800 K), ii) activation enthalpy of crystallization (DSC, magnetic balance), iii) Young modulus (vibrating reed apparatus, 300-800 K), iv) magnetic permeability (Maxwell-Wien bridge, H=0,5 A/m). Phase stability was studied by making use of magnetic after-effects-$\Delta\mu/\mu$ (difference in permeability after demagnetization).

It was shown that the heat transition of amorphous-crystalline state, the optimized magnetic permeability and the change of Young modulus due to structural relaxation depend on Nb content. In all cases a remarkable maximum for x=6 at % of Nb, is observed. In contrary to this $\Delta\mu/\mu$ continuously decreases with increasing Nb content. These results show a correlation between free energy (frozen during sample fabrication) and the intensity of structural relaxation. It makes possibly to optimize soft magnetic properties in the relaxed amorphous phase free of iron nanograins.

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17:20 Poster I-8

Two-dimensional phase diagram of In(0.5)Ga(0.5)P

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Non-homogeneity of In(x)Ga(1-x)N films is normally explained as spinodal decomposition. This origin does not seem reasonable since the spinodal decomposition descriptions did not take into account the coherency strain energy. As it was shown in [1] due to this energy spinodal decomposition of the wurtzite In(x)Ga(1-x)N is non-profitable in the total composition range at any temperature.

Non-homogeneity of the "conventional"epitaxial III-V alloys is two-dimensional in nature [2] and two-dimensional phase diagrams should describe it. The two-dimensional In(0.5)Ga(0.5)N phase diagram on the (0001)A surface is considered. This surface is the triangular cation lattice. The alloy is described as a regular solution with the interaction parameter between In and Ga equal to 22.7 kJ/mole for this lattice. Such solution is equivalent to the Ising model that has the exact solution (ES) in this case [3]. The strictly regular (SRA), quasichemical (QCA) and cluster variation (CVA) approximations are also applied. The temperatures of the miscibility gap and concentrations of In-Ga pairs at these temperatures, respectively, are given as at ES: T = 822 K, y = 0.1667; at CVA: T = 884 K, y = 0.25; at QCA: T = 1114 K, y = 0.4 and at SRA: T = 1348 K, y = 0.5. There is the great difference not only between the temperatures but between the concentrations as well. ES demonstrates that from the thermodynamics standpoint one-phase In(0.5)Ga(0.5)N is in the stable state and non-homogeneous at its growth temperatures. In fact, ES establishes an existence of the macroscopic phase separation at temperatures higher that the temperature of an occurrence of a miscibility gap.

References

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17:20 Poster I-9

Sn-Cu-Al-Si phase diagrams study for the growth of silicon thin film by liquid phase epitaxy

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Liquid Phase Epitaxy (LPE) method for growth of Si layers at low temperature (<800°C) can be an interesting technique for thin mono-

crystalline Si photovoltaic applications. LPE has been largely studied at high temperature using solvents like tin or indium but only few works deal with low temperature LPE. The main difficulty is the low solubility of silicon in usual solvent. In this work, we propose the use of a quaternary alloy Al-Cu-Sn-Si. It permits growth at lower temperature, each elements playing a key role: Al reduce Si native oxide prior growth [1]. Cu is used to increase Si solubility. Sn allows growth at low temperature.

Phase equilibria in the Al-Cu-Sn-Si system have been calculated using the Factsage software package and thermodynamic data coming from the SGTE solution database for all solid compounds and the binary interaction in the liquid phase. The ternary interaction has been altered for the calculations. Within the SGTE database, the symmetrical Muggianu model is used by default. This model has the disadvantage that it extrapolates no very well in an unsymmetrical case such as Cu-Si-Sn. Either ternary interaction parameters are needed (with no physical meaning: they are only necessary due to the model chosen) or another extrapolation model has to be chosen. So, the unsymmetrical Toop model, which is implemented in the software package, has been used. Results of these calculations and their implications with respect to Si growth are discussed. We compare effective Si epilayer thickness obtained with expected Si incorporated in the liquid phase, and influence of alloy composition to the growth rate. We also present some electrical characterisation and discuss the use of such epitaxial layer for solar cells.

1- F. Abdo and al. PVSEC-15, Shanghai 2005 pp. 746-747

17:20 Poster I-10

Effect of surface treatment on the microstructure of TA6V

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TA6V alloy belong to a group of materials applied in aircraft industry. Various methods of surface treatment are used to improve corrosion and wear resistance of these alloys. Hybrid method allows to obtain Ti-Al intermetallic layers on the surface of TA6V alloy. These layers are characterized by high microhardness and improved wear resistance.

Nevertheless, surface treatment influences not only on the chemical and phase composition of the layers but also the microstructure of the substrate. Phase transformations taking place during surface treatment have an effect on the microstructure homogeneity and phase composition of TA6V alloy. The microstructure changes can influence the mechanical properties such as fatigue resistance of surface treatment elements of TA6V alloy.

The aim of the investigation was to analyze changes in the morphology and phase composition of TA6V alloy after surface treatment by hybrid method. Hybrid method consisted of two stages. In the first stage, the titanium alloy was covered aluminum layer. In the second stage, coated specimens were treated under glow discharge conditions. The investigation focused on phase transformations occurring as a result of different parameters of surface treatments. The structure of layers and substrate microstructure were examined on a light and scanning electron microscope with Energy Dispersive Spectrometer (EDS). The phases fraction was defined using stereological method and XRD analysis. The surface topography was investigated using a profilometer.

17:20 Poster I-11

Effect of Directional Crystallization Rate on Microtexture of Al-$CuAl_2$ Eutectic Alloy

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The effect of crystallization rate on the microstructure and the orientation relationship of (Al) and $CuAl_2$ phases in the eutectic alloy was investigated using a step-by-step beam scan on the computer controlled transmission electron microscope.

The microstructure of this alloy changed from the approximately parallel lamellar structure into the eutectic colony structure depending on the rate of the crystallization.

On the basis of the measurement of the individual orientations in the sections perpendicular to the crystallization direction the orientation maps were constructed. The resulting data enabled to link the microstructure development in the analysed alloy to the orientation relationships between (Al) and $CuAl_2$ phases and the preferred orientations.

The dominant orientation relationship identified in all analysed samples was (111) (Al) II (2-11) $CuAl_2$, [-110] (Al) II [120] $CuAl_2$. In the small irregular areas the appearance of another orientation relationships have been found.

17:20 Poster I-12

Phase Diagrams of Water-rich Parts of the Rare Earth Chloride - Water Systems

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Rare earth (RE) chlorides are essential compounds used for production of the RE metals, their alloys and compounds, also as catalysts in organic synthesis and polymerization. Because in vicinity of room temperature the RE chlorides crystalize from aqueous solutions as hydrates, it was essential to know the phase relations in this region. The corresponding literature was compiled and critically evaluated what allowed to select the most reliable solubility results (liquidus line) and the equilibrium solid phases of the RE chlorides in water. The selected solubility data do not change much near room temperature whereas at elevated temperatures typical strong bends of the li-

quiduses are observed reflecting tendencies of the systems to immiscibility. The water-rich parts of the RE chloride - water phase diagrams were constructed for majority of the systems. Shapes of the phase diagrams showed evolutional changes of the invariant points and phase stabilities through the RE series. This fact allowed to predict the phase diagrams of the $HoCl_3$-H_2O and $TmCl_3$-H_2O systems (which were not investigated) as well as of the $PmCl_3$-H_2O system (which due to the high radioactivity cannot be treated experimentally at proper concentrations of $PmCl_3$). Dehydration reactions of the RE chloride hydrates at higher temperatures, decreased pressures and using strong dewaterers are multistage processes, however there are some discrepancies in an uniform description of the reaction stages.

(partly based on achievements of late Tomasz Mioduski)

17:20 Poster I-13

The features of birefringence of the linearly polarized light in the magnetoelectric single crystals $LiNiPO_4$

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The magnetization of the antiferromagnetic magnetoelectric LiNiPO4 single crystal along the crystallographic axes *a*, *b* and *c* was measured. The carried out researches have shown in it presence of a weak ferromagnetism. The weak ferromagnetic moment is directed along the antiferromagnetic axis of the crystal. The measured remanent magnetization in $LiNiPO_4$ is equal to about 0.005 G at 5K. With increase of a temperature of the sample up to the Neel temperature (T_N~21K) the remanent magnetic moment was decreasing up to zero. Furthermore, in $LiNiPO_4$ the intermediate antiferromagnetic phase was revealed in the narrow temperature interval ~1 K near Neel temperature. The magnetic structure of the basic antiferromagnetic state of $LiNiPO_4$ is uniform, but its antiferromagnetic intermediate state has a modulated structure. For more information about processes of magnetic ordering the behaviour of birefringence in this crystal was investigated depending on changes of temperature or intensity of a magnetic field. So, on the temperature dependences of birefringence is observed jump at T_1 and at T_2 (=T_1+1K) break of a curve. These features to make agree with presence intermediate of structure in a temperature interval T_1-T_2. But herewith the temperature hysteresis with width 6 K and the centre in the T_N is observed. In addition, we notice linear dependence of birefringence on intensity of a magnetic field in fields up to 15 kOe and further break of a curve. In higher magnetic fields, the dependence carries parabolic character. The attempt is made to connect these features with behaviour of magnetic structure in a magnetic field. (ilt.kharkov.ua/bvi/structure/depart_e/d04/resecher-en.html)

17:20 Poster I-14

Phase transition in five-component chalkogenides $(PbSe)_x(AgAsSe_2)_{1-x}$

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The development of modern crioelectronic requires the creation of new the semiconductors materials, including materials, which to combine the superionic properties at low temperatures with other properties (optical, magnetic et al). In USU the five-component chalkogenides $(PbSe)_x(AgAsSe_2)_{1-x}$ (x=0.4-0.9) were synthesised and their electrical properties by a method impedance spectroscopy are investigated.

The compound with x=0.4 has grey dim colour, with other values x - metal shine. On the data of x-rays analysis, the compound consist of two phases - PbSe and $AgAsSe_2$.

Godographs of an impedance are characterised by presence of two precisely divided areas - low-frequency (electrode processes) and high-frequency (processes in volume of a sample). Researches were carried out on frequency 1592Hz in the field of temperatures 78K-400K.

The property of compound is exchange in depending on a part of phases. For x=0.4 and 0.5 the change a conductivity with metal on semiconductor type in the regions of temperatures 220K and 300K accordingly is observed . For other values x we have the semiconductor type of temperature dependence of conductivity in all investigated interval of temperatures .

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17:20 Poster I-15

Electrical properties the $AgGeAsS_{3x}Se_{3(1-x)}$ (x=0.7-0.9) at pressure 15-45GPa

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In Ural State University five-component chalcogenides $AgGeAsS_{3x}Se_{3(1-x)}$ were synthesized and their electrical properties at temperatures 78-400K are investigated . In the present work were investigated their electrical properties at pressure 15-45GPa. Such research represents the large scientific and practical interest, allows to specify areas of probable application of these connections at high pressure. The compound have grey colour and metal shine. These

compounds are mixed electronic-ionic conductors. On temperature dependencesthe conductivity and dielectric permittivity the phase transitions are found out.

For generation of pressure up to 45GPa used the carbonado-diamond anvil cell. Electrical properties were investigated with investigated-analyser of impedance RLC-2000 in the field of frequencies 100Hz-200kHz.

The godographs of an impedance of compounds and the dependences of conductivity on frequency are investigated. The hysteresis of conductivity is investigated at gradual the unloading a sample. The analysis of hodographs makes possible to determine the values of dielectric tangent (tgd =ReZ/ImZ). From the data of conductivity and dielectric tangent the the region of existence of phase transitions in samples are received. The influence of structure of a sample on its electrical properties at high pressure is analysed .

This work in part was supported by CRDF (grant EK-005-X1) and grant CRDF and Min. of Education of the Russian Federation (Post Doctoral Fellowship, award EK-005-X1, annex 07, No Y1-E-05-09), and grant RFBR (No 06-02-16492-a)

17:20 Poster I-16

Phase Transformation Study of Aluminium Containing Ductile irons by Dilatometry

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The temperature for the start of martensite formation (M_S) for three Al-alloyed ductile irons containing 0.48%Al, 4.88%Al and 6.16%Al were determined by high-speed dilatatometry. Microscopic observation and hardness measurements also were used to indicate the effects of aluminium addition on the martensite transformation during quenching. The examination confirmed that an austenite/martensite transformation occurs for irons containing 4.88%Al and less. The results indicate that martensitic transformation occurs at about 173°C for 0.48%Al and 4.88%Al. Whereas there was no evidence of the martensitic transformation for 6.16% Al .

Keywords: Phase transformation, Dilatometry, Ductile iron, Aluminium, Martensite

17:20 Poster I-17

The Influence of Al Content on the Partial Fe-C-Si Equilibrium Phase Diagram

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MTDATA software was used in order to gain an insight into the effects of alloying addition on the stability of the various phases that are in equilibrium in the materials under study. This was achieved by performing complex equilibria calculations to produce isoplethal sections of temperature against alloy concentration. Thermodynamic data were retrieved from databases appropriate to the experimental alloys compositions. After retrieval of the data, a range of temperature and composition was chosen for each calculation. By performing global Gibbs energy minimisation calculations, the stable phases in the temperature and composition were determined and plotted as isoplethal sections. It can be seen the aluminium content modifies the iron-carbon equilibrium at this alloy composition. The austenite region decrease with increasing Al content, and for cast iron containing about 4.88%Al or more, there is no evidence of austenite zone. However, there was a difference with observation for the 4.88%Al.

Keywords: Phase diagrams, MTDATA software, Austenite, Phase transformation

17:20 Poster I-18

The influence of the structure of metastable τ - phase on magnetic hysteretic properties of $Mn_{55.5}Al_{44}C_{0.5}$ alloy

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Ferromagnetism of Mn-Al system alloys is determined by a metastable τ-phase, which is usually stabilized by small amount of C. This phase formed under specific thermal treatment, it has an ordered $L1_0$ type face centered tetragonal structure and exhibits rather high value of magnetic anisotropy constant. For a given chemical composition the as-cast state the coercivity value is usually low. The improvement of magnetic properties may be attained by plastic deformation. For studying domain structure and magnetic hysteretic hysteretic properties the samples with different structural states of τ-phase were processed by means of deformation and thermal treatment. After annealing at 900°C the cast samples were cooled in the oil. During this processing the coarse grained nonmagnetic ε-phase transformed into a needle type τ-phase. Subsequent extrusion under different temperature - strain rate conditions and additional annealing enables formation of τ-phase with submicron mean grain size (0.5-0.7 μm) and different dislocation density(10^9-$10^{11}$$cm^{-2}$). It is shown that coercivity increases from 303 to 368 kA/m as the grain size decreases in submicron grained samples, and does not depend on the dislocation density. In these samples most of the grains in the initial nonmagnetized state have multidomain structure and contain several domains with alternating antiparallel directions of magnetization divided by straight parallel Bloch type domain walls. The estimation of the criterion of single domain state suggests that magnetization occurs by means of nucleation of the reverse domains in submicron structural state and by means of domain walls displacement in nanocrystalline state. That means that in spite of significant refinement of τ-phase a single domain state has not been achieved in either of the structural states under study.

17:20 Poster I-19

Nanocrystalline materials manufactured by torsion under pressure of 2 GPa

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Improved mechanical properties of bulk materials with ultra-fine grained (UFG) structure (nanostructure) are mostly reported, which are manifested by greater strength, hardness and ductility in comparison with conventional coarse-grained materials; also the high-speed superplasticity flow is reported. In metal alloys with UFG structure manufactured by severe plastic deformation (SPD) the formation of metastable states or solid solutions in immiscible systems is observed.

SPD is commonly proposed as a method to obtain UFG microstructure. Such methods of processing by means of SPD like Equal-Channel Angular Pressing (ECAP), Accumulative Roll-Bonding (ARB), High Pressure Torsion (HPT) and Mechanical Alloying (MA) are commonly used recently. ECAP type of processing for example enables obtaining of bulk samples of relatively great dimensions and free from porosity in opposition to the methods based on powder metallurgy, rapid cooling or crystallization from gaseous phases. The ARB method in turns, can supply semi-final products in the form of sheets, which can be directly used for further work forming operation. High pressure torsion, although can't supply big dimension samples, it leads generally to highest degree of grain refinement. Mechanical Alloying (MA) by ball milling and successive compacting offers the method to extend the solubility limit.

The main objective of the paper is to present the influence of the phenomenon of intensive shear deformation during High Pressure Torsion (HPT) processing on the changes of structure, grain size, mechanical properties and solid solubility. Study is performed with metals and mostly metal alloys like Ti, Al and Cu alloys, severely deformed up to high deformation degree.

17:20 Poster I-20

Influence of metalloids content on magnetic properties and thermal stability of amorphous alloys based on iron

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The aim of the paper is to study the influence of metalloids content (Si, B), Nb and Cu on the enhancement of soft magnetic properties (ESMP) effect, the 1h optimization annealing temperature Top, the crystallization temperature Tx and the Curie temperature Tc. The following amorphous alloys (melt spinning ribbons) were examined: Fe87Nb2B11, Fe84Nb2B14, Fe76Nb2B22, Fe76Nb2Si13B9, Fe78B22 and Fe75Cu1Nb2Si13B9. As quenched samples were annealed for 1h at temperature Ta (300-900 K) and at room temperature the following measurements were carried out i) magnetic permeability μ (Maxwell-Wien bridge), ii) intensity of magnetic relaxation Δμ/μ, iii) coercive field, iv) magnetization M (magnetic balance) and v) resistivity ρ. The structural changes were examined by making use of X-ray and HRTEM techniques. Both characteristic temperatures Tc and Tx were determined using measurements of M(T) and ρ(T) with heating rate 0.5-10 K/min.

In all cases μ(Ta) show a maximum situated at the temperature Top. For the Fe87Nb2B11, Fe84Nb2B14 alloys (small amount of metalloids) the ESMP effect takes place at temperature range 450-600 K while for the Fe78B22, Fe76Nb2B22, Fe76Nb2Si13B9 alloys (higher amount of metalloids) at 550 - 800 K. An increase of the metalloids content leads to a slowing down of diffusion processes and what follows to an increase of the temperature Top. In all cases the ESMP effect takes place in the relaxed amorphous phase free of αFe or αFe(Si) nanograins. In contrary to this for the Fe75Cu1Nb2Si13B9 alloy the ESMP effect is observed at 600-850 K and is due to formation of nanostructure of αFe(Si).

17:20 Poster I-21

TEM study of iridium silicide contact layers for Low Schottky Barrier MOSFETs

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An influence of annealing temperatures on the iridium silicide nanolayer formation in reaction between 15 nm thick Ir metallisation and Si substrate during annealing by the rapid-thermal-annealing (RTA) process for 120 s, was analysed using the transmission electron microscopy (TEM). The silicide layers are used as source and drain contacts for a novel technology of Low Schottky Barrier MOSFETs on SOI. The high quality of the silicide/Si interface and of the silicide structure is essential for the electrical properties of the device. The studies enabled the determination of the silicide layer thickness and morphology, as well as the silicide/Si interface roughness. Annealing of the Ir/Si structure at 300 and 400 °C caused a formation of an amorphous iridium silicide layer between Ir and Si layers, which thickness is about 5 and 7 nm, respectively, and the Ir/Ir-Si/Si interfaces are smooth. At 500 °C the whole Ir layer completely reacted with Si, forming about 30 nm thick crystalline $IrSi_x$ layer. Irregular grains are visible in this layer. The $IrSi_x$/Si interface is slightly rough. When the annealing temperature increases the reaction depends more on the diffusion of Si atoms into the polycrystalline Ir layer than on the Ir diffusion into the Si substrate. The diffusion of Si atoms is already predominant at 500 °C. The diffraction analysis showed that the most stable phase at 500°C is IrSi.

17:20 Poster I-22

Sequence of phases formations in Al-Mg-Si alloy with Sc and Zr addition during heat treatment

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The addition of a small amount of Sc to aluminum alloys improves their properties, such as strength, heat resistance, welding characteristics, decrease grain size. The beneficial role of Sc is attributed to a high density of coherent precipitates of the Al_3Sc phase formed during decomposition of the solid solution at temperatures between 300 and 550°C. Alloying with Zr in addition to Sc improves both the grain-refining efficiency and thermal stability of the alloys. In the present work the phases and the sequences of their precipitation in Al-1%Mg-0.6%Si (in wt %) alloy with Sc and Zr additions has been studied. Two-step heat treatment was applied: the annealing in the temperature 350°C following by quenching from 540°C. Precipitation of the β′ (Mg_2Si), cubic β (Mg_2Si) and $Al_3(Sc,Zr)$ phases have been observed during the first step of annealing. Annealing at 540°C has led to dissolution of the Mg_2Si precipitates and growing the particles of the $Al_3(Sc,Zr)$ phase. Analytical transmission electron microscopy and high resolution microscopy have been applied for the identification of phases, determination of particle sizes, their distribution and relationship between the phases.

17:20 Poster I-23

Synthesis and characterization of nano-TiO_2 dispersed $Al_{65}Cu_{20}Ti_{15}$ amorphous matrix composite

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As strength to density ratio constitutes on important criterion for selection of materials for structural application in aircrafts, age-hardenable Al-alloy continues to find wide-ranging application in aerospace industry. It is predicted that the maximum strength level of these alloys (~ 500-600 MPa) could be more than doubled in nano-intermetallic dispersed amorphous matrix composite instead of age hardenable crystalline alloy. While several studies concerning development of in-situ nano-intermetallic dispersion in amorphous Al-alloys have been reported, further studies are warranted with regards to synthesis and characterization of ex-situ nano-ceramic oxide dispersed amorphous matrix composites, consolidation of the milled product to develop bulk components and carry out mechanical testing to determine the scope and level of improvement in mechanical properties. Thus, the present study is devoted to developing nano-TiO2 dispersed amorphous (partially/completely) Al65Cu20Ti15 alloy by mechanical alloying followed by a detailed microstructural characterization (by SEM, XRD, TEM) and mechanical property evaluation with samples compacted and sintered under special conditions of warm torsional compaction. Furthermore, differential scanning calorimetry was carried out to determine the transition temperatures and thermodynamic properties of the composite. The result suggests that the present system holds promise for developing a superior Al-based structural material with a novel microstructure. [Partial financial supports from the CSIR, New Delhi project no. 70(0048)/03-EMR-II and DST, New Delhi Indo-Polish project no. INT/POL/P-7/2004 are gratefully acknowledged]

17:20 Poster I-24

Reactivity of a Ti-46Al-8Nb Alloy in Air at 700 - 900 ^{o}C

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A titanium aluminide alloy, Ti-46Al-8Nb (at %), with a lamellar structure ($\gamma+\alpha_2$) was tested for oxidation resistance in air at 700, 800, 850, and 900 °C in isothermal and thermal cycling conditions (1-hour and 20-hour hot-dwell time). The oxidation behaviour was assessed by means of mass change measurements as a function of time. The X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS) were used to determine phase composition and chemical composition of the oxidation products. Surfaces of the oxide scales were examined by optical microscopy (OM) and scanning electron microscopy (SEM). Cross-sections of the selected samples were analysed using both secondary electron images and backscattered electron images (SEI and BEI) as well as transmission electron microscopy (TEM).
In isothermal tests the mass gains increased with temperature, the oxidation kinetics followed approximately a parabolic rate law. The calculated parabolic rate constants ranged from about 10^{-12} to 10^{-10} kg^2/m^4s. The oxide scales were built of Al_2O_3 and TiO_2, the former being the main component of the outermost layer. The innermost layer contained oxygen and all three elements of the alloy with varying local concentrations. The oxidation behaviour of Ti-46Al-8Nb was compared with a commercial titanium alloy, WT4 (Ti-6Al-1Mn), and with selected oxidation-resistant alloys.

17:20 Poster I-25

Intermetallic phase identification on the cast and heat treated 6082 aluminium alloy

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In the technical aluminium alloys besides the intentional additions, transition metals such as Fe and Mn are always present. Even not large amount of these impurities causes the formation a new phase components. The exact composition of the alloy and casting condition will directly influence the selection and volume fraction of intermetallic phases. During casting of 6xxx alloys, a wide variety of Fe-containing intermetallics such as Al-Fe, Al-Fe-Si and Al-

Fe-Mn-Si phases are formed among the aluminium dendrites. The type of these phases depends mainly on the cooling rate and Fe to Si ratio of the alloy. These intermetallics have different unit cell structures, morphologies, stabilities and physical and mechanical properties. As cas billets require require a homogenization treatment to make the material suitable for hot extrusion. During this homogenization several processes take place such as the transformation of interconnected plate like beta-Al_5FeSi intermetallics into more rounded discrete alpha-$Al_{12}(FeMn)_3Si$ particles and the dissolution of beta-Mg_2Si particles. Transformation of beta-Al_5FeSi to alpha-$Al_{12}(FeMn)_3Si$ intermetallics is important because it improves the ductility of the material. Dissolution of Mg_2Si is also important since it will give maximum age hardening potential for the extruded product. In this study, several methods was used such as: optical microscopy, transmission (TEM) and scanning (SEM) electron microscopy in combination with electron dispersive X-ray (EDX) using polished sample, and X-ray diffraction (XRD) to identification intermetallics compounds in 6082 aluminium alloy.

17:20 Poster I-26

Calculations of oxide inclusions composition in the steel deoxidized with Mn, Si and Ti.

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The non-metallic inclusions in steel of controlled size, distribution and composition play an important role in obtaining required structures. The $MnO\text{-}SiO_2\text{-}TiO_2$ inclusions, which are the result of steel deoxidization with Mn, Si and Ti, act as the nucleation centers of acicular ferrite from austenite. This structure offers good mechanical properties and may be obtained even in the case of large austenite grains, eg. in heat affected zone on welding. However, this function of inclusions requires proper chemical composition, which in turn results from the parameters of preceding operations. The aim of the present work is an attempt of designing the steel composition prior to casting, which would allow for obtaining proper $MnO\text{-}SiO_2\text{-}TiO_2$ inclusions during steel crystallization. This was accomplished on the basis of thermodynamic calculations of equilibrium distribution of components between liquid oxide phase $MnO\text{-}SiO_2\text{-}(Al_2O_3)\text{-}TiO_2(Ti_2O_3)$ and liquid steel for decreasing temperature, i.e. the conditions corresponding to the behavior in casting tundish and casting mould. The presence of Al_2O_3 in inclusions must be also considered, as aluminum is common deoxidizing agent and it strongly affects the inclusions composition. The calculations were carried out by means of both FACTSage and authors' own programs. The analysis of phase diagrams $MnO\text{-}SiO_2\text{-}Al_2O_3$ and $MnO\text{-}SiO_2\text{-}TiO_2$ allowed to decide, if oxide solution was liquid in the solidus temperature of steel, which is important for the action of inclusions as the nucleation centers.

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17:20 Poster I-27

Study on phase relations in high-temperature region of Ti-As, Zr-As and Ti-Cu-As systems.

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The copper based alloy containing lead, iron and arsenic is the intermediate product in the recovery of copper from the slag of flash smelting process. The arsenic removal from this alloy may be executed by means of the reaction with titanium or zirconium. In order to analyze this possibility the basic knowledge on As-Cu-Ti and As-Cu-Zr equilibrium diagrams is necessary. However, the data on high temperature regions of these systems are lacking. The aim of the present work is to estimate these phase diagrams based on own experiments, in which the compositions of phases in equilibrium under applied arsenic pressures in closed systems were determined. Arsenic pressure in the system was controlled either through the temperature of pure arsenic in nonisothermal experiment, or through its activity in As-Cu matrix alloy in isothermal experiment. The phases' compositions were then obtained from X-ray microanalysis of quenched samples. As a result, the points of liquidus lines for binary As-Ti and As-Zr alloys were determined and from these the arsenic activities calculated in the liquid phase. The provisional versions of binary As-Ti and As-Zr phase diagrams is suggested. The experiment for As-Ti-Cu system revealed, that AsTi compound is in equilibrium with copper rich liquid.

17:20 Poster I-28

Study on the Cu-Sn-Bi

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The toxicity of lead containing solders caused the strong need of development of other types solders. Although nowadays the most prominent lead-free solders are based on alloys of tin with small amounts of silver and copper, other types, based on Sn-In, Sn-Bi and Sn-Zn systems, are under development As microstructures formed during the solding are related to the phase equilibria of the solding materials and other metals, used as the substrates in electronic devices, the knowledge of thermodynamic properties of alloys is essential for the correct description of the technologic processes.Copper is one of the most popular materials for electronic devices, hence in this work, there are studied thermodynamic properties of Cu-Sn-Bi system. Isopiestic method is applied for the bismuth activity measurements and on the basis of the experimental results interaction parameters are calculated.

17:20 Poster I-30

The charge states of In impurity atoms in PbTe<In>/Si films prepared by modified HWE technique

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Heterostructures on Si substrates formed from AIVBVI semiconductors doped with III A Periodic system group metals will continue to attract a widespread attention both for their fundamental electronic properties and for the applications in infrared (IR) optoelectronic. It is well known that the way to increase the charge carrier density in PbTe, which is needed for photovoltaic IR detectors, is doping with In and Tl. For the preparation of PbTe/Si heterostructures the modified HWE technique has been used. The chemical quantitative composition, X-ray patterns, SEM results, and charge carrier densities of PbTe/Si films, which had been fabricated under different experimental conditions, have been compared in this work. For the first time in this work the amphoteric (donor or acceptor) behaviour of In impurity atoms has been analyzed with respect of Te atoms concentration. It has been detected that at 77 K the high values of charge carrier density of about 10^{18} - 10^{19} cm^{-3}, which are needed for photovoltaic IR detectors, appear only in *n*-type $Pb_{1-y}In_y$ Te/Si films with, if these layers were characterized with little deficiency of Te atoms with regards to stoichiometric relation. If $0.001 < y_{In} < 0.009$, the electron density shows the weak dependence upon the concentration of In atoms. On the contrary, in *p*-type $Pb_{1-y}In_y$ Te/Si films with little excess of Te atoms the hole densities decline fast with increase in In concentration at $y_{In} > 0.007$. For explanation of amphoteric behaviour of In the model based upon the different charge states of impurity atoms has been proposed within frameworks of crystal - quasichemical approximation.

17:20 Poster I-31

Pressure influence on thermoelectric properties of lead

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Investigations of high pressure properties of different materials attract considerable interest of solid matter physics.

We investigate pressure influence on phase transitions of lead.

High pressure has been generated in the diamond anvil cell with Brindgmen anvils of the "rounded cone-plane" type made of synthetic diamonds called «carbonado». Measurements were carried out at room temperature.

Thermoelectromotive force was used as a sensitive parameter. Thermo-emf measurement let to identify conditions of new structural state formation in metals and alloys. Pressure dependences of thermo-emf of lead and relaxation processes at structural modifications were investigated under pressures 5-50 GPa.

According to previous investigations phase transitions in lead take place at 16, 20 and 63 GPa [1], and also some authors report about structural modifications of lead at higher pressures.

During measurements it was shown that lead thermo-emf changed inconvertible after loading. At the analysis of the time dependences of thermoelectromotive force of sample a number of features was revealed. Near 15, 30 and 45 GPa relaxation times change drastically. It may be connected with probable transitions at these pressures. But further work is necessary to corroborate this claim and to determine the nature of these changes.

This work was supported in part by grant BRHE EK-005-X1 (Ural research educational center "Advanced materials").

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17:20 Poster I-33

Activities of interstitial elements inside heat-resistant steels, filler alloys, and weld electrode materials

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Steels are popular engineering materials for industrial use. They are usually alloyed with the other elements (Me: Cr, Mo, V, W, Nb, ... and N) in order to improve the mechanical and corrosion resistance properties.

The modern high-resistant steels (P91, P92, E911, VM12) [1] reached the high strength up to 100MPa at 575-650°C. It is no rare case that these steels have to be weld joined with the other steels, which have different compositions (T/P22, T/P23, T/P24, …). In the case of dissimilar weld joints [2], the mechanical properties usually take a turn for the worse because the phase microstructure changes caused by long-time diffusion during exploitation at high temperature [3]. The most important diffusing elements are the carbon and the nitrogen for the steels. The diffusion flux of these light elements is in close relation to their thermodynamic activities.

The heat-resistant steels, filler alloys, and welding electrodes can be thermodynamically treated as Fe-Me-N-C based closed systems. Phase diagrams and temperature dependent carbon and nitrogen activities of the steels can be calculated using CALPHAD approach [4].

In this contribution, the CALPHAD method is applied. Examples of phase diagrams, temperature dependences of the carbon and nitrogen activities are calculated, plotted, and discussed.

Acknovledgement: MSM/00216/22410 and GD 106/05/H008.

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17:20 Poster I-34

Phase polymorphism of $[Ni(DMSO)_6](ClO_4)_2$ and $[Ni(DMSO)_6](BF_4)_2$ compounds studied by differential scanning calorimetry

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Chlorate and tetrafluoroborate of heksadimethylsulfoxide of nickel (called later HNiC and HNTF respectively) indicate very rich and interesting phase polymorphism. The differential scanning calorimetry (DSC) measurements, which were made using Mettler Toledo DSC 821e apparatus in the temperature range of 253-630 K, show that the phase polimorphism of the title compounds is very similar. HNiC and HNTF melts gradually in two and three steps, with the final melting point at 526 and 614 K respectively. The investigated compounds decompose just above melting point. Their crystallization by cooling undergoes in a specific way because some of their phases can be very easily supercooled, and becomes metastable phases.

HNiC and HNTF indicate several anomalies on the DSC curves connected with the phase transitions, which number and kind depend on so called "thermal history" of the samples. Namely, in dependence of those how fast and how deep was the sample cooled, on DSC curve were observed from one to six anomalies connected with phase transitions between different stable and different supercooled, metastable phases. It was concluded from the values of the enthalpy changes, that some of these phases are so called "orientationally dynamically disordered crystals" (ODIC) and the others are more or less ordered phases.

17:20 Poster I-35

Phase polymorphism of $[Cd(DMSO)_6](ClO_4)_2$ studied by differential scanning calorimetry and transmitted light intensity methods and observed using thermal microscope

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The differential scanning calorimetry (DSC) measurements made for $[Cd(DMSO)_6](ClO_4)_2$ (called HC) in the temperature range of 298-473 K indicated very rich and interesting phase polymorphism of this compound [1]. On the basis of the obtained results we can conclude that the number and kind of the phase trasitions strongly depend on so called "thermal history" of the samples. The crystallization of HC undergoes in a specific way because some of the phases can be very easily supercooled. In the examined temperature range four solid phases of HC have been detected (two of them are stable and two metastable). The title compound melts at 465 K. In order to see if the observed phase transitions are connected with a change of the texture of the sample the transmitted light instensity (TLI) and thermal microscope (TMP) measurements were made.The TMP observation confirmed that all phases, investigated by the DSC method, are the solid ones, and that HC melts at 465 K. The TLI measurements show that some of the phase transitions are connected with a change of the sample texture, and can be observed as anomalies on the TLI curves.

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17:20 Poster I-36

Phase Regularities in the Quasi-Binary Thallium(I) Telluride - Zinc Group Metal Telluride Systems

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The title systems (Tl_2Te - (Zn, Cd, Hg)Te) are polythermal cross sections of respective ternary ones of the type Tl-M-Te, where M is any metal. There is a considerable probability of chemical compounds formation in such systems, while (according to former data of other authors) no compound is formed in the systems Tl_2Te-ZnTe and Tl_2Te-CdTe, and in the Tl_2Te-HgTe only one compound was found. The former data were obtained by differential thermal analysis (DTA). This is the reason why we decided to reinvestigate the systems employing another method namely common thermal analysis (TA). From our studies it appeared that in the Tl_2Te-ZnTe system one compound was formed of component molar ratio Tl_2Te : ZnTe = 9:1 melting congruently at 713 K; in Tl_2Te-CdTe two compounds: 6:1 melting congruently at 704 K and 1:3 melting incongruently at 1050 K; in the Tl_2Te-HgTe system three compounds: 5:1 melting congruently at 696 K, as well as 1:3 and 1:9 both undergoing decomposition at 653 K and 713 K, respectively.

From the comparison of above data some conclusions may be derived: 1^o tendency to compound formation in the series Tl_2Te - (ZnTe→CdTe→HgTe) increases with the increase of the metal atomic weight of the second component: one compound→two compounds→three compounds; 2^o the compound composition on the Tl_2Te side was the nearer to the pure thallium telluride, the greater stability of the second component: 9:1→6:1→5:1, respectively; 3^o the melting point of the compound decreases slightly: 713→704→696 K, respectively.

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Symposium J

Welcome

Over the last 15 years, biomaterials research is oriented to the activation of biomaterial surfaces in order to optimise tissue integration of medical devices. At the present state-of-the-art, functionalization is turning around the grating of polyacrylic acid or polyethylene glycol as spacer molecules for further binding of bioactive molecules such as extracellular matrix constituents, but without any eminent progress. - Since now about 5 years several laboratories have enhanced their competences to develop new technologies in order to obtain biocompatible materials with functionalised surfaces. Several means are available in particular the grafting of bioactive molecules (i) via new spacer molecules to stimulate the target to go to the material surface, and (ii) via cage molecules to provide controlled drug delivery systems. Most of these methods can be applied on all implantable materials, and they are in particular very useful and forthcoming for scaffolds, hybrid medical devices and tissue engineering.

The aim of this symposium is to generate more scientific and industrial co-operation between technical, biological and medical domains. It will subsequently largely be open to researchers from multiple disciplines such as material science and engineering, mineral and organic chemistry, (bio)physics, electro-chemistry, analytics, cell and molecular biology, pharmacology, medicine (orthopaedics, dentistry, ophthalmology, cardiology, urology, visceral surgery).

This symposium will cover wide multidisciplinary fields from Material Science (metals, ceramics, polymers), Surface Physicochemistry, Cell Biology, Biochemistry, Macromolecular Chemistry to achieve the improvement of interface interactions and subsequently of tissue integration of medical devices.
The main topics of the symposium will be:

1. Surface tailoring: physical and chemical surface characterizations; surface formulation, surface energy,
2. Physical and chemical molecular grafting: extracellular matrix constituents, spacer and/or cage molecules;
3. Cytochemical immune-labelling: cytoskeleton, extracellular matrix, focal adhesion contacts; cell membrane receptors, pro-inflammatory markers;
4. Nanopaterning and nanostructural devices;
5. Interaction between biomaterials surfaces with bioactive molecules and cells, cell adhesion;
6. Endogenous biomimetic coatings in appropriate fluids
7. Controlled drug delivery systems;
8. Immobilization of bioactive molecules;
9. Tissue engineering

Scientific Committee:

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- **Hartmut F. HILDEBRAND,** Biomaterials Research Laboratory, Lille Medical University, Lille (France)
- **Julio SAN ROMAN,** CSIC, Institute of Science and Technology of Polymers, Madrid (Spain)
- **Barbara NEBE,** Dept. of Internal Medicine/Clinical Research, Rostock Medical University, Rostock (Germany)
- **Joelle AMEDEE,** Inserm U577 - Biomaterials and Tissue Repair, University Bordeaux-2 (France)

Proceedings

The manuscripts submitted to this symposium and accepted on the basis of the referee procedure adopted for regular papers would be published in the international scientific journal **"Biomolecular Engineering"**.

Programme

Monday, 4 September

Scaffold Engineering
Monday morning, 4 September, 9:00
Chair: Roger Thull, Thomas J. Webster

9:00	Invited oral

Physicochemical, molecular and cellular events of the interaction between metallic implants and biological systems

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Almost all surfaces are electrified being in contact with the extracellular fluid or blood. Reversible or irreversible - structural changes of adsorbed substances can take place. In particular in organic macromolecules bonds together with oxygen bridge bondings may break down, giving rise to structural and/or conformational changes. Conformational changes may arise as a result of an exchange of charge carriers. However, changes can also be initiated by high field strengths, such as occur in consequence of the presence of local elements. A conception for the actual changes of the protein molecule during the adsorption process is up to now not available. The adsorption of hydrogen ions or hydroxyl groups respectively leads to changed potential gradients at the surface of the protein comparable with the potential gradient within the biomaterial/electrolyte interface. A criterion for the suitability of materials as a biomaterial for close tissue contact is the physico-chemical reactivity of the surface. As known, bulks and surfaces have either properties as a metal, a

semiconductor or an insulator. The differences are caused by the electronic structure of the materials described by the density of electronic states and their occupation by charge carriers. The interface of course, plays an important role for instance in the transfer of charge in the electron transport chain, the behavior of cell-material interaction, the adsorption of proteins and other chemical messengers on the material surface. As a summary it is allowed to say: Biocompatibility depends on the electronic structure of the material. Conformational unchanged macromolecules are the prior condition for biocompatibility and control the attachment and probably also the degree of attachment via adhesion proteins.

9:40 Invited oral

Outgrowth endothelial cells from human peripheral blood on nanometric fibroin nets

Sabine Fuchs[1], Antonella Motta[2], Claudio Migliaresi[2], Charles James Kirkpatrick[1]

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Outgrowth endothelial cells (OEC) isolated and expanded from human peripheral blood are an attractive cell source of autologous EC for applications in tissue engineering. On micrometric fibroin fibers OEC formed differentiated endothelial cell layers. After embedding in a wound healing matrix (fibrin hydrogel) OEC migrated from the fibroin scaffolds into the fibrin and formed a microvessel-like network. In this study we investigated the response of OEC to silk fibroin nets with fibers in the nanometric range, that is, the physical dimensions of fibers of the extracellular matrix. The viability of the cells, the formation of EC layers, focal contacts and cellular polarization on nanometric fibroin fibers were studied. The formation of cell contacts with the scaffolding material and cell migration on the fibers are highly important to understand and to be able to guide the process of endothelialization or vascularization of tissue engineered constructs based on newly designed biomaterials. On the nanometric nets OEC formed cell layers with focal adhesions on the single nanofibers (demonstrated by immunofluorescence for focal adhesion kinase). Other molecules relevant for endothelial cell polarization and response of endothelial cells, such as caveolin-1 and its phosphorylated form, were investigated in parallel and revealed polarized OEC, indicating that OEC can use the nanometric fibers as a matrix for cell polarization and migration. Further studies are currently underway to investigate the possible beneficial effects of nanometric fibroin nets on important endothelial cell functions. (This work was supported by grants HIPPOCRATES, EXPERTISSUES from the European Commission)

10:10 Keynote lecture

Microstructures materials for cell guidance

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Micro patterned surfaces containing geometrically defined bioadhesive and non-adhesive domains are useful tools to study cell attachment and groth. Patterned surfaces with different chemical and topographical heterogeneities have been realised by the photoimmobilization of polysaccharides on different substrates using a photomask containing features of defined dimensions and geometry. The cell response to the patterns was studied using several different cell lines which all arranged on the surface in accordance to the features geometry. The cytoskeleton stress fibres and the localisation of focal contacts demonstrated that cells adapted their shape to the pattern dimensions and geometry at least down to 5μm in width. The presence of integrin clusters along the cell borders and at the migrating leading edge together with the actin stress fibres polarisation indicated that the cell adhesion is integrin-mediated and the cell migration is guided by the micro-patterns. It has been also observed that the constrain of the cell in a defined shape, influenced by the pattern characteristics, induced changes in the gene expression. The role of plasma proteins in cell-surface interaction was assessed determining the type and the amount of the proteins adsorbed on the surfaces. Furthermore, a fibronectin coating was realized on the polysaccharide surfaces. The cell response was evaluated leading to the conclusion that the protein coating is essential for an optimal fibroblast adhesion.

Coffee break
Monday morning, 4 September, 10:30

Opening Ceremony
2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Scaffold Engineering (continued)
Monday afternoon, 4 September, 14:00
Chair: Roger Thull, Thomas J. Webster

14:00 Keynote lecture

Repair and regeneration of osteochondral defects in the articular joints

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People suffering from pain due to osteoarthritic or rheumatoidal changes in the joints are still waiting for a better treatment. Although some studies have achieved success in repairing small cartilage defects, there is no widely accepted method for complete repair of osteochondral defects. Therefore, there is room for a new medical approach, which outperforms currently used methods.

The aim of this study is to show potential of using a tissue engineering approach for regeneration of osteochondral defects. The critical review of currently used methods for treatment of osteochondral defects is also provided.

In this study, two kinds of hybrid scaffolds developed in Hutmacher's group have been analysed. The first biphasic scaffold consists of fibrin and PCL. The fibrin serves as a cartilage phase while the porous PCL scaffold acts as the subchondral phase. The second system comprises of PCL and PCL-TCP. The scaffolds were fabricated via fused deposition modeling (rapid prototyping method). Bone marrow-derived mesenchymal cells (MSC) were isolated from New Zealand White rabbits, cultured in vitro and seeded into the scaffolds. Bone regenerations of the subchondral phases were quantified via micro CT analysis and the results demonstrated the potential of the porous PCL and PCL-TCP scaffolds in promoting bone healing. Fibrin was found to be lacking in this aspect as it degrades rapidly. On the other hand, the porous PCL scaffold degrades slowly hence it provides an effective mechanical support.

This study shows that in the field of cartilage repair or replacement, tissue engineering may have big impact in the future. In vivo bone and cartilage engineering via combining a novel composite, biphasic scaffold technology with a MSC has been shown a high potential in the knee defect regeneration in the animal models. However, the clinical application of tissue engineering requires the future research work on scaffold design and cellular delivery.

14:30 Keynote lecture

Dextran and hyaluronan methacrylate based hydrogels as matrices for soft tissue reconstruction

Stephanie Möller[1], Jürgen Weisser[1], Sabine Kussmann[2], Matthias Schnabelrauch[1]

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Polysaccharide hydrogels have become increasingly studied as matrices in soft tissue engineering. Main advantages of these materials are their known cytocompatibility and the possibility to tailor the material properties chemical modification of the educts. In this work cross-linkable polysaccharide methacrylates based on dextran, amino dextrans, and hyaluronan were synthesized and their transformation into stable hydrogels were studied. In comparison to hyaluronan-based materials, the dextran gels showed a reduced water incorporation, better form stability, mechanical strength, and longer durability. The formed gels had no cytotoxic effects against CHO and 3T3 cells, but cells could only adhere inefficiently in long term experiments. Smooth gel surfaces, fibronectin addition and the use of mixed gels improved the adherence of cells. Different scaffold architectures usable as matrices for soft tissue engineering were studied in vitro including porous and perforated gels and a layer-by-layer assembly in which each layer was overgrown with cells. Embedding of cells between two layers inhibited their aggregation to spheroids and cells kept attached at the support. Selected hydrogels were examined in a rabbit model in vivo to study their biocompatibility, stability, and degradation. No signs of inflammation were seen and with prolonged duration the material was degraded and lacunas were formed obviously by immigrating or ingrowing cells. Additionally, in dextran gels an ingrowth of cells from the surrounding into little channels could be observed similar to the sprouting of small vessels. Optimizing their mechanical properties, the dextran hydrogels represent promising candidates as matrices for soft tissue reconstruction.

15:00 Oral

A bone bioengineering with nanocarbon molecules

Oleg Lysko[1], Roman V. Shein[1], Volodymir Frolov[1], Olena Gogotsi[1], Olena A. Kysil[1], Eugenia V. Buzaneva[1], Loredana Carta-Abelman[2], Uwe Ritter[2], Peter Sharff[2], V. L. Dubok[3]

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Now medical treatment for bone defects involves replacement of lost bone with an artificial material. Tissue engineering offers the perspective to regrow missing bone by culturing new cells on synthetic scaffolds.

The first step of our investigations is aimed to choose potential bone scaffold material, to develop models of nanocarbon molecule behaviour (fullerenes, multi-walled carbon nanotubes MWCNT) in environment of simulated body fluid (SBF) [1]. Main process was studied: nanocarbon molecules participation in the formation of hydroxyapatite crystals which are formed during bone mineralization. We found that modified C60 molecule and MWCNT scaffolds, having bio- positive or negative charge in nanofluids, provide the best environment for hydroxyapatite crystal growth in SBF with chemically synthesised hydroxyapatite Ca10(PO4)6(OH)N nanopowder. The perspective of the investigation area have been confirmed by pioneering results at University of California, Riverside that demonstrates the topography single bone forming cell on MWCNT [2].

References:
1. O. Lysko et al. Bulletin of the University of Kyiv. Series: Physics and Mathematics, 2002, 3, 323
2. Laura Zanello et al. Nano Lett. 2006, 6, 562.

15:20 Oral

Surface tailoring of nanocarbon molecules for photosensitizers: biomolecular engineering of nanocarbon fluids

Iryna Sporysh[1], Olena A. Kysil[1], Eugenia V. Buzaneva[1], Lars Weber[2], Loredana Carta-Abelman[2], Uwe Ritter[2], Peter Sharff[2]

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The advanced study of nanocarbon molecules as bioactive molecules allows fundamental investigation and developing biotechnologies to obtain biocompatible materials with immobilized surface. To use nanocarbone molecules (fullerene and short carbon nanotubes CNT) in biocells it is necessary to immobilize the surface of molecules for preparing aqueous and biosolution with high molecule content, which forms nanofluids. Fullerene or CNT fluids - several molecules C60 and CNT surfaces - novel water soluble fullerene and CNT derivatives with oxygen groups and mono and polynucleotides[2]. Using these results we developed biotechnology of nanocarbon fluids design in water and biosolutions as model cells. Comparative analysis photoluminescent spectra (PL) immobilized C60 and short multi-walled CNT fluids was carried out after identification tailoring molecules by methods of absorption spectroscopy at UV-vis range and vibration spectroscopy at IR range. The results demonstrate: PL spectra of nanocarbon molecules separately functionalised by mono- (adenosine monophosphate) and poly- (botanical ss- and ds-DNA) nucleotides have intensive band in 550-850nm range excited by light with wavelengths at 325-440nm range. The PL band narrows, intensity increases for nanocarbon fluids changed by the contents of tailoring biomolecules and oxygen, hydroxyl groups.

Coffee break

Monday afternoon, 4 September, 15:30

Biocoating and Cell Adhesion

Monday afternoon, 4 September, 15:50

Chair: Barbara J. Nebe, Charles James Kirkpatrick

16:00 Keynote lecture

Chemical and physical surface modifications of biomaterials to control adhesion of cells

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Colonisation of implants and scaffolds for tissue engineering is dependent on the presence of adhesive proteins, which represent specific cues for cellular adhesion receptors (integrins). Adsorption of proteins from surrounding liquids depends on the physicochemical surface properties of materials, which also control their conformation and hence functional activity. The majority of polymers for tissue engineering applications are quite hydrophobic and do not support the conformation of adhesive proteins. Therefore colonisation of scaffolds can be delayed or impaired. We explored different techniques to modify the surface of biomaterials to improve their cell contacting properties. Organosilanes have been applied to create self assembled monolayers of a defined molecular composition on inorganic materials. Striking differences in the activities of integrins particularly regarding signal transduction and extracellular matrix formation were observed. It turned out that negatively charged substrata such as carboxylic functions have superior properties compared vs. hydrophilic hydroxyl or amine surfaces. We modified also the surface of hydrophobic (polymeric) materials with poly (ethylene glycol) block copolymers (Pluronics). It turned out that low coating concentrations enhanced adhesion of cells on hydrophobic surfaces. The effect seems to be based on the stabilisation of protein conformation by neighbouring PEG molecules. Finally we explored the ability of the layer-by-layer technique to assemble biogenic macromolecules in matrix-like structures on charged surfaces. We applied chitosan, heparin and fibronectin to obtain control over the adhesive properties of the substratum. Overall, these generic techniques can be applied to different types of surfaces to obtain control over the adhesive properties of biomaterials.

16:30 Oral

Fibrils of Different Collagen Types Containing Immobilised Proteoglycans (PGs) as coatings: Characterisation and Influence on Osteoblast behaviour

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Introduction. Collagen is used as a coating on titanium implants and as a scaffold material in bone tissue engineering. Proteoglycans (PGs), consisting of a protein core connected to glycosaminoglycan chains, may be bound to collagen fibrils. PGs can affect osteoblast behaviour and also bind and influence the effect of growth factors such as TGF-β1, which stimulates extra-cellular matrix production. The aim of this work is to characterise fibrils of the collagen types I, II and III with regard to amount of decorin and biglycan bound and to study the reaction of primary rat (RO) and human (HO) osteoblasts to titanium surfaces coated with fibrils containing decorin and biglycan. The influence of PGs on the effect of TGF-β1 on osteoblasts was also examined. Adhesion, proliferation and collagen synthesis were investigated. Methods Fibrils of collagen types I, II and III containing decorin and biglycan were formed at 37ºC in a 30 mM phosphate buffer at pH 7.4 at different PG:collagen ratios. PG content of fibrils was determined colormetrically. Fibril morphology was studied by atomic force microscpoy (AFM). Results and Dis-

cussion. Decorin caused a reduction in fibril diameter, but biglycan did not. Collagen type II fibrils bound more PGs than type I and III fibrils, with more biglycan than decorin bound by all collagen types. Biglycan promoted the formation of focal adhesions, and also stimulated HO proliferation, as did decorin but not as strongly. Biglycan but not decorin inhibited RO collagen synthesis. TGF-β1 reduced the proliferation and enhanced the collagen synthesis of HOs. However the decrease in proliferation was lower and the increase in collagen synthesis was higher when decorin was present instead of biglycan. Conclusions. The ability of collagen fibrils to bind PGs varies according to PG and collagen type. Biglycan and decorin influence primary osteoblast behaviour by themselves, but also influence the effect of TGF-β1.

16:50 Oral

Plasma-assisted amino functionalized titanium - a surface for enhanced osteoblast functionality

Birgit Finke[1], Frank Lüthen[2], Karsten Schröder[1], Barbara J. Nebe[2], Claudia Bergemann[2], Rainer Bader[2], Joachim Rychly[2], Andreas Ohl[1]

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Titanium and its alloys are widespread applied in bone replacement. It is well known, that the behaviour of osteoblasts is determined by the surface characteristics like roughness and oxide composition. In more detail, surface properties control the adhesion of biomolecules, which induce a cellular response. Hyaluronan (HyA) could be a key substance for the design of the interface between implant and osteoblasts. HyA is a negatively charged molecule, which will preferably adsorb to a positively charged titanium surface. Therefore, a thin plasma polymerized film with positively charged amino groups is fixed at the titanium surface. The polished metal surface was activated by a low pressure oxygen plasma for the fixation of an about 50-100 nm thin layer of a plasma-polymerized allylamine. Therefore, a microwave-excited pulsed plasma was applied at a pressure of 50 Pa. Collagen coupling was applied to compare cell adhesion to the plasma polymer with cell response to ECM molecules.The coated surface was analyzed by XPS, FTIR, Zeta potential and contact angle measurements.Plasma polymerization only partially sustains the structure of the allylamine molecule. The nitrogen concentration at the film surface is N/C=25 % and therewith near the theoretical value of a chemically polymerized film (N/C=33 %). Amino groups are present, confirmed by Zeta potential, FTIR, and chemical derivatization XPS measurements. The film properties differ from chemically polymerized allylamine in oxygen content and nitriles, verified by XPS and FTIR spectra. Contact angle measurements demonstrate a medium hydrophilic film surface advantageous for cell adhesion. Osteoblast cell adhesion on allylamine plasma polymer coated titanium surfaces was superior to covalent coupled collagen.

17:10 Oral

Plasma surface modification of chitosan membranes using different monomers aiming to improve the adhesion of Osteoblast-like cells

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Surface is the first contact with the living tissues when an implant is placed in the body. Therefore, surface characteristics of biomaterials play an essential role in the performance of a potential device. The aim of this study was to modify the surface of chitosan membranes, prepared by solvent casting, and to study the effect of the modifications on osteoblast-like cells adhesion/proliferation. Plasma activation and then grafting by subsequent immersing in monomer solution was performed. Two monomers were used to compare the influence of different functional groups on cell adhesion; acrylic acid (AA) as a source of carboxyl groups and vinyl sulfonic acid (VSA) for sulfonic groups' introduction. All treatments resulted in more hydrophilic (contact angle) surfaces. The monomer grafting on the surface was also confirmed by X-Ray photoelectron spectroscopy (XPS); S peak appears in the VSA spectrum and C=O peak intensity increase after AA grafting. Fournier transform infrared spectroscopy (FTIR-ATR) showed no significant differences between treated and original materials. This was expected since the modification was performed only on very surface layer of the materials and not in the bulk. Scanning electron microscopy (SEM) analyzes has indicated that surface topography was not affected by the different treatments. Cell adhesion and proliferation tests (SaOs-2) revealed that the presence of sulfonic groups significantly increases both SaOs-2 proliferation (dsDNA quantification) and the activity of the osteoblasts marker Alkaline Phosphatase (ALP) compared to untreated or AA-grafted membranes. Cell morphological analysis (optical microscopy, SEM) confirmed these results.The present results might be very useful for tailoring cell adhesion/proliferation on chitosan biomaterials. This is particularly relevant, as it is known that cell adhesion and proliferation are major factors to consider when applying biodegradable polymers as scaffolds for tissue engineering.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Surface Nanoparticles

Tuesday morning, 5 September, 9:00
Chair: Halina Podbielska, Rolando Barbucci

9:00 Oral

Enhanced Bio-compatibility of Ti-6Al-4V by Laser Surface Engineering

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The present study aims at enhancing the wear resistance and bio-compatibility of Ti-6Al-4V by laser surface melting and nitriding. Laser surface treatment has been carried out by melting of sand blasted Ti-6Al-4V substrate using a high power continuous wave DI-ODE laser with argon and nitrogen as shrouding environment (at a gas flow rate of 5 l/min). Following laser surface treatment, a detailed characterization (microstructure, composition and phase) and property evaluation (wear, corrosion, bio-compatibility) of the surface have been carried out. Laser surface melting increased the volume fraction of acicular (quenched-in a) Ti and decreased the volume fraction of b phase in the microstructure (as confirmed by X-ray diffraction analysis). On the other hand, laser surface nitriding led to formation of dendrites of titanium nitrides. The microhardness is improved to a maximum of 450 VHN (in laser surface melting) and 900-950 VHN (in laser surface nitriding) as compared to 260 VHN of as-received substrate. Surface melting increased the corrosion potential (E_{corr}) and primary potential for pit formation (E_{pp1}) significantly as compared to as-received Ti-6Al-4V. However, processed under similar conditions, surface nitriding shifted E_{corr} marginally to the nobler direction, and increased E_{pp1} as compared to Ti-6Al-4V. Biocompatibility behavior showed a better cell viability due to surface nitriding and inferior cell viability due to surface melting as compared to as-received Ti-6Al-4V.

9:30 Oral

The Effect of Process Parameters on the Liquid Flame Spray Generated Titania Nanoparticles

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Nanoparticles have become important in many applications. It is essential to be able to control the particle size, because the properties of nanoparticles change dramatically with particle size.

An efficient way to create nanoparticles is to use aerosol processes. In this study a method called Liquid Flame Spray is used. In Liquid Flame Spray a high speed hydrogen/oxygen flame with liquid precursor is used. We have studied the effects of different parameters on particle size of titania. Titania is used as a photocatalyst, but the particle size and the crystal structure is important when looking at the efficiency. Using the flame methods dopants and sensitizers can easily be introduced in order to improve the photocatalytic activity. The generated nanoparticle size has been measured by aerosol instrumentation and the morphology has been verified with transmission electron microscopy.

In Liquid Flame Spray method there are several adjustable parameters. Those are precursor feed rate into the flame; concentration of the precursor; solvent used in the precursor; precursor material itself; mass flow of combustion gases and of course the torch used.

We used metal organic based titanium precursors. Alcohol solvents, predominantly ethanol and 2-propanol, were used in precursors. There were great differences in particle production between the precursors. Differences could also be seen for various solvents. As for precursor feed in the flame the more mass is introduced the larger the nanoparticles are i.e. precursor concentration and precursor feed rate have an impact on particle size. A similar phenomenon can be discovered for the combustion gases. Torch design also plays an important role in controlling the particle size.

9:50 Oral

The use of functionalized gold nanoparticle monolayers in the culturing of stem cells

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Embryonic stem (ES) cells are pluripotent cells with the ability to differentiate into all specialized cell types, making them a very promising foundation for stem cell-based regenerative therapeutics. Unfortunately, several obstacles have hindered the progress in the development of ES cell-based therapies. Perhaps the most debilitating hurdle is the lack of a reliable and robust cell differentiation system making it quite difficult to produce homogeneous and large scale cellular populations of a specific lineage. The present paper describes our work in using monolayers of gold nanoparticles to control the chemical environment for the growth of embryoid bodies (EBs), an intermediate state prior to the induction of lineage specific differentiation. We find these monolayers can be made directly within the commercial culture plates used for cell culturing. It is also possible to achieve surface functionalization with a wide variety of molecules (such as thiols, amine compounds, peptides). Through this method, the transparent nature of the monolayers enables the use of optical microscopy to monitor the stem cell culturing process and spectroscopic characterization with a plate reader can be used to screen for a variety of experimental conditions. Current protocols do not generate EBs with uniform size and shape, which is attributed to the lack of a robust differentiation strategy. Our preliminary experiments indicate that hydrophobic polymers like PDMS not only induce the uniform formation of cell aggregates but also may play a supporting role in the proliferation and development of EBs. We find that functionalized gold nanoparticle monolayers provide better defined surfaces in which to study the critical role of protein adsorption and control of subsequent cell attachment.

10:10 Oral

Ti-cp functionalisation by deposition of organic/inorganic silica nanoparticles

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Owing to its acceptable mechanical properties and excellent biocompatibility, titanium is the metal of choice in orthopaedics and cardiovascular surgery. In many surgical operations, chemicals and/or biomolecules (such as antibiotics or growth factors) are used in conjunction with prostheses, so as to avoid or stimulate targeted biological events. Often, immobilisation instead of release of such molecules is preferred to optimise their effects, thus avoiding ectopic transformations. A versatile method for the functionalisation of pure Ti is shown here, which should facilitate the preparation of purpose-built implants.

In order to avoid the hydrolysable Ti-O-Si bond found in directly silanised Ti, we use organic/inorganic silica colloids, derived from commercially available 25 nm Ludoxâ silica nanoparticles. Prior to deposition, the particles are functionalised by a propylsemicarbazide moiety, which confers them excellent reactivity towards carbonyl containing molecules (e.g. on appropriately designed linkers, or oxidised glycoproteins). Semicarbazide chemistry allows a highly chemoselective, covalent attachment of the molecules of interest to the colloids. After spin-coating onto the Ti substrates, the colloids were shown by SEM to form a uniform layer, and to be very strongly adsorbed; the reactivity of the supported semicarbazide functionalities being maintained.

Biological assays with MC3T3-E1 osteoblasts revealed an excellent cytocompatibility as shown by the assessment of cell viability, vitality and morphology.

In our further studies, growth factors will be bound on the functionalized surfaces, and their efficiency on the proliferation and differentiation of osteoblasts will be tested.

Coffee break

Tuesday morning, 5 September, 10:30

Physical and Chemical Surface Coatings

Tuesday morning, 5 September, 11:00

Chair: Qingling Feng, Roman V. Shein

11:00 Keynote lecture

Silica sol-gel matrix doped with photolon molecules for sensing and medical therapy purposes

Halina Podbielska, Agnieszka Ulatowska-Jarża, Gerhard Műller, Joanna Bauer, Uwe Bindig

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Photolon is one of the new photosensitizers that found applications in photodynamic therapy (PDT). Its chemical structure has a partial reduced porphyrin moiety and molecular structure is comparable to chlorine e_6, which can be isolated after hydrolysis of the 5-membered exocyclic β-ketoester moiety of pheophorbide. For this study we have produced Photolon doped sol-gel matrix in form of films deposited on silica fibers cores. The matrix was produced from sols prepared from the silicate precursor TEOS mixed with ethyl alcohol. The sol-gel material was prepared with factor R=20, where R denotes the solvent to precursor molar ratio. Hydrochloride acid was added as catalyst in proportion to ensure the acid hydrolysis (pH»2). The mixture was stirred at room temperature for 4 hours by means of magnetic stirrer (speed 400 rot/min). The coated fibers were placed in various environments; liquid and gaseous, with various pH and with various zinc cations concentrations. The reactions were studied by exploiting spectroscopic methods, whereas the fluorescence response was examined. It was demonstrated that Photolon immobilized in sol-gel matrix may react with the environment and shows visible changes depending on the external changes. Furthermore, it was observed that these reactions are reversible. These biomaterials are also examined as carriers for PDT. It was proved that they posses bacteria toxic activity.

11:30 Oral

Preparation of gradually componential metal electrode on solution-casted Nafion™ membrane

Ren-Jei Chung[1], Li-Chun Chen[1], Ming-Fa Hsieh[2], Tsung-Shune Chin[1]

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A typical ionic-polymer-metal composite consists of a thin perfluorinated ionomer membrane and noble metal electrodes plated on both surfaces. It undergoes a large bending motion when an electric field is applied. With proper arrangement and package, a great number of "smart devices" including artificial muscle are anticipated. In this study, a solution-cast route was used to prepare the electro-active polymer membrane, Nafion™ (DuPont Co.), and Pt electrodes were fabricated onto the membrane through electro-less plating. The results showed that weights facilitate the crystallnity of the solution-cast films. The Iamor.($778cm^{-1}$)/ Isymm($1156cm^{-1}$) in FTIR increased with loadings. The shift in WAXD peaks also supported this

fact. The number and size of the crystalline domains of solution-cast film decrease as studied by SAXS. The Young's Modulus of solution-cast film decreases as increasing weight because of the loss of crystallinity (180~140 MPa). A finely dispersed platinum particle deeper and gradient penetrating within the near-boundary region with a smaller average particle size and more uniform distribution could be obtained through a reverse electro-less plating. Its surface roughness is 3nm comparing to 52nm of a traditional process. But its surface resistance is too high (3.5 ohm) to activate the bending. To solve this problem, we coated the second Pt electrodes by a typical electro-less plating, and the resistance decreased to 0.7 ohm. The results depicted that the fabricated IPMC shows longer bending lifetime than typical IPMC. In a 0.09% NaCl solution, the device was able to vibrate for 8 hours under a 5V, 0.1 Hz actuation.

11:50 Oral

Microstructure analysis on the interface between zirconia/alumina composites and bio-coating glass

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The objective of this study was to analyze the microstructure on the interface between zirconia/alumina composites and bio-coating glass. Four kinds of zirconia/alumina ceramics were selected as the research substrate while VITA alpha glass-ceramic as the coating-glass. Energy spectrum analysis, optical and electron scanning microscope were observed on the junction between substrate and coating glass. The volume fraction of each phase, grain shapes and sizes were determined from SEM micrographs. From the experiment we can find zirconia particles migrated into grain boundaries and located at three or four grain junctions of alumina particles. The ceramic microstructure of superficial zone is permeated by the glass through grain boundaries and partially dissolves the grains. Neither on a macroscopic level in a polished section, nor on a microscopic level in a fracture surface were cracks or porous areas detected. The homogeneous bond between Vita alpha glass-ceramic and Z/A composites are distinct. Therefore, this system composed of zirconia/alumina composites and Vita alpha coating-glass would satisfy matching properties of dental requirements and it has a great future to be used as all-ceramic restoration materials.

12:10 Oral

Cellulose based micromotors; rotation in a magnetic field

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In the search for materials systems where molecular recognition is controllable, an exciting new phenomenon has been recorded on film under a cross-polarised optical microscope for the very first time. The synthesis of mesoporous silica by sol-gel mineralization of cellulose nanorod nematic suspensions has been studied in the past,[1] but the reaction under a magnetic field has never been investigated. The presence of a thermal gradient in a fluid mixture induces a relative matter flow of the components and is known as the Ludwig-Soret effect.[2] Cellulose microcrystals are helical in nature and align under a magnetic field.[3] By applying a magnetic field in the region of 50 mT to the cellulose suspension, a novel spectacle is observed. The effect displayed is confined within a microdrop and is similar to a Tornado vortice as it rotates at high speeds moving erratically around the drop. Like a contained storm system, a combination of factors governs the resultant rotations. The helicity of the ionic cellulose, together with the reacting of the pre-hydrolysed silanol solution evolving gases is not enough to explain the high speed rotation, neither is the presence of a thermal gradient in the fluid. The aim of this paper is to report the phenomenon and to propose hypotheses as to what is actually occurring and harness this new discovery. With further research, this micro-tornado could be developed into a new generation of chemical motor.

1: E. Dujardin et al. *J. Mater. Chem*. **2003**, 13, 696

2: R. Piazza et al. *Phys. Rev. Lett*. **2002**, 88, 208302

3: J. F. Revol et al. *Liq. Crystals***1994**, 16, 127

Lunch break

Tuesday afternoon, 5 September, 12:30

Biological Surface coatings and Hydroxyapatite

Tuesday afternoon, 5 September, 14:00

Chair: Thomas Groth, Wojciech Swieszkowski

14:00 Keynote lecture

Improved initial osteoblast's functions on amino-functionalized titanium surfaces

Barbara J. Nebe[1], Frank Lüthen[1], Birgit Finke[2], Karsten Schröder[2], Claudia Bergemann[1], Joachim Rychly[1], Rainer Bader[3], Andreas Ohl[2]

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Adhesion and spreading of cells on biomaterials are integrin-mediated processes. But recent findings indicate a key role of the matrix substance hyaluronan (HA) in interface interactions [Zimmermann E et al. 2002]. Because HA is a negatively charged molecule we assume that a biomaterial surface with an opposed charge could boost the cell's first contact to the surface until cells synthesize matrix proteins on its own to mediate the fundamental integrin contact. Polished titanium cp (Ra 0.19µm) was coated with a thin layer of a

plasma polymer with amino-groups (Ti PPA). For this purpose, allylamine was polymerized with a microwave excited, pulsed, low pressure, gas-discharge plasma. Collagen was immobilized on Ti PPA with PEG-DA. Human osteoblasts MG-63 were cultured in serum-free DMEM to avoid masking of the coated surface with adsorbed proteins. Adhesion and cell cycle phases were calculated by flow cytometry. Time dependent spreading of cells and stained actin cytoskeleton were measured using confocal microscopy. For quantification of gene expression of osteogenic markers, we used a real-time TaqMan RT-PCR assay.
Ti PPA is significantly advantageous concerning initial adhesion and spreading during the first hours of cell's contact to the surface. The proliferation of osteoblasts is positively influenced. Otherwise there is only a slight effect on gene expression of the differentiation markers alkaline phosphatase, collagen I and bone sialoprotein. Therefore, future experiments are directed on the focus if the advantage in the initial contact phase of cells on amino- functionalized titanium is still to be detect on the level of differentiated cell function like protein expression and mineralization. Our results demonstrate that functionalization of titanium with positively charged amino-groups is sufficient enough to significantly improve initial steps of the cellular contact to the material surface.

14:30 Oral

Photo-luminescent hydroxyapatite coatings through a bio-mimetic process

Ren-Jei Chung[1], Huai-Yu Cheng[1], Hsiao-Wei Wen[3], Ming-Fa Hsieh[2], Tsung-Shune Chin[1]

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Hydroxyapatite (HAp) is the major inorganic component in natural bones and has been widely studied and prepared in many forms for orthopedic and dental applications. In this study, we successfully manufactured crystallite HAp on to Si(100) through a bio-mimetic process. The deposition of hydroxyapatite two-dimensional films on Si substrate was carried out using a supersaturated solution containing Ca2+ and PO43- ions. All chemicals used were of analytical grade. The calcium ion and phosphate ion sources were hydrated Ca(NO3)2.4H2O and (NH4)2HPO4 respectively. All preparations were done at room temperature, i.e. 298K. In the previous studies, stimulated body fluid (SBF) was commonly used to produce the HAp coatings through a series of bio-mimetic processes. However, the precursor ions of SBF are complex and usually lead to a surface of specific crystalline with incisive edges, neither a uniform plane. Here we used a simplified precursor to insure pure and uniform HAp coatings. GIA-XRD diffraction pattern insured the crystalline structure of HAp coatings. FE-SEM images showed that the coatings were composed of granular islands of HAp with a roughness of 50nm after two days immersion. FTIR data showed that functional groups of HAp increased with the immersion days of preparation. And the absorption wavelength between 1420cm-1-1480cm-1 belonging to CO32- implied the existence of carbonated HAp. XPS results indicated that there were two kinds of binding structures of Ca2+ cation.Pulsed He-Cd laser with a wavelength of 325nm was used as the excitation sources of the photo-luminescent measurement with a scan rang from 350nm-900nm. Results showed that the coatings emitted specific light with a peak value of 465nm and 515nm. The intensity of emitting light was greater with the coatings were thicker. And a blue shift phenomenon was also observed. The PL properties can be useful in in situ bio-imaging or bio-chip application associated with bio-optical electronics.

14:50 Oral

Biological and physico-chemical assessment of hydroxyapatite (HA) with different porosity

Jean-Christophe Hornez[1], Feng Chai[2], Nicolas Blanchemain[2], Michel Descamps[1], Hartmut F. Hildebrand[2]

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HA with specific internal porosities was loaded with different antibiotics (ATBs) and then tested on its microbiological effectiveness. The HA purity was controlled with X-ray diffraction, IR and Raman spectrometry. Micro- and meso-porosities were obtained by varying the sintering temperature and/or adding graphite and PMMA as porogenous agents. The biological tests concerned cell viability, proliferation and morphology (SEM), and the cytochemical staining of actin and vinculin. The micro- and meso-porous HA samples had an internal pore size of 1-10 µm and 10-50 µm respectively. X-Ray Diffraction and FTIR confirmed the high purity of the HA. The *cell viability tests* with L132 cells confirmed the excellent cytocompatibility of HA, the graphite powder and the ATB Vancomycine. *Proliferation rate* was assessed with MC3T3-E1 osteoblasts. All HA samples produced a higher proliferation than the controls; the micro-porous HA inducing the highest cell growth. The ATB impregnated HA also stimulated cell proliferation but in lower extend. Cytochemical staining of osteoblasts revealed a well developed *cytoskeleton* with strong stress fibres. Labelling of the focal adhesion contacts with anti-vinculin showed a less developed adhesion process in the cells on the different HA substrates. It was possible to realize a highly pure Hydroxyapatite with different but controlled porosities by varying the sintering temperature and/or by porogenous agents. This purity and the micro-porosity stimulate significantly cell growth. These efficiencies will now be improved by other methods of functionalization for controllable drug delivery system in the HA porosity volume.

15:10 Oral

Antibacterial activation of hydroxyapatite (HA) with controlled porosity by different antibiotics

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In order to prevent the increasing frequency of per-operative infections, bioceramics can be loaded with anti-bacterial agents, which are released with respect to their chemical characteristics. HA was elaborated with specific internal porosities and then tested on its microbiological effectiveness. Unpolished dense HA wafers and with micro- and meso-porosities were used for the antibacterial tests on *S. aureus,S. epidermidis* and *E. coli.* HA samples were impregnated for 7.5, 15, 60 min and 24 h with Vancomycine, Ciprofloxacine and Gentamycine. The ATB effectiveness was studied in PBS and in human plasma by the disk and liquid agar diffusion tests. The impregnation time plays a significant role for the duration of release and for the antibacterial efficiency, which was longer but not higher for the higher concentration. The ATB impregnated micro-porous HA induced significantly stronger and longer anti-bacterial effects than meso-porous samples, the antibacterial effect of dense samples was very short for all ATBs. The most important difference in the duration of the ATBs effectiveness was observed with the release media. The ATB release in PBS is slow and the antibacterial effectiveness prolonged with respect to the ATB and/or the bacterial strain. When extracted in the physiologically more relevant human plasma, the antibacterial effect was not longer than 72 h. This arise the question about the relevance of PBS or other so-called physiological buffers. The micro-porosity stimulates significantly the increase the adsorption capacity, and prolongs the release time of bioactive molecules such as antibiotics, even under strongly realistic physiological conditions. These efficiencies will now be improved to prolong the effect of bioactive molecules by physical or chemical properties.

Coffee break

Tuesday afternoon, 5 September, 15:30

Biological Surface coatings for Drug Delivery Systems

Tuesday afternoon, 5 September, 15:50

Chair: Aidan M. Doyle, Matthias Schnabelrauch

16:00 Keynote lecture

New Polymer Drugs as Bioactive Surface Coatingsd for Drug Eluted Coronary Stents

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The application of minimally invasive surgery for the treatment of coronary disease by the implantation of metallic "stents" has become a revolutionary technique with relatively good results against myocardial infarction.The use of biocompatible polymeric coatings offers a good way for the isolation of the metallic wall of the stent and the vascular medium, as well as a tool for the controlled release of specific drugs to improve the antithrombogenic character of the device and to control the restenotic process that is produced in a noticeable number of patiens at medium or long term. We work with new bioactive polymeric systems incorporating specific drugs with potent antiaggregating properties for platelets. Hydrophobic systems based on flexible alkyl acrylate copolymer systems present excellent adhesion to the surface of metallic stents, very high stability and good control of the delivery of the corresponding antiproliferative drug during several days to weeks depending on the application of the coating with a barrier layer of the same polymer system. The polymer coating offers good properties as support of antiproliferative compounds. Several well known drugs based on taxol, simvastatine, and rapamycin have been estudied and the release behaviour has been tested in vitro.

16:30 Oral

Biomimetic apatite coatings-carbonate substitution and preferred growth orientation

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Biomimetic apatite coatings were obtained by soaking chemically treated titanium in SBF with different HCO_3^- concentration. XRD, FTIR and Raman analyses were used to characterize phase composition and degree of carbonate substitution. The microstructure, elemental composition, and preferred alignment of biomimetically precipitated crystallites were characterized by cross-sectional TEM analyses. According to XRD, the phase composition of precipitated coatings on chemically pre-treated titanium after exposure to SBF was identified as hydroxyl carbonated apatite (HCA). A preferred c-axis orientation of the deposited crystals can be supposed due to the high relative peak intensities of the (0002) diffraction line at 2Theta = 26° compared to the 100% intensity peak of the (2131) plane at 2Theta = 32°. The crystallite size in direction of the c-axis of HCA decreased from 26 nm in SBF5 with a HCO_3^- concentration of 5 mmol/l to 19 nm in SBF27 with a HCO_3^- concentration of 27 mmol/l. Cross-sectional TEM analyses revealed that all distances correspond exactly to the hexagonal structure of hydroxyapatite. The use

of an auto-correlation method indicates that most of crystallites c-axes are distributed in a cone with an apex angle of about 30° around a preferential orientation perpendicular to the titanium surface. The HCO_3^- content in SBF also influences the composition of precipitated calcium phosphates. Biomimetic apatites were shown to have a general formula of Ca10-x-yMgy(HPO4)x-z(CO3)z(PO4)6-x(OH)2-x-w(CO3)w/2. According to FTIR and Raman analyses, it can be supposed that as long as the HCO3- concentration in the testing solutions is below 20 mmol/l, only B-type HCA ($0 < z < 1$; $w = 0$) precipitates. At higher HCO_3^- concentration, it can be assumed that AB-type HCA ($z = 1$; $0 < w < 1$) forms.

16:50 Oral

Functionalization with Cyclodextrins of PVDF membranes for Controlled Delivery of Chlorhexidine-Diglyconate

Francois Boschin[1,3], Stephane Leprêtre[2], Nicolas Tabary[2], Nicolas Blanchemain[1], Elisabeth Delcourt-Debruyne[3], Bernard Martel[2], Michel Morcellet[2], Hartmut F. Hildebrand[1]

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Guided Tissue Regeneration is a technique, which promotes by the interposition of a membrane the proliferation of the periodontal cells. The results of this technique are limited by the bacterial contamination of the membranes. The evaluation of the polymerization and grafting conditions on the membranes of β-cyclodextrin (β-CD) and maltodextrin (MD) was performed to select the parameters of time and temperature of polymerization. Under the optimal conditions, a grafting rate of 12% of the weight of the membrane was obtained. The physical and chemical analyses showed that the Chlorhexidine-Diglyconate (dig-CHX) formed inclusion complexes with the β-CD and the maltodextrin. The *in vitro* studies of the dig-CHX release showed that β-CD prolonged the release duration and increased the released quantity. The biological tests showed a good biological tolerance with regard to polymers of β-CD and MD, and of the membranes functionalized by CD and MD.

The impregnation of the membranes by dig-CHX leads to an increase of the toxicity in cell cultures of the membranes proportionally to the quantity of the dig-CHX incorporated. Only weakly toxic amounts of dig-CHX for different cell types were selected according to the clinical application and to the biological results. These incorporated dig-CHX concentrations appeared to be ineffective in microbiological assays. The use of β-CD or MD provides new means to improve the release kinetics of bioactive molecules, but the use of the CHX does not seem suitable to impregnate RTG membranes.

17:10 Oral

Preparation and characterization of novel starch-poly-ε-caprolactone microparticles incorporating bioactive agents for drug delivery and tissue engineering applications

Elizabeth R. Balmayor, Kadriye Tuzlakoglu, Helena S. Azevedo, Rui L. Reis

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One limitation concerning the delivery of bioactive agents is their relatively short half-life when administered into the body. Incorporation of bioactive agents into depot vehicles provides a means to increase their persistence at the disease site in order to confine its availability to a targeted region and thus avoid stimulation at undesired sites. In this work, starch-polycaprolactone (SPCL) microparticles were developed to be used in drug delivery and TE applications.

SPCL microparticles were prepared by using an emulsion solvent evaporation technique and different experimental conditions were evaluated. They were characterized in terms of morphology (scanning electron microscopy) and size distribution (sieving). In addition, their chemical structure was studied (Fourier infrared spectroscopy). To evaluate the potential of developed microparticles as a drug delivery system, dexamethasone was used as model drug and as an osteogenic factor. DEX was entrapped into SPCL microparticles at different percentages. The loading efficiencies and release profiles were evaluated.

By using the methodology describes herein, it was possible to obtain particles with a spherical morphology, exhibiting different surfaces, from smooth to porous, and with sizes 100-550 micrometer. The release behavior of the entrapped molecules seems to be governed by diffusion and degradation of the polymeric matrix. Therefore, these microparticle systems seem to be quite promising for controlled release applications and, at the same time, as carriers of important differentiation agents in the field of TE.

Wednesday, 6 September

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session

2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

Cell Attachment
Wednesday afternoon, 6 September, 14:00
Chair: Jean-Christophe Hornez, Julio San Roman

14:00 Invited oral

Nanotechnology for Treating Damaged Organs: A Collection of in vivo Studies

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Nanobiotechnology is a growing area of research primarily due to the potentially numerous applications of new synthetic nanomaterials in engineering/science. Although various definitions have been given to the word "nanomaterials" by many different experts, the commonly accepted one refers nanomaterials as those materials which possess grains, particles, fibers, or other constituent components that have one dimension specifically less than 100 nm. For catalytic applications, it has been speculated that nanophase compared to conventional grain size materials increase the adsorption of species due to greater numbers of atoms at the surface, a higher surface area, increased grain boundaries at the surface, and less acidic OH- groups (due to a much larger proportion of edge sites to cause delocalization of electrons). For tissue engineering applications, the same novel properties of nanophase materials are being used to treat organ failure. Specifically, this talk will emphasize the numeorus advances both in vitro and in vivo that have been made towards increasing tissue regeneration using nanophase ceramics, metals, polymers, and composites of the above. Specifically, the talk will concentrate on studies which have focussed on the use of nanophase materials in orthopedic, dental, cartilage, bladder, vascular, and the central and peripheral nervous systems. In doing so, this talk will emphasize the promise that nanophase materials have for healing numerous damaged organs.

14:30 Oral

RGD peptide grafting onto micro-patterned PET

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PET has been most widely used as biomedical implant because of its desirable properties, such as strength and modulus. Nevertheless, the surface should be improved in order to enhance its biointegration.Some studies have shown that spatial distribution and density of bioactive molecules mainly govern success of biomimetic materials modifications. The aim of this study is to determinate impact of peptide RGD spatial repartition onto cell attachment.
RGD Peptides grafting: The four subsequent steps are: Hydrolyse with NaOH + water/acetonitrile; Oxidation with $KMnO_4$ in H_2SO_4; NHS and EDC grafting; RGDC grafting.
Micro-patterns creation: Ablation was performed by laser onto the graft surface. Moreover, chemical patterns were formed using conventional photolithographic techniques.
XPS results shown that modified surface exhibits the expected ratios. Grafting ^{3}H-Lysine on PET allow to quantify the density of grafted amino-acid onto the surface and to confirm the stability of the bond after intensive washing and sonicating. Moreover, this tool makes it possible to visualise micro-patterns. Micro-patterns created by laser ablation can also be visualised by optical profilometer.

14:50 Oral

Tribo-corrosion behavior of DLC coated 316L stainless steel under simulated body fluid condition

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Tribo-corrosion is defined as the degradation of materials that results from synergy of mechanical and electrochemical processes. A new apparatus was designed and built in order to study tribo-corrosion behavior of materials. This technique use an electrochemical set-up of the three-electrode type for controlling the potential of the surface of a conducting material subjected to rubbing in a tribometer. In this way it is possible to carry out friction and wear tests in electrolytic solution under well-defined electrochemical conditions determined by the applied electrode potential. In this paper, description of this apparatus was presented and the effect of PE-CVD DLC coatings on corrosion and tribo-corrosion behavior of 316L stainless steel under simulated body fluid condition was investigated. Potentiodynamic polarization tests have shown that DLC coated surfaces present higher corrosion potential (E_{corr}) and breakdown potential (E_b) than bare surfaces. The charge transfer resistance (R_{ct}) measured using electrochemical impedance spectroscopy (EIS) was also higher for the DLC coated surfaces. Porosity of DLC coated surfaces were shown to be critical for corrosive wear resistance.

Coffee break
Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony
(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster PJ-1

Apatite-forming ability of alumina and zirconia ceramics in a supersaturated Ca/P solution

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Alumina and zirconia ceramics are bioinert and therefore do not bond to living bone. It has been shown, however, that by a suitable chemical treatment the hydroxy groups can be formed on the surfaces, which are capable of inducing apatite nucleation in a simulated body fluid (SBF). The aim of our study was to verify the apatite-forming ability of zirconia and alumina ceramics in a supersaturated Ca/P solution, which was recently suggested as an effective and fast biomimetic method. Polished alumina and zirconia substrates were immersed in a supersaturated Ca/P solution for various periods of time at pH 7.2 and 36.5 °C. For comparison, a bio-glass was used as a reference material. After drying the specimens were verified by SEM/EDS, XRD, TEM and AFM analysis. The first isolated clusters were formed on the substrate surfaces after only 2 hours, which also confirms the effectiveness of the Ca/P solution in biomimetic apatite-forming. According to SEM/EDS analysis these clusters contain calcium and phosphorus. All substrates immersed in Ca/P solution for 27 hours showed uniform nanocrystalline layers. The XRD patterns of scratched coating layers indicated that the hydroxyapatite was formed, which was later confirmed by TEM analysis. Attempts were made to quantify the thickness of these layers as a function of time for various substrates, but no conclusive results could be obtained so far. The above results indicate the apatite forming ability of alumina and zirconia ceramics in supersaturated Ca/P solution.

17:20 Poster PJ-2

Carbon magnetic encapsulated nanoparticles for biomedical applications: thermal stability studies

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Carbon encapsulated magnetic nanoparticles can find many prospective biomedical applications, e.g., in drug and gene delivery systems, disease detection, cancer therapy, rapid toxic cleaning, biochemical sensing, and magnetic resonance imaging. Each of these applications hinges on the relationship between magnetic fields and biological systems. Herein we present the results on the thermal stability of carbon encapsulated Fe-Nd-B nanoparticles. The products were synthesized by using Radio Frequency thermal plasma. Thermal stability was investigated by thermogravimetry and differential thermal analyses. Carbon nanostructures were thermally stable up to 600-700 K.

17:20 Poster PJ-3

Sensoric effect of Langmuir - Blodgett films based on fluorene derivatives - TCNQ CT - complex

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The construction of conducting material with increasing complexicity exhibiting a precise architecture, size, shape and functionality is a challenging domain of rapidly growing interest. It founds its incentives in the increasing needs for molecular and sensoric devices which accompany the developmnet of nanotechnologies[1].

A major support for the advancement in the design of functional molecular objects is our improving ability to control their macromolecular structure in great detail often by using proper deposition methods (Langmuir - Blodgett). In the last two decades there has been an increasing interest in electroactive molecules arranged in thin films, due to their capability for the development of electronic and sensoric devices[2].

Continuing our considerable interest in precursors of conducting and sensing materials chemistry[3] stimulated us to present the results of research into the properties of LB films containing TCNQ. TCNQ as well as its derivatives focus on extesively use as electron acceptor in a large number of organic conducting system. The present work pointed on results of measurement of the effect of toxic gases on CT complex LB film gas sensor. The monitored changes of the surface dark conductivity of sensor made of fluorene derivatives with TCNQ due to adsorption of toxic gases.

1. Schappacher M., Deffieux A., *Macromolecules*, **2005**, *38*, 7209-7213

2. Hirano Ch., Imae T., Fujima S., Yanagimoto Y., Takaguchi Y., *Langmuir*, **2005**, *21*, 272 279

3. Cabaj J., Sołoducho J., Nowakowska A., Chyla A., *Electroanalysis Journal*, **2006**, *18 (8)*, 801-806

17:20 Poster PJ-4

Quantification of the longitudinal force necessary to detach osteoblastic cells from a glass substrate using ultrasounds.

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Understanding of the phenomena of cell adhesion and in particular,

understanding of cell adhesion on biomaterials is of crucial importance for the development of new biomaterials with excellent biocompatibility. One of the physical quantitative indexes to evaluate the quality of cell-material adhesion is its strength. Determining the strength of adhesive bonds requires applying external forces to the cells. Thus, a few methods have been developed to evaluate the strength of cell-material adhesion (micropipette, microplates, microcantilever…). These methods apply shear forces on adherent cells. The aim of our work is to propose the development of a new ultrasonic characterization method of cellular adhesion on substrates. With our method, longitudinal acoustic waves are applied on cell culture to impose a longitudinal strain on cells. Only the cells subjected to a sufficient level of strain will be detached from the substrate. The idea is to bind cell detachment rate to the longitudinal strain threshold supported by cells. From this result, we can deduce the critical force just sufficient to detach the cell. The technique is investigated for the 200 kHz ultrasound frequency. Tests were carried out with the MC3T3-E1 osteoblastic cell line on a glass substrate with different functionalizations. Our results to date provide the value of the necessary force to detach with reproducibility osteoblastic cells from glass. Other materials like dense hydroxyapatite substrates are under investigation.

17:20 Poster PJ-5

Nanocrystalline Hydroxyapatite Coating of Nanostructured Ti an Al Alloy

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Ti and Al alloys are extensively employed thanks to their excellent biocompatibility, corrosion resistance, mechanical properties and workability. Better performance and workability can be achieved through new compositions and/or modifications of the available Ti alloys. The deposition of nanocrystalline hydroxyapatite by a method previously described (Bigi et. Al 2005) onto nanostructured Ti13Nb13Zr substrates and standard Ti6Al4V substrates promote cells proliferation and adhesion. In order to investigate the potential application of these alloy substrates in tissue engineering , this study was conducted with the aim of using this materials for in vitro proliferation of human bone-marrow-derived mesenchymal progenitors (bmMSC). Coated and uncoated substrates were submitted to structural and morphological characterization. In order to determine the proliferation rate of cells cultured on biomaterials, the MTT test was performed. These in vitro studies showed that MSC proliferation progressively increases from 7 to 28 days of cultures showing lower MTT values of Al alloys vs Zr one. Molecular biology analyses for the principal bone markers, osteocalcin, osteopontin, collagen type I, confirm the correct commitment of the bmMSC in presence of coating of nanocrystalline hydroxyapatite. In particolar Zr alloys with coating of nanocrystalline hydroxyapatite show higher expressions. Morphological analyses performed by means of SEM analyses, showed that on the nanostructured surface of the alloys cells attached and spead well.This ultrastructural analysis supported evidence that MSC grown onto Zr alloy with coating of nanocrystalline hydroxyapatite show a phenotype osteocyte like.

17:20 Poster PJ-6

A simplified preparation technique of silicon-substituted calcium phosphates

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Silicon-contained calcium phosphates catch the attention of researchers and industrial manufacturers of bone grafts. Precipitation is the standard preparation technique that occurs by mixing of calcium- and phosphate-containing solutions at pH ~ 10 in presence of a silica, followed by aging, filtration, washing off, drying and sintering at 900 - 1200 °C. Here we present a simplified preparation technique of silicon-contained calcium phosphates.

Chemically pure $Ca(NO_3)_2 \cdot 4H_2O$, $NH_4H_2PO_4$, ammonium hydroxide (25% aqueous solution), and a very fine dry powder of silica-gel were used as initial chemicals. The chemicals were mixed and the mixture was placed into a alumina crucible. A small amount of ammonium hydroxide was added into the crucible. The crucible was placed into a furnace and the suspension was sintered at 1000 °C for 4 hours (ramp rate 3°C/min), followed by cooling down to ambient temperature. The resulting powder was analyzed by SEM, XRD, IR-spectroscopy and by chemical analysis

The experimental results revealed that depending on the initially selected Ca/P and Si/P molar ratios, the final compounds might have either the structure of a silicon-substituted hydroxyapatite or a silicon-stabilized mixture of α- and β-tricalcium phosphates. Therefore, silicon-contained calcium phosphates might be easily prepared by a single stage which makes unnecessary the stages of aging, filtration, washing and drying. Presumably, the biological behavior of the simplify-prepared silicon-containing calcium phosphates should not be very different from those prepared using the routine way. We believe that this simplified preparation technique will reduce the production costs and make artificial bone grafts more affordable to people.

17:20 Poster PJ-7

Artificial Extra-Cellular Matrices Based on Fibrils of Different Collagen Types Containing Immobilised Glycosaminoglycans (GAGs) for Titanium Implants

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Introduction. Collagen has been used as a coating for titanium implants and as a bone tissue engineering scaffold material. The goal

of this work was the characterisation of fibrils of collagen types I, II and III containing the following glycosaminoglycans (GAGs): chondroitin sulphates A, B and C (CSA,CSB,CSC), hyaluron and low and high molecular weight (MW) heparin and their influence in the form of coatings on human osteoblast (HO) behaviour. In addition, the effect of BMP-4 on HO behaviour was investigated. Methods. Fibrillogenesis took place at 37ºC in a 30 mM phosphate buffer at pH 7.4. GAG content of fibrils was measured colormetrically. Fibril morphology was studied by atomic force microscopy (AFM). Results and Discussion. Collagen type II fibrils bound more GAGs than fibrils of types I and III with the exception of hyaluron. GAGs were preferentially bound in the order CSC > high MW heparin > CSB > CSA > low MW heparin > hyaluron. Differences might be due to: a) differences in molecular weight; longer glycosaminoglycan chains may form more stable bonds with collagen by "bridging" more binding sites on collagen; b) different GAG charge densities; a higher charge density would lead to increased ionic binding. The amounts of GAG bound in this manner are comparable to those achieved by EDC/NHS crosslinking. HOs cultured on collagen-coated titanium surfaces containing CSA exhibited greater proliferation and collagen synthesis compared to those cultured on uncoated titanium. The addition of BMP-4 appeared to reduce proliferation and collagen synthesis, but seemed to stimulate the expression of osteoprotegerin and osteocalcin. Conclusions. The ability of collagen fibrils to bind GAGs varies according to GAG and collagen type. Coatings consisting of type I fibrils containing CSA and BMP-4 appear to modulate primary human osteoblast behaviour. These results may be of importance when designing collagen-based extra-cellular matrices.

17:20 Poster PJ-8

Antibacterial functionalization of an experimental self-etching primer by inorganic agents: microbiological and biocompatibility evaluations

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Antibacterial activities have previously been demonstrated on oral bacteria with inorganic antibacterial agents (ABAs) after their incorporation into an experimental self-etching primer (ESP) before its curing. The aim of the present study was to assess their biocompatibility and antibacterial activity after curing.

6 ABAs (basic magnesium hypochlorite, ZnOw AT-83, ZnOw AT-88, Longbei antibiotic, Antim-AMS2 and IONPURE-H) were incorporated respectively into ESP for treating specimens. After light-curing, their bactericidal activities on *Streptococcus mutans* and influences to the early bacterial colonization were assessed by direct contact and viable count. Systemic toxicity was evaluated in rats after short-term oral exposure and cytotoxicity was assessed by direct contact *in vitro* tests with NIH fibroblasts. Incorporation of ZnOw AT-83, Longbei antibiotic, Antim-AMS2 or IONPURE-H significantly enhanced the antibacterial effect of ESP after curing, even after 1 month aging. Specimens treated by ESP with ZnOw AT-83, Longbei antibiotic or Antim-AMS2 showed slightly less bacterial adhesion than control. Animal experiments revealed neither toxic manifestations nor significant differences in body weight gain between control animals and exposed groups. Cell vitality or proliferation rates were ranged from 76% to 100% with respect to controls. Basic magnesium hypochlorite, ZnOw AT-83 and ZnOw AT-88 were the less toxic agents. Toxicity only existed in areas beneath the specimens and/or in the direct vicinity of the specimen edge. There was no influence on the cell density over the limit of specimens.

The here tested ABAs can be effectively incorporated in ESP to provide antibacterial activity against *S. mutans*. ZnOw AT-83 was the most promising one.

17:20 Poster PJ-9

RGD nanodomains grafting onto titanium surface

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Titanium alloys exhibit excellent biocompatibility and corrosion resistance in the body fluid and possess favourable mechanical properties. In order to control the tissue-implant interface and to favor cell-adhesion onto titanium, our work deals with the grafting of cell-binding peptides containing the Arginine-Glycine-Aspartic acid (RGD) sequence

Whereas the influence of the peptide sequence is well studied, there are very few studies on the role of peptide clustering. Besides, numerous works give very different values of peptide density necessary for focal contacts formation.

In the present study, we focus on the elaboration of patterned biomaterial surfaces with highly functionalized nanodomains. The grafting of an aminosilane linker on a cleaned titanium surface was then undertaken. Each of the free amino moieties was used as an initiator core for either a dendrimer synthesis, an hyperbranched polymer synthesis or the attachment of functionalized nanoparticules, therefore multiplying the number of free groups available for RGD immobilization on the material surface.

The density of grafted molecules can be quantified by XPS (X-Ray Photoelectron Spectroscopy), Scanning electron micrograph, High Resolution β-Imager or by chemical ways.

Eventually, the free functionnal sites will be used to covalently link a peptide sequence mediating cell attachment.

17:20 Poster PJ-10

Corrosion resistance of nanostructured titanium

Halina Garbacz[1], Marcin Pisarek[1,2], Krzysztof J. Kurzydlowski[1]

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Pure titanium is more biocompatible with human fluids and tissue than other materials but the coarse-grained titanium is too weak for implants and prostheses. The strength of pure titanium is less than half the strength of TiAl6V alloy, which is widely used for medical applications. However, the small percentages of vanadium and aluminum atoms contained in the alloy are potentially toxic. Hydrostatic extrusion (HE) allows to obtain nano-grained titanium characterized by excellent mechanical properties. The strength of pure titanium with nanosize grains is even higher than in the case of conventional TiAl6V.

The present work studies corrosion resistance of pure Ti-Grade 2 after hydrostatic extrusion. The extrusion of titanium results in the grain size below 90 nm with high-angle grain boundaries. The main purpose of the investigation is to study the effect of nanostructure on oxide layer and corrosion resistance of titanium. A detailed knowledge of chemical composition of oxide layer on a nanometer scale may help to understand corrosion resistant of Ti. Surface analytical technique like AES combined with Ar+ ion sputtering allow to investigate the interface/interphases region and examine its chemical properties. The chemistry of the oxide film formed on the metallic Ti was analysed by Auger Electron Spectroscopy (AES).

17:20 Poster PJ-11

Simulation of microcantilevers sensor arrays for DNA detection

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Microcantilever based biosensors were born in 1941 and they are an adaptation of the atomic force microscope (AFM) in which preferential adsorption of target molecules to receptors coating one side of the cantilever leads to a measurable bending response. Microcantilevers, with force constants in the range of 0.008 N/m to 0.5 N/m have been shown to be highly sensitive transducers for a variety of environmental factors including chemical binding, temperature and humidity.Due to their sensitivity and small size, microcantilevers have proved to be useful in biosensing and many other applications. In this paper, I tried to optimize microcantilever design, in order to have good sensing and high performance.

17:20 Poster PJ-12

Synthesis of hydroxyapatite films

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The paper presents the first investigation results for the hydroxyapatite films received by hf magnetron sputtering.The thin films were investigated by the TEM and HEED methods, and the thick films by the methods of SEM, X-ray phase analysis and IR-spectroscopy. It has been stated that the deposited films is a composite, namely, in the amorphous matrix the crystallites are distributed. After the prolonged (10-15 min) beam treatment the formation of the block crystal structure with the block size as large as 5 mkm from the HA subgrains of the same orientation (a=0,942 nm, c=0,687nm) were observed. The small amount of the nanocrystalline phase is CaO. The structure of the HA film annealed in air on an object grid at about 1170 K for 15 min is formed by the blocks of HA nanocrystals. The crystal phase CaO was not revealed. The electron diffraction pattern of the HA film annealed in air on the object grid at 1170 K during one hour shows the whole set of diffraction maxima, corresponding to the HA lattice, which suggests the synthesis completion. According to the data of the x-ray phase analysis, the energy dispersion analysis (with Ca:P=1.5) and the IR-spectroscopy, the thick films composition corresponds to that of the HA.

17:20 Poster PJ-13

Mathematical correlation between biomaterial and cellular parameters - critical reflection of statistics

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For mathematical modelling of the biomaterial-cell contact it is necessary to find both parameters characterizing physical and chemical properties of the material surface and also such describing the reaction of the adhering cells. Only those material and cell parameters that correlate with each other are applicable to model this contact mathematically. Only few papers are dealing with this special problem. The aim of this paper is to present results of physical/chemical and biological investigations made on differently modified rough titanium implant surfaces in order to find out only the correlating parameters. Furthermore we discuss several ways to apply statistical methods to the correlation problem.

Only few ones of all investigated parameters both on material and on cellular side were applicable for correlation. For example we found in our studies that fractal structure parameter topothesy has influence on the spreading behaviour of the osteoblastic cells. However the correlation coefficient and its statistical significance heavily depend on the method of averaging the available data. Especially the biological data (spreading area) were afflicted with relatively high error up to 30%. Averaging of this data masks the true facts. That's why the correlation coefficient considerably decreases if the biological parameters are not averaged. On the other hand the statistical reliability increases due to the higher number of investigated cases.

Critical error discussion is necessary in statistical correlation between material and biological parameters. Often the results are heavily influenced by the statistical handling of data, especially if only few data are available. May be that new unconventional methods like bootstrap method can show a way out of this problem.

17:20 Poster PJ-14

Surface atomic structure of copper-alanine complex in solution

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In this contribution, we report about our study concerning the physical chemical surface properties of copper-amino acid complex crystal. Firstly, we will show the study of the copper-alanine growth kinetics in solution by means of X-ray surface diffraction. Finally, the atomic surface structure of dry and wet copper complex will discussed in correlation the their bulk structure. Copper is an essential metalloelement, needed for metabolic processes in cells. Many enzymes require copper in the active site to be biochemically active. A crucial question in many biological processes is concerning with correlation between atomic structure and the functionality. Most of the atomic structures known until today have been obtained from bulk crystals, where the packing produces a strong intermolecular interaction that is far from the real biological active conditions. Copper(II) complexes with amino acids and amino acid derivative ligands are good model compounds for the metal-ligand sites on proteins.

We have studied, by means of surface X-ray diffraction (SXRD), the surface atomic structure of copper complexes with Alanine ($Cu\text{-}Ala_2$). Although, the bulk structure is well known since many years, in the bulk, the intermolecular interactions inside the crystal are stronger that in possible biological conditions. However, at the crystal surface, molecules are less constrained. Closer biological conditions can be achieved if the atomic surface structure is determined in aqueous solution.Crystals were studied in a cell filled with saturated solution, controlling the temperature with a thermostatic bath. The growth of the crystal was followed in real time by measuring a rocking scan of the reflection (1 1 0.5), which gives more sensitivity to the surface modifications. The experiments were carried out on the SpLine beamline (BM25) at the ESRF in Grenoble (France) using a six-circle diffractometer in vertical geometry.

17:20 Poster PJ-15

Influence of manganese ions on the cellular behavior of human osteoblasts in vitro

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Divalent cations like Mn^{2+} are known to strongly influence the integrin affinity to ligands and - in consequence - cell adhesion to extracellular matrix proteins. Therefore, divalent cation supplementation of biomaterials could be a promising approach to improve the ingrowth and the integration of implants. We were interested, whether manganese ions affect cellular functions like spreading, proliferation as well as gene expression in human osteoblasts. MG-63 osteoblastic cells (osteosarcoma cell line, ATCC) were cultured in DMEM with 10% FCS. $MnCl_2$ was added at a concentration range of 0,01 - 0,5mM for 24 and 48 h. Spreading (cell area in μm^2) of PKH26-stained cells (cell membrane dye, Sigma) was analyzed using confocal microscopy (Carl Zeiss). Cell proliferation was measured by flow cytometry (BD Biosciences). Quantification of the phosphorylation status of signaling proteins was estimated using the Bio-Plex 200 system (Bio-Rad Laboratories GmbH). Gene expression of osteogenic markers at the mRNA and protein level was analyzed by quantitative real time RT-PCR and Western Blot, respectively.

The results demonstrated that at higher concentrations of Mn^{2+} cells revealed a spindle shaped morphology. Further analyses indicated a reduced spreading, proliferation as well as phosphorylation of signaling proteins due to the influence of Mn^{2+} in a concentration dependent manner. Although expression of Bone Sialoprotein (BSP) a the mRNA level increased both after 24 h and 48 h in the presence of manganese, no increased expression of BSP was detected at the protein level. The expression of ALP and collagen I mRNA decreased at > 0,1mM $MnCl_2$. We speculate that the effect of manganese cations on cell functions is strongly concentration dependent and the release of manganese when incorporated in a biomaterial surface has to be thoroughly adjusted.

17:20 Poster PJ-16

Spark plasma sintering synthesis of porous nanocrystalline titanium alloys for biomedical applications

Radu Nicula[1], Frank Lüthen[2], Manuela Stir[1], Barbara J. Nebe[2], Eberhard Burkel[1]

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The reason for the extended use of titanium and its alloys as implant biomaterials stems from their lower elastic modulus, their superior biocompatibility and improved corrosion resistance compared to the more conventional stainless steel and cobalt-based alloys. Nano-

structured titanium-based biomaterials with tailored porosity are important for cell-adhesion, viability, differentiation and growth. Powder metallurgy technologies like foaming or low-density core processing were recently used for the surface modification of titanium alloy implant bodies to stimulate bone in-growth and improve osseointegration and cell-adhesion, which in turn play a key role in the acceptance of the implants. We here report on preliminary results concerning the synthesis of mesoporous titanium alloy bodies by spark plasma sintering. Nanocrystalline cpTi, Ti-6Al-4V, Ti-Al-V-Cr and Ti-Mn-V-Cr-Al alloy powders were prepared by high-energy wet-milling and sintered to either non-porous (cp Ti, Ti-Al-V) or uniform porous (Ti-Al-V-Cr and Ti-Mn-V-Cr-Al) bulk specimens by spark plasma sintering in vacuum. Cellular interactions with the porous titanium alloy surfaces were tested with the osteoblast-like osteosarcoma cell line MG-63. Cell morphology was investigated using scanning electron microscopy (SEM). The results of the SEM analysis were further correlated with the alloy chemistry and the topographic features of the surface, namely porosity and roughness.

17:20 Poster PJ-17

The dynamics of cholesterol molecules near the surface of transmembrane protein - MD simulation.

Przemysław Raczyński, Aleksander Dawid, Zygmunt Gburski

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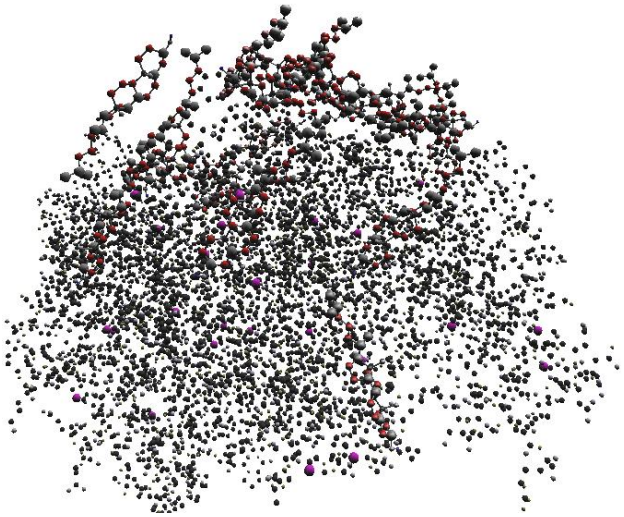
We have made the molecular dynamics (MD) simulations for the cluster of cholesterols localized near the transmembrane protein at the physiological temperature T = 309.75 K. We have observed that the cholesterol molecules form a lodgment on the surface of protein. Several physical charactertistics of the deposited cholesterol cluster have been calculated among those: the mean square displacement, diffusion coefficient, linear and angular velocity autocorrelation function and their Fourier transforms.



17:20 Poster PJ-18

The influence of the carbon nanotube on the structural and dynamical properies of cholesterol cluster.

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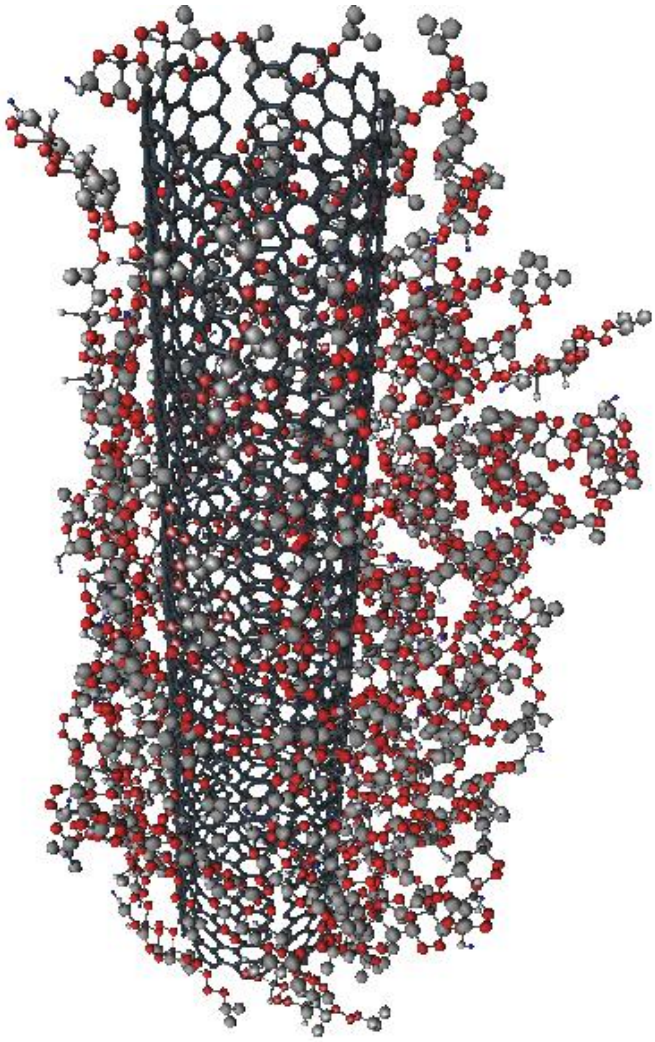
We have performed the molecular dynamics simulations for the free cholesterol cluster and the same cluster located near the carbon nanotube. We have found that the cholesterol molecules quite evenly cover the surface of single walled armchair (10, 10) carbon nanotube, forming the molecular layer. Moreover, the characteristic aligment of cholesterol molecules within the layer (along the nanotube) is observed. The comparision of the structural and dynamical observables characterizing cholesterol molecules are presented and discussed, both for the cluster with and without the presence of the nanotube.



17:20 Poster PJ-19

Molecular dynamics simulation of the properties of the homocysteine nanosystem.

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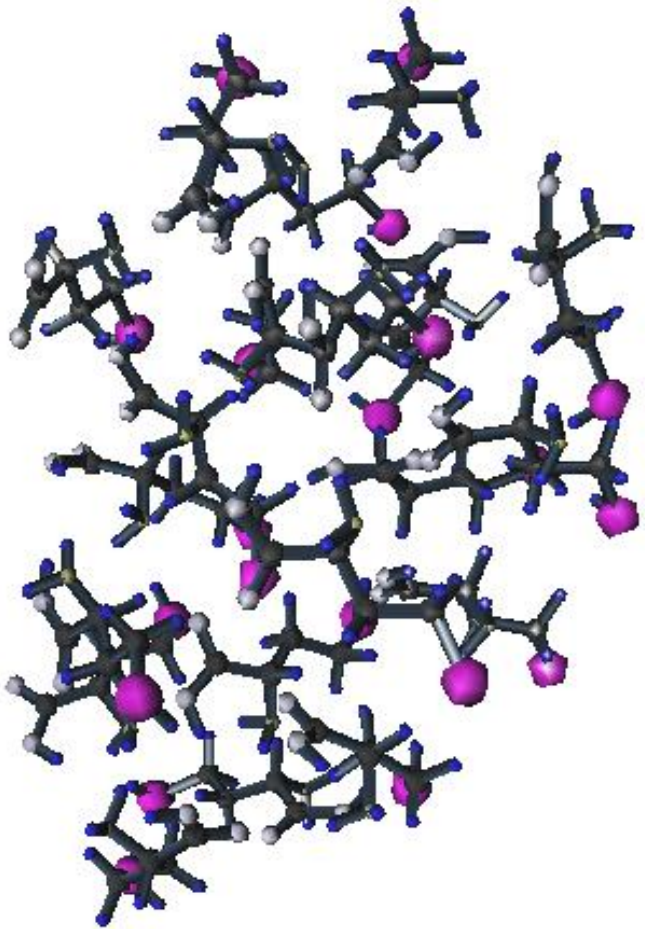
Excessive of homocysteine in the human body is recently considered as a factor increasing the risk of the cardiovascular system diseases. The nanosystem composed of finite number of homocysteine molecules (n = 20, 50, 80) have been studied by MD technique. We have also performed MD simulations with boundary conditions for macroscopic (bulk) sample of homocysteine molecules, as a reference. The specific physical properties of homocysteine nanosystem have been calculated and compared with their counterparts of bulk sample.



17:20 Poster PJ-20

Morphology and chemical characterization of Ti surfaces modified for biomedical applications

Malgorzata Lewandowska[1], Agata Roguska[1], Marcin Pisarek[1,2], Maria Janik-Czachor[2], Krzysztof J. Kurzydlowski[1]

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Titanium is known as a good biomaterial for various medical applications. In the form of porous structures, it is used as a support for living cells. In order to assure cell adhesion and growth, various modifications have been applied to such titanium structures. It has been discovered that the contact of cells with materials of different morphology and chemistry results in a modification of a shape and bioactivity of the cells. However, the morphological and chemical factors responsible for a stable growth of cells are still not well known. The aim of the present work is to characterize in detail the chemical composition and morphology of titanium surfaces subjected to various environments. The modifications consisted in exposure of Ti to acidic or alkaline solutions. These modifications result in chemical and morphological changes of the Ti surface. The special attention has been paid to identify the factors influencing cell adhesion and growth.

Surface analytical techniques such as AES or XPS combined with Ar+ ion sputtering allowed to investigate the chemical composition of Ti oxide layer and examine the chemical properties of Ti surface after the chemical pre-treatments. SEM examinations provided morphological characterization of the Ti samples. The results show large differences in morphology of Ti pretreated with different procedures whereas only minor differences in the chemistry of the surfaces. The possible influence of various surface features on surface biocompatibility is discussed.

17:20 Poster PJ-21

Polysaccharide microarrays for polysaccharide/growth factor interaction studies

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Sulfated polysaccharides are known to potentiate the in-vitro activity of certain proteins, such as growth factors. We have in our hands a library of 115 dextrans functionalised with various amounts of carboxymethyl, benzylamide and sulfate moieties (DMCBSu, Dextran methylcarboxylate benzylamide sulfate)[1] that mimic the properties of heparin. In order to identify rapidly sets of compounds that bind growth factors, we developed microarrays on which these large

polymers (above 40 kDa) are printed and immobilised by physisorption.

The library of DMCBSu is designed in such a way that it gives us an insight into which substitution patterns are most favorable for the interactions with given proteins. This identification method has already been validated for interactions with platelet derived growth factor-BB[2]. In order to evaluate the affinity of each dextrans with Bone morphogenetic protein-2[3], a growth factor involved in osteoinduction and bone regeneration, microarrays were incubated with a solution of the cytokine. Binding intensities were determined with a fluorescence-based detection technique, through consecutive incubations with biotinylated anti-BMP2, and Cy3-labelled streptavidin. After semi-automated data analysis, this strategy enabled us to isolate groups of compounds with different affinities for BMP2, which could be employed in various biomedical applications. Compounds isolated in such a way will soon be tested for their ability to potentiate the action of BMP-2 *in-vitro*.

1 Huynh et al, *Carbohydr Res*. **2001**, *332*, 75-83.

2 Carion et al, *Chembiochem*. **2006**, *7*, 817-826.

3 Chen et al, *Growth Factors*. **2004**, *4*, 233-41.

17:20 Poster PJ-22

Preparation of Photoelectric Dye-Immobilized Polyethylene Film for Artificial Retina

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The retina has photoreceptor cells which absorb light and convert photon energy to electric potentials on the cell membrane. In this study, in order to develop a prototype of retinal prostheses, a photoelectric dye was immobilized on a polyethylene film surface.

Carboxyl moieties were introduced to polyethylene film surface by reaction with 97% fuming nitric acid at 80C for 20 min in a flask. The fi1m was then washed with water until pH-neutral and dried in air. A photoelectric dye 2-{2-[4-(dibutylamino)phenyl] ethenyl} -3-carboxy methyl- benzothiazolium bromide was then linked to the film through ethylenediamine forming amides in the catalytic presence of dicyclohexylcarbodiimide in chlorobenzene at 35C for 24h.

Intracellular calcium elevation was used as a sign of cell activity response to the dye-immobilized films. Pieces of immature retinal tissues of 12-day-old chick embryos (loaded with Fluo-4 acetoxymethyl ester; a molecular probe of fluorescence emission) were observed with a fluorescent dissecting microscope in order to monitor intracellular calcium levels in the retinal tissues. Significant elevation of intracellular calcium was observed in the retinal tissues when they were placed on the dye-immobilized films, but without the film, it returned to a low level. This fact indicated that extracellular calcium ions flew into the retinal cells through voltage-gated calcium channels which the dye-immobilized film triggered on light irradiation.

17:20 Poster PJ-23

Acid-Base Properties of Hydroxyapatite: Potentiometric Titrations Coupled with Solubility Measurements

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The interactions of biomolecules with the surface of hydroxyapatite (HAP) is of great importance in systems where biological fluids are in contact with hard tissues. The characteristics of the electric double layer (EDL) developed between the surface of HAP and electrolyte solution, play a crucial role in these interactions. In the present study the acid-base properties of HAP in contact with KNO_3 solutions were investigated using potentiometric titrations. The novelty of this work is the analysis for calcium and phosphate of aliquots of samples withdrawn from the system every 0.5 pH unit during the titration procedure. It was thus possible to know precisely the composition of the solution at each point of the titration curve. It was found that, even for rapid titration experiments, a remarkable amount of H^+ ions ($H^+_{dissol.}$) participates to bulk solution equilibria with species coming from the dissolution of HAP. These ions must be taken into account in the determination of the point of zero charge (pzc) and consequently of the surface charge (σ^o). Finally, it was demonstrated that the intersection point of the titration curve of the suspension (HAP/indifferent electrolyte) with the titration curve of the respective blank solution (indifferent electrolyte), which has been modified to include the $H^+_{disol.}$ ions, corresponds to the pzc. Conclusively, a novel and easy methodology for determining the pzc and surface charge of HAP and other sparingly soluble solids is presented.

17:20 Poster PJ-24

Ferrimagnetic DP-Bioglass Particles Modified with Polyethylene Glycol and Folic Acid Used for Drug Delivery Process

Min-Hua Chen[1], Chung-King Hsu[1], Feng-Huei Lin[2], Leszek Stobinski[3], Jerzy Peszke[4]

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DP-bioglass particles are biodegradable material which can be used as bioactive material in soft tissue and bone treatment. It has often been used in orthopedics and plastic surgery. Recently, DP-bioglass was also used as a carrier for drug and gene delivery systems. Additionally, ferrimagnetic DP-bioglass can be a potential candidate for magnetic-induction hyperthermia, by using a magnetic field. The aim of this work is the preparation and characterization of surface-modified DP-bioglass particles. Their surface should be modified by

polyethylene glycol (PEG) and folic acid (FA) to improve its intracellular uptake and ability to target specific cells. The first PEG-FA complex was synthesized using carbodiimide (DCC) to link PEG with FA. Then PEG-FA molecules were immobilized on the surface of DP-bioglass particles, which were initially modified by amino-silane (AEAPS) as a coupling agent. FTIR, TGA and NMR were used to reveal this immobilization process. Biological studies have shown that ferrimagnetic DP-bioglass particles coated with PEG-FA complex significantly enhance their intracellular uptake by targeted cells.

17:20	Poster	PJ-26

Inverse Inkjet Printed Gold Micro Electrodes for the Structured Deposition of Epithelial Cells and Fibrin

Rolf Zehbe[1], Ulrich Gross[2], Helmut Schubert[1]

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The micro structured deposition of vital cells is an important challenge in tissue engineering, biosensor technology, and in all research dealing with cell-cell and cell-substrate contacts. Hence, an inkjet printing technology has been developed to manufacture Au-based micro electrodes by sputter coating inversely printed polyester foils. These electrodes feature minimal structure-sizes of 35 µm and consist of an anode and a cathode part. They were used with fibrinogenic epithelial cell suspensions to deposit human keratinocytes (HaCaT), mouse fibroblasts (L-929) and the protein fibrin by applying DC-voltage. Subsequently cells were electrophoretically attracted to the anode, following exactly its shape, while the insoluble fibrin was simultaneously precipitated due to the electrically mediated polymerization of the soluble fibrinogen molecule. Furthermore, it was demonstrated that this technique is suitable to co-deposit both epithelial cell-types in a layered fashion.

The lower voltage boundary for successful deposition was set at approx. 0.8 V needed for the conversion of fibrinogen into fibrin, while the upper voltage boundary was set at approx. 1.85 V, when commencing electrolysis inhibited deposition of vital cells.

Subsequent to the anodic cell-fibrin deposition, cells were cultivated for up to four days and then characterized by FDA+EB staining, methyl violet staining and SEM. Finally, the fibrinogen-fibrin conversion was studied using HPLC, SDS-gel-chromatography and ATR/FTIR.

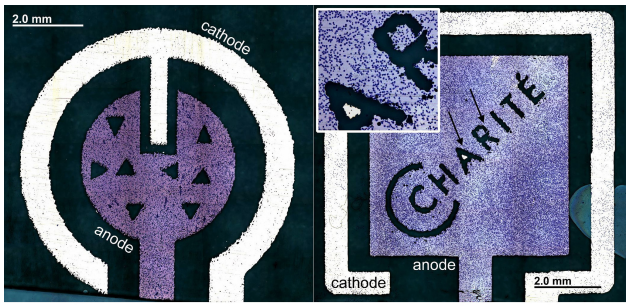

Figure:
Sample ellectrodes made by inverse inkjet printing and Au sputter coating with deposited epithelial cells and fibrin (methyl violett staining)

Reference:
R. Zehbe et al., Micro structured electrodes for the anodic immobilization of cells in a 2D fibrin-matrix, BIOmaterialien 6 (3), 2005, 254

17:20	Poster	PJ-27

Emulsion-based Synthesis of PLGA-Microspheres for the in vitro Expansion of Porcine Chondrocytes

Cornelius Schneider[1], Franziska Gabler[2], Simone Frauenschuh[2], Christoph Brochhausen[3], Peter Götz[2], Helmut Schubert[1], Rolf Zehbe[1]

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The in vitro cell expansion of autologous chondrocytes is of high interest in regenerative medicine since these cells can be used to treat joint cartilage defects. In order to preserve chondrocyte differentiation/ phenotype, while optimizing proliferation and adhesion on microspheres, several processing parameters were varied. In this study three different polylactide-co-glycolides were used with differing lactide-glycolide ratios (85:15 and 50:50) and in case of the 50:50-polymer with differing residual monomer contents (0.83% and 0.9%). An emulsion route was established, where the polymer was dissolved in $CHCl_3$ and then injected in 30g of stirred 0.5% PVA/H_2O solution at different concentrations (0.05, 0.10, 0.15 g/ml) and different stirring velocities (200, 300, 400 rpm) to produce microspheres with varying diameters. The spheres were washed in 2-propanol and were UV irradiated for sterilization purposes. The sphere size distribution and morphology was analyzed using image analyzing software (NI Vision 7.1) on SEM pictures. Linking the size distribution to the cell cultivation experiments, three optimum samples were selected for further investigations. The degradation of the carriers was determined in a long term experiment in culture medium for 4 month by gathering the weight loss every 20 days. Adherent cells were characterized after 0.5h up to 5 days by FDA+EB staining and SEM.

In ongoing investigations we test these microspheres as carriers for prostaglandins, since these molecules have a proliferative effect on chondrocytes, this could open exciting possibilities to enhance the expansion of autologous chondrocytes.

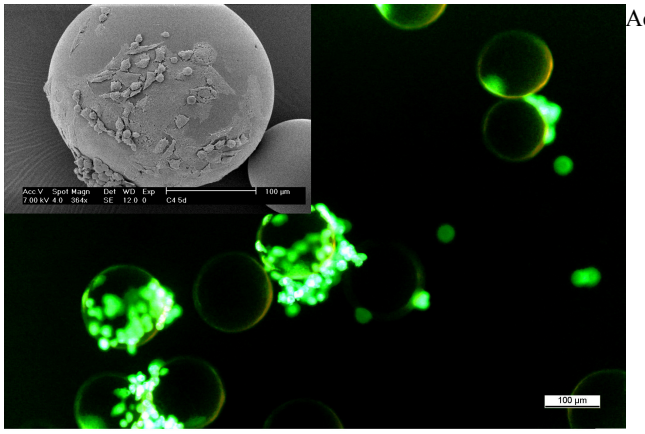

Figure:
Microspheres with adherent Chondrocytes - FDA+EB vital staining and SEM (cut-out)

knowledgments:
The authors would like to thank Dr. H. Liedtke of Boehringer Ingelheim Pharma for the polymer samples.

17:20 Poster PJ-28

Effect of phase transformation on the junction between zirconia/alumina ceramics and commercial coating-glass

Jiuyu Song[1,2], Bin Zhang[1,2], Jihua Chen[1], Jiqiang Gao[3], Guanjun Qiao[3]

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The object of this research was to find the influence of zirconia's phase transformation on the junction between zirconia/alumina ceramics and commercial coating-glass. Effect of phase transformation was observed through coefficient curves of thermal expansion in four kinds of zirconia/alumina composites: pure zirconia, zirconia toughened alumina, alumina toughened zirconia, pure alumina. Energy spectrum analysis, optical and electron scanning microscope were observed on the junction of zirconia/alumina ceramics with coating glass. Furthermore, pre-sintering process was executed to find the heat softening point of the coating-glass. Through experiments, we find phase transformation was only observed in the pure zirconia group. There is no obvious effect of phase transformation in the other three groups. Therefore, we can conclude that adding alumina into pure zirconia may restrict the transformation of zirconia particles and result in the decrease even the disappearance of transformation phenomenon. Therefore, VITA alpha coating glass is a kind of adoptable bio-glass matching with zirconia and zirconia composites. In the mean time, thermal expansion coefficient of two matching materials should also be considered.

Poster Session 2 / Poster Award Ceremony

(continuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Wednesday, 6 September

Symposium K

Welcome

The Symposium is going to address new oxide materials - namely these exhibiting high- T_c superconductivity (HTSC) and colossal magnetoresistance (CMR), as well as ferromagnetic oxide semiconductors and magnetic oxide nanoparticles. The phenomena of HTSC and CMR, especially their microscopic mechanisms, are still hot topics in the solid state physics. In spite of the very intensive studies performed by the best research groups in the field - both phenomena are not fully understood. In materials exhibiting both of these phenomena, the co-existence of various structural, magnetic, charge and orbital states is observed, due to strong electron-electron and electron-lattice couplings. The so called parent compounds for these two groups of materials are antiferromagnetic insulators and compounds exhibiting HTSC or CMR are obtained by proper doping of them. In both groups of materials characteristic phenomena are associated with spatial inhomogeneity related to coexistence of different phases, known as the phase separation. The phase separation manifests itself in formation of phases with different magnetic and conductive properties. By using chemical doping or applying external factors, e.g., light, pressure, and ionizing radiation, various ground states of oxides materials can be realized and various fascinating collective phenomena can be observed. Ferromagnetic oxide semiconductors, magnetic oxide nanoparticles, as well as, materials exhibiting HTSC and CMR can be used in the new technologies for production of sensors of new generation, for information storage and for production of heterostructures needed for spintronics.

The main topics of the Symposium will be:

- fabrication and studies of thin films and heterostructures of HTSC and CMR materials
- nature of superconductivity in strongly correlated systems and search of new superconducting materials
- ferromagnetic oxide semiconductors
- magnetic oxide nanoparticles
- studies of the mixed state in HTSC and in other superconductors relevant to the applications
- magnetic and transport properties of CMR materials
- studies of phase separation in HTSC and CMR materials

Scientific Committee:

- Prof. H. Szymczak (Chairman) - Institute of Physics, PAS, Warsaw, Poland
- Prof. A. F. Andreev - Kapitza Institute of Physical Problems, RAS, Moscow, Russia
- Prof. T. Dietl - Institute of Physics, PAS, Warsaw, Poland
- Prof. H. U. Habermeier - Max Planck Institute, Stuttgart, Germany
- Prof. H. W. Weber - Atomic Institute of Austrian Universities, Vienna, Austria

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- **Prof. Marta Z. Cieplak**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Dr. Ryszard Żuberek**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Proceedings

The Symposium Proceedings will be published in the journal **"Acta Physica Polonica A"**.

Programme

Monday, 4 September

Opening Ceremony
2nd floor, Small Hall (237)
Monday morning, 4 September, 11:00

The Czochralski Award Ceremony
Monday morning, 4 September, 11:30

Lunch break
Monday afternoon, 4 September, 12:30

Parallel Session
Monday afternoon, 4 September, 14:00

14:10 Invited oral

Novel Properties of Atomically Arranged Perovskites

Bogdan Dabrowski

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Owing to unusual chemical versatility and structural intricacy, perovskites can be designed to attain unique electronic, magnetic, ferroelectric, thermoelectric, mixed-conducting, and other properties through selection of the A- and B- site ions, their fractions, ionic sizes and valences, spin states, and orbital orderings, as well as the oxygen content and vacancy ordering. The best-known examples are spectacular high temperature superconducting cuprates, colossal magnetoresistive manganates, ferroelectric titanates, and recently discovered remarkable cobaltites with spin-dependent conductivity and thermoelectric power, and ferrites with dramatic metal-insulator transitions. We will illustrate our systematic exploration of the effects of composition, temperature, pressure, and oxygen content on

thermodynamic stability and physical properties of manganites and cobaltites. The focus of this presentation will be on kinetically stable compounds with decreased structural distortions and atomically ordered layered- and double- perovskites with enhanced useful properties.

Supported by the NSF-DMR-0302617 and by the US Departments of Education and Transportation

14:40 Invited oral

Ferromagnetic oxide heteroestructures for spin filtering

Josep Fontcuberta[1], Ulrike Lüders[1,5], Martin Gajeck[1,2], Agnes Barthélémy[2], Manuel Bibes[3], Karim Bouzehouane[2], Stéphane Fusil[4], Eric Jacquet[2], Jean-Pierre Contour[2], Jean François Bobo[5], Albert Fert[2]

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The need of spin-polarized current sources has fuelled the research on half-metallic ferromagnetic oxides during the last decade. Indeed, development on spintronics mainly relies on the availability of sources of spin-polarized carriers that could be ultimately injected into semiconductors of other functional structures. Unfortunately, so far, the half-metallic character of the celebrated manganites can not be exploited in room-applications as the magnetoresistance of magnetic tunnel junctions or related devices decays rapidly with temperature and becomes unpractical. Therefore, efforts have to be directed to development of alternative spin-polarized sources. In this presentation we shall overview our recent developments in this direction. We will illustrate that heterostructures involving thin ferromagnetic oxides tunnel barriers can be successfully used to spin-filter a paramagnetic charge current. We will discus results, and promises of these devices as candidates as key building blocks of magnetoelectric devices.

15:10 Oral

Suppression of interface induced electronic phase separation in $La_{2/3}Ca_{1/3}MnO_3/SrTiO_3$(110) : ^{55}Mn NMR study

Marek Wojcik[1], Ewa Jedryka[1], Ingrid Canero Infante[2], Florencio Sanchez[2], Vladimir Laukhin[2,3], Josep Fontcuberta[2]

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Optimally doped mixed valence manganites are considered to be a good source of spin polarized electrons in oxide based magnetic tunneling junctions. However, numerous experiments have shown that magnetic and magnetotransport properties of manganite films deviate strongly from those observed in bulk materials. The presence of ferromagnet/insulator interface seems to influence significantly their half metallic properties. Indeed, ^{55}Mn NMR experiments carried out on series of epitaxial $La_{2/3}Ca_{1/3}MnO_3$ (LCMO) films grown on (001) $SrTiO_3$ (STO) substrates revealed a complex electronic phase separation and an insulating nature of the thinner films. Contributions from manganese atoms in ionic state corresponding to the localized spin states (Mn^{4+}) as well as $Mn^{3+/4+}$ mixed valence state have been identified and attributed to the intrinsic properties of interface, but the driving force behind this phenomenon remained unclear. In this work we present a new approach to this problem: phase separation has been studied as a function of the growth direction of LCMO film. A comparative studies of magnetic properties as well as ^{55}Mn NMR study of LCMO films grown on (001) and (110) STO substrates, respectively, have been performed. It has been found that films grown on (110) substrates invariably show improved magnetic properties as compared to their (001) counterparts. In contrast to the results obtained for LCMO(001) films, the ^{55}Mn NMR spectra recorded for LCMO(110) films show only a mixed valence $Mn^{3+/4+}$ resonance line without any contributions from localized (Mn^{4+}) charge states. These findings can be interpreted in the context of possible interface reconstruction driven by elastic and electrostatic energies resulting from the lattice mismatch and polarity discontinuity at the LCMO/STO interface.

Coffee break

Monday afternoon, 4 September, 15:30

Parallel Session

Monday afternoon, 4 September, 15:50

15:50 Invited oral

Magnetism, charge order and ferroelectricity in ternary oxides

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Magnetic materials are well known for applications e.g. in data storage, but ferroelectric materials have important applications as well, such as being used for sensors. For a material combining both magnetism and ferroelectricity a number of additional applications may be envisaged. Unfortunately, "conventional" ferroelectricity and magnetism seem to be diametrically opposed, and in the rare instances when they occur simultaneously the coupling between magnetic and electric order parameters is weak. Recently, ferroelectricity originating from certain spiral magnetic structures has been observed and described. These materials exhibit a large magneto-electric coupling, but due to the key role of the Dzyaloshinskii-Moriya interaction the ferroelectric polarization is small.
An alternative ferroelectric mechanism, allowing in principle for

both large polarization and a large magneto-electric coupling, is a suitable charge-ordering. Such a mechanism has been proposed for $LuFe_2O_4$. The problem with this compound is that it forms with varying oxygen content. As will be shown, both charge order and magnetic properties depend heavily on the oxygen stoichiometry. More generally, it is important to understand charge-ordered materials. A compound for which (as for $LuFe_2O_4$) an electrostatically driven charge-order has been found is Fe_2OBO_3. This material has been shown to exhibit charge-order, but the structure of the charge-ordering is unclear. The synthesis of single crystals of Fe_2OBO_3 allowed us to study this compound in greater detail, and results from, among others, Mossbauer and electron microscopy, will be presented. A number of materials with a magnetic mechanism of ferroelectricity will be discussed as well.

16:20 Invited oral

Enhancing the superparamagnetic blocking temperature of nanoparticles with bi-magnetic core-shell structures

German Salazar-Álvarez[1], Jordi Sort[2,3], Abdusalam Uheida[4], Mamoun Muhammed[4], Santiago Suriñach[3], María Dolores Baró, Josep Nogues[2,3]

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Magnetic nanoparticles are being extensively studied due to their wide range of potential applications, spreading from biomedicine to magnetic recording. While some applications require superparamagnetic particles, others, like recording, need the particles to remain ferromagnetic. Moreover, the use of magnetic nanoparticles for sensing purposes calls for the particles to be magnetically soft without being superparamagnetic. In this context, we have investigated oxide bi-magnetic core-shell nanoparticles with ferrimagnetic cores and antiferromagnetic (or ferrimagnetic) shells. Two examples of core-shell structures will be discussed. These have been fabricated by two different post-synthesis methods using previously prepared ferrimagnetic γ-Fe_2O_3 cores: i) Co(II) ions were adsorbed on the cores to form a shell with a composition near $CoFe_2O_4$ and ii) a MnO layer was formed by using the cores as seeds for the heterogeneous growth. It is shown that both ferrimagnetic and antiferromagnetic shells allow to controllably increase the blocking temperature, T_B, of the system with respect to that of untreated particles. The presence of the shells also brings about a remarkable increase of the coercivity. Moreover, in the case of antiferromagnetic shells a shift of the hysteresis loop along the field axis, i.e., exchange bias, is also observed after field cooling from above the Néel temperature. These results open the door to the possibility to synthesize a single, generic, type of particle, which can later be modified to adapt to the needs of a specific application.

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

Tuesday, 5 September

Parallel Session

Tuesday morning, 5 September, 9:00

9:00 Invited oral

MgB_2 single crystals, influence of magnetic and non-magnetic ions substitution on superconducting properties and structure

Janusz Karpinski[1], N. Zhigadlo[1], K. Rogacki[1,2], Bertram Batlogg[1], Goetz Schuck[1], Roman Puzniak[3], A. Wisniewski[3], R. Gonnelli[4]

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Pure and substituted single crystals of the two-gap MgB_2 superconductor have been grown at a pressure of 30 kbar at temperature 1800-2000°C using the cubic anvil technique. High crystals' quality expresses as low residual resistivity ρ_o (40 K) = 0.5 μΩcm. Magnetic (Mn, Fe) and non-magnetic (Li, Al, C) ions have been substituted to study the substitution effects in a two-band superconductor and it's influence on the impurity scattering in and between σ and π bands. $Mg_{1-x}Al_xB_2$ and $MgB_{2-x}C_x$ crystals were grown for x=0-0.3. Al and C dope MgB_2 with additional electrons, causing similar decrease of T_c. Li dopes MgB_2 with holes without significant decrease of T_c. Magnetic ion Mn^{2+2} for Mg^{2+} leads to very rapid decrease of T_c. This indicates strong pair breaking by magnetic scattering centers. For Mn substituted crystals superconductivity is completely suppressed for x=0.02. Fe at low concentration up to x=0.03 is nonmagnetic impurity in MgB_2 therefore decreases T_c less rapidly than Mn. However, crystals with higher Fe concentration show rapid decrease of T_c, which indicates that Fe can be a magnetic impurity. Carbon substitution increases the upper critical field twice for x=0.05-0.10, while Al, Fe and Mn substitutions decrease this field. The upper critical field anisotropy γ decreases with all substitutions, but temperature dependence of γ is different, which indicates different scattering rates for different substitutions in π and σ bands. For Mn and Al, both energy gaps exist up to the highest substitution rate, while for C substitution, merging of two energy gaps was observed indicating influence of the interband scattering.

9:30 Invited oral

Magnetic interactions in cuprate-manganite heterostructures and superlattices

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Ferromagnetism and superconductivity are long range ordering principles with a mutual exclusion in homogeneous systems. However, if they are spatially separated as in thin film heterostructutres and superlattices both properties are appearing simultaneously and their interaction can be studied. Heterostructures and superlattices consisting of the half-metal ferromagnet $La_{0.67}Ca_{0.33}MnO_3$ and superconducting $YBa_2Cu_3O_7$ layers were fabricated by pulsed laser deposition techniques and their magnetic as well as electronic interaction were studied by a variety of techniques ranging from transport measurements to magnetic, neutron diffraction and XMCD analysis. It turns out that at the interface interaction effects are taking place at two different length scales. One is based on the self-injection of spin-polarized quasiparticles with a length scale of ~10 nm, the other is a short range exchange interaction with a length scale of ~3 nm. Furthermore, it could be shown that an so far unknown coupling of adjacent magnetic layers occurs when the superlattices are cooled through the superconducting transition temperature.

10:00 Oral

Magneto-optical investigation of the electromagnetic interaction in twinned $YBa_2Cu_3O_{7-d}/La_{1-x}Sr_xMnO_3$ bilayers

Roberto Gerbaldo[1], Gianluca Ghigo[1], Laura Gozzelino[1], Francesco Laviano[1], Enrica Mezzetti[1], Bruno Minetti[1], Piotr Przyslupski[2], Andrei Tsarou[2], Andrzej Wisniewski[2]

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An extensive study of the electromagnetic interaction in magnetic/superconducting bilayers is presented. The direct visualization of the magnetic pattern in manganite/cuprate superconductor bilayers is achieved by means of the magneto-optical imaging technique [1]. Quantitative evaluation of the supercurrent distribution in patterned YBCO layers grown on LSMO films with different doping levels is performed, in order to study the local coupling between the magnetic and the superconducting layers [2]. We deal with different phenomena occurring because of both the electronic and the magnetic couplings between the layers. In particular, the electronic coupling is shown to cause the enhancement/depression of the local superfluid density in correspondence with the magnetic structure topology in the manganite film. Possible charge-transfer mechanisms occurring between the manganite and the superconducting layers in dependence on the observed local magnetic order are discussed. Moreover, we present evidence of the spontaneous vortex nucleation and of the vortex guidance phenomenon due to the magnetic interaction exerted on the superconductor by the local manganite domain arrangement [1]. In this respect, we discuss about reliable approaches to engineer the vortex guidance phenomenon in all perovskite heterostructures by means of structural defects and/or by suitable external magnetic field.

[1] F. Laviano et al., Appl. Phys. Lett. 86 (2005) 152501.
[2] L. Gozzelino et al., Supercond. Sci. Technol. 19 (2006) S50.

Coffee break

Tuesday morning, 5 September, 10:30

Parallel Session

Tuesday morning, 5 September, 11:00

11:00 Invited oral

Metastable states in manganites - experimental aspects

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The spin, charge, lattice and orbital degrees of freedom in doped manganites are strongly coupled to each other, leading to rich phase diagram with a variety of competing phases. External stimuli such as electric current/field may affect the topology of coexisting phases, leading to difference between transport properties of various metastable resistive states (MRS) in the same sample. MRS in $La_{1-x}Ca_xMnO_3$ (x=0.18,0.2) crystals can be created by a current treatment. They are characterized by long-term memory surviving even thermal cycling to room temperature. Only heating to T>350 K erases the imprinted state of the system. The nature of phase competition can be understood in terms of glass like transition at low temperatures and to the appearance of Griffith-phase regime at temperatures $T_C<T<T_G$, where T_C and T_G are the Curie and Griffith temperatures, respectively.

In the pristine state polycrystalline $Sm_{0.1}Ca_{0.84}Sr_{0.06}MnO_3$ exhibits phase separation below T_C=105 K comprising ferromagnetic (FM) and antiferromagnetic phases. The metastable states with reduced magnetization or diamagnetic (DIA) response were obtained by successive number of quick cooling of the sample placed in container with kerosene-oil mixture. The observed field and temperature dependences of the magnetization in DIA state are reversed in comparison with ordinary FM ones. Only after some storage time at room temperature, the abnormal magnetic state is erasable. It seems plausible that localized electron orbits with radius of tens nanometers, confined within dislocation network, may give rise to the DIA effects at temperatures 105 K<T<240 K. At T<105 K the magnetic response to external magnetic field is controlled mainly by the coupling of FM clusters with DIA matrix.

11:30 Oral

I-V characteristics of resistive oxides: DC versus pulsed measurements

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Nonlinear conductivity of resistive materials has been a popular topic of investigations over many years. During the past decade great interest was aroused by the dramatic electric-field-induced current switching in some charge-ordered manganites.[1] The thermal theory of non-ohmic conductivity, negative differential resistance and dielectric breakdown goes back over a century.[2] Nevertheless, most recent I-V characteristics were obtained using DC. Joule heating was supposedly ruled out by calculating the expected temperature increment or monitoring it close to the sample surface, while disregarding the inhomogeneous temperature and current distribution.

An old technique for preventing and confirming absence of Joule heating errors consists of applying pulsed currents and following the time dependence of the response on an oscilloscope. Joule heating is negligible, as long as the response remains independent on time. We investigated pulsed and DC I-V characteristics of various resistive oxides: polycrystalline and granular ferromagnetic manganites, double perovskites, charge ordered manganites and related materials (see Reference 3). The DC I-V characteristics were measured up to the negative resistance regime. In most cases the DC characteristics mask a perfectly ohmic or a moderately non-ohmic conductivity obtained by pulsed measurements. *This demonstrates that the widely used DC I-V measurements in the high current regime are usually misleading*. A review of the results obtained for the non-ohmic regime of the systems investigated will be presented.

1. for a brief review see: S. Mercone et al, J. Appl. Phys. **98**, 23911 (2005).

2. H. Froehlich, Rep. Prog. Phys. 6, 411 (1939); D. M. Kroll, Phys. Rev, B **9**, 1669 (1974) and references therein.

3. B. Fisher et al, Appl. Phys. Lett. **88**, 152103 (2006).

11:45 Oral

Structural, 1/f noise and MOKE characterization of vicinal $La_{0.7}Sr_{0.3}MnO_3$ thin films

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This paper presents structural, magnetic and electrical properties of 40 nm thick $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) thin films deposited on vicinal (001) $SrTiO_3$ susbrates. The vicinal angles were 2, 4, 6, 8 and 10° from the [001] direction towards [110]. Structural properties were studied by X-ray diffraction, which indicated that the LSMO films grew with their (001) axis coincident with the (001) axis of the substrate. The surface morphology was carefully studied by Atomic Force Microscopy (AFM) and Scanning Tunnelling Microscopy (STM). The root mean square roughness measured in 2 µm x 2 µm images was in the 0.132 - 0.328 nm range, which is comparable to what is obtained on (100) $SrTiO_3$. STM confirmed the AFM pictures, showing regular steps of width in the 20 - 50 nm range. SQUID magnetometer measurements revealed a Curie temperature of 350 K and Kerr Magneto-Optical microscopy enabled the magnetic domain imaging. As previously reported in [1] for 10°, an uniaxial easy magnetization direction was obtained for angles above 4°, with the easy axis along the steps. In the case of 10° substrates, the magnetization reversal process was studied in both easy and hard directions. It occurs by propagation of magnetic domain walls in the easy axis direction while it shows a coherent reversal process in the hard direction. Finally, resistivity versus temperature characteristics in a magnetic field of 7 T and preliminary 1/f noise measurements will be presented for bias currents along and perpendicular to the step direction. Devices making use of the anisotropy of the magnetization are envisaged for low field magnetometry.

[1] M. Mathews et al., Appl. Phys. Lett. 87 242507 (2005)

Lunch break
Tuesday afternoon, 5 September, 12:30

Wednesday, 6 September

Plenary Session
2nd floor, Small Hall (237)
Wednesday morning, 6 September, 9:30

Coffee break
Wednesday morning, 6 September, 10:30

Plenary Session
2nd floor, Small Hall (237)
Wednesday morning, 6 September, 11:00

Lunch break
Wednesday afternoon, 6 September, 12:30

Parallel Session
Wednesday afternoon, 6 September, 14:00

14:00 Invited oral

Opto-magnetism and coherent control of spin dynamics in magnetic oxides

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The ever-increasing demand for the density and speed of information storage has triggered an intense search for ways to control the magnetization of a medium by means other than magnetic fields. The control of magnetism by light is one of the potential approaches to this problem since a laser pulse is one of the shortest man-made events. It has been shown how femtosecond laser pulses can thermally change the magnetization on a sub-picosecond time scale.

Far more exciting is the possibility of the non-thermal control of spins. This is possible, for example, via the nonlinear inverse Faraday effect, which does not require absorption and is based on a Raman-like coherent optical scattering process. Because the photons conserve their angular moments and loose only very small part of energy in the process, they can in principle participate in many consecutive excitation events.

In this talk I will consider such opto-magnetic effects and demonstrate, how circularly polarized femtosecond laser pulses can non-thermally excite and coherently control the magnetization in antiferromagnetic and ferrimagnetic oxides. The effect of such 100 fs optical pulse on a magnetic system was found to be equivalent to the effect of a magnetic field pulse with an amplitude of up to 1 T. In addition, different non-thermal mechanism was shown to simultaneously modify the magnetic anisotropy.
Coherent control of spin precession can be achieved by using multiple pulses in a rapid succession. Thus the precession can be amplified or stopped by sending a second pump pulse even or odd number of half-periods after the first one. Note that in the case of stopping, the energy of magnetic precession is taken away by the light pulse, which is an equivalent of the magnetic cooling.

14:30 Oral

Light-induced magnetism in polycrystalline and thin films of Cr_2O_3

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We studied on a chromium oxide Cr_2O_3 to investigate near-infrared (NIR) light-induced magnetism function at room temperature. It is well known that the Cr_2O_3 has a corundum crystal structure with antiferromagnetic (AF) transition at $T_N \sim 308$ K. A X-band electron-spin resonance (ESR) spectrometer and a SQUID magnetometer were employed to clarify magnetic behavior in the Cr_2O_3 under the illumination of NIR light ($\lambda = 1064$ nm). Under the light illumination, the light-induced ESR signal appears above 250 K and is remarkably enhanced around room temperature. Contrary, with visible green light ($\lambda = 532$ nm), the ESR profile does not show any change. The light-induced effect is strongly dependent on the irradiation light wavelength. We conclude the light-induced ESR below T_N may come from light-excited carriers associating with a reentrance from antiferromagnetic to paramagnetic spin order. Thin films were successfully prepared by a RF magnetron sputtering and a sol-gel method. The T_N of the thin film was increased from 308 K to about 400 K. We discuss intriguing properties on light-induced effect for both polycrystalline and thin films and state the chromate is a candidature to be applied to NIR detection industrial devices.

Keywords: light-induced magnetism, Perovskite structure, Spin State transition, ESR

14:45 Oral

Magnetic phase transition in pure and cobalt-doped geometrically frustrated $Ni_3V_2O_8$ single crystals

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The recent interest in geometrically frustrated systems is spurred by the new phenomena predicted such as: noncollinear Neel long-range order, "order by disorder", magnetization plateaus and magnetization jumps. Of particular interest is magnetism on the two-dimensional kagome lattice which consists of corner-sharing triangles. Recently, magnetic phase transitions in isostructural kagome-type compounds $Ni_3V_2O_8$ [1] and $Co_3V_2O_8$ [2] have been studied in details. Since, traditionally, it is assumed that both randomness and frustration lead to spin-glass effects we decided to introduce chemical disorder by Co- doping $Ni_3V_2O_8$ single crystals. In this paper we describe the results of magnetic measurements performed on $Ni_3V_2O_8$ and $Ni_{2.91}Co_{0.09}V_2O_8$ single crystals. Although no spin-glass effects have been observed nevertheless a strong effect of 3 at% cobalt doping has been evidenced. In particular, Co-doping leads to new, magnetic field induced, phase transitions for $H \| a$ and $H \| c$. It means that a strong ion-lattice coupling expected for Co^{2+} in octahedral sites is responsible for the change of magnetocrystalline anisotropy in $Ni_3V_2O_8$ but also for the local deformations related to a Jahn-Teller effects, which influences the exchange interactions in the system.

[1] G.Laves et al., Phys. Rev. Lett. 93 (2004) 247201.
[2] R.Szymczak et al., Phys. Rev. B 73 (2006) 094425.

Coffee break

Wednesday afternoon, 6 September, 15:30

Poster Session 2 / Poster Award Ceremony

(contiuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

Thursday, 7 September

Parallel Session
Thursday morning, 7 September, 9:00

9:00 Invited oral

Layered cobaltites: synthesis, oxygen nonstoichiometry, transport and magnetic properties

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Complex cobalt oxide perovskites with general formula $RBaCo_2O_{5+x}$ (R=rare earth) have been attracted considerable interests because of their interesting properties: magnetic and metal-insulator transitions, giant magnetoresistance, ionic conductivity and a structural similarity to high temperature superconductors. All the compounds are oxygen non-stoichiometric ($0 < x < 1$) and the cobalt cations can adopt different oxidation and spin states. Compounds with $x \approx 0.5$ display a metal-insulator transition. We have found that this transition is affected by an oxygen isotope substitution [1] and is accompanied by structural changes and melting of the orbital-ordering [2]. Measurements performed on series of polycrystalline samples with different oxygen contents showed that magnetic properties depend not only on oxygen stoichiometry but also on synthesis history. Some preliminary measurements made on $TbBaCo_2O_{5.5}$ crystals grown by Traveling Solvent Floating Zone method will be also presented.

[1] K. Conder, E. Pomjakushina, V. Pomjakushin, M. Stingaciu, S. Streule, A. Podlesnyak, J Phys.: Condens. Matter **17** (2005) 5813

[2] E. Pomjakushina, K. Conder, V. Pomjakushin, Phys. Rev. B **73** (2006) 094203

9:30 Oral

Structure and magnetic properties of $(Gd, Y)_{0.33}Sr_{0.67}CoO_{3-\delta}$ compounds

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We have studied the structure and magnetic properties of the $A_{0.33}Sr_{0.67}CoO_{3-\delta}$ (A=Y, Gd) samples. X-ray diffraction and Rietveld refinement show that the structure is the perovskite-type structure with layered alternate ion of CoO_6 octahedron and CoO_4 tetrahedral polyhedron. In the Y-doped sample, a magnetization jump was observed around 180 K below the Curie temperature T_C = 304 K during the magnetic field cooling. The magnetization jump may be associated with the onset of antiferromagnetism appeared as the Co^{3+} ion orbital ordering state with the intermediate spin state. The Gd-doped sample exhibits higher magnetization and lower Curie temperature than Y-doped sample. The field magnetization vs temperature curve reveals a reentrant paramagnetic behavior below 100 K, which comes from the spin state transition of Co^{3+} ion from intermediate to low spin configuration.

Keywords: Strontium doped cobaltite, Perovskite structure, Spin State transition, Thermal hysteresis

9:45 Oral

$Co_{3-x}Mg_xV_2O_8$ crystals grown by the optical floating zone method

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Kagome staircase lattice of Co^{2+} ions in $Co_3V_2O_8$ is attractive due to complex magnetic structure involved by geometric frustration. $Mg_3V_2O_8$ is nonmagnetic compound with the same crystal structure and with the lattice parameters very close to that of $Co_3V_2O_8$. Due to incongruent melting of these compounds one of the useful method of crystallization is a growth by the floating zone method in the traveling solvent zone configuration.

In this work we discuss process of growing of the mixed crystals $Co_{3-x}Mg_xV_2O_8$. In the Co-reach region it is interesting to investigate the influence of perturbation of the Co system (by replacing part of Co by Mg ions) on magnetic properties. On the other hand, the magnesium reach region is interesting for EPR and optical measurements for determination of crystal field parameters of Co ions. Solid state synthesis has been performed by sintering the pressed mixture of stoichiometric amounts of CoO, MgO and V_2O_5. Single crystals have been grown from polycrystalline feed rods by floating zone technique carried out in the optical image furnace under O_2 atmosphere.

Crystal structure of these materials is based on slightly deformed cubic close packing of oxygen ions. 3/8 of existing octahedral positions are occupied by Co^{2+} or Mg^{2+} ions and 1/8 of tetrahedral positions by V^{5+} ions. Possible migration of cations may involve the perturbation of crystal symmetry resulting the twinning effect sometimes observed in grown crystals.

The work was supported by the Polish State Committee for Scientific Research (KBN) under the project 1 P03B 038 27.

10:00 Oral

Electrical and magnetic properties of p-n diode structures based on lanthanum manganites and Nb-doped $SrTiO_3$

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There is increasing interest in p-n junctions formed between hole-doped manganite films and conducting n-type $SrTiO_3$<Nb> (STON). In this work, high quality $La_{2/3}Ba_{1/3}MnO_3$ (LBMO), $La_{2/3}Ca_{1/3}MnO_3$ (LCaMO) and $La_{2/3}Ce_{1/3}MnO_3$ (LCeMO) thin films were grown *in-situ* at 750°C on Nb 0.1 wt % doped conducting STON substrates by pulsed laser deposition and dc magnetron sputtering. Our attempts were undertaken to elucidate a role of dopant element (Ba, Ca and Ce), deposition conditions and possible lattice mismatch at the interface on electrical and magnetic properties of the heterostructures. Current perpendicular to plane geometry was used to investigate the junction resistance, magnetoresistance, the current-voltage (*I-U*) and capacitance-voltage (*C-U*) characteristics of the prepared LBMO/STON, LCaMO/STON and LCeMO/STON heterostructures. Asymmetric *I-U* relations measured at T=78÷300 K for all the heterostructures including LCeMO/STON certified hole doping of the films. The characteristic values of diffusion voltage, corresponding to a steep current increase at a forward bias have been estimated. Magnetoresistance and magnetic field-dependent *C-U* characteristics were measured at various temperatures to model charge distribution in the vicinity of the p-n junctions. It was found in this work that the effect of epitaxial strain on band gap of the manganites (LCaMO/STON) as well as enhanced phase separation phenomenon at the (LCeMO/STON) interface influence strongly nonlinear *I-U* characteristics of the heterojunctions. Microstructure of the manganite films, possible strains and oxygen diffusion in the interface region were found to be of key importance for the controlled fabrication of the manganite heterostructures.

Coffee break

Thursday morning, 7 September, 10:30

Parallel Session

Thursday morning, 7 September, 11:00

11:00 Invited oral

Local assessment of the current flow in large bulk superconductors and in coated conductors by the newly established magnetoscan technique - theory and experiment

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Large (RE-123) bulk superconductors (with a size of several centimeters) are promising materials for applications, since they provide high trapped magnetic fields. Scanning such fields by a Hall probe leads to rather "global" information on the sample quality, since currents from the whole sample volume contribute to the trapped field. Inhomogeneities and defects can strongly reduce the trapped fields, but details usually cannot be identified by this method. The situation is similar in coated conductors. Currently, a lot of research on Y-123 based conductors is carried out with the aim to produce long tapes with high critical current densities (jc's). Measuring jc over large lengths results in a global value determined by that cross section of the conductor that carries the weakest current. Local properties are not addressed in this way. The position dependence of the superconducting parameters can be obtained from small samples cut from larger pieces. To access the local properties without destroying the original samples, a new method, called magnetoscan, was recently introduced. A small permanent magnet scans the sample surface and locally induces currents. The field of these currents is recorded by a Hall probe fixed below the magnet. We show that the magnetoscan technique indeed reveals a significant local structure of the field map, indicating inhomogeneities and defects in both large bulk samples and long coated conductors. Several experimental results are shown and discussed. Artificial defects are introduced to analyze their effects. Additionally, we provide a theoretical description to confirm our interpretation. We numerically calculate the current dynamics during a magnetoscan applying a 3 dimensional model in the case of bulk materials, and a 2 dimensional model in the case of coated conductors. Defects and inhomogeneities are introduced by a position dependent jc. Experiment and theory are in good agreement for both types of application.

11:30 Oral

Vortex motion and quasiparticle resistivity in superconductors at microwave frequencies

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When analyzing experimental data of the complex microwave resistivity in superconductors in presence of an applied magnetic field, one has to face the problem of correctly taking into account all the relevant contributions. In fact, at nonzero frequencies both the normal and superfluid fractions, as well as moving vortices, contribute to the electrical transport, resulting in intricate expressions for the resistivity. We present here an extended study of microwave resistivity data measured by means of a broadband technique between 2 and 20 GHz and of a resonant system at 50 GHz. Data are collected on several superconducting materials (YBaCuO, SmBaCuO, MgB_2). We discuss the main experimental fingerprints that allow to identify the relevance of the different contributions from the measured microwave response as a function of frequency, temperature and magnetic field. We show that vortex motion and quasiparticle dissipation have different weight and different origins in different materials. In HTCs vortex motion prevails at high fields, while at low fields a significant quasiparticle contribution exists, that we ascribe to the presence of lines of nodes in the gap. In MgB_2 the two contributions are comparable even at high fields. For the latter material, we are able to quantitatively take into account vortex motion and quasiparticle response. In particular, we show that a two fluid model applies well to MgB_2 in a relatively large region of the H - T phase diagram. In this region an excellent agreement is found between derived superfluid parameters (superfluid density, upper critical field), theoretical predictions and independently measured parameter. This result, never obtained on this material by using standard expressions, clearly

demonstrates the necessity of taking into account all contributions to the overall microwave resistivity.

Lunch break
Thursday afternoon, 7 September, 12:30

Parallel Session
Thursday afternoon, 7 September, 14:00

14:00 Invited oral

Ferromagnetic oxide heterostructures for spin electronics

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For spintronic devices ferromagnetic materials with high spin polarization and Curie temperatures above room temperature are required. Promising candidates are transition metal oxides such as the doped manganites, magnetite (Fe_3O_4), or the double perovskites ($A_2BB'O_6$ with A= Sr, Ba, Ca and BB' = CrW, CrRe, FeMo, FeRe). In addition, transition metal doped oxide semiconductors such as Mn or Co doped ZnO or TiO_2 have attracted considerable interest. We have grown epitaxial thin films and complex heterostructures of various ferromagnetic oxides using UHV pulsed laser deposition. By process optimization a layer-by-layer growth mode could be established resulting in high quality epitaxial films. With respect to device applications heterostructures consisting of different materials are required. Here, strain accomodation, interdiffusion, oxygen stoichiometry and proper choice of termination layers are important factors in the engineering and optimization of interface properties. The heterostructures have been used for both the clarification of the underlying physics in half-metallic transition metal oxides and the realization of simple device structures. Among our various activities we discuss (i) the exchange mechanism and effect of doping and strain in the double perovskites [1], (ii) the tunnel magnetoresistance in magnetite based magnetic tunnel junctions, and (iii) the exchange mechanism and magnetotransport properties of Mn and Co doped ZnO [2].

This work is supported by the DFG within SPP 1157 (project GR 1132/13).

[1] P. Majewski et al., Phys. Rev. B 72, 132402 (2005), Appl. Phys. Lett. 87, 202503 (2005). [2] K.-W. Nielsen et al., Superlattices and Microstructures 37, 327 (2005).

14:30 Invited oral

Ferromagnetism of semiconducting compounds: oxides vs. arsenides

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There is currently widespread interest in the development of novel semiconductors which are ferromagnetic above room temperature. Much of this work has been stimulated by predictions of high temperature ferromagnetism in p-type semiconductors doped with Mn [1]. For GaN, ZnO, and diamond the above room temperature T_c has been predicted, providing simultaneous incorporation of 5% of Mn and $3x10^{20}$ holes per cm^3 can be achieved. Despite the p-type doping alone, the other big issue in preparation of transition metals rich compounds is an avoiding their inhomogeneous distribution, clustering and precipitations of foreign chemical/magnetic phases. Concerning oxides, several reports on Co, Fe and Mn enriched ZnO, or even in pure, normally nonmagnetic dielectric layers of HfO_2 and VO_2, showed evidence for ferromagnetic behaviour. But there are other that have found no ferromagnetic order originating from the semiconductor itself. Our results decidedly conform to the latter findings in that sense, that occasionally observed ferromagnetic coupling can be attributed to precipitations, in-situ or ex-situ contaminations, not mentioning technical flaws during experimental procedure.

Diametrically different situation is met in arsenides. Here recent advances of MBE resulted in routine fabrication of high and homogenous Mn concentration. Since the same Mn contributes also a hole to the system, carrier mediated ferromagnetism can be easily born. In canonical ferromagnetic semiconductor (Ga,Mn)As T_c above 170 K and a uniform magnetisation corresponding full atomic Mn magnetic moment has been observed. The hole density and Mn concentration dependence of T_c, magnetic anisotropy and other micromagnetic properties are astoundingly well reproduced by model theoretical calculation [1], indicating that (Ga,Mn)As may be the best understood ferromagnet.

[1] T. Dietl et al., Science 287, 1019 (2000), T. Dietl et al., Phys. Rev. B 63, 195205 (2001).

15:00 Oral

$LaNiO_3$ - a high temperature electrode studied by in situ spectroscopic ellipsometry

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$LaNiO_3$ is a metallic perovskite with suitable electric properties to be used as an conductive electrode in an all oxide hetero-structure. The fabrication of a such material on a $SrTiO_3$ (100) substrate by pulsed laser deposition has been studied in function of the oxygen partial pressure and substrate temperature. The variation of these two growth parameters results in changes in structure, morphology, optical and electrical properties of the thin films. An epitaxial growth has been obtained for an optimal set of parameters. High temperature in-situ spectroscopic ellipsometry (SE) was used to study the growth and the thermal variation of the optical properties in the temperature range of 300K to 1070K.

The characteristic feature of a conducting medium is the presence of free electric charges. The adjustement of the ellipsometric parameters has been realized with a Drude-Lorentz model. The Drude part corresponds to the oscillations of free electrons whereas the Lorentz

part is due to the bound electrons. Both free and bound electrons contribute to the optical properties. The refractive index *n* and the extinction coefficient *k* are obtained from the adjusted optical model and they are consistent with the literature for measurements at the ambient temperature. The resistivities can be calculated from the Drude part of the parameters and they are in agreement with the measures by a 4 probes techniques on the films. During the growth, an evolution of the optical properties is observed and the film thickness is given until the skin depth is reached. More interesting is the thermal evolution of both *n*, *k* during the cooling. The main observation is a change in thermal variation of *n* and *k* at a temperature of about 524K. The analysis of the model parameters indicates a change in the electric properties essentially related to the free carriers. Implications to the use of $LaNiO_3$ as a high temperature electrode will be discussed.

Coffee break
Thursday afternoon, 7 September, 15:30

Joint session Symposia E, F & K, Room 219
Thursday afternoon, 7 September, 15:50

15:50 Invited oral

Ferromagnetic oxide semiconductors: using off-stoichiometry to tune low-dimension magnetism and consequently the iron valency

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Today's research in diluted magnetic semiconductors (DMS) is concentrated on large band gap semi-conductors with the scope to obtain carrier mediated high temperature ferromagnetism by substitution of appropriate transition metal ions into the host matrix. This study investigates the possibility of alternative approach, such as the creation a of DMS starting from a ferromagnetic oxide. The control of stoichiometry during growth of thin oxide film will be used as a tool to modify the physical properties of the oxide such as magnetism, valence state and consequently transport properties. In particular, ferrimagnetic iron oxides like garnets or illmenites show already high intrinsic Curie temperatures (T_C > 400 K) and are suitable candidates for this case study. Among the different systems, the Yttrium Iron Garnet (YIG) will be presented in detail. Pulsed laser deposition of Yttrium Iron Garnet thin films allows to explore a new part of its phase diagram, e.g. controlled stabilization of iron and yttrium vacancies within the oxygen sub-lattice, and to tune magnetism by off-stoichiometry. Magnetization and Curie temperatures are measured by polar magnetic circular dichroism (MCD). A significant increase of the Curie temperature (+10%) indicates changes of the super-exchange coupling through the variation of the Fe-O-Fe distance. Simultaneously, an important increase of the magnetization (up to 120%) is observed. The temperature dependent MCD measurements demonstrate that the increase of magnetization is due to a preferential occupation of the iron vacancies on the octahedral sites. Iron valence presumably changes from Fe^{3+} to Fe^{4+} accompanied by the presence of iron and yttrium vacancies. Its consequences to the carrier doping in application to spintronics will be discussed.

16:35 Invited oral

Origin of ferromagnetism and phase separations in diluted magnetic semiconductors

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A considerable effort has been devoted to understand the origin of ferromagnetism, often persisting up to above room temperature, in a number of semiconductors doped with transition metals. In the talk, I will argue that ferromagnetic DMS can be divided into three categories. The first consists of (Ga,Mn)As and related compounds. Here, theory built on Zener's model of carrier-mediated ferromagnetism and the Kohn-Luttinger *kp* theory of semiconductors describes thermodynamic, micromagnetic, optical, and transport properties. To the second group belong compounds, in which the proximity of the localisation boundary and/or a competition between long-range ferromagnetic and short-range antiferromagnetic interactions leads to an electronic nanoscale phase separation that results in characteristics similar to colossal magnetoresistance oxides. Finally, in a number of compounds a chemical nanoscale phase separation is observed, reminiscent of spinodal decomposition. Mechanisms accounting for this effect in particular materials will be discussed.

The work is supported by NANOSPIN E.C. project (FP6-2002-IST-015728), by Humboldt Foundation, and carried out in collaboration with M. Sawicki, K. Osuch, H. Przybylińska, M. Kiecana, and A. Lipińska in Warsaw, as well as with groups of H. Ohno in Sendai, S. Kuroda in Tsukuba, K. Trohidou in Athens, J. Cibert in Grenoble, J. Jaroszyński in Tallahassee, and A. Bonanni in Linz.

Posters

Monday, 4 September

Poster Session 1

Main Hall
Monday afternoon, 4 September, 17:20

17:20 Poster K-1

Flux penetration in a ferromagnetic/superconducting bilayer

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The Hall sensor array is a useful tool for measuring local magnetic fields. An array of miniature Hall sensors has been used to study the flux penetration in a ferromagnetic/superconducting (F/S) bilayer consisting of Nb or YBaCuO as the S layer and the Co/Pt multilayer with perpendicular magnetic anisotropy as the F layer, separated by an amorphous Si layer to avoid proximity effect. The F layer is first premagnetized to different magnetization reversal stages to obtain various magnetic domain patterns. The effect of these domain patterns on the flux behavior in the S layer is then studied at various temperatures in the superconducting state. We have observed in the F/S bilayer with Nb that some domain patterns strongly increase the first penetration field indicating the formation of barrier at the sample edge. Further flux penetration shows a complicated pattern, including the regions of flux jumps separated by flat terraces. These effects are not detectable by the global SQUID magnetometry and persist to high temperatures, close to the superconducting transition temperature. Effects observed in the bilayer with YBaCuO are quite different. We discuss the origins of the observed flux penetration pattern.

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17:20 Poster K-2

The impedance of the melt-textured YBaCuO superconducting slab in high dc magnetic field

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We have performed ac impedance measurements of the melt-textured YBaCuO superconducting slab in a wide range of external dc magnetic field (from zero to 12 T). We have observed a peak in the real part of the impedance. This peak is connected with a skin size effect. It appears when ac penetration skin-size depth is equal to the half of the slab thickens.

In our work we analyze changes of this peak caused by variations of dc magnetic field and temperature. The changes of the peak are connected with some peculiarities of the average conductivity of the superconductor. In high temperature superconductors there are three characteristic ranges of the conductivity: flux-flow, flux-creep and thermally assisted flux-flow (TAFF) range. We analyze the conductivity of the YBaCuO sample by using the impedance measurements.

17:20 Poster K-3

EPR in kagome staircase compound $Mg_{2.997}Co_{0.003}V_2O_8$

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The paramagnetic resonance studies of Co^{2+} ions in $Mg_{2.997}Co_{0.003}V_2O_8$ single crystals are reported. The single crystals of $Mg_{2.997}Co_{0.003}V_2O_8$ were grown from polycrystalline feed rods by floating zone technique. This compound have the same (Cmca) crystal structure and very similar lattice parameters to the kagome staircase compound $Co_3V_2O_8$. The EPR measurements were carried out using Bruker EMX spectrometer working on fixed frequency (9.25 GHz) with Oxford-made helium flowing cryostat in temperature range from 3.8K to 300K. The samples were the roentgenographically oriented disks of 3 mm diameter and thickness of 0.5 mm cut from the single crystalline rod. The EPR spectrum shows two groups of resonance lines associated with two crystallographically nonequivalent Co ions positions that are known in the kagome staircase system as "cross-tie" and "spine" sites. The ground state was described by spin-Hamiltonian with an effective electronic spin S=1/2 and nuclear spin I=7/2. The local symmetry of oxygen octahedron surrounding Co ions, main values of the g-factors and hyperfine structure were determined for both cobalt positions.

17:20 Poster K-4

Synthesis and electrical properties of La-Pr-Mn-O thin films and heterostructures

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Perovskite manganites are widely investigated for their huge magnetoresistance and many interesting properties. Hole-doping is usually achieved in the $La_{1-x}A_xMnO_3$ compounds by substituting a bivalent cation such as Ca, Ba or Sr at a rare earth site. During the last few years, there was increasing interest in electronically doped manganites prepared by partial replacing La by a tetravalent Ce, Te, Zr as well as mixed valence Pr ions. Such electro-doped system could be very promising for fabrication of *p-n* junctions in novel spintronics devices. Here we report on the synthesis and study of both $La_{0.7}Pr_{0.3}MnO_3$ (LPMO) ceramic samples and thin films and related heterostructures composed of LPMO and *p*-type $La_{2/3}Ca_{1/3}MnO_3$ (LCMO). Ceramic LPMO samples were prepared by a conventional solid state reaction technique. Thin LPMO films and LPMO/LCMO

heterostructures were grown on lattice-matched perovskite $SrTiO_3$, $NdGaO_3$ substrates by pulsed laser deposition. Electron-doping has been indicated for LPMO ceramic samples and thin films from thermopower data. Both LPMO ceramics and thin films exhibited resistivity of 0.1 Ωcm at 300 K and semiconductor-like behaviour. Meanwhile, LPMO/LCMO interface resistance showed an anomalous peak at 185 K. The heterostructures exhibited nonlinear current-voltage characteristics. Series of post-deposition annealing experiments demonstrated a crucial role of annealing temperature and ambience on electrical, magnetic and optical properties of LPMO material and the heterostructures.

17:20 Poster K-5

Effect of post annealing on $La_{0.7}Sr_{0.3}MnO_3$ thin films

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Stability of $La_{0.7}Sr_{0.3}MnO_3$ thin films fabricated by pulsed laser deposition has been investigated. LSMO films were deposited on (100) LAO substrates at 650 ^{o}C with the films thickness about 50 nm. The oxygen pressures used to fabricate the films were 50 and 100 mTorr. Then in situ annealing was performed at 100 and 50 mTorr, respectively. Curie temperature (T_c) of the films was estimated from the peak of the temperature dependent resistivity (RT) data. For the films deposited at 100 mTorr, T_c of the film without post annealing is about 360 K. After annealing at 50 mTorr, T_c slightly dropped for short annealing time and recover to 360 K for 30 mins annealing. Resistivity at room temperature of the film annealed for 30 mins is $2x10^{-5}$ Ωm. Resistivity change between T_c and 20 K is 85%. For the films deposited at 50 mTorr, it maintains semiconducting behaviors without transition after annealing for different time imply that films are oxygen deficient. These effects are related to the structural and chemical properties of the films.

17:20 Poster K-6

Heat capacity analysis of $YBa_2Cu_3O_{7-\delta}$ superconductors: role of lattice and electron contribution

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Observed temperature dependent heat capacity $C(T)$ behavior of high-T_c $YBa_2Cu_3O_{7-\delta}$ cuprate superconductors, has been theoretically analysed in the temperature domain $70 < T < 110$ K. Calculations of $C(T)$ have been made within the two component scheme: one is the Fermionic term and the other is the Bosonic (phonon) contribution. While estimating the electronic term, we use a mean field step and follow two fluid model below and above T_c. Later on, the lattice heat capacity is estimated within harmonic approximation for high temperature expansion ($T > \theta/2\pi$), the model has only one free parameter, the moments of phonon density of states. Within the two fluid model for electronic specific heat along with reported γ value leads to a sharp discontinuity at T_c. The Coulomb correlations and electron-phonon coupling strength have significant implications on the γ. Henceforth, the present numerical analysis of specific heat from the present model shows similar results as those revealed from experiments. The accurate fitting of the specific heat data reveals that it is possible to decompose the documented specific heat into dominant lattice contribution and electronic channel. However, the specific heat from electronic term is only a fraction of lattice specific heat in $YBa_2Cu_3O_{7-\delta}$ high-T_c superconductors.

17:20 Poster K-7

Scattering processes of normal and fluctuating carriers in $REBa_2Cu_3O_{7-\delta}$ (RE=Y, Ho) single crystals with unidirectional twin boundaries

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The effect of twin boundaries on the normal and fluctuation conductivity of $REBa_2Cu_3O_{7-\delta}$ (RE=Y, Ho) single crystals has been investigated. The temperature dependence of the excess conductivity can be satisfactory described by the Lawrence-Doniach theoretical model. The twin boundaries are efficient scattering centers of normal and fluctuation carriers. The predicted values of the coherence length perpendicular to the ab-plane $\xi_c(0)$ are in good agreement with the values determined from the magnetic measurements of stoichiometric YBaCuO crystals.

17:20 Poster K-8

Intrinsic defect mechanisms and dopant substitution in $RBa_2Cu_3O_{6.5}$ (R=Y, Al and Ho)

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We have applied an atomic scale simulation technique based on classical ionic potentials to study the structure and defect chemistry of a range of orthorhombic $RBa_2Cu_3O_{6.5}$ compounds. The technological significance of the rare-earth cuprate superconductors has been briefly reviewed whereas the efficiency of the approach has been demonstrated through comparison of the predictions to previous ex-

perimental and theoretical results. The internal energies for the intrinsic defect reactions have been calculated. A number of divalent and trivalent defects have been dissolved into the Cu^{2+} and R^{3+} sublattices respectively. The calculated solution and binding energies demonstrate a systematic variation as a function of the cation dopant radius. Particular bond distances and the critical temperature (T_c) have been correlated suggesting that the superconducting properties of RBaCuO are related to the local structural and electronic environment.

17:20 Poster K-9

Defect chemistry and clustering in $Y_{1-x}Pr_xBa_2Cu_3O_{6.5}$

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Atomic scale simulation techniques based on energy minimization have been applied to study the structure and defect chemistry of a range of orthorhombic $Y_{1-x}Pr_xBa_2Cu_3O_{6.5}$ (x=0, 0.25, 0.5, 0.75, and 1) compounds. The technological significance of the $Y_{1-x}Pr_xBa_2Cu_3O_{6.5}$ and related high temperature superconductors has been reviewed. The efficiency of the model has been demonstrated through comparison of the predictions with previous experimental and theoretical results. A number of divalent and trivalent defects have been dissolved into the Cu^{2+} (or Ba^{2+}) and Y^{3+} (or Pr^{3+}) sublattices respectively. The calculated solution and binding energies demonstrate a systematic variation as a function of the praseodymium content.

17:20 Poster K-10

Excess conductivity of $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-x}$ single crystals

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In this work the in-plane conductivity of YBaCuO and $Y_{1-z}Pr_zBa_2Cu_3O_{7-x}$ single crystals with a system of unidirectional twin boundaries was investigated. The results indicate that the partial substitution of Y by Pr results to the formation of two superconducting phases with different critical temperatures. The Pr dopants behave as efficient scattering centers of normal and fluctuating carriers. For this, a slight doping with praseodymium (till $z \approx 0.05$) results to a significant narrowing of the temperature interval in which the pseudo-gap regime is realized in the ab-plane of YBaCuO single crystals.

17:20 Poster K-11

Magnetization, resistivity, and IR spectra of (Tb-Dy)$BaCo_2O_{5.5}$ single crystals

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We report on the investigation of magnetic, magnetoresistive and IR-spectra of the single crystals of (Tb-Dy)$BaCo_2O_{5.5}$ grown by the spontaneous crystallization method. This layered perovskite exhibits a very rich phase diagram including two different paramagnetic phases with MI transition at 340K as well as ferromagnetic and antiferromagnetic phases. The strictly pronounced anisotropy of magnetization and resistivity was observed at the temperatures below T_c. The relationship of R_{ab} / R_c varies from 1 to 20 for T = 260÷100K and MR effect value exceeds 10% at 100K in 1T. Infrared absorption(transmission) spectra were measured between 300-4000 cm^{-1} using IR Fourier-spectrophotometer Nikolet-460 (Varian, USA). The IR-spectra are sensitive to the O-Co-O bonding length and angles. The frequencies of bending and stretching modes are connected with 250-420 cm^{-1} and 400-800 cm^{-1}, respectively. We assume that the display of a few splitting components in the system of the bending mode (377 and 429 cm^{-1}) as well as in the system of the stretching mode (522, 580, 634 and 664 cm^{-1}) are due to the existence of different local distortions of the fragments of the single crystal matrix owing to the presence of (Tb-Dy) rare earth ions.

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17:20 Poster K-12

Magnetic anisotropy and structural properties of ferromagnet/MgO/ferromagnet system

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Magnetic tunnel junctions (MTJ) with MgO barriers so far have shown the highest tunelling magnetoresistance effect (TMR). Such structures are potentially important for device applications. The tunneling mechanism in MTJ with MgO barrier is different from these where amorphous AlO_x is used.

We have fabricated and studied the role of the bottom and upper electrode on the growth mechanism of the epitaxial MgO barriers in M_1/MgO/M_2 trilayer structures by means of XRD, NMR and TEM techniques. As an M_1 and M_2 electrodes we used Co, Fe and CoFe layers. Our preliminary results by means of NMR method indicated different chemical short range ordering in bottom and upper electrodes.

17:20 Poster K-14

Quantitative magneto-optics of patterned $YBa_2Cu_3O_{7-d}$ / $La_{1-x}Sr_xMnO_3$ perovskite bilayers

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Magneto-optical imaging of the magnetic field distribution in magnetic and superconductor perovskite heterostructures is presented. The real-time high-resolution imaging of the magnetic field distribution by the magneto-optical technique [1] allows the direct visualization of the local electromagnetic interaction between materials with different electronic orders [2]. We report on new nonlinear calibration and background treatment leading to the quantitative imaging of the local magnetic pattern in the magnetic layer and of the local supercurrent density in the superconducting one, with sub-micrometric resolution. In particular, we present the quantitative measurement of the enhancement/depression of the local superfluid density due to the electronic interaction with the underlying magnetic structure exhibited by the manganite layer.

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17:20 Poster K-15

Inhomogeneous magnetic state in $LaMn_{0.5}Fe_{0.5}O_3$

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Magnetization and neutron diffraction studies on $LaMn_{0.5}Fe_{0.5}O_{3+d}$ perovskites have been performed. In order to exclude the influence of Mn^{4+} ions from concideration the as-prepared one has been annealed in vacuumed tube that led to nearly stoichiometry of the sample that has been confirmed by computer refinement of the NPD data. The oxygen reduction led to certain changes in the magnetic properties of the sample.

Both of the samples have smeared magnetic transition and branching point near the same temperature (~ 180 K) thus demonstrating inhomogeneous magnetic states of the compounds. Although the reduced sample has a less pronounced divergence between FC and ZFC curves, possibly confirming a stronger ferromagnetic component in comparison with the as-prepared one. The reduced sample most likely has certain ferromagnetic order which is seen from the neutron measurements, whereas the as-prepared compound demonstrates dominated spin glass component. The NPD measurements revealed an antiferromagnet component in the both samples. Most likely an origin of the antiferromagnet ordering is similar to that discussed for $LaCo_{0.5}Fe_{0.5}O_3$ compound thus confirming a certain phase separation. An essential role of competed phases is supported by the field magnetization measurements, thus even in relatively high magnetic fields no saturation was observed.

Fitting of the NPD data revealed that differences in the Mn-O distances are almost neglected thus indicates a removing of the static Jahn-Teller distortions. Magnetic properties of the stoichiometric solid solution can be well described in the terms of superexchange model assuming positive Mn^{3+} - O - Mn^{3+} dynamic interactions whereas Fe^{3+} - O - Fe^{3+} interactions are strongly antiferromagnetic.

17:20 Poster K-16

Interpretation of anomalous optical conductivity in the ferromagnetic metallic state of manganites

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The observed frequency dependent optical response of hole doped $La_{0.8}Ca_{0.2}MnO_3$ manganites, has been theoretically analysed. Starting from an effective two-dimensional (2D) interaction potential for hole doped manganites, the spectral function is developed. Calculations of the optical conductivity $\sigma(\omega)$ have been made within the two-component scheme: one is the coherent Drude free carrier excitations. The other is incoherent motion of carriers from one site to other leading to a polaron formation, originated from interband transitions between the Hund rule split bands (Mn-Mn) and due to charge-transfer transitions between the O 2*p* and the Mn e_g bands. The approach successfully accounts for the anomalies reported in the optical measurements for the ferromagnetic metallic state. Estimating the effective mass from heat capacity measurement, the model has only one material parameter, the relaxation rate. The frequency dependent relaxation rates are expressed in terms of memory functions and the coherent Drude carriers from the effective interaction potential leads to a sharp peak at zero frequency and a long tail at higher frequencies, i.e. in the infrared region. While to that the hopping of carriers from Mn site to Mn and Mn site to O (incoherent motion of doped carriers) yields two-peak value around 0.4 and 4.0 eV in the optical conductivity centred at mid-infrared region. We find that both the Drude and hopping carriers in the manganites will contribute to the optical process of conduction in the Mn-O planes and shows similar results on optical conductivity in the mid-infrared as well as infrared frequency regions as those revealed from experiments.

Keywords: Optical conductivity, layer interactions, Drude carriers, and hopping carriers.

17:20 Poster K-17

Electroresistance and magnetoresistance of polycrystalline $La_{0.67}Ca_{0.33}MnO_3$ films

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Polycrystalline $La_{0.67}Ca_{0.33}MnO_3$ films were grown by pulsed laser deposition technique on 100-faces of a cleaved of MgO crystal demonstrating arrays of nanoscale defects. The films with average thickness of about 150 nm showed a granular structure of submicron size (as found by AFM) and exhibited paramagnetic-ferromagnetic phase transition temperature at 145-220 K. Ag electrodes were deposited onto the masked tape-like films with 1 mm in width to perform electrical and magnetic field-dependent electrical resistance measurements. Either dc current or rectangular shape electric pulses of nanosecond time duration and repetition rate of 100 Hz were used for the investigations. Influence of electrical field on film transport properties was investigated systematically at various temperatures, various applied magnetic field and using a gap between the adjacent patterned electrodes ranging from 10 μm to 360 μm. The highest values of both negative magnetoresistance (CMR) and electroresistance (ER) of the films were indicated in the vicinity of their characteristic temperature T_m corresponding to a maximum value of low-field resistance. Slight shift of the resistance-vs-temperature curve R=f(T) to higher temperatures was indicated for the films with applied magnetic field. However, our measurements using nanosecond pulses revealed absence of any shift of the characteristic peak (at T_m) with applied strong electrical field. Furthermore, occurrence of additional resistance anomaly at $T>T_m$ has been indicated in a case of strong pulsed electrical field. Additional investigations are in progress to reveal correlation between ER and CMR and explain the origin of the observed high field resistance anomaly.

17:20 Poster K-18

Magnetoresistance of polycrystalline LCMO films in microwave magnetic field

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Rare earth manganites demonstrate unusual electrical properties due to a strong interplay between spin, charge and orbital degrees of freedom. During the last years, major attention was paid to resistance change of both manganite films demonstrating various crystalline quality and related heterostructures by applying a permanent magnetic field (colossal magnetoresistance, tunneling magnetoresistance, grain boundary magnetoresistance etc). Recent investigation of polycrystalline manganite films in a microwave electrical field gave insightful information about their grain boundaries. However, up to now, microwave radiation-induced resistance change of the manganite films was studied taking into account a key role of high frequency electrical field on electrical properties of the manganites. Here we present new experimental evidence indicating importance of magnetic field component of microwave field (f = 9.4 GHz) on magnetoresistive properties of polycrystalline manganite films. Polycrystalline $La_{0.7}Ca_{0.3}MnO_3$ films were grown on lucalox substrates by on-axis pulsed laser deposition. Microwave measurements revealed different character of the temperature-dependent electrical resistance of polycrystalline LCMO films placed in various positions of a waveguide. In the center of the waveguide, microwave electric field should determine resistance change under the influence of H_{10} wave. Near the narrow wall of the waveguide, where the electric component of the H_{10} wave decreases notably, the longitudinal microwave magnetic component should take part in the magnetoresistance of the polycrystalline LCMO film that was proved experimentally. Additional experiments with applied external permanent magnetic field confirmed the influence of microwave magnetic field on magnetoresistance of polycrystalline LCMO films.

17:20 Poster K-19

Half metallic $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) films for spintronic applications

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In the emerging field of spintronics, information is carried by the carrier spin instead of their charges. Introduction of spin degree of freedom in addition to the carrier charge gives the potential advantage of non-volatile data storage, increased processing speed, lower power consumption and higher information density. LSMO, a well known half metallic manganite has been successfully used as a spin injecting electrode in many inorganic/organic spintronic devices. LSMO film properties are greatly influenced depending on substrate properties, deposition parameters, film thickness, post-annealing treatments etc. and hence their spin injection capability is also immensely modified. So, our present study concentrates on optimizing the different parameters for achieving the best spin injector for our LSMO based spintronic devices. We have deposited LSMO films by pulsed laser deposition (PLD) on 3 different substrates i.e. MgO (100), $SrTiO_3$ (STO) (100) and $NdGaO_3$ (NGO) (001), using different deposition temperature, laser fluence and different thicknesses. The films were characterized by Atomic Force Microscopy (AFM), X-ray Diffraction (XRD), magnetization, transport and magnetoresistance (MR) measurements. The effect of oxygen and vacuum annealing treatments on the films were also studied. Our study indicates that films deposited at 700°C are c-axis oriented epitaxial films with a very smooth surface while the films deposited at higher temperatures shows polycrystalline growth with higher surface roughness. Comparative study of the magnetic and transport properties of the LSMO films and the spin - valve devices based on these different LSMO films will also be discussed.

17:20 Poster K-20

Studies on ionic conductivity and electrical relaxation in nanocrystalline cubic-zirconia using complex impedance analysis

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6-10 mol% of yttria, scandia, dysporsia and ceria stabilized nanocrystalline cubic-zirconia was prepared by chemical co-precipitation technique at room temperature. The co-precipitated powders were weakly agglomerated with an average crystallite size of 50-100 nm, surface area of 8.49 m^2/g and average particles size of 150-300 nm. The activation energy of formation, calculated using different approaches, ranged 0.90-1.30 eV. The frequency dependent conductivities and dielectric constants were measured in the frequency range from 50 Hz to 1 MHz and at a temperature between 300 and 900 K. Complex impedance spectra, complex modulus formalism and complex conductivity spectra were carefully analyzed to separate grain (bulk) conductivity from grain boundary contribution and electrode-electrolyte effects. Analysis of ac impedance data using complex impedance plane representation gives the dc and ac resistance showing a typical negative temperature coefficient of resistance behavior analogous to a semiconductor. Relaxation time obtained from complex conductivity spectra are well matched with the loss impedance plots and loss modulus spectra. Impedance analysis suggests the presence of temperature dependent electrical relaxation process in the material.

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17:20 Poster K-21

Dynamics of thermomagnetic avalanches in melt-textured YBaCuO superconductors

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Thermomagnetic avalanches are commonly observed in hard type-II superconductors. From the viewpoint of practical applications of type-II superconductors thermomagnetic avalanches are problematic, because they may drive the superconducting sample into a normal or, at least, into a resistive state. The dynamics of the thermomagnetic avalanche depends on the processes of thermal and magnetic diffusion. The equations describing dynamics of the thermomagnetic avalanche are nonlinear, because many of the parameters influencing diffusion processes changes nonlinearly with temperature and with the magnetic field. At certain conditions, this may lead to unusual behavior of the thermomagnetic avalanche dynamics, e.g. to the development of dendritic flux structures in superconducting films [1].

In present work we have investigated dynamics of thermomagnetic avalanches in melt-textured YBaCuO samples. Our experiments were performed by using a miniature Hall probe placed on the surface of the investigated sample. Samples of different shapes were investigated. We have found that in melt-textured YBaCuO superconductor the thermomagnetic avalanche is accompanied by giant convergent oscillations of the surface induction. Similar experiments we have performed on other type-II superconductors. Convergent oscillations of the surface induction were also observed in the case of a slab of conventional NbTi superconductor. In the case of the slab of $Bi_2Sr_2CaCu_2O_{8+\delta}$ high temperature superconductor the dynamics of the thermomagnetic avalanche is also complex, consisting of two processes of different time scales. However, in this case no convergent oscillations were found.

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17:20 Poster K-22

Magnetic and Magnetotransport Properties of $(Co,Fe)S_2$ thin films

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Recently $Co_{1-x}Fe_xS_2$ has been proposed as a tunable source of highly spin-polarized electrons, based on the fact that the Fermi level may be fine-tuned by solid solution alloying with the isostructural diamagnetic semiconductor FeS_2 having the pyrite structure. Anisotropic magnetoresistance measurements have been used as a probe of the sign of carrier polarization.

In this study we report on the preparation, magnetic and magnetotransport properties, of $(Co,Fe)S_2$ thin films. The films have been prepared by thermal-sulfuration of $Co_{1-x}Fe_x$ films in a sulfur atmosphere at 500^0C, at a sulfur pressure of 1 atm for 12 hrs. $Co_{50}Fe_{50}$ films prepared by Pulsed Laser Deposition (PLD) as well as Magnetron Sputtered $Co_{30}Fe_{70}$ films have been used. Proper adjustment of heat treatment conditions is crucial in order to achieve the correct sulfur stoichoiometry throughout the film. Thicker films show a cusp in the magnetization versus temperature curves around 100K which indicates an inhomogeneous nature.

The resistivity decreases monotonously as a function of temperature and shows a plateau at the Curie temperature. In the MR curves a small (2%) low field extrinsic contribution is observed at 5K which decreases rapidly with temperature and disappears well below the Curie temperature of 135K. This extrinsic MR is increased (4%) for thinner films. Above the Curie temperature a quadratic MR term dominates. The coercivity decreases linearly with temperature. The Hall effect measurements show a substantial Anomalous Hall Effect (AHE) contribution that is superimposed at the ordinary Hall effect and disappears at the T_C.

17:20 Poster K-23

Influence of pressure on magnetic properties of (Sr, La)(Ru, A)O_3 for A = Cr, Mn

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An influence of hydrostatic pressure on phase transition temperature, T_C, and spontaneous magnetisation, M_0, of selected perovskite ruthenates ($SrRuO_3$, $La_{0.2}Sr_{0.8}RuO_3$, $SrRu_{0.9}Mn_{0.1}O_3$ and $SrRu_{0.9}Cr_{0.1}O_3$) has been determined from magnetic measurements performed in the pressure interval of 0-12 kbar. Decrease of T_C with pressure has been found for all of the samples. The $M_0(P)$ remains unchanged for most of the samples, except the $La_{0.2}Sr_{0.8}RuO_3$ sample, where M_0 decreases with pressure. For $La_{0.2}Sr_{0.8}RuO_3$, the pressure coefficient dT_C/dP = -0.67 K/kbar is very similar to that of $SrRuO_3$ (-0.68 K/kbar). For $SrRu_{0.9}Cr_{0.1}O_3$ and $SrRu_{0.9}Mn_{0.1}O_3$ the pressure coefficient dT_C/dP is equal to about -0.5 K/kbar. The decrease of T_C in $SrRuO_3$ was reported earlier and explained in terms of strain effects. For La-substituted compound, a similar decrease of T_C with increasing pressure is probably caused also by the strain effects. The decrease of T_C with increasing pressure for both Cr-substituted and Mn-substituted samples indicates an overall band structure effect on T_C for these hole-doped compounds. Since the pressure affects the Ru-O-Ru bond angles and the Ru-O bond lengths, and the ferromagnetism in ruthenates is sensitive to these parameters, a weakening of ferromagnetic interactions is, thus, consistent with complex band structure effects related to the modulation of the Ru-O hybridisation by the change of structural distortion.

17:20 Poster K-24

Influence of manganese doping on transport properties in thin LCMO films

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The massive resurgence of interest for manganites thin films is connected with hope for applying these materials in commercial devices. The goal of this work is to establish the influence of temperature, applied magnetic field, current and time on the most industry-important property - resistivity.

In this paper we present the influence of manganese doping on thin manganite films properties. The measurements were carried out on 100 nm thin films family $(La_{0.7}Ca_{0.3})_{1-x}Mn_{1+x}O_3$, x=0, 0.1 and 0.2. The samples were deposited onto $LaSrGaO_4$ substrates using PLD method. All films are single-phase, oriented along the pseudo-cubic axis [100] and have the same structural symmetry.

Three types of experiments were conducted. In the first one R(T) curves were registered for five magnetic field and four current values (B=0, 0.1, 1, 4, 7T and I=10nA, 0.1μA, 1μA, 10μA). The shift of T_c, as well as, the resistivity changes are observed. In the $(La_{0.7}Ca_{0.3})_{0.8}Mn_{1.2}O_3$ sample, the CMR reaches the value of ~80% and a resistivity switching in low field, e.g., 0.2T, is observed. This switching occurs only at low I values, e.g., 10nA, and may be connected with percolative conduction. The time and history characteristics are also done. Thus, in the above sample an emergence of a new, in comparison with the x=0 composition, insulating and metastable ground state has been identified. According to the Mott's model, carriers in this insulating state are highly correlated. To prove it, the resistivity magnetic loops R(B) were studied. The hysteresis occurs at low temperatures, where the ground state changes. The I-V characteristics were also measured. Their evolution before and after applying magnetic field at several temperatures is observed. The current induced phenomena and their discussion will be presented.

17:20 Poster K-25

Evidence for strong electron-phonon interaction in doped Bi-2212 from Josephson spectroscopy

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According to Abrikosov [1] a high transition temperature T_c in HTSC occurs because of the presence near the Fermi level of an extended van Hove singularity. Due to Abrikosov's model optical phonons with small wave vectors play a leading role in pairing. A strong electron-phonon coupling in HTSC was confirmed by studies of the effect of generation of Raman-active optical phonons by AC Josephson current [2, 3], by photoemission spectroscopy data [4], by studies of the isotop effect [5], and the effect of renormalization of the quasi-particle density of states at temperatures below T_c [6]. In the present investigation an interaction between the AC Josephson current and Raman-active phonon modes in the entire range of phonon frequencies (up to 20 THz) was observed in doped Bi-2212 single crystals. It has been found that in the current-voltage characteristics of doped Bi-2212 Josephson junctions the structure related to generation of optical phonon modes can be observed in both underdoped and overdoped single crystals, with the doping level having only a negligible effect on the frequency of the main phonon modes. This means that the electron-phonon coupling in BSCCO does not change significantly over the entire region where superconductivity exists. At the same time we have not observed any structure which could be related to the "magnetic resonance" mode whose energy is expected to change significantly on doping. Thus the role of magnon modes in pairing is questionable.

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17:20 Poster K-26

Scaling of superconducting gap and critical temperature in doped Bi-2212, Bi-2201 and Hg-1201

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High-temperature superconductivity in cuprates occurs in CuO_2 planes within a relatively narrow interval of impurity hole concentration p [1]. It is well established that the transition temperature T_c varies with p according to a parabolic law. At the same time there is a conflicting information about a doping dependence of a superconducting gap Δ. In the present investigation we have measured Δ in doped Bi-2212, Bi-2201 and Hg-1201 by tunneling, Josephson and Andreev spectroscopy using a break junction technique. In particular we have studied the intrinsic Josephson effect (IJE) on natural ultrathin steps (with a height from 1.5 to 30 nm) which are always present on cryogenically cleaved surfaces of doped HTSC single crystals. For a stack of n equivalent junctions the value of the gap voltage V_{gn} corresponding to a sharp increase of a quasi-particle current (**J**||**c**) is given by: $V_{gn} = (2\Delta/e)n$. The experimental dependence $V_{gn}(n)$ has been used to determine the gap parameter Δ with high accuracy. The unusual sharpness of a gap feature in case of a highly anisotropic gap parameter could be caused by an extended van Hove singularity [1], the presence of which significantly enhances the gap structure in current-voltage characteristics for **J**||**c** [2, 3]. We have used the data of tunneling, intrinsic tunneling and Andreev spectroscopies to derive the dependence of the superconducting gap Δ on the impurity hole concentration p in Bi-2212, Bi-2201 and Hg-1201. In all cases the superconducting gap Δ(4.2 K) was found to scale with the transition temperature T_c in the entire doping range.

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17:20 Poster K-27

Surfactant effect on synthesis of nanocrystalline $La_xSr_{1-x}MnO_3$ by hydrothermal method

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Crystalline $La_xSr_{1-x}MnO_3$ (LSMO) has been prepared by a hydrothermal route in the presence of surfactant. The cationic surfactant of cetyltrimethylammonium bromide (CTAB) as a template is used to regulate the nucleation and crystal growth. The as-synthesized product has been characterized by X-ray diffraction and transmission electron microscopy. Nanosized and uniform rod-like monocrystals of LSMO have been obtained. Their magnetic properties have been studied using a vibrating sample magnetometer. Our results reveal that the use of surfactant helps to lower the processing temperature and to speed up the formation and crystallization of LSMO. The surfactant also provides us an additional means of controlling the nanocrystalline size.

17:20 Poster K-28

Anharmonicity and weak modes assignment in LaSrCuO with oxygen isotopic substitution

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The oxygen isotope effect on the first and second order Raman scattering of high-quality $La_{2-x}Sr_xCu^{16,18}O_4$ (80% substitution ^{16}O by ^{18}O) polycrystalline compounds with x=0.00, 0.015 has been investigated. Measurements were obtained on microcrystallites in the approximate (*zz*) and (*xx/yy*) scattering configuration, at low temperatures (down to 10K). The soft mode that appears in the first order *zz* spectra related with the orthorhombic-to-tetragonal phase transition shows a deviation from the harmonic law at all temperatures studied. The apex oxygen mode follows quite well the mass harmonic law, even at low temperatures. A broad band of apparent B_{1g} symmetry in the region of 300-330 cm^{-1} of the *(xx)* polarization spectra seems to consist of two modes, one of which shifts with the isotopic substitution and the other is not shifted and therefore is due to La atoms vibrations. Concerning the second order Raman scattering several weak and strong peaks have been observed and categorized to those that involve vibrations of the oxygen atoms or the heavy elements.

17:20 Poster K-29

Specific heat, magnetocaloric effect and phase diagram of $La_{1-x}Sr_xMnO_3$ system for $0 \le x \le 1$

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Majority of research of $La_{1-x}Sr_xMnO_3$ manganites was performed for $x < 0.4$ compositions. Only a few papers (~5) described properties of the $0.6 < x \le 1.0$ samples. This was caused by an inherent difficulty in synthesizing samples with $x > 0.5$. In the present studies, the phase diagram for the whole range of compositions ($0 \le x \le 1$) was investigated by means of the specific heat measurements, that were performed for 11 selected compositions, over the temperature range 2-390 K, in magnetic field up to 9 T. As a result, (1) two controversies concerning the phase diagram have been clarified, i.e., it was shown that the order of the Jahn-Teller transition does not depend on x and that the phase transition occurring below the Curie temperature for the $0.155 \le x \le 0.17$ samples is the Jahn-Teller transition, not a charge ordering one. (2) The ferromagnet-paramagnet, antiferromagnet-paramagnet, and antiferromagnet-ferromagnet

transitions have been thoroughly studied. It was evidenced that the shape of the specific heat anomaly and the kind of critical behavior of the specific heat at the antiferromagnet-paramagnet phase transition strongly depended on the type of the antiferromagnetic configuration (A, C, and G). The temperature of the first order antiferromagnet-ferromagnet transition occurring in the x = 0.55 composition was found to be strongly lowered by the magnetic field (by ~33 K for 9 T). (3) Two parameters characterizing magnetocaloric effect, i.e., the change of temperature of an adiabatically isolated sample and the change of entropy of a sample under isothermal conditions, induced by the change of magnetic field, have been determined. The values of these parameters, allowing to assess applicability of the studied material for magnetic refrigeration, suggest that the magnetocaloric effect in (La,Sr)MnO_3 is substantial but not outstanding in comparison with other refrigerant materials.

17:20 Poster K-30

Substrate effect on the ground state of the magnetic order in NSMO/YBCO superlattices

Andrei Tsarou, Roman Minikayev, Wojciech Paszkowicz, Maciej Sawicki, Piotr Dluzewski, Piotr Przyslupski

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One of the purposes of research into the growth of high quality superlattices is to develop structures applicable in electronic devices, another to study proximity effect between magnetic and superconducting layers. We report here our recent results on structure, transport and magnetic properties of $Nd_{0.5}Sr_{0.5}MnO_3$/$YBa_2Cu_3O_7$ superlattices. Up to now among manganite/cuprate structures mostly heterostructures with ferromagnetic ground state of manganite were studied. In the bulk form $Nd_{0.5}Sr_{0.5}MnO_3$ compound is charge ordered insulating antiferromagnet. The NSMO/YBCO superlattices were deposited on (110) $NdGaO_3$ and (100) LSAT substrates. Our preliminary results demonstrate significant differences in the magnetic properties of superlattices deposited on $NdGaO_3$ and LSAT substrates.

17:20 Poster K-31

Strain relaxation in thin films of LaSrCuO grown by pulsed laser deposition

Iryna Zaytseva, O. Abal`oshev, Marta Z. Cieplak, Viktor Domukhovski, Marek Berkowski

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The studies of the growth of thick films of $La_{1.85}Sr_{0.15}CuO_4$ (LSCO) by pulsed laser deposition on the $SrLaAlO_4$ substrates indicate the presence of variable built-in strain, ranging from compressive to tensile. While the compressive strain is induced by the mismatch to the substrate, the origin of tensile strain is unclear, and we have suggested recently that it may be caused by the thin layer of the oxygen or strontium-deficient material which grows in the initial stages of the deposition [1]. To check this hypothesis in the present study we investigate the properties of several groups of LSCO films of thicknesses ranging from 20 to 300 nm. The X-ray diffraction and the atomic force microscopy are used to evaluate the strain and the microstructure, and the superconducting properties are measured using dc resistivity and ac susceptibility. For each group of films of given thickness, several films are studied. The films in each group grow with variable degree of strain. In thin films the strain ranges from compressive to close to zero indicating variable compressive strain relaxation. In thick films the strain ranges from zero to tensile strain. Surprisingly, the maximum Tc observed in each group is almost independent of the film thickness. This observation rules out the possibility that the tensile strain is caused by the oxygen or strontium-deficient layer close to the substrate interface. Instead, it is possible that the grain-to-grain misalignment in thick films contributes to the building of tensile strain inside the these films.

1. M. Z. Cieplak, M. Berkowski, A. Abal'oshev, S. Guha and Q. Wu, Supercond. Sci. Technol. **19**, 564 (2006).

Wednesday, 6 September

Poster Session 2 / Poster Award Ceremony

(contiuation of Monday's Poster Session), Main Hall
Wednesday afternoon, 6 September, 15:50

EMRS 2006 Fall Meeting Exhibition

Programme

Abstracts

Unscheduled abstracts

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Exhibition

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Exhibition

National Institute for Materials Science

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Abstracts

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Satellite events

Fall School on Thermal Analysis

Programme

Abstracts

Unscheduled abstracts

Author alphabetical order

Poster

Thermal Conductivity analysis of Y-124 Copper oxide Superconductors

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We investigate the anomalies reported for the thermal conductivity (k) of the Copper Oxides superconductors by developing a model Hamiltonian that incorporates the scattering of phonons with defects, grain boundaries, charge carriers, and phonons in the lattice. The lattice thermal conductivity dominates in superconductors is an artifact of strong phonon-defects and -impurity scattering mechanism below T_c. Later on, the scattering of electrons with impurities is investigated in order to assess their role towards thermal conduction. The anomalies are well accounted in terms of interaction among the phonons-impurity and the carrier-impurity. It is concluded that the behaviour of the thermal conductivity is determined by competition among the several operating scattering mechanisms for the heat carriers and a balance between electronic and phononic contributions.

Posters

Abstracts

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Poster

Thermal Conductivity analysis of Y-124 Copper oxide Superconductors

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We investigate the anomalies reported for the thermal conductivity (k) of the Copper Oxides superconductors by developing a model Hamiltonian that incorporates the scattering of phonons with defects, grain boundaries, charge carriers, and phonons in the lattice. The lattice thermal conductivity dominates in superconductors is an artifact of strong phonon-defects and -impurity scattering mechanism below T_c. Later on, the scattering of electrons with impurities is investigated in order to assess their role towards thermal conduction. The anomalies are well accounted in terms of interaction among the phonons-impurity and the carrier-impurity. It is concluded that the behaviour of the thermal conductivity is determined by competition among the several operating scattering mechanisms for the heat carriers and a balance between electronic and phononic contributions.

Thin-layered materials workshop

Warsaw, 3 September 2006

The event is organised as a continuation of the tradition of workshops organised and sponsored by the AMAS-ISN: International Scientific Network for Advanced Materials and Structures.

Lectures on:

· Technology,
· Characterisation techniques,
· Modelling,
· Application

of contemporary materials produced and applied as thin films will be delivered by renowned experts.

Although the lectures, reviewing the state-of-art in the particular fields, will be addressed especially to young researches and students everybody interested in the subject-matter is cordially invited to attend the Workshop.

Participation in the workshop is free of charge.

Contact and registration - deadline: May 15 2006:
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Programme

Sunday, 3 September

Lectures
Sunday morning, 3 September, 9:00

9:00 Oral

Chemically and Electrochemically Deposited Thin-layered Materials

Maria Trzaska

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The lecture is focused on fundamentals and mechanisms of processes for producing thin-layered materials by the electroless and electrocrystalization methods. Features of various techniques and technologies with using these methods are presented. Possibilities of forming such different structures as: amorphous, nanocrystalline, microcrystalline as well as composite are exhibited. Influences of the process parameters on the material structures and properties are described. Identification of morphologies of the thin-layered materials by using SEM, AFM, RTG, TEM techniques as well optic microscopy are presented. Examples of various structures of materials
Produced by the electroless and electrostalization methods are given. Properties and applications of the thin-layered materials are described.

9:30 Oral

TEM Characterization of Thin-Layered Materials

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The progress in physical vapor deposition techniques opens possibilities to produce more and more complicated multilayer coatings of greatly improved properties. The detail microstructure control of such coating is very important especially as a malfunction or lower than expected properties result. Due to fine features of such systems frequently practically only transmission electron microscopy (TEM) guarantees the required high spatial resolution. This resolution is however realized only on very thin foils which are both a problem to prepare but also to handle.
The present overview summaries the existing sample preparation techniques including argon ion milling and gallium ion cutting (so called Focused Ion Beam technique). The last technique is realized through two mine ways, i.e. “H bar technique” and “*in-situ* welding”. The effect of sample preparation on results of TEM investigations including both microstructure and analytical results will be discussed. It shows that even very good high resolution electron microscopy (HREM) images of multilayer TiN/Ti multilayer coatings might be recorded working with properly prepared FIB samples. However, the analytical investigations realized using energy dispersive systems (EDS) are very liable to errors caused by artifacts in case when “H bar” specimens are used practically excluding quantitative measurements. The more reliable in such situations are simple maps which however supply less precise qualitative description of material.

10:00 Oral

Characterization of Thin-Layer Materials by Auger Electron Spectroscopy (AES) Combined with Ion Etching

Marcin Pisarek

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Studies of the chemical and physical nature of passive oxide films

formed on metals and alloys is a central subject of corrosion research. Passive oxide layers, typically 2-5 nm in thickness, protect many metals and alloys from an aggressive influence of the corrosion environment. Most of present knowledge on the chemical composition of passive films comes from ex-situ surface analytical methods. In particular AES (Auger Electron Spectroscopy) technique provide fundamental information on the chemical composition and state of components of thin surface layers, and particularly on surface atoms. AES is closely related technique which involve the energy of electrons emitted from small depth (several atomic layers) at the surface of a material. These fundamental information ensured the usefulness of AES for practical surface analysis. The composition surface layers is often quite different from that of the bulk material due to contamination, oxidation, or technological processing. This method give an insight into important problem concerning nature of passive films / oxide layer. Notable they provide an answer to the following question:

a) Which elements are present at the surface?
b) What is chemical state of these elements?
c) How much of each chemical state of each element is present?
d) What is the spatial distribution of the materials in three dimensions?
d) Is material present as a thin film at the surface?
- how thick is the film?
- how uniforms is its thickness?
- how uniform is the chemical composition of the film?

Many interesting information about structure and chemical composition of passive films / oxide layer can be obtained using combination of ion sputtering with AES method. Depth profiling is usually accomplished by inert gas (Ar+) ion bombardment to remove successive layers of materials from the surface. AES composition profiles are particularly useful since they can be acquired fast and with high depth resolution. Sputter profiling, however, damages the sputtered oxide films and can lead to composition changes by preferential sputtering and/or ion mixing.

Coffee break

Sunday morning, 3 September, 10:30

Lectures

Sunday morning, 3 September, 11:00

11:00 Oral

Thermal Properties of Thin Films and Problems with Their Determination

Jerzy Bodzenta

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Nowadays thin films are widely used in many fields. Thin films cover: cutting tools, medical implants, optical elements, integrated circuits, etc. Metal-insulator-semiconductor (MIS) layered structures form a base for operation of electronic devices. This is why knowledge of physical properties of thin films is very important. Of course different properties define usability for different applications. Thin films used for passivation should be tight, mechanically and chemically stable. Optical properties of thin films decide a quality of optical coatings. Electric properties are crucial for thin films used in electronic devices. But more often and often thermal properties become important. It is frequently connected with growing integration scale of optic and electronic devices. Thermal properties of thin films can be completely different than these properties of bulk materials. Let us consider a diamond as an example. The thermal conductivity of high-quality single crystals of diamond is about 2 200 W m-1 K-1 at room temperature. But the thermal conductivity of the best 1 mm thick diamond films is not higher than 100 W m-1 K-1. Value of this quantity for very thin diamond films is still lower. The main reason of this fact is that the heat transport in solids is very sensitive to inner structure and thin films contain defects which restrict heat transport by phonons. Additionally highly disordered interfacial layer between the film and a substrate forms a thermal barrier and lowers the effective thermal conductivity of the film. Determination of thermal parameters (the thermal conductivity or the thermal diffusivity) of thin films is a complicated problem. Submicron layers cannot be obtained as free-standing membranes and must be investigated on o substrate. The influence of the thin film on "average" thermal properties of the sample is very weak. This is why sophisticated measuring methods are used for determination of the thermal conductivity and the thermal diffusivity of thin films. Non-contact methods which do not require any preliminary sample preparation are preferable. It is experimentally shown, that even very thin layer additionally deposited on investigated sample may considerably change results of measurements.

11:30 Oral

Stress Evolution in Metallic Thin Films

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Intermittent deposition of metallic thin film.

Copper, silver and gold thin films were deposited on 100μm Si(001) substrates at room temperature in vacuum system (p= 10^{-9} hPa). Deposition rate ranged from 0.2 up to 0.5 Å/s. The process was intermittent in a periodic way – for each several nm thick sublayer (7÷18 nm) the deposition was interrupted for about 10 minutes. The total force per unit depth (F/w) in films was *in-situ* determined by the use of the substrate curvature measurement method with laser scanning technique [1]. The evolution of the general trend in F/w for each metal is similar to that observed for continuous deposition. However, a significant stress evolution during the interruption periods was observed. Characteristic changes of F/w during 10 minutes interruption periods were not observed until the layer reached thickness about 20 nm. Similar effect was also observed for silver layers, whereas for gold layers no evolution of F/w was observed for interruption periods. Evolution of the total force per unit depth (F/w) for each studied material will be presented and discussed in detail.

Thermal treatment.

Thermal treatment is one of the stress modification methods. The

stress measurements during thermal cycles provide quantitative data on mechanical stability of thin films. When very thin layers are annealed a hysteresis loop may have a difficult to explain shape. The experiments have been performed by the use of the curvature measurement optical system [1]. To interpret annealing results additional characterization by X-ray diffraction has been performed and results will be discussed.

Ion irradiation.

Ion irradiation modifies a stress in thin films. In most cases the stress relaxation effect is observed for tensile and compressive stresses, however the build up of compressive stress was also observed. Stress evolution during ion implantation of 210 keV Ar and 300 keV Kr ions into the Mo thin films deposited on Si substrate as well as silicon substrate without a film will be presented and discussed.

References.

[1] P.A.Flinn, D.S.Gardner and W.D.Nix, IEEE Trans. Electron. Dev. ED-34 (1987) 689.

12:00 Oral

Application Nanotechnology and Micro-Nanoparticles Impregnation to Produce PM Parts

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The technologies of powder metallurgy provide vast possibilities of forming powder products with desired mechanical, physical and service properties. Considering cost minimization, an important advantage of those technologies is low specific energy consumption in serial and mass production, as well as almost wasteless production process.The authors of the article present methods of production of sintered powder parts developed in the Poznań Metal Forming Institute and discuss research results. The powder forging of the precision high performance structural components are of great importance to achieve a definite material density. This study is focused on deformation PM- parts. e.g. sleeve bearing during precision cold forging. The results presented relate to sleeve bearing made by the method of forging powder parts impregnated with oil-solid lubricant mixtures. A proper strain distribution during plastic deformation of the particulate material has been achieved by the optimization of the technology parameters. The results of the impregnation examination were evaluated from the viewpoint of optimization of tribological properties of composites.

Lunch break

Sunday afternoon, 3 September, 12:30

Lectures

Sunday afternoon, 3 September, 14:00

14:00 Oral

Monte Carlo Simulation of Structural Transformations in Intermetallic Nano-Layers

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"Order-order" kinetics in $L1_0$-ordered FePt nano-layers have been studied by Monte Carlo (MC) simulation implemented with Glauber dynamics and vacancy atomic-jump mechanism. In the presented preliminary approach the effects of the substrate character and of the tetragonal distorsion of the superstructure were neglected. Atomic pair-interaction energies in two co-ordination shells were evaluated on the basis of Cluster-Expansion "ab-initio" calculations carried out for Fe-Pt binary system. Nano-layers limited by two (001)-type free surfaces were simulated by imposing two-dimensional (x-y) periodic boundary conditions upon a sample of FePt with different variants of $L1_0$ superlattice (different orientations of monoatomic planes). Selective effect of free surfaces on the stability of $L1_0$ variants was observed. While no effect was detected in the case of the variants with (100)- and (010)-type Fe and Pt planes (x- and y- $L1_0$ variants, respectively), the $L1_0$ stability was strongly reduced in the case of z-$L1_0$ variant ((001)-type monoatomic planes). As a result, the initially homogeneous FePt films ordered in z- $L1_0$ variant showed a complex "order-order" process resulting in a microstructure of x- and y-$L1_0$ variant domains. Kinetics of the process was analysed in atomistic scale.

14:30 Oral

STM/STS Investigations of Cu-phthalocyanine Molecules on $Al_2O_3/Ni_3Al(111)$

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Low temperature scanning tunneling microscopy (LT-STM) and spectroscopy (STS) have been used to investigate the growth of Cu-phthalocyanine (C32H16N8Cu) molecules on an ultrathin Al2O3 oxide film grown on the Ni3Al(111) surface [1] as a function of coverage and annealing temperature. For sub- and monolayer coverage and a deposition temperature of 140 K two different planar molecular adsorption configurations rotated by 30o with respect to each other were observed with sub molecular resolution in the STM images. For Q CuPc» 1 ML, before completion of the first layer, the growth of a second layer was already observed. The measured distance of 3.5 Å between 1st and 2nd layer corresponds to the distance between layers in the a modification of crystalline CuPc. By the use of STS' the lowest unoccupied molecular orbital (LUMO) of the adsorbed CuPc molecules has been identified at an energy of 1.2 eV. The lateral distribution of the electronic states of the CuPc has been analysed and mapped by STS. The C32H16N8Cu molecules grown at 140 are thermal stable up to 350 K against shape changes upon

prolonged annealing to temperatures up to 350 K. A template effect of the underlying oxide film on the CuPc growth, as has been observed for metals like Pd [2], has not been found.

[1] S. Degen, A. Krupski, M. Kralj, A. Langner, C. Becker, M. Sokolowski and K. Wandelt, Surf. Sci. Lett. 576 (2005) L57-L64.
[2] S. Degen, C. Becker and K. Wandelt, Faraday Discuss. 125 (2003) 343.

Coffee break
Sunday afternoon, 3 September, 15:00

Lectures
Sunday afternoon, 3 September, 15:30

15:30	Oral

Positron Depth Profiling in Solid Layers

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We shall start with a brief review of the principles of the Doppler Broadening of the positron annihilation radiation line, the technique most frequently used in defect depth profiling in solids and relevant to dc-beams. We will then focus on some specific examples of Slow Positron Implantation Spectroscopy (SPIS) investigations related to technological applications such as i) the sub-surface damage production by ion implantation in metals or ceramics, ii) the phase transition in tungsten coatings possibly induced by internal stresses, and iii) the dependence on substrate preparation of defect profiles in deposited layers. We will try to elaborate on the possibility of deriving some criteria from SPIS results in order to assess adhesion in metal/polymer or polymer/metal couples.

Posters

COST Action D30. Meeting of Working Group D30/002/03 "High Pressure Synthesis and Processing of Nanopowders"

The working group research area is as fiollows:
The production and processing of nanocrystalline powders, with size in the range 5 – 100 nm is an important field of nanotechnology. The difference in physical properties of such ultra-dispersed powders comparing to microcrystalline crystalline ones opens great opportunities in functional applications. The key issue for application of nanopowders is the engineering of crystallographic structure, chemical composition and the chemistry of their surfaces. Application of high pressures is of crucial importance in the synthesis of nanopowders during reactions taking place in fluids. Application of high pressures permits to increase the synthesis temperature comparing to atmospheric pressure reactions, thus accelerate the reaction and increase the perfection of the crystal lattice. High pressure continuous flow reactors are used for industrial production of nanopowders. It permits also to carry out reactions in supercritical conditions, where the solubility of solids in liquids can be strongly varied by changing the process parameters. In the present project we want to focus attention on hydrothermal, amonothermal and solvothermal reactions.

We are going to develop the fundaments of technologies for luminescent, piezoelectric, magnetic, and semiconducting nanopowders of oxides and nitrides, including coated powders, of precisely controlled size distribution, chemical composition and surface structure. We are also committed to link our research to industry needs both in terms of the application of nanopowders as well as of the high pressure equipment for their production.

The meeting is a satellite event of Symp. C "Doped nanopowders.....

The group is part of COST Action D30 "High Pressure Tuning of Chemical and Biochemical Transformations" . More information about COST can be found under http://www.cost.esf.org/index.php

Programme

Sunday, 3 September

Posters Cost D30 Sunday WGM

Posters at Unipress
Sunday afternoon, 3 September, 13:00
Chair: Agnieszka Opalińska

Sunday Afternoon COST D30

Short presetnations by COST Action Particpants
Sunday afternoon, 3 September, 14:00
Chair: Witold Lojkowski

Monday, 4 September

COST D30 WG Meeting Overview Lectures

Overview lectures in frame of Working Group Meeting COST Action D30
Monday afternoon, 4 September, 14:00
Chair: Agnieszka Opalińska

14:00	Oral

Novel Methods for the Fabrication of Nanomaterials and Their Applications

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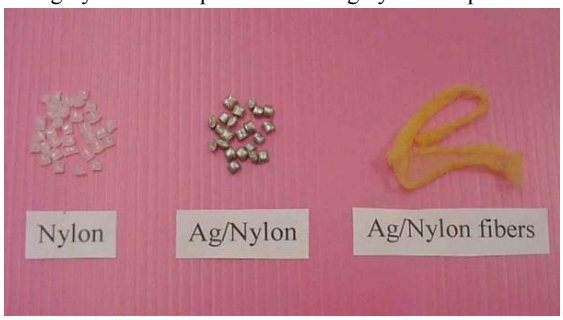
The four methods by which nanomaterials are being produced in my laboratory will be described briefly. The methods are: sonochemistry, microwave dielectric heating, sonoelectrochemistry, and RAPET (**R**eaction under **A**utogenic **P**ressureat **E**levated **T**emperatures). I will concentrate in my talk on the application of these nanomaterials for various applicative or industrial projects.

For example sonochemistry have been employed for coating nanosilver particles on Nylon 6, 6 chips. The nanocrystals of pure silver, 50-100 nm in size, are finely dispersed on the polymer surface without damaging the Nylon 6,6 structure. This Ag/nylon nanocomposite is stable to many washing cycles, and thus can be used as a master batch for the production of nylon yarn by melting and spinning processes.. The fabric knitted from this yarn has shown excellent antimicrobial properties. In Figure 1 we present three stages in the preparation of the final product.

Some other applicative examples of nanomaterials prepared by the microwave and RAPET techniques will be presented.

Photo of nylon chips before and after sonochemical coating with silver, and Ag/nylon fibers spun from the Ag/nylon composite.



14:45 Oral

Hydrothermal synthesis of doped oxide nanomaterials: a review

Robert R. Piticescu, Roxana M. Piticescu

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Hydrothermal method represents a soft chemical procedure with high potential impact in the synthesis of new nanomaterials due to its versatility, ability to control nucleation and growth mechanisms and possibility to get nanomaterials in one step with lower energy consumption and low environmental impact than other synthesis routes.

An important feature of the hydrothermal method is the high potential in producing a wide range of doped oxide nanomaterials with controlled compositional and structural homogeneity at the nano level.

The first part of the paper presents the thermodynamic methods enabling controlling the synthesis process of doped oxide nanomaterials.

In the second part some examples are discussed are discussed based on original results regarding the hydrothermal synthesis of some functional doped oxide materials based on zirconia, barium strontium titanate and zinc oxides. The advantages and problems related to the hydrothermal synthesis of such nanomaterials are finally discussed.

Acknowledgements: Romanian Ministry for Education and Research in the frame of National Programme for New Materials, Micro and Nanotechnologies-MATNANTECH, Prof. Claude Monty from CNRS-PROMES, Prof. Witold Lojkowski from UNIPRESS Warsaw, EGIDE-France and ESF-COST Programme for supporting the researches.

15:50 Oral

Structure and "Ferromagnetism" of ZnO

Jianzhong Jiang

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Structure and "Ferromagnetism" of ZnO

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Zinc oxide, ZnO, is a wide band gas semiconductor with a range of technological applications including electronic and electro-optic device, catalysts, chemical sensors, piezoelectric transducers, and conductive solar cell window layers. ZnO has the wurtzite hexagonal structure at ambient condition. Bates et al first discovered a high pressure phase with the rocksalt or NaCl cubic structure. The quenchability of the high pressure phase is a very important problem in the field of materials science. In this talk, we will address 1. How can we quench the high-pressure cubic ZnO phase to ambient condition? 2. How stable is the cubic ZnO phase? 3. What kind of properties does the cubic ZnO phase have? 4. How does the cubic-ZnO phase transfer into hexagonal ZnO phase? 5. is there ferromagnetism of Co-doped ZnO phase?

16:20 Oral

Characterization of Nanomaterials by Scattering and Spectroscopy

Markus Winterer

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Crystals are distinguished from other states of matter by the existence of long range order or periodicity. Glasses or amorphous materials lack long range order. Nevertheless, it is possible that bond distances and coordination numbers exhibit narrow distributions. The disorder in glasses is homogeneously distributed whereas in nanocrystalline solids a heterogenously distributed disorder is generated by interrupting the periodicity of crystals by interfaces. A simple model of nanocrystalline materials distinguishes between crystalline grains and a network of interfaces, i.e. grain boundaries and pore surfaces.

Scattering techniques provide structural information on ordered as well as disordered materials. Diffraction by the crystal lattice emphasizes the long range order which may obscure the diffuse scattering from disorder whereas spectroscopy by a local probe is dominated by short range order. Spectroscopic techniques and EXAFS (Extended X-ray Absorption Fine Structure) spectroscopy in particular provide complementary information on the structure of nanocrystalline materials where order and disorder coexist.

Diffraction methods such as X-ray, electron or neutron diffraction rely on the long range order of materials to extract structural information. This is easily seen by the Scherrer broadening of the diffraction peaks when the crystal size decreases, i.e., the accuracy of the structural information decreases with decreasing grain size. Scattering experiments using the same probes can also provide structural information in case of highly disordered materials.

Spectroscopic methods such as NMR (Nuclear Magnetic Resonance) or EXAFS provide structural information down to the molecular level. However, in case of large disorder the data analysis is very demanding. It is possible to investigate the local structure of nanomaterials by XAFS .

16:50 Oral

Soft solution processes: application to hybrid nanostructured materials

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Hydrothermal procedures are one of the few methods enalbing the synhtesis of new materials in the present age. We will demonstrate the influence of the processing parameters for the synthesis of new hybrid organic-inorganic nanostructured materials with controlled morphology (spheres, whiskers....) using hydrothermal reactions at low temperatures. The applications are related to regeneraive medicine and tissue engineering.

17:20 Oral

Synthesis, sintering and properties of doped nanocrystalline powders of zirconia, zinc oxide and yttrium aluminum garnet (YAG)

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The results of research carried out in the Institute of High Pressure Physics in collaboration with partners within COST action D30 and D32 is presented. The main results are highlighted:

microwave driven hydrothermal synthesis is well suited for synthesis of doped nanopowders

- doping on nanopowders is an effective tool to reach high doping levels
- it seems that hydrothermal synthesis permits to reach high doping levels
- new phenomena have been discovered as far as optical properties of the nanopowders.
- application of high pressure permitted to obtain dense transparent nanoceramics of YAG
- new methods of nanopowders characterization have been developed:

- measurements of density of nanopowders
- measurement of grain size distribution with the use of fine analysis of x-ray diffraction profiles and determination of lattice constants of nanopowders

Posters Cost D30 Sunday WGM

Posters at Unipress
Sunday afternoon, 3 September, 13:00
Chair: Agnieszka Opalińska

Posters

Sunday, 3 September

Science & Technology Commercialization Marketplace NOTUS

Programme

Posters

Modeling real materials from first principles

Point defectsSolid state diffusionInterfaces & Grain boundaries

Two days workshop on Sept. 5-6, 2006in ICM, Warsaw University, Poland(satellite event to E-MRS Fall Meeting 2006)

Programme

Posters

Index

A

B

C

D

E

F

G

H

I

J

K

L

M

N

O

P

Q

R

S

T

U

V

W

X

Y

Z