Programme and Book of Abstracts

E-MRS Fall Meeting 2005

Programme and Book of Abstracts: E-MRS Fall Meeting 2005

Published August 2005, ISBN 83-89585-07-3

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Revision: 44.4.14, 2005-08-22 12:56 GMT

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The European Materials Research Society

2005 FALL MEETING

Warsaw University of Technology
Warsaw (Poland)
5th - 9th September, 2005

Conference Organisers:

Polish Materials 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 (***)
European Materials Research Society, France (****)

THE E-MRS FALL MEETING CHAIRMEN:

K. Kurzydłowski (*)
W. Łojkowski (**)
A. Mycielski (***)
P. Siffert (****)

The European Materials Research Society

The European Materials Research Society (E-MRS) is a non profit scientific association founded in 1983 with its Headquarters in Strasbourg, France. The Society focuses its activity on creating synergy from the interactions between interdisciplinary, innovative fields of materials research. Great attention is given to spreading and exchanging information and to 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 about the scientific and technological developments in their fields of interest,taking advantage of the society's links with other Materials Research Societies belonging to the International Union (IUMRS).The 2005 Fall Meeting will include 9 symposia. The conference will also include an exhibition of products and services of interest to participants as well as training activities for young researchers or scientists wishing to extend their expertise to new fields.

E-MRS General Secretary P. Siffert

Address: **E-MRS BP20**
67037 Strasbourg Cedex 2, France

Telephone: **+33 3 88 10 65 43**
+33 3 88 10 62 55

Fax: **+33 3 88 10 62 93**

e-mail: **emrs@phase.c-strasbourg.fr**

E-MRS European Co-ordination Group:

- H. Grimmeiss E-MRS President
- P. Siffert E-MRS General Secretary
- A. Slaoui E-MRS Vice-President

Welcome to Warsaw!

We are pleased to welcome delegates to the 2005 Fall Meeting of the European Materials Research Society, E-MRS, being held at Warsaw University of Technology for the fourth time, in addition to the Annual Spring Meeting, held each May/June in Strasbourg, France. The organizers have followed the scheme developed over the years by the Society to host a wide ranging multi-disciplinary conference to bring established scientists with a world reputation together to share experiences and state of the art knowledge. A very important philosophy of E-MRS is to facilitate scientific and social contacts between established researchers, young scientists and doctoral students. The 2005 Fall Meeting will endeavour to continue these themes and further enhance the contacts between scientists from Western and Eastern Europe, North America and Asia.

The 2005 Fall Meeting comprises nine parallel symposia and 3 Plenary Sessions which are being held in the morning on the first three days of the Conference 5th, 6th and 7th September.

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.

The Development of Warsaw University of Technology

At the end of the 19th century, Poland was partitioned by three invader powers: Russia, Prussia, and the Austro-Hungarian Empire. Warsaw was then the capital of the Kingdom of Poland which was a part of the Russia Empire. At that time, efforts were made by Polish scientists to establish a technical university in Warsaw. In 1897 a special committee drafted a memorandum to the General Governor, Prince Alexander Imeretyński, advocating the need for an institution of

higher technical education in the Kingdom of Poland. In January 1898, the Governor endorsed the initiative in his own memorandum submitted to Tzar Nicholas II. During the Tzar's visit to Warsaw, he was presented with one million gold roubles donated by members of Polish society, with the suggestion that the money should be used to found a technical university in Warsaw. Tzar Nicholas II expressed official approval for the establishment of a technical university provided that the additional 2.5 million roubles needed to build and furnish it, would be contributed by Polish society. The royal decree sanctioning the establishment of the Tzar Nicholas II Warsaw Polytechnical Institute was published in June 1898. The ceremony to lay the foundation stone of the Institute's Main Building took place in August 1898. Thanks to the rapid rate of the construction work, classes began in the new building in the autumn of 1901 although the Physics and Electrical Engineering Building, Mechanics Building, and the Chemistry Building were still under construction.

During the First World War, when the Russian army was in retreat, most of the movable property of the Polytechnical Institute - libraries, experimental and laboratory equipment, etc.- was taken to Russia. In the interwar period, a few thousand engineers graduated from the Polytechnical Institute, now renamed as the Warsaw University of Technology. During the Second World War, the University of Technology buildings were largely destroyed, especially during the Warsaw Uprising of August 1944. The University Buildings were reconstructed and modernized to meet the then current needs in the immediate post-war period, 1945-48.

Organisers

Local Organising Committee:

- J. R. Blizzard (4)
- Ł. Ciupiński (1)
- H. Garbacz (1)
- M. Lewandowska (1)
- R. Pielaszek (3)
- M. Ryszkowska (1)
- A. Szadkowski (2)
- M. Chmielecka (3)

(1) Warsaw University of Technology
(2) Institute of Physics, Polish Academy of Sciences
(3) Institute of High Pressure Physics, Polish Academy of Sciences
(4) European Materials Research Society, E-MRS

Conference Secretariat:

Faculty of Materials Science and Engineering
Warsaw University of Technology
Wołoska 141, 02-507 Warsaw, Poland
Phone/Fax: +48 22 660 87 94
e-mail: warsaw.fall@e-mrs.org

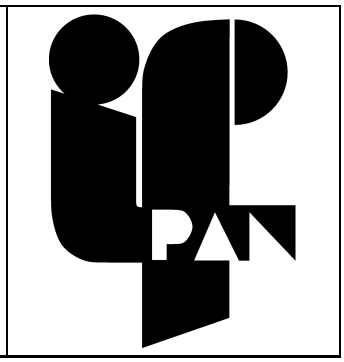
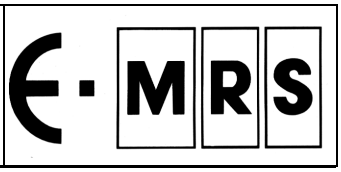
Hotel accomodation & tourist programme:

Dorota Korpaczewska
"CONGRESS-OR" Congress & Tourism Bureau
47 Żurawia Street Apt. 304
Phone/Fax: +48 22 621 31 16; +48 22 628 45 48
e-mail: d.korpaczewska@congressor.com.pl

Acknowledgements

The E-MRS FALL MEETING 2005 is supported by:

Polish Ministry of Science and Information Technology	kbn STATE COMMITTEE FOR SCIENTIFIC RESEARCH
The Polish Materials Society (PMS)	PTM POLSKIE TOWARZYSTWO MATERIAŁOZNAWCZE
The Polish Materials Science Society	PMS SOCIETY
Faculty of Materials Science, Warsaw University of Technology	

Institute of Physics, Polish Academy of Sciences	
Institute of High Pressure Physics, Polish Academy of Sciences **Network Nano and Micro-technology NAMIC**	
Center of Excellence PRENABIO, The European Commission Contract: No. G1MA-CT-2002-04055 Polish Ministry for Science and Information Technology	
EUROPEAN MATERIALS RESEARCH SOCIETY	

The Polish Materials Society

The Polish Materials Society (PMS) was founded in 1995 with the aim of promoting and propagating materials sciences and engineering. Currently the PMS unites 160 specialists in materials engineering employed in various centres in Poland and Europe. The Society has two Sections: one established in Kraków and the other in Gdańsk. Under the patronage of the PMS President, an annual competition is organised for the best doctoral thesis in the field of materials engineering. The Society is a co-publisher of the scientific journal entitled Inżynieria Materiałowa (Materials Engineering) which publishes papers (subject to a referee's evaluation) in both Polish and English. The journal is devoted to materials problems, and is particularly concerned with metallic materials, various forms of plastics, ceramics and composites related to applications. The journal also promotes modern developments in surface engineering. During recent years, the PMS has been the co-organiser of numerous conferences.

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. As part of the E-MRS 2005 Fall Meeting the Polish Materials Science Society is organising a round table discussion on Future Visions on Europe's University - Based Research. The discussion will be held on Thursday 8th of September from 9.00 to 10.30 and will include participation by eminent representatives from the Polish Universities. The PMSS will also establish a prize for the two best posters presentations at the E-MRS Fall Meeting made by Ph.D. students. In the near future the Society will take on the responsibility for planning and organising the 2006 and subsequent E-MRS Fall Meetings.

Programme

The E-MRS 2005 Fall Meeting will consist of the three Plenary Sessions, and 9 separate scientific symposia which will run concurrently.The Opening Ceremony will take place on Monday 5th September at 10:00 am. To facilitate the exchange of ideas and information, especially between the experienced scientists, and between the researchers from different areas of interest - Poster sessions, common for all the symposia, will be organized on Monday, September 5, 15:50-17:20 and Wednesday, September 7, 15:50-17:20.

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

PLENARY SESSIONS:

1. Monday, September 5, 10.30 - 11.15, 11:45 - 12:30
2. Tuesday, September 6, 11:00 - 11.45
3. Wednesday, September 7, 11:00 - 12.30

POSTER SESSIONS:

1. Monday, September 5, 15:50 - 17:20
2. Wednesday, September 7, 15:50 - 17:20

SYMPOSIA

	ROOM	TOPIC
A	134	INDIUM NITRIDE AND INDIUM RICH RELATED ALLOYS: CHALLENGES AND OPPORTUNITIES
B	219	MULTI-COMPONENT ALLOYS AND INTERMETALLIC COMPOUNDS FOR MAGNETIC APPLICATIONS AND NANOTECHNOLOGY
C	315	SHAPE MEMORY MATERIALS FOR SMART SYSTEMS
D	219	MAGNETOELECTRONICS
E	213	HYBRID POLYMER SYSTEMS OF INDUSTRIAL IMPORTANCE
F	231	INTERFACIAL PROCESSES AND PROPERTIES OF ADVANCED MATERIALS
G	226	ADHESION IN BUILDING BONDS - MACRO-, MICRO- AND NANOSCALE
H	144	FILLER METALS, SOLDERS AND JOINTS OF THE NEXT GENERATION
I	Small Hall (Mała Aula)	6TH HIGH PRESSURE SCHOOL: HIGH PRESSURE TECHNOLOGY OF NANOMATERIALS

Satellite events coordinated with the E-MRS Fall Meeting

- 1st International Laser Ceramic SymposiumWorkshop, 5th and 6th September, Room 206
- Future Visions on Europe's University - Round table discussion
- Workshop on Nanostructured Photonic Sensors
- Centre of Excellence NanoCentre workshop and microsymposium
- Modern Methods of Materials Science Modelling Workshop
- Nanomechanical Characterization Workshop

Timetable of Events

Sunday, 4th September	
19:00 - 21:00	WELCOME RECEPTION, Main Hall

* * *

Monday, 5th September	
10:00 - 10:30	OPENING CEREMONY Small Hall (Mała Aula)
10:30 - 11:15	PLENARY SESSION 1 Small Hall (Mała Aula) **Kazimierz Rzążewski** Polish Academy of Sciences, Center for Theoretical Physics, Warsaw, Poland *Physics of Quantum Gases*
11:15 - 11:45	Coffee Break Main Hall (Duża Aula)
11:45 - 12:30	PLENARY SESSION1 (Continued) Small Hall (Mała Aula) **Völker Haerle** OSRAM Opto Semiconductors GmbH, Regensburg, Germany *Optoelectronic Devices based on Nitride Semiconductors*
12:30 - 14:00	Lunch break
14:00 - 15.30	INDIVIDUAL SYMPOSIA See plan of WUT for the location of each symposium
15:30 - 15:50	Coffee Break Main Hall (Duża Aula)
15:50 - 17:20	MONDAY POSTER SESSION (Main Hall)

* * *

Tuesday, 6th September	
9:00 - 10:30	INDIVIDUAL SYMPOSIA
10:30 - 11:00	Coffee break
11:00 - 11:45	PLENARY SESSION 2 Small Hall (Mała Aula) **Philippe Dubois** Laboratory of Polymeric and Composites Materials (LPCM), University of Mons-Hainaut, Mons, Belgium *Performant clay/carbon nanotube polymer nanocomposited*
11:45 - 12:30	Presentation of diplomas and prizes for Symposia Chairperson and best Ph.D. posters Main Hall (Duża Aula)
12:30 - 14:00	Lunch break
14:00 - 15:30	INDIVIDUAL SYMPOSIA
15:30 - 15:50	Coffee break
15:50 - 17:20	INDIVIDUAL SYMPOSIA
20:00	Conference Dinner (InterContinental Hotel)

* * *

Wednesday, 7th September	
9:00 - 10:30	INDIVIDUAL SYMPOSIA
10:30 - 11:00	Coffee break
11:00 - 11:45	PLENARY SESSION 3 Small Hall (Mala Aula) **Helmut Dosch** Max-Planck-Institut fur Metallforschung, Stuttgart, Germany *Advanced Analysis in Materials Science: From Fundamental Research to New Technologies*
11:45 - 12.30	PLENARY SESSION 3 (Continued) Small Hall (Mała Aula) **Jaroslav Fabian** Institute of Theoretical Physics, University Regensburg, Germany *Spintronics: concept and challenges*
12:30 - 14:00	Lunch break
14:00 - 15:30	INDIVIDUAL SYMPOSIA
15:30 - 15:50	Coffee break
15:50 - 17:20	POSTER SESSION (Main Hall)

* * *

Thursday, 8th September	
9:00 - 10:30	INDIVIDUAL SYMPOSIA

Thursday, 8th September	
9:00 - 11.30	Round Table Discussion: **Future Visions on Europe's University** Chair: Marcel Van de Voorde Participation on invitations only.
10:30 - 11:00	Coffee break
11:00 - 12:30	INDIVIDUAL SYMPOSIA
12:30 - 14:00	Lunch break
14:00 - 15:30	INDIVIDUAL SYMPOSIA
15:30 - 15:50	Coffee break
15:50 - 17:20	INDIVIDUAL SYMPOSIA

* * *

Friday, 9th of September	
9:00 - 10:30	INDIVIDUAL SYMPOSIA
10:30 - 11:00	Coffee break
11:00 - 12:30	INDIVIDUAL SYMPOSIA
12:30 - 14:00	Lunch break
14:00 - 15:30	INDIVIDUAL SYMPOSIA
15:30 - 15:50	Coffee break
15:50 - 17:20	INDIVIDUAL SYMPOSIA

* * *

Saturday, 10th of September	
10:00 -- 13:30	SYMPOSIUM I **Attention: not in WUT main building** but in the Building of the Faculty of Materials Science, WUT, Woloska 141.

Exhibitors

HYSITRON INCORPORATED

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Part I. Symposia

Plenary session

Programme

Monday, 5 September

Plenary Session
Monday morning, 5 September, 10:30
Main Building, Small Hall (Mała aula)
Paul Siffert presides

10:30 invited oral

Physics of Quantum Gases

Kazimierz Rzążewski

Polish Academy of Sciences, Center for Theoretical Physics (CFT PAN), al. Lotników 32/46, Warszawa 02-668, Poland

Contact: kazik@theta1.cft.edupl

This year has been declared the World Year of Physics. We are commemorating seminal papers written by Albert Einstein in 1905. Nearly 20 years later he strikes again. He discovers (following preliminary results by Indian physicist Satyendranath Bose) a new type of the phase transition, the one entirely due to the indistinguishability of atomic particles. This phase transition, known today as the Bose-Einstein condensation, according to the Organizing Committee of this Conference deserves to be a subject of yet another lecture celebrating the Year of Physics.

A weakly interacting, nearly ideal gas composed of bosons exhibits quantum features when cooled to nanokelvin temperatures. At a critical temperature it spontaneously collapses to the lowest energy state of the potential well holding the gas. Amazing advances in the techniques of cooling and trapping of neutral atoms made it possible to achieve this phase transition in 1995. The Nobel Prize for Eric Cornell, Karl Wieman and Wolfgang Ketterle quickly followed in 2001. Today dozens of laboratories all over the world perform experiments with quantum degenerate dilute gases and the whole area is a prime example of the emerging "quantum engineering".

Remaining at the level of presentation of a popular lecture I will explain the concept of Bose-Einstein phase transition. I will tell how to cool the system to such low temperatures, first using lasers and then using a method that resembles cooling a morning coffee in your mug. Finally, I will also review some recent experiments with the condensate.

Plenary Session
Monday morning, 5 September, 11:45
Main Building, Small Hall (Mała aula)
Pierre Ruterana presides

11:45 invited oral

Optoelectronic devices based on nitride semiconductors

Volker Haerle

OSRAM Opto Semiconductors GmbH, Wernerwerkstrasse 2, Regensburg D-93049, Germany

Contact: volker.haerle@osram-os.com

Tuesday, 6 September

Plenary Session
Tuesday morning, 6 September, 11:00
Main Building, Small Hall (Mała aula)
Peter Glasow presides

11:00 invited oral

Performant clay/carbon nanotube polymer nanocomposites

Philippe Dubois

Laboratory of Polymeric and Composites Materials (LPCM) University of Mons-Hainaut, Place du Parc 20, Mons 7000, Belgium

Contact: philippe.dubois@umh.ac.be

Homogeneously dispersing rigid particles in polymer matrices is a very common and widely used method that allows for readily increasing the stiffness of the so-obtained composite materials. Depending on the intrinsic nature of the added filler, other properties can also be enhanced such as fire resistance, electrical and thermal properties. However, such improvements of composite materials performances usually require high filling levels, detrimental to the ultimate mechanical properties of the resulting materials.

Polymer nanocomposites represent a new class of composite materials, i.e., particle-filled polymers for which at least one dimension of the dispersed particles is in the nanometer range. This variety of nanofillers provides the related composite materials with significantly improved properties most often at filler content as tiny as 3 to 5 wt%. Interestingly, remarkable thermo-mechanical performances have been recorded using organo-modified layered silicates (organoclays) as nanofiller precursors. Actually, upon incorporation of such organoclays in a polymer matrix, intercalation of the polymer chain in-between the clay platelets or complete exfoliation of the silicate platelets can be observed. Exfoliated nanocomposites usually exhibit significantly higher improvement in properties such as mechanical, thermal and flame retardant properties. However in many polymeric matrices, reaching such large extent of organoclay exfoliation and dispersion still remains a difficulty.

More recently discovered carbon nanotubes constitute another family of potential nanofillers for polymer matrices. Carbon nanotubes are mainly subdivided in two families: single-walled nanotubes (SWNTs) or multi–walled nanotubes (MWNTs) where several nanotubes of decreasing diameter are interlocked. Carbon nanotubes find applications in various fields such as in field emission devices, electrically and thermally conductive materials, hydrogen storage devices and molecular sieves. Even though carbon nanotubes have already been blended within various polymers, homogeneous dispersion of the nanotubes in the polymer matrix remains one major challenge, since bundles of aggregated SWNTs or MWNTs most often persist and therefore limit the performances of the recovered composite materials.

This contribution aims at reporting on very recent developments in syntheses, properties and (future) applications of polymer-based nanocomposites filled with either organoclays and/or carbon nanotubes. Undoubtedly, the key-challenge remains to reach a high level of nanoparticle dissociation (i.e., either to delaminate the silicate nanoplatelets or to break down the bundles of aggregated carbon nanotubes) and their fine dispersion upon melt blending within the selected polymer matrix. In that context, the in situ polymerization/grafting process as catalyzed directly from the nanofiller (organoclay or carbon nanotube) surface proved highly efficient allowing for the complete destructuration of the native filler aggregates. Dissociated nanoparticles were accordingly recovered, their surface was homogeneously coated/grafted by the in situ grown polymer chains as generated by this so-called "Polymerization-filling technique" (PFT). Interestingly enough, such surface-coated organoclays and/or carbon nanotubes were further added as "masterbatch" in commercial polymeric matrices by twin-screw extrusion. As a result of the pre-destructuration of the nanofillers by PFT, it comes out that the resulting polymer nanocomposites displayed much higher thermo-mechanical properties even with a nanofiller loading as tiny as 1wt%.

Wednesday, 7 September

Plenary Session

Wednesday morning, 7 September, 11:00
Main Building, Small Hall (Mała aula)
Krzysztof J. Kurzydlowski presides

11:00 invited oral

GENNESYS Grand European Initiative on Nanoscience and Nanotechnologies using Neutrons and Synchrotron Radiation Facilities

Helmut Dosch

Max-Planck Institut fuer Metallforschung (MPI-MF), Heisenbergstr. 3, Stuttgart D-70569, Germany

Contact: Claudia.Sussdorff@mf.mpg.de

Little happens in industrialised countries without the use of high-tech materials which are the building blocks of all modern technologies ranging from information, communication, health, energy to environment and transport. Through our microscopic insight into the atomistic structure of condensed matter, the development of novel materials has progressed at a breathtaking pace during the last decades.

Breakthroughs in the future development of advanced materials and novel technologies are facing key barriers in the destruction-free in-situ analysis of nanomaterials and nanomaterial systems under industrially and environmentally relevant conditions. These barriers pose critical challenges onto the European Research Infrastructure for the fine analysis of matter, i.e. the modern European Synchrotron Radiation and neutron facilities which have developed an impressive analytical potential and which are ready to offer and adjust their analytical technology to the advancement of nanoscience and nanotechnology.

The GENNESYS initiative, launched in 2003, is catalyzing this process in Europe.

Plenary Session

Wednesday morning, 7 September, 11:45
Main Building, Small Hall (Mała aula)
Józef Barnaś presides

11:45 invited oral

Spintronics: concept and challenges

Jaroslav Fabian

Institute of Theoretical Physics University Regensburg, Regensburg 93040, Germany

Contact: jaroslav.fabian@physik.uni-regensburg.de

Spintronics [1] is a rapidly developing field which promises novel electronic devices that combine charge transport and spin memory, to bring new functionalities to conventional semiconductor electronics. The goals are manipulation of charge by spin, to control the current flow by magnetic means, as well as manipulation of spin by electronic transport, to modify the information stored in spins by electric means. I will review basic concepts of spintronics and the progress made in satisfying its three requirements: spin injec-

tion, spin transport, and spin detection. I will also review recent progress in fabricating materials and materials structures (novel magnetic semiconductors) that can be employed in active regions of electronic devices to achieve the stated spintronics goals. Several proposals for spintronics devices will be critically presented, among them the Datta-Das spin field effect transistor, hot-electron spin transistors, as well as bipolar spintronic devices (the magnetic bipolar transistor). Challenges to the field as well as novel trends, such as nanospintronics, in which individual electron spins are manipulated for quantum information processing, will be discussed.

[1] I. Zutic, J. Fabian, and S. Das Sarma, Spintronics: fundamentals and applications, Rev. Mod. Phys. 76, 323 (2004).

Symposium A

Indium nitride and indium rich related alloys: challenges and opportunities

Welcome

Nitride semiconductors have revolutionized many areas of technology and increasingly in everyday life. To mention a few, blue/green LEDs; white LEDs, replacing incadescent bulbs; violet laser diodes used in high density data storage; UV emitters for detection of bio-materials; transistors for high-power, high-frequency electronics.

From the A1N-GaN-InN family, the latter binary compound is still a mysterous material, as many of its properties have not yet been adequately measured. InN bulk crystals do not exist and epi-layer are still of relatively poor quality. However, InN attracts very large interest because of a vivid debate on its band gap, and still unexplored properties of the ternary and quaternary compounds of (AlGaIn)N.

The aim of this Symposium is to present and discuss the recent advances in research on InN and Inrich related alloys. The topics will include:

- growth
- crystallographic properties
- optical and electronic properties
- electronic and optoelectronic devices

Scientific Committee:

O. Ambacher (TU. Ulmenau, Germany), V. Yu. Davydov (IOFFE, Russia), P. Gibart (CRHEA, France), B. Gil (U. Montpellier, France), M. Heuken (Aixtron, Germany), T. Inushima (Japan), Mike Leszczynski(Unipress, Poland), B. Monemar (U. Linköping, Sweden), K. P. O'Donnell (U. Strathclyde,UK), P. Perlin (Unipress, Poland), J. Redwing (Pennstate,USA), P. Ruterana (SIFCOM, France), T. Steiner (AFOSR, USA), M. Stutzmann (TU. Munich, Germany), E. Weber (UC Berkeley, USA), C. Wood (ONR,USA), C. Wetzel (RPI, USA)

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- **Dr. P. Ruterana**, SIFCOM-ENSICAEN,Caen, France
- **Prof. M. Stutzmann**, W. Schottky Institute TU Munich, Garching, Germany
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- **Prof. Mike Leszczynski**, Institute of High Pressure Physics ,UNIPRESS and TopGaN Ltd, Warsaw, Poland

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Programme

Monday, 5 September

Heterostructures: M. Leszczynski/R. Ruterana
Monday afternoon, 5 September, 13:30
Main Building, room 134

14:00 invited oral

Epitaxy Control and Characterization of InN, InN-based Ternary Alloys, and Their MQW-structures

Akihiko Yoshikawa, Song-Bek Che, Yoshihiro Ishitani, Xinqiang Wang, Wataru Terashima

Chiba University, Department of Electronics and Mechanical Engineering, 1-33 Yayoi-cho, Inage-ku, Chiba 263-8522, Japan Chiba University, Center for Frontier Electronics and Photonics, 1-33 Yayoi-cho, Inage-ku, Chiba 263-8522, Japan Chiba University, InN-Project as a CREST-program of JST, 1-33 Yayoi-cho, Inage-ku, Chiba 263-8522, Japan

Contact: yoshi@faculty.chiba-ujp

We have pointed out for the epitaxy of InN and InN-based ternary alloys that N-polarity growth regime is preferable than In-polarity one in the viewpoint of getting higher-quality epilayers as well as constructing fine structure InN-based heterojunctions. This situation is very much different from that of other typical III-nitrides of GaN, AlN and Ga(Al)-rich ternary/quaternary alloys including InGaN, where Ga- or Al-polarity is better than N-polarity. The reason for the different situation in epitaxy of InN-based III-nitrides is attributed to that of about 100 deg higher possible-epitaxy temperatures in N-polarity than In-polarity (~600 deg for N-polarity but ~500 deg for In polarity). The higher epitaxy temperatures result in both (a) much easier epitaxy control under around unity surface stoichiometry to avoid the appearance of In-droplets on the surface during growth and (b)better matching in epitaxy temperatures between InN-well and GaN and/or AlN-based barriers when fabricating InN-based nano-heterostructures.

In this paper, we report our recent results on the epitaxy of N-polarity InN-based ternary alloys of InGaN and AlInN both on GaN and InN underlayers. Further, we have investigated to fabricate InN-based nano-heterostructures including InN/

GaN, InN/InGaN, and InN/AlInN MQWs under the N-polarity growth regime.

The InN-based epilayers and MQWs were characterized by HR-XRD, AFM, TEM, Hall and PL measurements. We have succeeded for the first time in fabricating very fine InN-based nano-heterostructures which were revealed by the clear satellite diffraction peaks and sharp hetero-interfaces by XRD and TEM, respectively.

Finally we conclude that the N-polarity growth regime is very important in the epitaxy of InN itself, InN-based ternary alloys and fine MQWs structures.

14:30 invited oral

Determination of concentration, strain and internal electric fields in InN and InGaN quantum well and quantum dot structures

David J. Smith[1], Martha R. McCartney[2], Ted D. Moustakas[3], Papo Chen[4], Tao Xu[5], Lin Zhou[6], Masaki Takeguchi[7]

1. *Arizona State University, Department of Physics and Astronomy, Tempe 85287-1504, United States* **2.** *Arizona State University, Department of Physics and Astronomy, Tempe 85287-1504, United States* **3.** *Boston University, Department of Electrical and Computer Engineering, Boston MA, 02215, United States* **4.** *Boston University, Department of Electrical and Computer Engineering, Boston, United States* **5.** *Boston University, Department of Electrical and Computer Engineering, Boston, United States* **6.** *Arizona State University, Department of Physics and Astronomy, Tempe 85287-1504, United States* **7.** *National Institute of Material Science (NIMS), Tsukuba, Japan*

Contact: david.smith@asu.edu

The combination of high-resolution electron microscopy (strain field), Z-contrast imaging with the scanning transmission electron microscope (In concentration), convergent-beam electron diffraction (local lattice parameter), and electron holography (internal electric field), represents a powerful approach for studying the effects of compositional differences and local fluctuations in InGaN-related materials. All of these techniques have been used separately or in tandem in our recent studies to investigate phase separation and the effects of local segregation in InGaN quantum dots and quantum well structures. Note that tilting away from exact zone-axes orientations is required for both CBED and EH in order to avoid complications arising from strong elastic and inelastic scattering, but lateral composition changes on the nm-scale can still be measured with +0.5% accuracy using the CBED technique. In compositional fluctuations causing local electric field inhomogeneities seemed to be more pronounced near the onset of InGaN layer growth, suggesting strain relaxation as a strong contributing factor.

15:00 invited oral

Ultra-thin In-rich InGaN/GaN multiple quantum wells grown by metalorganic chemical vapor deposition

Euijoon Yoon[1], Soon-Yong Kwon[1], Hee Jin Kim[1], Seong-Il Baik[1], Young-Woon Kim[1], Jung-Won Yoon[2], Do-Young Park[2], Hyeonsik Cheong[2], Dai-Sik Kim[1], Yoon-Soo Park[1], Yudong Jang[3], Ki-Ju Yee[3], Donghan Lee[3], Fabian Rol[4], Le Si Dang[5]

1. *Seoul National University (SNU), School of Mat. Sci. Eng., Seoul 151742, Korea, South* **2.** *Sogang University, Seoul 121742, Korea, South* **3.** *Chungnam National University, Daejon 305764, Korea, South* **4.** *University J.Fourier-Grenoble-I, Lab. Spectrometrie Physique (CEA-CNRS), 17 avenue des Martyrs, Grenoble 38054, France* **5.** *Laboratoire de Spectrométrie Physique - CNRS UMR 5588 (SPECTRO), Université J. Fourier, BP87, Saint Martin d'Hères 38402, France*

Contact: eyoon@snu.ac.kr

We successfully grew In-rich InGaN/GaN multi-quantum well (MQW) structures using growth interruption (GI) by metal-organic chemical vapor deposition (MOCVD). The quality of overgrown InGaN/GaN QW layer in MQWs was largely affected by the crystalline quality and interfacial abruptness of underlying QW layer. Introduction of 10 sec GI was very effective in improving the crystalline quality and interfacial abruptness of InGaN QW layer, and we successfully grew 10 periods of In-rich InGaN/GaN MQW with 10 sec GI and obtained very strong near-ultraviolet (UV) emission (~390 nm) at room temperature. We believe that use of ultra-thin In-rich InGaN QW layer can be a new candidate for near-UV source, which might replace the conventional low-indium content (<10%), thicker InGaN QW layer.[1] Time-resolved PL measurement of the ultra-thin In-rich InGaN/GaN MQWs showed that the radiative lifetime was 1.75 ns and that the peak position as well as the lifetime did not change with changes in pump power, suggesting that there is negligible piezoelectric effect and it is highly efficient in capturing carriers.

By adoting a two-step growth method during the growth of InGaN quantum well (QW) layer, we obtained strong near-UV (~400 nm) as well as blue (~450 nm) emissions at room temperature from 1-nm-thick In-rich InGaN/GaN MQW structures. Temperature-dependent photoluminescence and high-resolution transmission electron microscopy study show that the 400 nm peak is attributed to band-to-band transition in 1-nm-thick InGaN QW, whereas the 450 nm peak is attributed to localized centers induced by the second-step InN growth and growth interruption. The thermal stability of the 450 nm peak is much better than that of the 400 nm peak. Detailed optical properties of the In-rich InGaN/GaN MQWs

will be reported.

[1] S.-Y. Kwon, S.-I. Baik, Y.-W. Kim, H. J. Kim, D.-S. Ko, E. Yoon, J.-W. Yoon, H. Cheong, and Y.-S. Park, Appl. Phys. Lett. 86, 192105 (2005).

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster A-1

Anomalous Temperature Dependence of Photoluminescence of Self-assembled InGaAsN Quantum Dots

Zhifeng Wei[1], S.J. Xu[1], W. J. Fan[2], S. F. Yoon[2]

1. *The University of Hong Kong, Hong Kong, Hong Kong* **2.** *Nanyang Technological University, Singapore, Singapore*

Contact: zfwei@hkusua.hku.hk

Dilute nitride alloy III-V semiconductors are being paid intense attention, due to the novel physics induced by the nitrogen incorporation in small percentages. Following the development of quaternary InGaAsN quantum wells, lower-dimensional InGaAsN quantum dots (QDs) have attracted an increasing interest and been regarded as promising structures for extending the emission wavelength to 1.55μm and beyond. In this report, we present variable-temperature photoluminescence (PL) spectra of self-assembled InGaAsN/GaAs QDs grown on GaAs substrate with molecular beam epitaxy. Two kinds of $In_{0.3}Ga_{0.7}AsN_{0.005}$ and $In_{0.5}Ga_{0.5}AsN_{0.006}$ QDs have been studied. Anomalous temperature dependence of their PL spectra was found. A newly developed luminescence model for localized states was employed to quantitatively interpret the PL results. The carrier re-distribution within different QDs is found to be responsible for the observed anomalies.

15:50 poster A-2

InN explained within chemical trends

Louis F. J. Piper[1], Tim D. Veal[1], Paul H. Jefferson[1], Chris F. McConville[1], William J. Schaff[2]

1. *University of Warwick, Gibbet Hill Road, Coventry CV4 7AL, United Kingdom* **2.** *Cornell University, 425 Philips Hall, Ithaca, NY 14853, United States*

Contact: L.F.J.Piper@warwick.ac.uk

A few experimentally observed properties of wurtzite InN initially appear to be unusual and controversial. However, InN merely represents a material lying at the extreme of the band-edge endpoints and thus can be explained within existing chemical trends. Both the In and N act to pull the conduction band minimum (CBM) and valence band maximum down with respect to the universal Branch-point energy (E_B), which lies close to the `average' mid-gap position of the semiconductor [1]. In fact the CBM of InN lies ~ 0.9 eV below E_B, resulting in electron accumulation [2], unlike for almost all III-V semiconductors where E_B lies within the Γ-point band gap, except for InAs which also accumulates.

Since the bulk Fermi level of InN generally lies far below E_B, even for degenerate n-type material, donor-like defects are energetically favourable, as described by the amphoteric defect model [3]. Here, it is shown that even low-energy N ion bombardment and annealing can result in the formation of amphoteric defects that stabilise the Fermi level at E_B. In contrast, at surfaces prepared by atomic hydrogen cleaning, the Fermi level is pinned slightly below E_B, allowing the electron accumulation to be facilitated by unoccupied donor-like surface states [4].

Finally, the effect of the electron accumulation on the opto-electronic properties of InN is considered. The electron accumulation results in an overestimation of the bulk electron density. By correcting this overestimation, the Moss-Burstein shift can be described using a band gap of 0.64 eV and a corresponding band-edge effective mass of 0.045 m_0 based on chemical trends [5].

[1] J. Tersoff, Phys. Rev. B, 32, 6968 (1985).

[2] I. Mahboob et al., Phys. Rev. Lett. 92, 036804 (2004).

[3] W. Walukiewicz, Appl. Phys. Lett. 54, 2094 (1989).

[4] L. F. J. Piper et al., submitted to ICNS-6 Conference Proceedings (2005).

[5] L. F. J. Piper et al., Phys. Rev. B, submitted (2005).

15:50 poster A-3

Conduction band anisotropy of InN and GaN studied by synchrotron ellipsometry

Munise Rakel[1,2], Christoph Cobet[1,2], Ruediger Goldhahn[3], Friedhelm Bechstedt[4], William J. Schaff[5], Wolfgang Richter[1], Norbert Esser[2]

1. *Berlin University of Technology, Hardenbergstr. 36, Berlin 10623, Germany* **2.** *Institute for Analytical Sciences (ISAS), Albert-Einstein-Str. 9, Berlin 12489, Germany* **3.** *Technical University Ilmenau, Center of Micro- and Nanotechnologies (ZMN), Gustav-Kirchhoff-Str. 7, Ilmenau 98693, Germany* **4.** *Friedrich-Schiller-Universitaet (IFTO), Max-Wien-Platz 1, Jena 07743, Germany* **5.** *Cornell University, Electrical and Computer Engineering, Ithaca, NY 14853-540, United States*

Contact: rakel@physik.tu-berlin.de

The complex dielectric function (DF) of wurtzite InN and GaN in a spectral region between 12 and 30eV was determined by spectroscopic ellipsometry with synchrotron radiation. Using a-plane (11-20) InN and M-plane (1-100) GaN, measurements were performed with the electric field vector parallel and perpendicular to the c-axis of the crystal. The dir-

ect determination of the DF with ellipsometry provides full access on the linear optical response and gives valuable information about the electronic structure since the imaginary part is associated with the joint density of states. Comparing to published EELS and XAS measurements ellipsometry yields more detailed information and has a better accuracy.

Above the plasmon frequency the DF is dominated by the In4d/Ga3d core level transitions to the unoccupied states. The main contribution for those transitions are described by the nondispersive (well localized) part of the cation d-states, thus the imaginary part gives a characteristic view of the conduction band states with p-symmetry. The comparison to calculated DOS by DFT-LDA of the conduction band agrees well in line shape and energetic positions. A considerable polarisation dependence of the DF, indicating the anisotropy of the conduction band, was observed. This anisotropy is induced by a direction dependent mixing of the empty p-states and can be discussed in terms of symmetry consideration in hexagonal crystals. Finally, the detected spin-orbit splitting of the In4d- and the Ga3d-band was determined to be 0.82eV and 0.41eV.

15:50 poster A-4

Full-potential study of d-electrons effects on the electronic structure of wurtzite and zinc-blende InN

F. Litimein[1], Bachir Bouhafs[2,4], Pierre Ruterana[3]

1. *Modelling and Simulation in Materials Science Laboratory (MSMSL), University of Sidi Bel-Abbes, Sidi Bel-Abbes 22000, Algeria* **2.** *Abdus-Salam International Center for Theoretical Physics (ICTP), strada costiera, 11, Trieste 34014, Italy* **3.** *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **4.** *Modelling and Simulation in Materials Science Laboratory (MSMSL), University of Sidi Bel-Abbes, Sidi Bel-Abbes 22000, Algeria*

Contact: bbouhafs@ictp.trieste.it

In this paper, we focus on the role played by the relativistic and d-electron effects on the electronic structure of both wurtzite (α-InN), as well as, the zincblende (β-InN) modifications of indium nitride. Here we perform all-electron full-potential linearized-augmented plane-wave calculations with spin-orbit interaction. The In 4d electrons are treated both as core or as valence electrons. We compare the performance of the local-density approximation (LDA), the generalized gradient approximation (GGA) and the recently proposed non-empirical meta-generalized gradient approximation (Meta-GGA) in calculations of the structural and electronic properties of InN.

Band structures, densities of states, orbital-resolved densities of states, total and partial valence charge densities and ionicity factors are analysed in great detail. The calculated values of the energy gaps, bandwidths, spin-orbit and crystal-field splittings and the correct band degeneracies are compared to experimental and/or ab initio results.

The role played by relativistic effects and meta-GGA functional on the band structure is discussed. For the structural properties meta-GGA is more accurate and gives the best description of InN.

We found that several features of α-InN resemble those of β-InN. Most of the calculated band parameters, of band gaps, total and upper-valence bandwidths, and antisymmetric gap for α-InN are close to those of β-InN to within 1%. The charge distributions have similar features meaning that this material has the same ionicity factor in both structures even when In 4d were treated both as core or as valence electrons.

15:50 poster A-5

Scanning near-field optical microscopy (SNOM) analysis of MOVPE InN

Hiroshi Miwa, Yasuhiko Nagai, Akihiro Hashimoto, Akio Yamamoto

University of Fukui (U. Fukui), 3-9-1 Bunkyo, Fukui 910-8507, Japan

Contact: e043874@icpc00.icpc.fukui-u.ac.jp

Recently, we have attained a mobility of 1100 cm^2/Vs and carrier concentration of $4.5\times10^{18}cm^{-3}$ for InN films grown by the atmosphere pressure MOVPE [1]. However, compared with MBE InN, MOVPE InN still has inferior electrical and optical properties. FWHM for PL peaks for MOVPE InN [2] is considerably larger than that for MBE InN [3], in spite of the similar PL peak energies. Therefore, MOVPE InN seems to have a large nonuniformity in their properties. In this paper, we report the scanning near-field optical microscopy (SNOM) analysis of MOVPE InN to get information about the nonuniformity. Samples of InN are grown at 600ºC on nitrided (0001) sapphire substrates without or with a GaN buffer by the atmospheric-pressure MOVPE. In the SNOM system (Type NFS-220FK, JASCO Corp., Japan.), a green laser (l= 532 nm) is used as an excitation source. A probe with an aperture size of 100-500 nmφ is used to illuminate the sample surface and collect near-field light (illumination-collection mode). A near-field PL spectrum and its intensity mapping are successfully obtained for InN films at room temperature for the first time. By monitoring a probe movement at each point of measurement, surface topography is also obtained. A large nonuniformity in the near-field PL mapping is observed for samples grown without buffer. Compared with films grown without buffer, those grown with a GaN buffer have a excellent uniformity in near-field PL intensity. Thus, the use of the buffer is found to improve effectively the in-plane uniformity of the near-field PL intensity. This seems to be due to the uniform nucleation of InN on the buffer. [1] A. Yamamoto et al., (will be presented in ICNS-6). [2] A.

Yamamoto et al., J. Cryst. Growth 261 (2004) 275. [3] F. Chen et al., Physica E 20 (2004) 308.

15:50 poster A-6

Contactless electroreflectance spectroscopy of InN layers grown by MBE

Marcin Motyka

Wroclaw University of Technology, Institute of Physics, ul. Wybrzeże Wyspiańskiego 27, Wrocław 50-370, Poland

Contact: marcin.motyka@pwr.wroc.pl

Contactless electroreflectance spectroscopy of InN layers grown by MBE

M. Motyka, R. Kudrawiec, J. Misiewicz

Institute of Physics, Wrocław University of Technology,

Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland

M. Kurouchi, T. Araki, Y. Nanishi

Dept. of Photonics, Ritsumeikan University, 1-1-1 Noji-Higashi,

Kusatsu, Shiga 525-8577, Japan

Contactless electroreflectance (CER) spectroscopy is a powerful tool to investigate optical properties of semiconductors. The derivative nature of this experimental method enables observation of a large number of sharp spectral features including this related to the band-gap transition splitted into individual lines associated with particular valence subbands. The issue of the bandgap energy for InN is still not fully understand, especially the structure of the valence band. In this paper two InN samples (S1 and S2) grown by molecular beam epitaxy (MBE) are studied in CER spectroscopy. The electron concentration for sample S1 and S2 is 2.7 and 6.0 10^{18} cm^{-3}, respectively while the electron mobility for samples S1 and S2 is 1330 and 870 cm^2/Vs, respectively. For the two samples CER features related to the band-gap energy are observed around 0.7 eV at room temperature. However, these features cannot be fitted by a one CER resonance. Note that the fine structure of the band-gap transition is visible by nagged eye. It suggest that the valence band at k=0 is splitted into individual subbands. The issue of the valence band structure for InN at k=0 is discussed in this work.

15:50 poster A-7

Investigation of InN layers grown by MOCVD and MBE using analytical and high resolution TEM

Pierre Ruterana, Morad Abouzaid

SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France

Contact: pierre.ruterana@ensicaen.fr

Among the nitride semiconductors InN, shows the smallest effective mass and the highest electron drift velocity thus setting it as a candidate for applications in ultrahigh speed electronic devices. The band gap of 0.7 eV obtained for InN makes it also suited for high efficiency solar cells. In good quality layers hall mobility of 1000 cm^2/s and residual carrier concentrations as low as 10^18 /cm^3 have been reported.

In this work we investigate the microstructure of InN layers grown by MOCVD on different buffer layers using TEM (AlN, directly on sapphire, GaN). The polarity of the layers is determined by Convergent Beam Electron Diffraction (CBED). We analyze the typical defects that form in these layers as well as the interfacial relationships between the substrate/buffer and active layers at atomic scale. The large mismatch between InN and sapphire or GaN leads to particular interface structures which critically depend on the growth conditions. It has been possible to point out typical interface structures from optimized growth conditions, and this will be discussed in the light of the PL emission intensity and peak position.

In MBE layers, we also confirm the growth polarity of the layers and it is pointed out that the layers contain a huge amount of dislocations, but no In precipitates, by cross section and planar view observations.

Our local analysis at atomic scale allows to clearly detemine the origin of the PL emission below 0.8 eV as related to the local chemical composition and structure of the InN layers.

15:50 poster A-8

A comparative study on MOVPE InN films grown on 3c-SiC/Si(111) and sapphire substrates

Takahiro Kobayashi[1], Myung Soo Cho[1], Naoki Sawazaki[1], Akihiro Hashimoto[1], Akio Yamamoto[1], Yoshifumi Ito[2]

1. *University of Fukui (U. Fukui), 3-9-1 Bunkyo, Fukui 910-8507, Japan* **2.** *Wakasa-Wan Energy Reseach Center (WERC), 64-52-1 Nagatani, Tsuruga 910-0192, Japan*

Contact: e043873@icpc00.icpc.fukui-u.ac.jp

We have previously reported [1] that a single-crystalline wurtzite InN is grown on a 3c-SiC/Si(111) template, which is formed by the carbon (C^+)-ion implantation into Si(111) followed by the annealing at a high temperature. However, the quality of InN is inferior to that grown on sapphire. In this paper, InN films are grown on 3c-SiC/Si(111) templates nitrided just before the buffer growth, and their properties are found to be improved to the level for InN films grown on sapphire substrates. The procedures to make 3c-SiC/Si(111) templates was reported elsewhere [1]. MOVPE growth of InN is performed at 600ºC with a GaN buffer layer grown at 550ºC. Just before the GaN buffer growth, a 3c-SiC/Si(111) template is nitrided at 900ºC for 30 min. It is found that the GaN buffer layer grown on the nitrided template is very uniform, while the buffer grown on an un-nitrided template shows the island growth. The uniform GaN buffer growth is due to the improved wetting of GaN layer on the 3c-SiC/Si(111) template.

No significant difference is found surface morphologies between InN films grown on the template and those on a sapphire substrate. Both films grown on the template and the sapphire show an intense photoluminescence with peak energy of 0.7eV at room temperature. The carrier concentration and Hall mobility of the InN films grown on the template are 7.6×10^{18} cm^{-3} and 630 cm^2/Vs, respectively. These data are comparable to those for MOVPE InN grown on the sapphire (6.5×10^{18} cm^{-3} and 870 cm^2/Vs) and for plasma-assisted MBE films grown on Si substrates (6.8×10^{18} cm^{-3}, 690 cm^2/Vs) [2]. Thus, the nitridation process of the 3c-SiC/Si(111) template is effective to obtain high-quality InN film. The 3c-SiC/Si(111) wafer will open up a variety of device application. [1] A. Yamamoto et al., Phys. Stat. Sol. (c) 2, 2281 (2005). [2] H. Ahn et al., Appl. Phys. Lett. 86, 201905 (2005).

15:50 poster A-9

Growth of N-polarity InN by ArF-laser assisted MOVPE

Akio Yamamoto[1,2], Ken Kasashima[1], Masayoshi Miyanishi1, Akihiro Hashimoto[1]

1. *University of Fukui (U. Fukui), 3-9-1 Bunkyo, Fukui 910-8507, Japan* **2.** *CREATE-Fukui, JST (CREATE-Fukui), 61-10 Kawai-Washizuka, Fukui 910-0102, Japan*

Contact: yamamoto@fuee.fukui-u.ac.jp

To overcome the problem of the low decomposition rate of NH_3, we have developed the laser-assisted MOVPE (la-MOVPE) where an ArF excimer laser (λ=193 nm) dissociates NH_3 photolytically [1]. A film can be grown in a wide range of growth temperature, from RT to 700ºC. This growth technique provides a growth rate of more than 0.5 μm/h just by flowing a very little amount (100 sccm) of NH_3. Thus, the la-MOVPE is promising as a new growth technique for InN. In this paper, we report that, by employing ArF-laser assisted nitridation of sapphire substrate, an N-polarity InN film can be grown by the ArF-laser assisted MOVPE. Using a la-MOVPE system, an InN film is grown at 600ºC on (0001) sapphire substrates. An ArF excimer laser with an energy 50 mJ/pulse and a repetition rate 20 Hz is introduced parallel to the substrate surface. The surface morphology of a grown InN film is found to be markedly dependent on the nitridation conditions for sapphire substrate. When no nitridation is made, the surface of a grown InN film is very rough and many In droplets are seen on it. A film with a mixture of rough-surface regions with In droplets and smooth-surface regions is grown on the substrate nitrided thermally at 1000ºC for 30 min. By employing the ArF-laser irradiation to the NH_3 flow during the substrate nitridation at 1000ºC for 30 min, a smooth surface region is expanded to the entire surface of a film. The smooth surface is found to be stable at a growth temperature higher than 700ºC. The KOH etching of grown films reveals that the smooth surface has a 5-10 time higher etching rate than the rough one. These facts indicate that a film with the smooth surface has N-polarity. Thus, the ArF-laser assisted nitridation of sapphire substrate results in the successful growth of N-polarity InN by MOVPE. [1] A. G. Bhuiyan et al., Jpn. J. Appl. Phys. 42, 7284 (2003).

15:50 poster A-10

Mass flow and reaction analysis of the growth of GaN layers by HVPE

Pawel Kempisty[1], Stanisław Krukowski[2], Izabella Grzegory2, Bolesław Łucznik[2], B. Pastuszka[2], Michał Boćkowski[2], Sylwester Porowski[2]

1. *Warsaw University of Technology, Department of Technical Physics and Applied Mathematics, Warszawa, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: kempes@unipress.waw.pl

HVPE growth in horizontal flow reactor has been analyzed using finite element calculation and molecular estimates of the reaction rates. The reaction rate of the HCl with liquid Ga has been estimated using ideal gas approximation for HCl vapor. The conversion HCl to GaCl rate has been obtained in function of the pressure, temperature, flow velocity and the geometry of the reactor.

Finite element code FIDAP (commercially available from Fluent Inc.) has been used to calculate the flow pattern in the reactor. In the first approximation it was assumed that the flow pattern is weekly dependent on the temperature distribution in the reactor. It was also assumed that the volume reaction rates can be approximated by temperature independent reaction constants.

Using these calculation it was possible to obtain the reaction rates on the susceptor surface. The reaction rate leads to different growth rates depending on the geometry of the susceptor and the distance between the flow outlets. The calculated reaction rates were compared with the measured growth rate in horizontal, normal pressure HVPE reactor.

Additionally , the parasitic reaction on the pipe close to the gas inlets was observed. It was shown that the reaction is strongly inhomogeneous, depending on the geometry of the inlets. The reaction depends on the flow velocity and the pressure. The obtained reaction rates were compared with the estimates drown from the growth experiments.

15:50 poster A-11

Optical properties of InN films and the influence of surface contaminations

Christoph Cobet[1,2], Patrick Vogt[1], Munise Rakel[1,2], Ruediger Goldhahn[3,4], Massimo Drago[1], Colm Stephens[5], Antje Vollmer[6], Wolfgang Richter[1], Norbert Esser[2]

1. *Berlin University of Technology, Hardenbergstr. 36, Berlin 10623, Germany* **2.** *Institute for Analytical Sciences (ISAS), Albert-Einstein-Str. 9, Berlin 12489, Germany* **3.** *Technical University Ilmenau, Center of Micro- and Nanotechnologies (ZMN), Gustav-Kirchhoff-Str. 7, Ilmenau 98693, Germany* **4.** *Technische Universität Ilmenau, Institut für Physik, PF 100565, Ilmenau 98684, Germany* **5.** *Trinity College, Dept. Physics, College Green, Dublin 2, Ireland* **6.** *Berliner Elektronenspeicherring-Gesellschaft für Synchrotronstrahlung m. b. H. (BESSY), Albert-Einstein-Str, Berlin 12489, Germany*

Up to the late 90's, InN properties were measured mostly on sputtered thin films of low quality. The fundamental bandgap of these samples was assumed to be around 2eV. But new theoretical investigations, as well as experimental progress in the epitaxial growth of more perfect layers by MBE and MOVPE, have shown that the fundamental band gap is more likely located around 0.7eV. Therewith, the bandgaps of group III–Nitrides could cover a very wide spectral range from the far ultraviolet (AlN) to the near infrared (InN). But the reasons for the contradicting results concerning the fundamental band gap and the VIS-VUV dielectric functions are still under discussion.

Here we report on measurements of the dielectric function of hexagonal InN in a broad spectral range from 0.5-12eV by means of ellipsometry. An a-plane InN(11-20) layer grown by molecular beam epitaxy in the Cornell University, was utilized to determine the ordinary and the extraordinary part of the dielectric tensor. We find a huge anisotropy between both components. The specific absorption structures differ concerning the energy position or disappear in the extraordinary component. The ellipsometric measurements also indicate a strong influence of surface contaminations, which finally could effect the position of the measured band gaps as well. In order to analyze the surface degradation in more detail, we performed SXPS before and after a thermal annealing of hexagonal MOVPE-grown InN samples in UHV. The InN(0001) surface after transfer into UHV gives rise to clear C1s and O1s core-level contributions, the latter originating from hydroxides due to the water contamination of the surface in air. Thermal annealing at 573K is sufficient to remove the carbon and hydroxide components. Stable oxide contributions could not be removed even at the highest annealing temperature (733K) possible for InN. These results may explain partly, at the end, the wrong conclusions drawn about the fundamental band gap.

15:50 poster A-12

Investigation of Deformation Fields Anisotropy in Multilayered (In,Ga)As/GaAs Structures with Quantum Wires by HRXRD.

Oleksandr N. Yefanov[1], Vasyl P. Kladko[1], O Y. Gudymenko[1], V V. Strelchuk[1], Yu I. Mazur, Zh V. Wang[2], G. J. Salamo

1. *V.Lashkaryov Institute of Semiconductor Physics NAS Ukraine (ISP), Nauky prosp., Kyiv 03028, Ukraine* **2.** *University of Arkansas, Fayetteville 72701, United States*

Contact: efa@hotbox.ru

The results of investigation of $In_{0.3}Ga_{0.7}As$/GaAs superlattices with quantum wires by High-Resolution X-Ray Scattering are presented. The influence of lattice distortion on rocking curves (RC) was analyzed with dynamical theory of diffraction. It allowed explaining azimuth dependence of experimental rocking curves. Anisotropic changes in the shape of InGaAs lattice unit cell were shown and measured.

The influence of smooth borders between hetero-layers was analyzed. Comparative analysis of influence of different gradient functions in the hetero-border on rocking curves was done. Parameters of hetero-interfaces in our samples were determined with the help of RC modeling.

The influence of shot time annealing (30sec) with different temperatures on quantum wires was observed. Increasing of lateral mass transfer with increasing of annealing temperature was observed.

If smooth hetero-interface and anisotropic distortions in unit cell are not considered, wrong determination of structure parameters (both period of superlattice and ratio of layers thicknesses) may occur.

The result obtained allowed us to find the regularity in structure changes under the influence of annealing and to investigate features of elastic deformations relaxation in space ordered InGaAs quantum wires. It is very important for quantum-size objects physical properties understanding.

15:50 poster A-13

Rietveld refinement for polycrystalline indium nitride

Roman Minikayev[1], Wojciech Paszkowicz[1], Sławomir Podsiadło[2], Stanisław Krukowski[3], Mike Leszczynski[3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: minik@ifpan.edu.pl

Indium nitride is an attractive semiconductor material for optoelectronic applications. It is a potential material for high-speed electronics and solar cells. The structural data of the bulk material provide a necessary basis in development of such devices. In the present work, the structural information on indium nitride is obtained from high-quality powder diffraction patterns collected at a laboratory diffractometer. Polycrystalline InN powders studied in this paper were prepared from high-purity components. Powder diffraction data were collected at a laboratory Bragg-Brentano diffractometer equipped with a copper X-ray tube, a focusing incident-beam monochromator and a semiconductor strip detector. The applied instrument, described in more detail in Ref. [1], allows for acquiring the data with excellent statistics and resolution. During sample mounting, efforts were made for reduction of possible preferred orientation effects. The applied structure models include minor-impurity phases. Rietveld refinements were performed using the Fullprof.2k (v. 2.70) program [2]. Values of refined lattice parameters and the free positional parameter of nitrogen are consistent with earlier literature data. The present data complete the recent refinements for AlN and GaN [3].

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15:50 poster A-14

LATTICE PARAMETERS OF INDIUM NITRIDE IN THE 22-310 K TEMPERATURE RANGE

Wojciech Paszkowicz[1], Pawel Piszora[2], Roman Minikayev[1], Michael Knapp[3], Carsten Baehtz[4], Sławomir Podsiadło[5]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Adam Mickiewicz University, Faculty of Chemistry, Grunwaldzka 6, Poznań 60-780, Poland* **3.** *Technische Universität Darmstadt, Institute of Materials Science, Petersenstr. 23, Darmstadt 64287, Germany* **4.** *Hamburger Synchrotronstrahlungslabor HASYLAB (HASYLAB), Notkestrasse 85, Hamburg D-22603, Germany* **5.** *Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland*

Contact: paszk@ifpan.edu.pl

Variation of unit-cell size of indium nitride with temperature has been studied in Refs. [1-3]. The low-temperature range has been only partially explored up to now. In the present study, the lattice parameters for indium nitride were determined using X-ray powder diffraction at a synchrotron radiation source (beamline B2, Hasylab/DESY, Hamburg) in the temperature range from 22 K up to 310 K. An image-plate detector was applied for data collection. The wavelength was calibrated using internal diamond standard, according to the method described in Ref. [4]. The lattice parameters a and c behave in a similar way: they are virtually constant below about 130 K. In the range 200 to 300 K the variation is close to a linear one. Discussion of the results will involve the earlier studies of thermal expansion of indium nitride.

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Tuesday, 6 September

Band Structure I: A. Yoshikawa/D. Alexandrov

Tuesday morning, 6 September, 9:00
Main Building, room 134

9:00 invited oral

Band Structure and Properties of InN and In-rich In1-xGaxN Alloys

Wladek Walukiewicz[1], Kin M. Yu[1], Sonny X. Li[1,2], Rebecca E. Jones[1,2], Joel W. Ager III[1], Eugene E. Haller[1,2], Hai Lu[3], William J. Schaff[3]

1. *Lawrence Berkeley National Laboratory (LBNL), 1 Cyclotron Road, Berkeley 94720, United States* **2.** *University of California, Berkeley 94720, United States* **3.** *Cornell University, 425 Philips Hall, Ithaca, NY 14853, United States*

Contact: W_Walukiewicz@lbl.gov

In this presentation we review the results of our recent studies aimed at understanding the electronic structure and its effects on electrical and optical properties of InN and In-rich $In_{1-x}Ga_xN$ alloys. We have investigated MBE-grown films, unirradiated as well as irradiared with high energy particles. Measurements of the fundamental absorption edge give the value of 0.64 eV for the room temperature energy gap of InN. The low energy gap and a strong kp interaction leads to a small electron effective mass and a distinctly nonparabolic conduction band. In samples with high electron concentra-

tions, a large increase in the absorption edge energy is observed due to the Burstein-Moss shift. In addition to having a narrow gap, InN also has an extremely high electron affinity of 5.8 eV, which places the conduction band edge of this material 0.9 eV below the average energy of dangling bond defects represented by the Fermi level stabilization energy, E_{FS}. This unusual band alignment explains the extreme proclivity for n-type conduction and the large surface electron accumulation densities in InN and In-rich alloys. As grown, undoped InN is always n-type with electron concentrations ranging from mid 10^{17} cm^{-3} to as high as 10^{21} cm^{-3}. A similar range of electron concentrations can be achieved by irradiation with 2 MeV He^{+} ions. At a sufficiently high dose, the electron concentration saturates. In agreement with the amphoteric defect model the saturation concentration n_s, corresponds to Fermi energy pinned at E_{FS}. In $In_{1-x}Ga_xN$ the conduction band shifts towards the E_{FS} resulting in reduced n_s with increasing x. For x>0.66 E_{FS} moves below the conduction band edge and acceptor like defects are preferentially formed, leading to a decrease of electron concentration in irradiated samples. Photoluminescence results show that samples with x<0.6 are two orders of magnitude less sensitive to irradiation damage than standard III-V compounds.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

9:30 oral

Valence band structure of InN from x-ray photoemission studies

Louis F. J. Piper[1], Tim D. Veal[1], Paul H. Jefferson[1], Chris F. McConville[1], F Fuchs[2], J Furthmüller[2], Friedhelm Bechstedt[2], Hai Lu[3], William J. Schaff[3], Hiroyuki Naoi[4], Y Nanishi[4]

1. *University of Warwick, Gibbet Hill Road, Coventry CV4 7AL, United Kingdom* **2.** *Freidrich-Schiller-Universitat Jena, Jena 07743, Germany* **3.** *Cornell University, Electrical and Computer Engineering, Ithaca, NY 14853-540, United States* **4.** *Ritsumeikan University (Rits), Kusatsu 525-8577, Japan*

Contact: L.F.J.Piper@warwick.ac.uk

The band structure of InN has been investigated by comparing the calculated valence-band density of states with x-ray photoemission spectroscopy (XPS) of the valence-bands. The band structure of InN has previously been calculated using density functional theory (DFT) within the local density approximation (LDA) [1]. Such calculations result in an overlap of the conduction and valence bands around the Γ-point, giving rise to negative band gaps. The overlap is due to the overestimation of the pd-repulsion within the DFT-LDA [2]. Pseudo-potentials accounting for the self-interaction corrections of the In4d electrons were used to revise the amount of pd-repulsion, avoiding the overestimation [1]. The percentage contribution of the pd-repulsion was determined by comparing the experimental and theoretical positioning of the In4d valence-levels with respect to the valence band maximum (VBM). Here, the true experimental In4d - VBM separation is reported as 16.0 ± 0.2 eV, in contrast to a previous value of 14.9 eV [3]. The valence-band density of states was calculated using DFT-LDA, with the revised pd-repulsion for the self-interaction corrections included. Good agreement between the experimental and theoretical valence-band density of states was obtained. Previous such studies of InN were hampered by the difficulty in preparing InN free surfaces. Conventional methods of surface preparation are severely limited for InN. Here atomic hydrogen cleaning (AHC) cycles were used, which have been shown to successfully prepare clean, electronic-damage free InN surfaces [4]. A combination of core-level XPS, scanning electron microscopy and atomic force microscopy confirmed that AHC produced clean, flat, featureless surfaces.

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9:45 oral

Band Gap Energy of a-InN Thin Films

Jebreel M. Khoshman[1], Martin E. Kordesch[2]

1. *Al-Hussein Bin Talal University (AHU), P. O. Box 20, Ma'an, Jordan* **2.** *Ohio University (OU), Dept. of Physics & Astronomy, Athens, OH 45701, United States*

Contact: khoshman@phy.ohiou.edu

Recently there has been an exciting conflict on the optical band gap of InN. The determination of the band gap energy of amorphous (a) InN thin film presented a challenge and added another dimension to the E_g conflict. At temperature < 325 K, we deposited InN thin films onto different substrates (c-Si (111), glass, and quartz) by using RF magnetron sputtering at a rate between 0.03 and 0.04 nm/s. X-ray diffraction patterns revealed that the films grown on the substrates have an amorphous nature over a wide range of diffraction angles (20^{o}- 100^{o}). The optical E_g of a-InN have been determined by three independent methods, spectroscopic ellipsometry (SE), spectrophotometric (SP), and photoluminescence (PL) over the energy range 0.88 - 4.1 eV. The absorption coefficient, α, was obtained by the analysis of the measured ellipsometric spectra through the Tauc-Lorentz model. The optical bandgap was determined using the modified Tauc and Cody extrapolations. The corresponding Tauc and Cody optical bandgaps were found to be 1.73 and 1.72 eV, respectively. These values were in excellent agreement with the values of the bandgap energy obtained as fitting parameters in the Tauc-Lorentz model: $1.72 \pm 5.9 \times 10^{-3}$ eV. Using the SP method, α was cal-

culated according to the Beer-Lambert law and it showed that the value of the bandgap (1.74 eV) was much closer to that by the analysis of SE data, and also to the values of E_g as a fitting parameter in the chosen model. The PL spectra of the films showed only one strong band edge at 1.69 eV while it exhibited only one weak band edge PL emission at 1.63 eV. The spectral dependence of the polarized absorptivities was investigated at different angles of incidence. We found that there was a higher absorptivity for wavelength < 725 nm. This wavelength, ~ 725 nm, therefore indicated that the absorption edge for a-InN film is about 1.70 eV.

10:00 invited oral

Plasmonic effects in InN-based structures with nano-clusters of metallic indium

Tatiana V. Shubina

Ioffe Physico-Technical Institute (Ioffe), Polytechnicheskaya, 26, Saint-Petersburg 194021, Russian Federation

Contact: shubina@beam.ioffe.ru

Plasmonic effects in metal/semiconductor composites have recently received a lot of attention. Many exciting applications of them are expected for light amplification and conversion. To reveal optical peculiarities of InN related to plasmonic resonances, a comparative study has been performed of the films grown by MBE (i) with obvious inclusions of metallic indium, (ii) without the massive inclusions, and (iii) with intentionally formed sheets of the metallic nano-particles. Additionally, thin In insertions (10-50 nm) in a GaN matrix have been fabricated to study interband absorption in metallic In, whose onset occurs near 0.7 eV. In the first type of InN-based structures, the spectra of thermally detected optical absorption (TDOA) measured at 0.35 K show pronounced peaks near 1 eV, ascribed to the Mie resonances. Emission intensity is strongly enhanced in the vicinity of the metallic inclusions. This phenomenon is discussed in terms of coupling of radiative states with the surface plasmons. In the state-of-art InN films grown outwardly without such inclusions, the TDOA spectra also exhibit a sharp peak, but near 0.7-0.75 eV. Theoretical estimation shows that this peak position may correspond to the surface plasmons in an InN composite with thin oblate metallic insertions. Generally, the optical properties of such films are similar to those of the layers with the intentionally introduced sheets of the In nano-particles. In both types of structures, PL excitation spectra have pronounced peaks in the same 0.7-0.75 eV range, as in the TDOA. The emission is strongly p-polarized with the degree of polarization as much as 40%. PL intensity is increased when exciting at higher angles to the surface normal. The emission in InN frequently consists of two peaks. The origin of the PL splitting is considered taking into account different states involved into recombination process and their coupling with plasmonic modes.

Band Structure II: W. Walukiewicz/T. Shubina

Tuesday afternoon, 6 September, 14:00
Main Building, room 134

14:00 invited oral

Dielectric function of InN: Nonparabolicity and excitonic effects

Friedhelm Bechstedt

Friedrich-Schiller-Universitaet (IFTO), Max-Wien-Platz 1, Jena 07743, Germany

Contact: bech@ifto.physik.uni-jena.de

The InN semiconductor is a challenge for the theoreticians. There are several pecularities in the electronic band structure and, hence, in the resulting electrical and optical properties: (i) Extremely shallow In4d electrons which influence the valence bands, e.g. by the pd repulsion. (ii) One of the largest electron affinities which is responsible for the small fundamental gap, the small electron effective mass, and the nonparabolicity of the conduction band. (iii) A charge neutrality level in the conduction band being responsible for the n-accumulation layer at the surface. (iv) Strong excitonic effects because of the small electron mass and dielectric constant.

All effects are included in the calculation of the electronic band structure and the many-body effects accompanying electronic excitations. The consequences are demonstrated for the ordinary and extraordinary dielectric functions. After inclusion of the pd repulsion, the quasiparticle band shifts, the attractive electron-hole interaction as well as the electron-hole exchange, we find excellent agreement with recent ellipsometry measurements concerning peak positions and lineshape. There are however deviations concerning the peak intensities above photon energies of 5eV.

The imaginary parts of the dielectric functions show a characteristic lineshape. After a steep onset of the absorption around 1eV, a plateau follows until about 4.5eV. Several pronounced peaks occur in the high-frequency region. We demonstrate that the absorption onset and the plateau-like region are dominated by the nonparabolicity of the conduction band. The interpretation of the high-energy absorption peaks only in terms of optical transitions at critical points is insufficient. The Coulomb interaction leads to an intermixing of transitions from different k-space regions. As a consequence oscillator/spectral strength is redistributed from higher to lower photon energies.

14:30 invited oral

Band transitions in the InGaN system

Petra Specht

University of California, Berkeley 94720, United States

Contact: specht@berkeley.edu

The InN – GaN ternary system is subject of ongoing research efforts because of the materials ability to tolerate an unusual high point – and extended defect density while exhibiting unique optoelectronic properties. Obviously, the large defect density must affect device performance. This experimental study in a transmission electron microscope addresses the need of imaging the host crystal without the effect from extended defects or other crystallographic phases. Valence electron energy loss spectroscopy (VEELS) with a monochromated electron beam is utilized allowing for high spatial (2 nm) and high energy (< 200 meV) resolution. The bandgap of various InN epilayers, deposited by molecular beam epitaxy, was measured to 1.7 eV (hexagonal system) and 1.4 eV (cubic system). Initial results on band transitions in ternary InGaN will also be given.

15:00 invited oral

Electron band structure and optical properties of InN and related alloys

Dimiter Alexandrov[1], K. Scott A. Butcher[2], Trevor L. Tansley[2]

1. *Lakehead University, 955 Oliver Road, Thunder Bay P7B5E1, Canada* **2.** *Macquarie University, Sydney 2109, Australia*

Contact: dimiter.alexandrov@lakeheadu.ca

Investigations of the electron band structures of InN and related alloys are presented. Theoretical method for calculation of electron band structures based on interactions between atomic orbitals belonging to nearest neighboring atoms is developed. The method is based on the Hartree-Fock model of electron interactions together with the corresponding Kohn-Sham corrections. The one-electron Schrödinger equation describing the interaction between neighboring orbitals is derived. It requires determination of the electron correlation energies. The energy terms, which are solutions of this equation, have further application for the calculation of the LCAO electron band structure of the corresponding compound alloy. The method is applied for the calculations of electron band structures of the following alloys: InN, InGaN, InAlN, InP, InAs, InSb, GaN, AlN and non-stoichiometric InN (with In atoms substitutional on the N site and N atoms substitutional on the In site). The calculated LCAO electron band structures of the semiconductors InN, InGaN, InAlN and non-stoichiometric InN are used for explanation of their optical properties. It is discovered existence of tunnel optical absorptions in InGaN, in InAlN and in non-stoichiometric InN. This phenomenon is investigated in details and it determines optical absorption edge in these structures to vary in interval 0.2 – 1.62 eV. It is found existence of energy pockets in the conduction band and in the valence band respectively due to the defects of these structures and it is discovered excitons of the structure formed by electrons and holes occupying corresponding pockets. The properties of these excitons are investigated in details. It is found that the annihilations between electrons and holes belonging to these excitons determine that PL spectra of these alloys to have maxima in range 0.18 – 0.82 eV. The theoretical results are compared with experimental data.

Band Structure III: T. Suski/A. Klochikhin

Tuesday afternoon, 6 September, 15:50
Main Building, room 134

15:50 invited oral

Optical anisotropy of InN from near-IR to deep-UV

Ruediger Goldhahn

Technische Universität Ilmenau, Institut für Physik, PF 100565, Ilmenau 98684, Germany

Contact: ruediger.goldhahn@tu-ilmenau.de

We present a detailed analysis of the ordinary and extraordinary dielectric tensor components (DF) of InN corresponding to electric field polarization perpendicular and parallel to the c-axis, respectively. The data were determined by spectroscopic ellipsometry and cover the energy range from 0.72 up to 30 eV. Results for both c-plane and a-plane InN films grown by MBE are compared.

The data indicate unambiguously two absorption edges below 0.9 eV depending on the light polarization direction. Such a behavior is typical for wurtzite material around the band gap. It arises from the different optical selection for transitions from the three valence bands at the center of the Brillouin zone into the conduction band. The extraordinary absorption edge is found 25 meV higher than the ordinary one, which corresponds to the splitting between the uppermost and the lowest-lying valence bands. Assuming a spin-orbit energy of 5 meV we estimate an experimental value for the crystal-field parameter of 24 meV. The extrapolation of the ordinary tensor component emphasizes a band gap of ~0.65 eV at room temperature for wurtzite InN, if carrier induced band gap renormalization, conduction band occupation as well as its non-parabolicity are taken into account.

The shape of the experimental DF's and the polarization dependence in the energy range from 4.5 up to 9.5 eV are in excellent agreement with theoretical calculations. Low-temperature studies as well as a third-derivate based line shape analysis allow us to determine the transition energies

for a large number of critical points of the band structure with highest precision. Finally, we discuss the polarization dependence of the semi-core-level excitations above 16 eV. The data analysis yields a value of 830 meV for the spin-orbit splitting of the InN 4d-bands.

16:20 invited oral

THEORETICAL INVESTIGATION OF THE InN BAND GAP ANOMALY

Su-Huai Wei

National Renewable Energy Laboratory (NREL), 1617 Cole Blvd, Golden 80401, United States

Contact: swei@nrel.gov

III-N semiconductors and their alloys are important materials for optoelectronic device applications such as light-emitting diodes and lasers. Recently, InN has attracted significant attentions because experimentally measured band gaps of InN show a wide range of variation from 0.6 to 2.0 eV. The origin of this variation is currently still under debate. Using a band-structure method that includes the correction to the band gap error in the local density approximation [S.-H. Wei et al., Phys. Rev. B 67, 165209 (2003)], we find that the band gap for stoichimetric InN is 0.8 ± 0.1 eV, in good agreement with recent experimental data, but is much smaller than previous experimental value of ~1.9 eV. The unusually small band gap for InN is explained in terms of the high electronegativity of nitrogen and consequently the small band gap deformation potential of InN. To understand the origin of some of the experiments, which show large band gap of InN, we have performed detailed analysis of the band structure of InN. The possible origins of the measured large band gaps are discussed in terms of the non-parabolicity of the bands, the Moss-Burstein shift, and the effect of oxygen. We find that the Moss-Burstein shift plays the dominant role in determining the measured InN band gap. The formation of InN_xO_{1-x} alloys reduces the band gap, whereas the formation of $(InN)/(In_2O_3)$ superlattices increases the band gap with respect to InN. The effect of non-stoichimetric InN will also be discussed.

Work was done with P. Carrier and supported by the U.S. DOE, under contract No. DE-AC36-98-GO10337

Rump Session I: Band Gap and Band Structure of InN
Tuesday afternoon, 6 September, 16:50
Main Building, room 134

Wednesday, 7 September

Properties, Optical, Electronic I: R. Goldhahn/ T. Veal
Wednesday morning, 7 September, 9:00
Main Building, room 134

9:00 invited oral

Localized donor states resonant with the conduction band in InN and GaN

Tadeusz Suski

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: tadek@unipress.waw.pl

This presentation is devoted to a comparative studies of a donor impurities contributing significantly to n-type conductivity in unintentionally doped GaN and InN binary nitrides.

One question crucial for understanding physicals properties of these compounds concerns the chemical nature of the donors present often in concentrations exceeding 1019 cm-3. The second one is related to their character and location of the related impurity levels in the band structure of both materials. In particular we discuss the conditions determining a role of these impurities as shallow or localized donors.

It has been found in GaN that oxygen forms an effective-mass-like donor state with the level situated » 30 meV below the conduction band (CB) minimum. Under sufficiently high pressures or alloying with Al, localized donor state can be formed by Oxygen and eventually it takes a role of a donor ground state. At ambient conditions this localized state of O is situated few hundreds of meV above the CB minimum.

In case of InN a situation is much less clear. It concerns an origin of high electron concentration which can exceed 1021 cm-3. Hydrostatic pressure studies show a strong transfer of electrons to the localized donor state in samples with electron concentration below about 5´1018 cm-3. Though the chemical nature of the donor state is unknown, its localized level (at ambient conditions) is located about few tens of meV above the CB minimum. The considered donor supplies about 5´1017 cm-3 electrons to the conduction band. It is suggestive to associate our findings with observations of an involvement of the localized donor state in radiative recombination of these materials. We discuss such a possibility in our presentation.

9:30 oral

Surface band bending at n-type and p-type InN by Auger Electron Spectroscopy

Volker Cimalla[1], Merten Niebelschütz[1], Gernot Ecke[1], Oliver Ambacher, Ruediger Goldhahn[3], Hai Lu[2], William J. Schaff[2]

1. *Technical University Ilmenau, Center of Micro- and Nanotechnologies (ZMN), Gustav-Kirchhoff-Str. 7, Ilmenau 98693, Germany* **2.** *Cornell University, 425 Philips Hall, Ithaca, NY 14853, United States* **3.** *Technische Universität Ilmenau, Institut für Physik, PF 100565, Ilmenau 98684, Germany*

Contact: volker.cimalla@tu-ilmenau.de

Thin films of InN show high n-type conductivity, for which the origin is not completely identified up to date. InN layers with carrier concentrations down to 10^{17} cm^{-3} have been prepared, however, no p-type InN could be demonstrated. Electron accumulation at the surface was verified on air [1], and on clean InN surfaces in ultrahigh vacuum [2]. It was explained as an intrinsic property of InN layers ascribed to its band structure [2]. In a previous work [3] we showed by Auger electron spectroscopy (AES) depth profiling and simultaneous conductivity measurements the correlation between the oxygen content and the electron accumulation. In this work we extend this study by analyzing the peak energy shift in AES on both undoped and Mg-doped InN. The position of the Auger peaks is sensitive to the position of the Fermi level [4]. This capability was used to obtain information about the type of conductivity and the band bending. On all InN surfaces a strong increase of the resistivity within the first 5 nm confirms the existence of a highly conductive n-type surface layer. A strong Auger peak shift of about 2 eV was observed due to the formation of a wide band gap In_2O_3. After reaching equilibrium of the peak shift after the removal of about 50 nm a difference of 0.3 eV was observed between undoped and Mg-doped InN. Consequently, Mg-doped InN layers have indeed p-type conduction; however, the high n-type surface conductivity is overlaying it and the InN layer appears to be n-type.

[1] H. Lu, W.J. Schaff, et al, Appl. Phys. Lett. 82, 1736 (2003)

[2] I. Mahboob, T.D. Veal, et al, Phys. Rev. B 69, 201307 (R) (2004).

[3] V. Cimalla, et al, phys. stat. sol (c) 2,

[4] R. Kosiba, Thesis, Technical University Ilmenau, 2004

9:45 oral

Multiple photoluminescence peaks from mixed-phase indium nitride thin films

Phillip A. Anderson[1], Robert J. Kinsey[1], Zongwen Liu[2], Simon P. Ringer[2], Roger J. Reeves[3], Steven M. Durbin[1]

1. *Department of Electrical and Computer Engineering, The MacDiarmid Institute for Advanced Materials and Nanotechnology, University of Canterbury, Christchurch, New Zealand, Christchurch, New Zealand* **2.** *Australian Key Centre for Microscopy & Micranalysis, University of Sydney, NSW, Australia, Sydney, Australia* **3.** *Department of Physics and Astronomy, The MacDiarmid Institute for Advanced Materials and Nanotechnology, University of Canterbury, Christchurch, New Zealand, Christchurch, New Zealand*

Contact: paa24@student.canterbury.ac.nz

The recent debate over the bandgap energy of InN has been based on measurements of the wurtzite phase of the material. Several attempts have been made to grow the metastable zincblende phase on cubic substrates, but the tendency of the material to revert to the wurtzite phase has prevented a bandgap being assigned to the cubic phase. [1,2] Measurement of the zincblende bandgap of InN would shed light on the current bandgap energy debate as typically the zincblende phase of III-V semiconductors has a slightly narrower bandgap energy than the wurtzite phase. For example α-GaN (wurtzite) and β-GaN (zincblende) have bandgaps of 3.4 and 3.2 eV respectively, while α-AlN and β-AlN have gaps of 6.2 and 5.1 eV respectively.

Indium nitride thin films have been grown on GaN buffer layers atop sapphire substrates by plasma assisted molecular beam epitaxy. Two films were investigated using high resolution transmission electron microscopy and both were observed to be majority wurtzite, although isolated zincblende grains were also identified within both films. Quantifying the amount of zincblende phase within each film proved difficult due to the small sample sizes investigated within the high resolution scans. However, it was clear that one film had a significantly higher cubic content and corresponded to n-type carrier concentrations and Hall mobility of 3×10^{19} cm^{-3} and 200 cm^2/Vs, respectively, as opposed to 1×10^{19} cm^{-3} and 500 $cm2/Vs$ for the lower cubic content. Photoluminescence studies revealed strong emission near 0.7 eV from both films, but the higher cubic content PL exhibited a low energy shoulder at 0.63 eV. The emission at the low energy shoulder persisted at comparable intensities to the 0.7 eV peak at temperatures from 10 to 300 K.

[1] A. Tabata, A. P. Lima, L. K. Teles et al. Appl. Phys. Lett. 74, 362 (1999)

[2] V. Cimalla, J. Pezoldt, G Ecke et al. Appl. Phys. Lett. 83,

3468 (2003)

10:00 invited oral

Acceptor states in photluminescence of n-InN

Albert A. Klochikhin[1], V. Y. Davydov[2], V. V. Emtsev[2], A V. Sakharov[2], V. A. Kapitonov[2], B. A. Andreev[4], Hai Lu[3], William J. Schaff[3]

1. *St.Petersburg Nuclear Physics Institute RAS (PNPI), Orlova Roshcha, Gatchina 188300, Russian Federation* **2.** *Ioffe Physico-Technical Institute, RAS, Saint-Petersburg, Russian Federation* **3.** *Cornell University, Department of Electrical and Computer Engineering, United States* **4.** *Institute for Physics of Microstructures RAS, Russian Federation*

Contact: Albert.Klochikhin@mail.ioffe.ru

The first observations of near-infrared interband optical transitions in InN were reported for samples with carrier concentrations close to $1*10^{19}$ cm^{-3}. Recent progress in the growth techniques has made it possible to grow n-InN films with improved characteristics. For example, the best samples are characterized by carrier concentrations of about $4*10^{17}$ cm^{-3} and room temperature Hall mobilities higher than 2100 cm^2/Vs.

We present results of the recent optical studies of the interband photoluminescence (PL) and absorption spectra of n-InN samples with Hall concentrations from $3.6*10^{17}$ to $6*10^{18}$ cm^{-3}. The well resolved structure consisting of three peaks was observed in the PL spectra of the high-quality samples in the energy interval from 0.50 to 0.67 eV at liquid helium and nitrogen temperatures. We attributed one of two low-energy features of the spectra to the recombination of degenerate electrons with the holes trapped by deep acceptors with a binding energy of E_{da}=0.050-0.055 eV and the other one is attributable to the LO-phonon replica of this band. The higher energy PL peak is considered as a complex band formed by two mechanisms. The first one is related to the transitions of electrons to the states of shallow acceptors with a binding energy of E_{sh}=0.005-0.010 eV and/or to the states of Urbach tail populated by photoholes. The second mechanism contributing to this band is the band-to-band recombination of free holes and electrons. Relative intensities of two higher energy PL peaks were found to be strongly dependent on temperature and excitation power.

Properties, Optical, Electronic II: F. Bechstedt/A. Yamamoto

Wednesday afternoon, 7 September, 14:00
Main Building, room 134

14:00 invited oral

Stoichiometry related point defects in InN

K. Scott A. Butcher, Patrick P. Chen, Marie Wintrebert-Fouquet

Macquarie University, Sydney 2109, Australia

Contact: sbutcher@ics.mq.edu.au

The Moss-Burstein effect has been invoked by a number of groups to explain variations in the band-edge absorption for InN samples of different carrier concentration. However, the variation of band-edge values reported in the literature is simply too broad to be explained solely by this effect. At least 5 separate apparent Moss-Burstein trends have been reported over the years [1-5], these trends appear to be related to the different methods of film growth used. We have examined samples that fall along one of the recently reported lines and find that it is dominated by a particular type of defect, and further suggest that the variations observed in the absorption properties of materials nominally referred to as InN are largely determined by stoichiometry related point defects.

[1] B. R. Natarajan, A.H. Eltoukhy, J. E. Greene and T. L. Barr, Thin Solid Films, 69 (1980) 201.

[2] T. L. Tansley, and C. P. Foley, J. Appl. Phys. 59 (1986) 3241.

[3] V. Yu Davydov, A. A. Klochikhin, V. V. Emtsev, S. V. Ivanov, V. V. Vekshin, F. Bechstedt, J. Furthmuller, H. Harima, A. V. Mudryi, A. Hashimoto, A. Yamamoto, A. J. Aderhold, J. Graul, E. E. Haller, Phys. Stat. Sol. B, 230 (2002) R4.

[4] W. Walukiewicz, S. X. Li, J. Wu, K. M. Yu, J. W. Ager III, E. E. Haller, Hai Lu, W. J. Schaff, J. Crystal Growth 269 (2004) 119.

[5] B. Maleyre, O. Briot, S. Ruffenach and B. Gil, Phys. Stat. Sol. C 2 (2005) 1379.

14:30 oral

Recombination processes with and without momentum conservation in degenerate InN

Evgenia Valcheva[1], S. Alexandrova[2], S. Dimitrov[1], H. Lu[3], William J. Schaff[3]

1. *Sofia University, Dept. of Solid State Physics and Microelectronics, 5 J. Bourchier blvd., Sofia 1164, Bulgaria* **2.** *Institute of Solid State Physics, Bulgarian Academy of Sciences (ISSP-BAS), 72, Tzarigradsko Chaussee blvd., Sofia 1784, Bulgaria* **3.** *Cornell University, Electrical and Computer Engineering, Ithaca, NY 14853-540, United States*

Contact: epv@phys.uni-sofia.bg

The evaluation of InN fundamental properties and parameters like band gap, effective mass values, phonon modes, etc., of both poly- and monocrystalline InN layers is a subject of per-

manently growing number of reports. Photoluminescence experiments are widely used to investigate the nature of the recombination processes. Applied to high concentration material the experiment is usually used for the determination of the effective optical band-gap assuming band filling and renormalization effects.

We report on a theoretical approach in which two cases of recombination in optically excited high concentration n-InN are considered: an electron occupying a conduction state can only decay to a valence state with the same wave vector, and the other, in which the probability is essentially independent of the wave vectors of the two states (no momentum conservation). The latter is applicable when the donors have random distribution in the real space. For high impurity concentrations the periodicity of the lattice is perturbed and the momentum conservation is partly lifted so that all available carriers can contribute to the total radiative recombination. The calculations are used to fit emission spectra of unintentionally doped ($\sim 1\times10^{18}$ -1×10^{19}cm^{-3}) InN layers studied in the temperature range 10-300K with excitation wavelength of 488 nm and recorded with PbS detector. The spectra peak at around 0.7eV and can be well fitted disregarding the momentum conservation law.

14:45 oral

Superconductivity of InN

Takashi Inushima

Department of Electronics, Tokai University, Hiratsuka, Kanagawa 259-1292, Japan

Contact: inushima@keyaki.cc.u-tokai.ac.jp

When we reported the observation of the superconductivity of InN in 2000 for the first time, we could not discuss its mechanism because we could not regard the superconductivity as a fundamental property of InN. At that time it was well known that the crystal growth of InN was very difficult and a minor change of the growth condition would easily produce a metal-In phase in InN. In the succeeding experiments, however, we made clear that neither the metal-In phase nor the In precipitation was the cause of the superconductivity.

We, then, considered that there was an optimum carrier density for the occurrence of the superconductivity. The scenario was as follows; when the lattice imperfection becomes large enough, but not too much, to produce high carrier density, a metal-In network will form in InN and produce superconductivity originating from the metal In; when the carrier density becomes small enough to produce a clear Fermi sphere of a degenerate semiconductor, the superconductivity will vanish. This scenario, however, was found to be wrong in the recent experiments done at GHMFL.

What is still obvious is that InN of small carrier density and with a well-defined Fermi surface shows superconductivity, and neither the electron density nor the crystal quality is the main cause of the superconductivity. We also know that the superconductivity is of the second kind and the upper-critical field determined from the magnetic field dependence of zero-resistance temperature is disturbed by the thermal and quantum fluctuations of the vortex, which suggests that the coherent length is short and the penetration depth is long. Neither the surface electron accumulation layer nor the metal-In precipitation has any contribution to the superconductivity.

Following these results, we propose a mechanism of the superconductivity of InN based on the interaction between the electrons with special distribution in wurtzite structure.

15:00 invited oral

Quantized Electron Accumulation, Inversion Layers and Fermi Level-Stabilization in Indium Nitride

Tim D. Veal[1], L. F. J. Piper[1], Chris F. McConville[1], M. H. Zareie[3], Matthew R. Phillips[3], Hai Lu[2], William J. Schaff[2]

1. *University of Warwick, Department of Physics, Gibbet Hill Road, Coventry CV4 7AL, United Kingdom* **2.** *Cornell University, Electrical and Computer Engineering, Ithaca, NY 14853-540, United States* **3.** *University of Technology (UTS), PO Box 123, Broadway, NSW 2007, Sydney, Australia*

Contact: Tim.Veal@physics.org

The large size- and electronegativity-mismatch between cation and anion in InN results in the conduction band minimum lying far below the universal branch point energy (E_B) [1]. This property is the common origin of the phenomena investigated here: the proclivity of InN towards donor-type impurities and defects; the quantized electron accumulation layer at the surface of n-type InN; and the inversion layer at the surface of p-type InN. High energy (1-2 MeV) particle irradiation of InN and InGaN alloys has recently been shown to stabilize the Fermi level at E_B, in agreement with the amphoteric defect model [2]. Here, it is shown that even low-energy (400 eV) N-ion bombardment and annealing at 550 K results in the formation of donor-type defects that significantly increase the electron density in the top ~15 nm of the InN. This stabilizes the Fermi level at E_B in the near-surface region, resulting in no surface space-charge layer. Conversely, at the surface of undamaged InN, electron accumulation layers exist. Meanwhile, tunnelling spectroscopy of such native electron accumulation at the surfaces of n-type InN reveals that these layers constitute a quantized two-dimensional (2D) electron gas system. The tunnelling spectra show structures reflecting the 2D electronic subbands in the surface quantum well. The tunnelling spectra are compared with calculations of the quantum well potential, the subband energies, and the charge-profile. Lastly, calculations of the properties of inversion layers at the surfaces of p-type InN are compared with

experimental data from Mg-doped InN.

[1] I. Mahboob, T. D. Veal, C. F. McConville, Hai Lu, W. J. Schaff, J. Furthmüller, and F. Bechstedt, Phys. Rev. B 69, 201307(R) (2004).

[2] S. X. Li, K. M. Yu, J. Wu, R. E. Jones, W. Walukiewicz et al., Phys. Rev. B 71, 161201(R) (2005).

Growth I: E. Yoon/P. Specht

Wednesday afternoon, 7 September, 15:50
Main Building, room 134

15:50 invited oral

Growth and properties of InN, InGaN, and InN/InGaN quantum wells

Yasushi Nanishi[1], Hiroyuki Naoi[2], Tsutomu Araki[3], Masahito Kurouchi[4], Daisuke Muto[5], Takao Miyajima[6]

1. *Department of Photonics, Ritsumeikan University, Kusatsu 525-8577, Japan* **2.** *Center for Promotion of the COE Program, Ritsumeikan University, Kusatsu 525-8577, Japan* **3.** *Department of Photonics, Ritsumeikan University, Kusatsu 525-8577, Japan* **4.** *Department of Photonics, Ritsumeikan University, Kusatsu 525-8577, Japan* **5.** *Department of Photonics, Ritsumeikan University, Kusatsu 525-8577, Japan* **6.** *Optoelectronics Laboratory, Materials Laboratories, Sony Corporation, Atsugi 243-0014, Japan*

Contact: hinaoi@se.ritsumei.ac.jp

InN and related alloys are very attractive materials for future photonic and electronic devices. Extensive studies on InN and In-rich $In_xGa_{1-x}N$ have been hindered for a long time by the difficulties of growing high-quality crystals because of high vapor pressure of nitrogen and low dissociation temperature. Recent developments of high-quality InN by MOVPE and MBE, coupled with discussion on true band-gap energy of InN, revived extensive attentions on this material system.

We already reported high-quality InN growth by RF-MBE. Comprehensive characterization of the grown films using XRD, TEM, EXAFS and Raman scattering clearly demonstrated that InN films grown in this study had ideal hexagonal wurtzite structure. We also reported successful growth of $In_xGa_{1-x}N$ films in full compositional range without noticeable phase separation.

In this presentation, we will report on our recent developments of InN and $In_xGa_{1-x}N$ growth. We have found that the insertion of a high-temperature-grown InN layer as a growth template was very effective for improving the surface morphology and crystalline quality of In-rich $In_xGa_{1-x}N$ layers. We have also succeeded in dramatically improving the crystalline quality of InN films by optimizing nitridation conditions of (0001) sapphire substrates. These high-quality InN films showed their excellent c-axis orientation with the FWHMs of (0002) XRCs as narrow as 1 arcmin without deteriorating their a-axis orientation. By using these high-quality InN layers as growth templates, the crystalline quality of $In_{0.8}Ga_{0.2}N$ layers was dramatically improved. Based on these studies, an InN/$In_{0.8}Ga_{0.2}N$ multiple quantum well structure and InN/$In_{0.8}Ga_{0.2}N$ single quantum well structures with various well widths were successfully grown on these high-quality InN templates. The MQW structure showed clear 1st and 2nd satellite peaks of XRD. The SQW structures exhibited photoluminescence emission from their well layers.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

16:20 oral

InGaN and InAlN alloys grown in the entire composition range by plasma assisted molecular beam epitaxy

Eleftherios Iliopoulos[1], Alexandros Georgakilas[1], Emmanouil Dimakis[1], Adam Adikimenakis[1], Katerina Tsagaraki[1], Maria Androulidaki[1], Nikolaos T. Pelekanos[2]

1. *Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology-Hellas, and University of Crete, Physics Department (MRG), Heraklion 71110, Greece* **2.** *Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology-Hellas, and University of Crete, Department of Materials Science and Technology, Heraklion, Greece*

Contact: iliopoul@physics.uoc.gr

InGaN and InAlN ternary alloy films, spanning the entire composition range, were grown successfully by radiofrequency plasma assisted molecular beam epitaxy and their properties were investigated by high resolution x-ray diffraction (HR-XRD), photoluminescence (PL), optical transmission and reflection spectroscopies.

Phase separation was fully suppressed kinetically in the RF-MBE grown films. XRD spectra exhibited well defined, narrow single diffraction peaks in all cases. However, growth of high structural quality thick films proved difficult for the case of InAlN with compositions close to 50% AlN.

In the case of InGaN films the determined in-plane biaxial strain exhibited a change of sign, from positive (compressive) to negative (tensile) for compositions larger than 60% InN and reached the value of 2.6×10^{-3} for 90% InN, a value close to the one measured for InN films grown under similar conditions.

Optical bandgap data of InGaN films were fitted well in the entire composition range with a constant value of bowing coefficient, equal to 2.4 eV. On the other hand, InAlN exhibited a dependence of the bowing coefficient on the AlN composition. In both cases the results were extrapolated to an InN bandgap between 0.9 and 1.0 eV, which is higher than the 0.7 to 0.8 eV value of the photoluminescence observed in our InN

samples.

16:35 invited oral

RF plasma sources for III-nitrides growth: influence of operating conditions and device geometry on active species production and InN film properties

Phillip A. Anderson[1], Roger J. Reeves[2], Steven M. Durbin[1]

1. *Department of Electrical and Computer Engineering, The MacDiarmid Institute for Advanced Materials and Nanotechnology, University of Canterbury, Christchurch, New Zealand, Christchurch, New Zealand* **2.** *Department of Physics and Astronomy, The MacDiarmid Institute for Advanced Materials and Nanotechnology, University of Canterbury, Christchurch, New Zealand, Christchurch, New Zealand*

Contact: paa24@student.canterbury.ac.nz

RF plasma sources are now the active nitrogen source of choice for III-nitrides growth by molecular beam epitaxy. One of the key process controls for III-nitrides film growth is the III:N flux ratio. Particularly for GaN growth, this has been shown to have a critical bearing on the morphology and electronic properties of films. However, due to the combination of species emitted from an RF plasma source, these ratios can be difficult to interpret. Active nitrogen species can include neutral and ionic forms of both atomic and excited molecular nitrogen. Factors which can influence which species are produced and in what quantity include, flow rate and RF power, as well as device features such as the aperture plate and cavity design. In this study we monitor the optical emission from an Oxford Applied Research HD-25 RF source at various powers and flow rates in the 500 – 900 nm range. Indium nitride films grown on GaN buffers atop sapphire substrates were grown under a range of plasma operating conditions to investigate the effect of active nitrogen species on film properties. Hall effect measurements revealed a carrier concentration of 5×10^{18} cm^{-3} for films grown where molecular nitrogen transitions dominated the spectrum as opposed to 5×10^{19} cm^{-3} when atomic nitrogen dominated. In-situ monitoring by reflection high energy electron diffraction showed that the a plane lattice constant relaxed fully within 2 nm of growth for high atomic flux, but does not fully relax until films are approximately 30 nm thick for high molecular flux. Emission spectra from the HD-25 are compared to similar measurements from the Veeco UNI-Bulb. It is found that both sources produce relatively more atomic nitrogen at high RF powers and low flow rates. However, for given operating conditions the HD-25 is found to produce a higher content of atomic and 2nd – positive series excited molecular nitrogen than the UNI-Bulb.

17:05 oral

Properties of MBE-grown InN (0001) films

Emmanouil Dimakis[1], Eleftherios Iliopoulos[1], Katerina Tsagaraki[1], Gladkov Peter[3], Philomela Komninou[2], Andreas Delimitis[2], Thomas Kehagias[2], Alexandros Georgakilas[1]

1. *Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology-Hellas, and University of Crete, Physics Department (MRG), Heraklion 71110, Greece* **2.** *Dept. of Physics, Aristotle University of Thessaloniki, Thessaloniki 54 124, Greece* **3.** *Institute for Radio Engineering and Electronics, Czech Academy of Sciences, Prague, Czech Republic*

Contact: dimakis@physics.uoc.gr

In-face InN films were grown heteroepitaxially on (0001) GaN/Al_2O_3 templates by radio-frequency plasma-assisted molecular beam epitaxy (RF-MBE). Films' properties were investigated by high resolution x-ray diffraction, photoluminescence, atomic force microscopy, transmission electron microscopy, scanning electron microscopy and Hall effect measurements.

A physical model of InN growth by RF-MBE is presented and the optimization of InN growth conditions is discussed in the model's context. High quality InN films were grown under optimized conditions. They exhibit a step-flow morphology with surface rms roughness as low as 0.20 nm, x-ray diffraction rocking curve linewidths of approximately 350 and 300 arcsec for the symmetric (0004) and the asymmetric (10-15) reflections, respectively, and electron mobilities in the range of 1500 $cm^2/V \cdot sec$ for carriers concentration in the low 10^{18} cm^{-3}.

The c and α lattice parameters were dependent on the growth conditions. The c versus a relation exhibited a linear behavior indicative of the presence of biaxial strain. From the data a Poisson ration equal to 0.42+-0.05 was determined for the case of InN.

All samples exhibited intense photoluminescence with clear evidence of the Burstein-Moss effect. Transitions to acceptor states were identified in several samples. The intensity of luminescence depended on the growth conditions and it was enhanced of the case of films grown with a two-step process and with near stoichiometric III/V flux ratio.

Electron microscopy observations revealed the abruptness of the InN/GaN interface and the relaxation of most of the misfit stress by a network of misfit dislocations at the InN/GaN interface. Determination of lattice constants and misfit dislocation periodicity were in agreement with the X-ray measurements.

17:20 invited oral

MOVPE growth of InN on Sapphire

Wolfgang Richter[1], Massimo Drago[1], Udo W. Pohl[1], Christoph Werner[1], Patrick Vogt[1], Norbert Esser[2], Markus Pristovsek[1]

1. *Berlin University of Technology, Hardenbergstr. 36, Berlin 10623, Germany* **2.** *Institute for Analytical Sciences (ISAS), Albert-Einstein-Str. 9, Berlin 12489, Germany*

Contact: massimo@gift.physik.tu-berlin.de

Today, the challenge for InN is the growth of epitaxial materials with low defect density and high electron mobility. MBE achieved first good layers in 2003 (Shaff et al.), but recently also MOVPE has shown encouraging results (Briot et al., 2004). But even then, the necessity of a material with uniform properties is reflected in the high dispersion of InN bandgap values proposed up to now. Good crystal quality is also the prerequisite for the fabrication of promising InN based electronic devices. Sapphire is presently the most used substrate for both epitaxial techniques, due to the experience gained in GaN epitaxy during the last 15 years. It is well known, for both GaN and InN, that sapphire nitridation is a necessary step to improve the layer quality. Since InN peculiarities require a careful and dedicated study, the effects of different sapphire nitridation procedures on the properties of InN epitaxial layer will be the focus of this presentation.

In-situ spectroscopic ellipsometry (SE) is used here for the first time to reveal and control the nitridation dynamics of sapphire in MOVPE. Sapphire nitridation is performed at 1050C with ammonia using nitrogen as carrier gas. SE identifies two distinct process-steps occurring during sapphire nitridation: (i) a fast surface reaction occurring within the first 30s and thereafter (ii) a slow nitridation reaction into the sapphire bulk with a simultaneous roughening of the surface. A set of InN samples was then prepared using the same growth procedure but with different nitridation times for the sapphire substrates. Our growth procedure involves high temperatures (580C) and low V/III ratio (NH_3/TMIn: 10000) resulting in growth rates as high as 350 nm/hour and in layers with specular surfaces. The InN samples were characterised ex-situ with respect to their electronic and crystallographic properties. The optimal nitridation times coincide with the completion of the fast surface reaction (30s).

Thursday, 8 September

Growth II: M.A. Poisson/A. Doolittle
Thursday morning, 8 September, 9:00
Main Building, room 134

9:00 invited oral

Inhomogeneities in MOVPE InN

Akio Yamamoto[1], Hiroshi Miwa[1], Wen Jun Wang[2], Akihiro Hashimoto[1]

1. *University of Fukui (U. Fukui), 3-9-1 Bunkyo, Fukui 910-8507, Japan* **2.** *University of Fukui, Venture Business Laboratory (U. Fukui, VB), 3-9-1 Bunkyo, Fukui 910-8507, Japan*

Contact: yamamoto@fuee.fukui-u.ac.jp

This paper reports in-depth and in-plane inhomogeneities in MOVPE InN studied using a different kind of PL measurement. Samples of InN are grown on sapphire substrates without or with a GaN buffer, using an atmospheric-pressure MOVPE system. The lowest carrier concentration and the highest mobility obtained for grown films are 4.5×10^{18} cm^{-3} and 1100 cm^2/Vs, respectively. For the in-depth inhomogeneity analysis, the macroscopic PL measurement is made from both the front surface and the film/substrate interface sides of an InN film using an excitation source with a different wavelength (325, 442 and 633 nm). PL peak energy near the front surface is almost independent of the excitation wavelength, implying that the property near the front surface is not changed with depth. PL peak energy from the interface side is higher than that near the front surface and is markedly increased with decreasing the excitation wavelength. Especially, a sample grown without buffer and then annealed in an NH_3 flow shows a higher peak energy by about 100 meV than those measured from the front side. Such a higher PL peak energy is due to the Burstein-Moss shift and shows the existence of a higher carrier concentration region near the interface. In-plane inhomogeneity of samples is studied using the Scanning Near-Field Optical Microscopy (SNOM) system (Type NFS-220FK, JASCO Corp., Japan). Employing the illumination-collection mode with a fiber probe of a 100-500 nmφ aperture size, a near-field PL spectrum and its intensity mapping are successfully obtained for InN films at room temperature. By monitoring a probe movement at each point of measurement, surface topography is also obtained. Inhomogeneities detected by the near-field PL mapping are dependent on the growth conditions of samples. Especially, the use of the buffer is found to improve effectively the in-plane uniformity of the near-field PL intensity. This seems to be due to the uniform nucleation of InN on the buffer.

9:30 invited oral

InN Polycrystalline Films: Growth, Structure and Optical Characterization

Vladislav Y. Malakhov

Institute for Problems of Materials Science, 3, Krzhizhanovsky, Kyiv 03142, Ukraine

Contact: vlad.malakhov@ipms.kiev.ua

The present survey deals with some results of researches of InN polycrystalline films which the author has obtained during last 30 years.

And though recent discovery of single crystalline layers of hexagonal InN have already changed our representations about bandgap of this compound having transformed it from wide bandgap to narrow one semiconductor, nevertheless many fundamental parameters have not under-gone essential changes but that have changed still demand to be understood and explained. In the paper arguments pro and contra concerning possible revision of the fundamental gap value of InN are resulted. On a basis of long-term experience of growing InN polycrystalline films and also the carried out complex researches of both optical and electronic properties of nitride films, the debatable issues are considered in connection with discovery of the narrow InN bandgap.

10:00 invited oral

Single crystalline InN narods by H-MOVPE

Olga Kryliouk, Hyun Jong Park, Joshua Mangum, Tim Anderson

University of Florida (UF), 227 Chemical Engineering Bldg., Gainesville, FL 32611, United States

Contact: olgak@grove.ufl.edu

Nanomaterials are experiencing a rapid development in recent years due to their exciting potential applications in different areas. InN has been investigated intensively as a constituent of $Ga_xIn_{1-x}N$ and $Al_xIn_{1-x}N$ which has been used as active layer in optoelectronic and electronic devices for over a decade. Although InN itself is still less studied material compare to GaN and AlN, the interest in the InN has been increased remarkably. The optical characterization of improved wurtzite InN crystals, although still a subject of debate, has a reported band gap in the range 0.65 to 0.9 eV. This band gap energy is considerably lower than the previously accepted value (Eg = 1.9 eV), and thus some additional applications of InN, such as solar cell and infrared laser diode, are possible. The addition of Ga (Eg = 3.4 eV) makes it possible to create a band gap range of 0.7 to 3.4 eV, which covers virtually the entire solar spectrum.

InN nanorods were grown by H-MOVPE technique. This technique is promising for the development of high efficiency III-Nitrides-based nanosize optoelectronic and electronic devices. Growth of InN using MOVPE has several challenges. The growth temperature is limited to the range of 450~650 °C because of the high vapor pressure of nitrogen over InN and low dissociation rate of NH_3. A high V/III ratio (>10^4) is usually required to prevent In droplet formation at growth temperature. The basic growth parameters, such as growth temperature, V/III and HCl/TMI ratios, and the growth mechanisms were investigated. A novel method was proposed to prevent In droplets formation. Equilibrium thermodynamic calculations of the In-Ga-N-Cl-H system, reaction pathways studies are complemented by experimental work. A wide range of characterization techniques, such as: XRD, AFM, SEM/EDS, TEM, AES, XPS, and PL were employed in this study.

Structure, Miscellanous: C. Kisielowski/S. Wei

Thursday morning, 8 September, 11:00
Main Building, room 134

11:00 oral

Characterization of chrystallographic properties and defects via X-ray microdiffraction in GaN(0001) layers

Rozaliya I. Barabash, G E. Ice[1], C Roder, S. Figge, Sven Einfeldt, Detlef Hommel[2], T. M. Kantona, J. S. Speck, S. P. DenBaars[3], R. F. Davis[4]

1. *Oak Ridge National Laboratory, Oak Ridge, United States* **2.** *University of Bremen, Institute of Solid State Physics, P.O. Box 330440, Bremen 28334, Germany* **3.** *University of California at Santa Barabara (UCSB), Santa Barbara, United States* **4.** *North Carolina State University (NCSU), Raleigh, United States*

Contact: barabashr@ornl.gov

Intrinsic stresses due to lattice mismatch and high densities of threading dislocations and extrinsic stresses resulting from the mismatch in the coefficients of thermal expansion are present in almost all III-Nitride heterostructures. Stress relaxation in the GaN layers occurs in conventional, cantilever (CE) and in pendeo-epitaxial (PE) films via the formation of additional misfit dislocations, domain boundaries, elastic strain and wing tilt. Polychromatic X-ray microdiffraction, high resolution monochromatic X-ray diffraction and finite element simulations have been used to determine the crystallographic properties, distribution of strain, dislocations, sub-boundaries and crystallographic wing tilt in uncoalesced and coalesced GaN layers grown by PE and CE. Tilt boundaries formed in the uncoalesced GaN layers at the column/wing interfaces essentially depend on the width-to-height ratio and growth conditions. Sharper tilt boundaries were observed at the interfaces formed by the coalescence of two laterally growing wings. The wings tilted upward during cooling to room tem-

perature for both the uncoalesced and the coalesced GaN layers. It was determined that finite element simulations that account for extrinsic stress relaxation can explain the experimental results for uncoalesced GaN layers. Relaxation of both extrinsic and intrinsic stress components in the coalesced GaN layers contribute to the observed wing tilt and the formation of sub-boundaries.

11:15 oral

Effects of Si doping position on the emission energy and recombination dynamics of GaN/AlGaN multiple quantum wells

Hamid Haratizadeh[1], Bo Monemar[2], H. Amano[3]

1. *Shahrood University of Technology, University Bolvd., Shahrood 3619995161, Iran* **2.** *Linköping University, Dept. of Physics and Meas. Techn., Linkoping, Sweden* **3.** *Department of Electrical and Electronic Engineering, Meijo University, Shiogamaguchi, Tempaku-ku, Nagoya 1-501, Japan*

Contact: hamha@walla.com

The effects of Si doping on the emission energy and recombination dynamics in a set of GaN/Al0.07Ga0.93N multiple quantum wells (MQWs) samples with different position of the dopant layer were studied by means of photoluminescence (PL) and time-resolved PL measurements. When the doping is in the barrier and in both barrier and well, the MQW emission appears above the GaN bandgap, while the sample doped in the well shows a redshifted emission. The redshift is attributed to the self-energy shift of the electron states due to the correlated motion of the electrons exposed to the fluctuating potential of the donor ions. At low temperature a longer PL decay time, 760 ps, was measured for the sample doped in the well, a factor two longer than for the barrier doped case. The difference is explained by the effect of interplay of free carriers and ions on the screening of the polarization field in these doped structures.

PACS: 78.47.+p, 78.55.Cr, 78.67.De

[ABSTRACT TRUNCATED TO 2000 LETTERS]

11:30 oral

Structural and optical characterization of thick InN epilayers grown on GaN templates by plasma assisted molecular beam epitaxy

Andreas Delimitis[1], Peter Gladkov[2], Thomas Kehagias[1], John Arvanitidis[1], Sotirios Ves[1], Maria Katsikini[1], Emmanouil Dimakis[3], Alexandros Georgakilas[3], Philomela Komninou[1]

1. *Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece* **2.** *Institute for Radio Engineering and Electronics, Czech Academy of Sciences, Prague, Czech Republic* **3.** *Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology-Hellas, and University of Crete, Physics Department (MRG), Heraklion 71110, Greece*

Contact: andel@auth.gr

The structural and optical properties of improved quality thick InN films grown by plasma assisted radio frequency molecular beam epitaxy (rf-MBE) on GaN/Al_2O_3 templates were investigated. The InN epilayers, up to 1.1 μm in thickness, were grown either by a single-step mode at 300 ^{o}C or by a two-step mode, with nucleation at the same temperature and overgrowth at 400 ^{o}C. The films were characterized by High Resolution Transmission Electron Microscopy (HRTEM), temperature dependent photoluminescence (PL) and Raman spectroscopy. At low temperatures the main PL emission was at 0.8 eV and 0.767 eV for samples with single-step and two-step growth mode, respectively. The improved properties of the two-step grown InN were outlined by the characterisation results. Such films exhibited lower threading dislocation density values, higher by a factor of two PL intensity spectra and sharper Raman peaks compared to single-step films. Both types of InN films exhibit tensile residual strain due to thermal origin and/or structural defects present in the epilayer. The amount of the residual strain deduced by the Raman spectra correlates well with that calculated from electron diffraction analysis through the variation of InN lattice constants. The difference in the main emission peak of the PL spectra between the single-step and the two-step samples by 33 meV provides further evidence of the different residual strain present in InN epilayers.

11:45 invited oral

Compositional modulation in the InxGa1-xN layers; relation to their optical properties

Zuzanna Lilental-Weber[1], K.M Yu[1], Sonny X. Li[2], Joel W. Ager III[1], Wladek Walukiewicz[1], Eugene E. Haller[1,2], H. Lu[3], William J. Schaff[3]

1. *Lawrence Berkeley National Laboratory (LBNL), 1 Cyclotron Road, Berkeley 94720, United States* **2.** *University of California, Berkeley 94720, United States* **3.** *Cornell University, 425 Philips Hall, Ithaca, NY 14853, United States*

Contact: z_liliental-weber@lbl.gov

Transmission Electron Microscopy (TEM) and x-ray diffraction (XRD) have been used to study compositional modulation in InxGa1-x N layers grown with compositions close to miscibility gap. The samples ($0.34 < x < 0.8$) were deposited by molecular beam epitaxy using either a 200-nm-thick AlN or GaN buffer layer grown on a sapphire substrate. Periodic compositional modulation leads to extra electron diffraction spots and satellite reflections in XRD in the q-2q coupled geometry. The ordering period D measured along c-axis was about D = 45 Å for x=0.5 and D = 66Å for x=0.78 for samples grown on AlN buffer layer. TEM and XRD determinations of D were in good agreement. Compositional modulation was not observed for the sample with x=0.34 grown on a GaN buffer layer. Larger values of D were observed for layers with higher In content and for those having larger mismatch with the underlying AlN buffer layer. The ordering starts on polar 10-11 planes where we had previously observed formation of V-defects (pinholes) in InGaN. Also, there was a theoretical prediction that In could preferentially accumulate on these polar planes. The possibility that the roughness of the AlN growth surface promotes strong In segregation on particular crystallographic planes leading to compositional modulation is considered.

This study show an extraordinary possibility to grow InGaN layers uniform in composition for high In content (x = 0.34) by MBE, and also the possibility to grow naturally formed thin layers on nanometer scale. The relationship between structural properties, compositional ordering, and optical properties of the In1-xGaxN films, in particular the presence or absence of a Stokes shift between absorption and photoluminescence, will be discussed.

The work in LBNL is supported by the U.S. Department of Energy under contract No. DE-AC03-76SF00098,

Properties, Structure I: Z. Liliental/S. Butcher
Thursday afternoon, 8 September, 14:00
Main Building, room 134

14:00 invited oral

Does electron microscopy produce In clustering in InGaN?

Colin J. Humphreys[1], Tim M. Smeeton[2], Mark J. Galtrey, Jonathan S. Barnard, Menno J. Kappers

1. *University of Cambridge, Department of Materials Science and Nanoscience Centre, Pembroke Street, Cambridge CB2 3QZ, United Kingdom* **2.** *Sharp Laboratories of Europe Ltd., Edmund Halley Road, Oxford OX4 4GB, United Kingdom*

Contact: colin.humphreys@msm.cam.ac.uk

High resolution electron micrographs (HREM) of InGaN/GaN quantum wells from many research groups appear to demonstrate very indium-rich clusters on a nanometer scale and PL results suggest that the excitons in InGaN quantum wells have a localisation of about 2 nm. However, we have studied bright blue and green LEDs grown by our Thomas Swan MOCVD reactor and from elsewhere, and our HREM results using a very low intensity electron beam show no evidence for such strong indium clustering. However, nanometre-scale strain inhomogeneities are introduced into the quantum wells by radiation damage very soon after exposing the material to the electron beam. The image features caused by the radiation damage are similar to those expected to be caused by indium-rich regions so we therefore suggest that reports of indium clustering may be influenced by electron beam damage artefacts. We find that in each quantum well the lower InGaN/GaN interface is atomically smooth, but that the upper interface has atomic height steps, and the lateral scale of these steps is on a nanometer scale. Because of the strong piezoelectric effect in GaN, an atomic size step in an InGaN quantum well is sufficient to cause exciton localisation. It is suggested that this may be the mechanism, or a contributory mechanism, to the exciton localisation observed in InGaN/GaN quantum wells.

14:30 invited oral

Low and high indium fluctuation in MOCVD grown InGaN/GaN as determined by quantitative HRTEM

Pierre Ruterana[1], Protima Singh[1], Florence Gloux[1], Slawomir Kret[2], E K. Suh[3], Pawel Dluzewski[4], Euijoon Yoon[5]

1. *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **2.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **3.** *ChungJu University, ChungJu, Korea, South* **4.** *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT PAN), Świętokrzyska 21, Warszawa 00-049, Poland* **5.** *Seoul National University (SNU), School of Mat. Sci. Eng., Seoul 151742, Korea, South*

Contact: pierre.ruterana@ensicaen.fr

The knowledge of local strain fields associated with defects is important for the determination of the local atomic configuration. This is particularly the case in GaN based heterostructures where densities of defects are large, and the correlation between properties and structure needs to be understood. Image processing and image simulations help to extract quantitative information. In this work, we discuss our results on InGaN/GaN quantum wells. For the GaN/InxGa1-xN/GaN QW's, the mechanism of the light emission is not yet clearly understood. Narukawa et al. suggested that indium rich cluster may help to localise the carriers and will be a centre for radiative recombination. Using pattern recognition on HRTEM images, Kisielowski et al. have shown that the indium may segregate into 1-3 nm diameter areas inside the QW. Ruterana et al., based on a strain distribution extracted from HRTEM images and 2D FEM modelling, pointed out that the In composition fluctuation areas may depend on the growth technique. In this work, two types of samples were investigated, in the first, the measured strain was always high, the thickness of the wells being in the 3-5 nm range. The second set of samples was prepared as pure InN/GaN quantum wells, with thicknesses in the range of a few monolayers, we find that the exctrated local strain does not reflect the nominal composition in these structures and we suggest the origin of the discrepency. In the two types of structures, the In composition fluctuations are clearly pointed out from the HRTEM observations. By a combination of 3D FEM and HRTEM observation, we show that the observed samples exhibit indium composition fluctuation. In many instances, the indium content can reach x=1 in the clusters inside the core. In these MOCVD QWs, we attempt to connect the Quantum dot density, composition, and shape, to the growth conditions.

15:00 invited oral

Quantitative Electron Microscopy of the InN-GaN Ternary Alloy System

Christian Kisielowski

Lawrence Berkeley National Laboratory (LBNL), 1 Cyclotron Road, Berkeley 94720, United States

Contact: CFKisielowski@lbl.gov

Over the last years quantitative Electron Microscopy of InxGa1-xN quantum wells and thin films contributed significantly to the development of electronic and optoelectronic devices that find numerous applications. The significant size difference between the indium (atomic radius = 0.20 nm) and gallium atoms (atomic radius = 0.18 nm), however, is known to induce substantial strain during growth of layered structures resulting into an indium distribution that can be inhomogeneous on a nanometer scale. Obviously, it is of concern that the observation of the material using high energetic electron beams can alter the as-grown structures and stimulate a redistribution of the chemically different elements. This contribution summarizes experiments that address such concerns. They range from refined sample preparation techniques to time resolved experiments that are quantitatively analyzed. It will be shown that experiments can be performed such that quantitative High Resolution Transmission Electron Microscopy, Scanning Transmission Electron Microscopy, and Electron Spectroscopy can be applied to produce a consistent set of data with no indication of beam induced damage. In contrast evidence will be given that the material exhibits spinodal decomposition. It is argued that this process influences the formation of indium rich clusters.

Properties, Structure II: D. Smith/H. Naoi

Thursday afternoon, 8 September, 15:50
Main Building, room 134

15:50 invited oral

Finite element modelling of nonlinear elastic and piezoelectric properties of InN and InGaN QDs

Paweł Dłużewski[1], Grzegorz Jurczak[1], Pierre Ruterana[2,3], S. P. Lepkowski[4]

1. *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT PAN), Świętokrzyska 21, Warszawa 00-049, Poland* **2.** *Laboratoire CRISMAT - UMR 6508, ISMRA et Universite de Caen, 6 Boulevard de Marechal JUIN, Caen 14050, France* **3.** *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **4.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: pdluzew@ippt.gov.pl

We investigate elastic and electric properties of wurtzite InGaN/GaN heterostructures with inhomogeneous indium composition. Distortion field and continuous fluctuation of chemical composition extracted by means of digital processing of the HRTEM images are used as the input data for the nonlinear FE calculations. This data are input to FE mesh in the form of nodal variables for extended nodal freedom degrees taking into account the fixed: nodal source distortions, chemical distribution and electric charge distribution as well as the wanted: nodal displacements and electrostatic potential distribution. To model a whole considered crystal region we use 27-node (brick) elements with quadratic shape functions assuring the compatibility condition between coupled fields (source distortions, displacements, chemical composition and electrostatic potential). This is an essential condition to avoid the extra effects including artificial residual stresses and caused by FE incompatibilities. We solve a boundary-value problem for piezoelectricity to calculate residual stresses and strains existing in a such heterostructure for the given (fixed) electric charge distribution. We obtain also the resulting electrostatic potential distribution. To do this we use finite element method based on nonlinear anisotropic piezoelectricity. During calculation we take into account piezoelectric polarization as well as the spontaneous polarisation of the wurtzite crystals.

16:20 invited oral

Strain state analysis of InGaN/GaN – sources of error and optimized imaging conditions

Andreas Rosenauer

University of Bremen, Institute of Solid State Physics, P.O. Box 330440, Bremen 28334, Germany

Contact: rosenauer@ifp.uni-bremen.de

Enormous progresses regarding the growth of group-III nitrides were achieved in the past years and commercial InGaN-based light emitting diodes and laser diodes have been realised. However, kinetic growth effects such as phase separation and segregation governing epitaxial growth of InGaN quantum wells are still not understood well enough.

In InGaN/GaN the local lattice parameter in growth direction can be used to obtain the local In-concentration. However, recent investigations e.g. by Smeeton et al. showed that InGaN layers are extremely sensitive to the electron beam in the microscope and that strain fluctuation form after a couple of minutes. Therefore, methods of composition determination that require acquisition of defocus series or precise adjustment of imaging conditions at the area under investigation cannot be used. In contrast, imaging conditions are needed that require minimum adjustment and which allow an accurate but sufficiently quick measurement of the strain distribution in the specimen.

In our contribution, we will show that strain state analysis in $In_xGa_{1-x}N$/GaN using a three- or two-beam condition can lead to substantial artefacts. We evaluated simulated images in dependence of specimen thickness, specimen orientation and objective lens defocus. We observed that the measurement is in agreement with the true strain profile for certain conditions only. An analysis of error sources revealed, that artefacts are mainly caused by a combination of delocalization and the composition dependence of the phases of the beams contributing to the image formation. Due to the delocalization effect, interference of the undiffracted beam with an 0002 beam works best. The chemical shift of the phases is minimized by using the 000+2 beam and a strong excitation of 000+4. Images simulated for these conditions taking into account lattice plane bending and strain using finite-element calculations revealed a good agreement of evaluated and true strain profiles.

Devices: C. Humphreys/C. Wood

Thursday afternoon, 8 September, 16:50
Main Building, room 134

16:50 invited oral

III-Nitrides semiconductor compounds for microwave devices

Marie-Antoinette di Forte-Poisson

Alcatel-Thales III-V lab (ATL III-V), Domaine de Corbeville, Orsay 91404, France

Wide Band Gap semiconductors, such as SiC and GaN, exhibit many attractive properties far beyond the capabilities of Si and GaAs. GaAlN/GaN high electron mobility transistors (HEMTs) with very impressive power densities up to 11.2 W/mm at 10 GHz have been reported by Cornell, and recently up to 30 W/mm at 4 GHz by Cree Research. This rapid progress in the performance of microwave power transistors, as compared to results published in early 1998 (2 W/mm), has been obtained thanks to the use of SiC substrates instead of sapphire. In this paper, we report on a comparative study of the physical properties of GaAlN/GaN HEMT structures grown respectively on sapphire and silicon carbide substrates . The critical steps of the MOCVD growth process of GaN on SiC (substrate surface preparation, nucleation layer composition and growth parameters) are described and their effects on the physical properties of the HEMT structures and the associated device performances will presented.

On the other hand, the GaAlN/InGaN/GaN double heterostructure, in which the channel layer is made of InGaN instead of GaN, is expected to significantly enhance device performance due to a stronger carrier confinement and a higher two-dimensional electron gas (2DEG) density at the interface in comparison with an GaAlN/GaN single heterostructure. However a strong alloy disorder scattering in GaInN and a significant interface-roughness scattering of the GaAlN/

GaInN heterointerface lead to low 2DEG mobility GaInN channel structure as compared to conventional GaN channel structure (Typically 1600 cm2V-1s-1). An improvement of the 2DEG mobility (1100cm2V-1s-1 at RT) has been recently demonstrated by using specific GaInN channel design to suppress alloy disorder scattering [1].

GaInN based channel HEMT heterostructure appears to be promising for microwave applications. A review of the main published results will be presented together with the last GaInN based HEMT developments in our laboratory.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

17:20 invited oral

Indium Nitride: A Material with Photovoltaic Promise and Challenges

William A. Doolittle, Elaissa Trybus, Gon Namkoong, Walter Henderson

Georgia Institute of Technology (GIT), 777 Atlantic Dr., Atlanta 30332-0250, United States

Contact: alan.doolittle@ece.gatech.edu

With the revision of the bandgap of InN down to about ~0.65eV, the III-Nitride material system has been touted as a promising photovoltaic material system. There is clearly potential for new and promising photovoltaic devices. However, several key challenges exist to reach this goal. Issues with mismatched tandem cells creating minority carrier recombination enhancing defects, sufficient p-type doping to demonstrate tunnel junctions, dealing with limited minority carrier diffusion lengths, and many other issues will be addressed.

It will be shown that some of these issues are best addressed by alternative substrate options such as germanium and substantially novel tandem interconnect technologies. Growth issues involving the use of Ge substrates will be identified and data for InN on Ge will be detailed.

Finally, the structural and optical behavior of InN on sapphire and Ge for various buffer topologies and oxygen content will be described. It will be shown that there can be, depending on buffer configurations used, significant evolution of the InN film defect structure with increasing thickness. This behavior is much more pronounced than in other III-Nitride materials and unfortunately is counter to the properties needed for good tandem solar cell performance.

Rump Session II: Indium Composition Fluctuations, Devices

Thursday afternoon, 8 September, 17:50
Main Building, room 134

Symposium Reception

Thursday evening, 8 September, 20:00

Symposium B

Multi-component alloys and intermetallic compounds for magnetic applications and nanotechnology

Welcome

Magnetic materials play an essential role in technology. Currently, several categories of magnetic materials are being extensively investigated extensively including soft magnetic materials, hard magnetic materials, magnetoelectronic materials, magnetomechanical materials and magnetocaloric materials. Some materials consisting of rare earth and transition metals are characterized by a large spontaneous magnetization, strong magnetic anisotropy, and a relatively high Curie temperature; some of them also have an extremely large coercivity. Small additions of light elements improve the primary magnetic properties of some rare earth-3d transition metallic compounds. Prospective soft magnetic materials are intermetallics based on light elements with transition metals. Recent investigations have proved that the magnetic characteristics might be significantly improved by using nanoscale materials.

The development of magnetoelectronic materials is important for data storage and sensor technology. Investigations of new ferromagnetic metals as a source of spin-polarized current injectors have also attracted recent attention. Magnetomechanical materials include giant magnetostrictive rare earth-transition metal intermetallics, magnetic shape memory alloys and magnetostructural phase transition intermetallics. Magnetocaloric materials can be used as magnetic refrigerants, even at room temperature, and are highly efficient and environmentally friendly.

A detailed understanding and improvement of all these properties, is impossible without knowledge of their electronic structures, and therefore investigations in this direction are highly justified.

Scope and topics:

- soft magnetic materials
- hard magnetic materials
- magnetoelectronic materials
- magnetomechanical materials
- magnetocaloric materials
- magnetic recording.

Scientific Committee:

D. Fruchart (Grenoble, France), B. Idzikowski (Poznań, Poland), O. Kalogirou (Thessaloniki, Greece), O. Moze (Modena, Italy), D. Niarchos (Athens, Greece), W. Suski (Wrocław, Poland), A. Szytuła (Cracow, Poland), R. Troć (Wrocław, Poland), K. Yvon (Geneva, Switzerland), G. Chełkowska (Katowice, Poland), A. Ślebarski (Katowice, Poland) and W. Zarek (Katowice, Poland)

Invited lecturers:

O. Bodak (Ivan Franko L'viv National University),"Crystal-chemical approach in search of new rare-earth intermetallics for magnetic applications"

R. Černý (University of Geneva),
"Structure of 2:17 compounds in Yb-Fe-Al system"

D. Fruchart (CNRS Grenoble),
"Magnetocaloric effect in d-metal phosphides and arsenides"

O. Kalogirou (Aristotle University of Thessaloniki),
"Influences of Co on structural and magnetic properties of $R_3(Fe_{1-x}Co_x)_{29-y}M_y$ (R = rare earth metal, M = transition metal) intermetallic compounds"

O. Moze (Univesity of Modena),
"Small angle neutron scattering investigation of spin disorder in nanocomposite soft magnets"

R. Troć (ILT SR Polish Academy of Science, Wrocław),
"Magnetoresistivity studies of intermetallic compounds and their impact on the understanding of exhibited phenomena"

A.Ślebarski (University of Silesia, Poland),
"Half-metallic ferromagnetic ground state in CePdSb"

Organisers

- Ewa Talik, University of Silesia, Poland
- Adam Guzik, University of Silesia, Poland
- Jan Heimann, University of Silesia, Poland
- Oksana Bodak, Ivan Franko National University of Lviv, Ukraine

Proceedings

Papers will be published in Journal of Alloys and Compounds.

Acknowledgements

Symposium is supported by:
Rector of University of Silesia in Katowice
Director of Institute of Physics, University of Silesia
Scientific Network MAG-EL-MAT
Polish Society of Crystal Growth

Programme

Monday, 5 September

Parallel Session
Monday afternoon, 5 September, 14:00
Main Building, room 219
Daniel Fruchart presides

14:00 invited oral

CRYSTAL-CHEMICAL APPROACH IN SEARCH OF NEW RARE-EARTH INTERMETALLICS FOR MAGNETIC APPLICATIONS

Oksana I. Bodak

Ivan Franko Lviv National University, Department of Inorganic Chemistry (LNU), Kyryla i Mefodiya str., 6, Lviv 79005, Ukraine

Contact: bodak@franko.lviv.ua

Magnetic systems have occupied the central position for the explanation most of physical phenomena, however, there is no significant progress in the development of new materials for permanent magnets in the last years. The researchers try to improve the properties of the known before phases but do not find the really novel compounds. For example, these are investigations on the R_2Fe_{17}-based solid solutions with Th_2Zn_{17} structure. In other words, progress in magnetism is limited by internal magnetic properties of existing magnetic materials. For the qualitatively new "jump" it is necessary to carry out searching of new compounds with magnetic properties, based on which it is possible to make new materials. It is carrying out insufficiently today.

The efforts necessary to make for adding any new series of magnetic intermetallics must be considered presently. For the successful solution of these tasks it is necessary to create the methodological approach, which must be based on physico-chemical character of component interaction in known binary and ternary systems and, principally, on crystal-chemical analysis – generalization of structure types with unit-axial direction, prediction of phases' formation of these types, etc.

The main idea is searching for a new group of multicomponent compounds based on Fe(Co) and Rare Earths, which have no similarity among known binary and ternary compounds and can be used as basic for new permanent magnets' materials. Despite of the fact that in R-Fe(Co)-X (X – p-element) systems the iron(cobalt)-rich compounds with unit-axial tetragonal $ThMn_{12}$, $BaCd_{11}$ and $CeNi_{8.5}Si_{4.5}$ structures are not known (except for some cases), one can expect an existence of the phases with such structure in multicomponent R-Fe(Co)-X-X` systems. These compounds would possess high-performance magnetic properties with very high level of probability.

14:45 oral

Effect of high-magnetic-field processing on the coercivity of sintered Nd-Fe-B magnets

Hiroaki Kato[1], Takahiro Akiya[1], Masato Sagawa[2], Keiichi Koyama[3], Terunobu Miyazaki[1]

1. *Department of Applied Physics, Tohoku University, Aoba-yama 6-6-05, Sendai 980-8579, Japan* **2.** *Intermetallics Co., Ltd, Kyoto 615-8206, Japan* **3.** *Institute for Materials Research, Tohoku University (IMR), Sendai 980-8577, Japan*

Contact: kato@mlab.apph.tohoku.ac.jp

Sintered Nd-Fe-B magnets are the most promising material for a driving motor of the electric vehicle (EV). In this kind of application, very large value of room-temperature coercivity H_c is required owing to the high-temperature circumstance of the EV-motor. Current Nd-Fe-B magnets commercially available for such high H_c applications, therefore, contains huge amount (~10 wt%) of Dy to enhance H_c. But, this method has a crucial disadvantage. That is, the magnetization must be sacrificed owing to the antiparallel coupling of Dy and Fe moments, resulting in smaller energy product values.

It is well known that a heat treatment around 600 C is indispensable to obtain a high coercivity in the Nd-Fe-B sintered magnets. We have been investigating an effect of high magnetic field in this annealing process on the magnetic properties of Nd-Fe-B sintered magnets. We already reported that the 37% increase of H_c occurs, when the sample containing 3.1 wt% Dy and 0.1 wt% Al was annealed at T_a = 550 C under a magnetic field of H_a = 140 kOe [1]. Here, we report on the high-magnetic-field processing work for a series of Dy-free Nd-Fe-B magnets.

The annealing treatments were carried out for samples with the dimensions 5x5x5 mm^3 by using a furnace installed in a cryocooled superconducting magnets which generate magnetic fields of up to 140 kOe. Six different values of T_a were selected between 500 and 625 C. In the sample containing small amount of Al and Cu, highest H_c value of 14.7 kOe was obtained for T_a = 550 C and H_a =140 kOe. This value of H_c is 16% larger than that of the control sample (T_a =550 C and H_a =0). These results were discussed in terms of a field-induced change in the interface matching between the main $Nd_2Fe_{14}B$ and intergranular Nd-rich phases.

[1] H. Kato, M. Sagawa, K. Koyama and T. Miyazaki, Appl. Phys. Lett. 84 (2004) 4230.

15:15 oral

Directly quenched bulk nanocrystalline (Pr,Dy)-(Fe,Co)-B-Zr-Ti hard magnets

Piotr Pawlik[1], Katarzyna Pawlik[1], Hywel A. Davies[2], Jerzy J. Wysłocki[1], Waldemar Kaszuwara[3], Nicola J. Harrison[2], Iain Todd[2]

1. *Institute of Physics, Częstochowa University of Technology, Al. Armii Krajowej 19, Częstochowa 42-200, Poland* **2.** *University of Sheffield, Department of Engineering Materials, Mappin Street, Sheffield S1 3JD, United Kingdom* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: pawlik@mim.pcz.czest.pl

The addition of 1 at.% of Zr to $(Pr,Dy)_x(Fe,Co)_{80-x}B_{20}$ type alloys (x up to 4.5 at.%) led to significant improvement of their glass forming abilities, so that amorphous 1mm dia. rods and thin walled tubes having outer diameters up to 4 mm could be produced. Subsequent annealing of the amorphous samples resulted in formation of a nanocrystalline structure containing soft magnetic Fe_3B, and the hard magnetic $(Pr,Dy)_2(Fe,Co)_{14}B$ phases. However, the low concentration of the RE elements, together with the detrimental effect of Zr on the magnetic properties, resulted in relatively low values of coercivity $_JH_c$ and of $(BH)_{max}$ for the alloys investigated. Recently, for as-cast Nd-Fe-B-type ribbon samples with Nd contents of ~9 at. % and high boron contents of ~15 at. %, significant coercivities were demonstrated. This has stimulated the present investigation of the processing of hard magnetic nanocrystalline bulk samples with improved magnetic properties. Nanocrystalline Fe-Co-Pr-B-Ti-Zr alloy rods of diameter 1 mm and tubes of outer diameter 3 mm, were produced by die casting. Good enhancement of remanence J_r to values>$0.6J_s$, in combination with unusually high $\mu_0 {}_JH_c$ of ~0.65T, were achieved for samples in the as-cast state. Subsequent annealing of the samples at various temperatures led to even better hard magnetic properties (i.e.$\mu_0 {}_JH_c$ increased to ~1T for samples annealed at 300^{o}C for 30 min).

Work supported by Polish Scientific Research Committee (KBN)(projects no. 3T08A 063 27 and 3T08C 008 28) and by the U.K. Engineering and Physical Sciences Research Council.

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster B-1

Transition from ferro to mictomagnetism in rich Fe_xAl_{1-x} alloys. A magnetoresistivity study.

M M. Amado[1], M. E. Braga[1], D. S. Schmool[1], Andre M. Pereira[1], J. M. Moreira[1], Joao P. Araujo[1], J. B. Sousa[1], J. S. Garitaonandia[2], F. Plazaola[3]

1. *Departamento de Fisica and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal* **2.** *Elektrika eta elektronica saila, Euskal Herriko Unibertsitatea, Bilbao, Spain* **3.** *Fisika Aplikatua II saila, Euskal Herriko Unibertsitatea, Spain*

Contact: mmamado@fc.up.pt

The $Fe_{1-x}Al_x$ system exhibits unusual magnetic properties around x~3 where ferromagnetism (FM) first sets in (for x<xc). In particular the magnetization under low applied fields (H <100 Oe) decreases with temperature below ~150K, as the FM state goes into a superparamagnetic (super-PM) structure. The understanding of the different phases below T_C still continues to present problems [1].

We report a magnetoresistance (MR) study on $Fe_{1-x}Al_x$ samples with x=0.295, 0.300 and 0.305, prepared by melt spinning and exhibiting a polycrystalline A2 structure (from X-ray diffraction). A four-point probe dc technique was used for the MR measurements, with the magnetic field applied parallel to the electrical current. The residual resistivity (ρ_0) increases dramatically with x in our samples ($d\rho_0$/dx~181μΩcm per 1%Al addition), reaching 353 μΩcm for x=0.305. This reflects a drastic increase in structural and magnetic disorder within a narrow range of composition, in a scale of the order of the electron mean free path.

Magnetoresistance and coercive field measurements clearly indicate three magnetic regimes below room temperature, with transition points around 50K and 150K. In fact from T_{Cinv} ~150K down to T_{iso} ~50K, the MR behavior indicates the presence of super-PM. Below T_{iso} the samples exhibit full super-PM, but the increase of the coercive field as T decreases indicates enhancement of local magnetic anisotropy.

The MR behavior can be understood using a dynamical model based on FM clusters, whose sizes vary with temperature within a PM matrix. From T_C to T_{Cinv} we have overlapping FM clusters leading to the highest degree of FM behavior. Below T_{Cinv} such coalescence disappears (smaller FM clusters) but the separate clusters are still magnetically interacting. Below T_{iso} ~50K, inter-cluster magnetic interactions fade away and full super-PM emerges.

[1] D.Schmool et al J. Mag. Mag .Mat. 272-276 (2004) 1342

15:50 poster B-2

Architecture and Electronic Structure of K-Phase in Fe-Al-C Alloys

Vladyslav A. Andryuschenko, Dmytro I. Oliferuk, Dmitriy A. Bogaychuk, Tatyana A. Shapoval

G.V. Kurdyumov Institute for Metal Physics National Academy of Sciences (IMP), Vernadsky Blvd. 36, Kyiv UA03680, Ukraine

Contact: vaavandr@i.kiev.ua

The electronic structure calculations of antiperovskite compounds $Fe_{4-y}Al_yC_x$ with hypothetical crystal structures have been carried out using the full-potential linearized augmented plane wave method (FLAPW).

The analysis of an electronic structure has shown, that reduction of concentration of atoms of iron which are replaced with aluminium causes reduction of density of electronic states of a valent zone. Increases in concentration of carbon predetermines their significant asymmetry near the Fermi level, and also presence of significant p-d hybridization of electron shells of iron and carbon.

The magnetic moments on atoms of iron essentially decrease at presence in the nearest crystal environment of atoms Fe atoms of carbon. Values of the calculated magnetizations of modelling crystal structures $Fe_{4-y}Al_yC_x$, correlate with the values established experimentally at 70 K for saturation magnetization in particles of K-phase which were extracting from alloys Fe-Al-C with a different chemical composition.

15:50 poster B-3

Magnetic, electric and electronic structure properties of the $Gd(Ni_{1-x}Co_x)_3$ compounds

Grażyna Chełkowska, Marzena Kwiecień, Katarzyna Rabijasz

University of Silesia, August Chełkowski Institute of Physics, Department of Solid State Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: gchelkow@us.edu.pl

We report results of measurements of the magnetic susceptibility, the electrical resistivity, the crystal and electronic structures of the polycrystalline intermetallic Gd $(Ni_{1-x}Co_x)_3$ compounds. The influence of substitution of Co for Ni atoms is reflected in an increase of the lattice parameters and unit cell volume. The Curie temperature T_C changes between 114 K for $GdNi_3$ and 615 K for $GdCo_3$. The electronic structure have been studied by using X-ray Photoelectron Spectroscopy (XPS). The XPS spectra in the valence band region show a hybridization of the states near the Fermi level (E_F). We also observe a remarkable decrease of the density of states near E_F and the shift of 4f Gd peaks in the energy scale for the Co – rich compounds.

15:50 poster B-4

Structure and magnetic properties of $R_4(Co,Ti)_{41}$ (R= Gd, Pr, Sm) alloys

Margarit Gjoka[2], Charalambos Sarafidis[1], Orestis Kalogirou[1], Dimitris Niarchos[2]

1. *Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece*
2. *Institute of Materials Science, NCSR "Demokritos", 153 10 Ag. Paraskevi, Attiki, Greece, Athens 153 10, Greece*

Contact: gjoka@ims.demokritos.gr

$Nd_3(Fe,Ti)_{29}$-type structure, widely studied during the last years, consists of a combination of Th_2Zn_{17} (2:17) rhombohedral and $ThMn_{12}$ (1:12) tetragonal segments in a ratio of 1:1. The existence of this composition indicates that more intermediate phases should be possible: e.g. a ratio 1:2 resulting to a 4:41 phase. The synthesis of a new phase with nominal composition $Tb_4(Fe,Si)_{41}$ has been recently reported [1]. In a previous work we have investigated the alloys with starting nominal composition $R_4(Fe,Ti)_{41}$ (R=Nd, Er; T=Ti, V) [2]. In this work we extend the previous study for the $R_4(Co,Ti)_{41}$ alloys (R=Gd, Pr, Sm). Alloys with starting composition $R_4(Co1\text{-}xTix)_{41}$ (x=0.037, 0.051, 0.061) were prepared and annealed in the temperature interval of 1133-1438 K. Thermomagnetic analysis (TMA) for the samples annealed at 1133 K showed one magnetic transition at 1025 K and 1058 K respectively for Gd (x=0.051) and Pr (x=0.037) which, according to the X-ray spectra, correspond to the disordered $CaCu_5$-type structure. The saturation magnetization, MS, of the first samples is 77.3 Am^2/kg at room temperature and both exhibit uniaxial anisotropy. The $Gd_4(Co,Ti)_{41}$ (x=0.051) alloy annealed at 1438 K crystallizes into the disordered 2:17 hexagonal phase with cell parameters a=8.350(1) and c=8.224(1) angstrom. In contrary, the $Pr_4(Fe,Ti)_{41}$ samples annealed at 1438 K crystallize into the rhombohedral 2:17R-type structure. The study of the magnetic properties and of the microstructure of this series is in progress and will be reported.

1. Ivanova, G.M. Makarova, Ye.V. Shcherbakova and Ye.V. Belozerov, J. Alloys Compounds 309 (2000) 141.

2. M. Gjoka, D. Niarchos, C. Sarafidis and O. Kalogirou, Proceedings of 17th International Workshop on Rare-Earth Magnets and their Applications, pp. 216-220 (2002).

15:50 poster B-5

Electronic structure of $Ce_2Rh_3Al_9$

Jerzy Goraus, Andrzej Ślebarski

University of Silesia, Institute of Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: jg@xps2.zfcst.us.edu.pl

We have investigated an electrical transport properties and electronic structure of $Ce_2Rh_3Al_9$. Ce-3d and Ce-4d XPS measurements showed a clear mixed valence behavior, confirmed also by the TB-LMTO-ASA and FP-LAPW band structure calculation. A non-magnetic ground state expected from LMTO calculations seem to be in a good agreement with experimental data, however, a predicted gap of 3 meV width is not observed in resistivity ρ(T) data. Our calculations carried out for ~3% larger or smaller lattice parameters in comparison to that, experimentally obtained at room temperature lead us to conclusion that the gap is still present. We argue, that an atomic disorder could be a reason that activated behavior in ρ(t) is not observed for $Ce_2Rh_3Al_9$.

15:50 poster B-6

Magnetic properties of the GdTiSb and GdTiSn - the new intermetallic compounds.

Adam Guzik

University of Silesia, August Chełkowski Institute of Physics, Department of Solid State Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: aguzik@us.edu.pl

The ternary intermetallic compounds from families of RTSb and RTSn (R- rare earth, T- transition metal) orders magnetically below a few Kelvin and are antiferromagnets, the ordering temperature is e.g. 3.5 K and 11 K for GdNiSb[1] for GdNiSn[2], respectively.

The new GdTiSb intermetallic compound possesses the very high ordering temperature reaching the 268 K. It crystallizes in a tetragonal type of structure with lattice parameters a=652 pm and c= 935 pm. The magnetic susceptibility in the paramagnetic region follows the Curie-Weiss law with the effective magnetic moment of 8.1 μB (7.94 for Gd^{3+}). The ferromagnetic transition occurs at 275 K under the magnetic field of 0.01 T. AC susceptibility measurements made in very weak magnetic field verifies it down to 268 K. The tin counterpart compound - GdTiSn orders magnetically below 108 K.

The GdTiSb resistivity at the room temperature is of the value of 110 μΩ*cm and shows a negative curvature in the temperature dependence, what can suggest a strong interaction between the d and the f electrons systems. The main ferromagnetic transition is slightly visible on the resistivity. Paradoxically, the reorientation effect occurring at 30 K emerges as an upturn on the resistivity. The mirror fingerprint of magnetic phenomena is visible in the GdTiSn temperature dependence of resistivity, at about 110 K and 15 K.

[1] R.V.Skolozdra et al, Acta Physica Polonica A, 92(1997) 343-346

[2] Y.Adosh et al., J. Magn. Magn. Mater 177-181(1998) 1063-64

15:50 poster B-7

Magnetic state in RAgAl (R = Dy, Ho, Er)

Jan Heimann, Dawid Dunikowski

University of Silesia, August Chełkowski Institute of Physics, Department of Solid State Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: Heimann@us.edu.pl

The intermetallics DyAgAl, HoAgAl and ErAgAl were obtained by the Czochralski method from a levitated melt. X-ray powder diffraction measurements have shown that the investigated samples crystallize in $CeCu_2$-type structure (Imma).

The X-ray photoelectron spectra were obtained with a Physical Electronics PHI 5700/660 XPS spectrometer using monochromatized Al Kα radiation.

The magnetic measurements (in static fields of 30-2500 Oe) were carried out in the temperature range 4.2-600 K by the Faraday method. Above 50 K magnetic susceptibility follows Curie-Weiss law with θ parameter 35, 16, and 8 K for DyAgAl, HoAgAl and ErAgAl respectively. The effective magnetic moments per rare earths atom are few percent higher than their free ion values. At low temperatures thermomagnetic effects between ZFC and cooled in field of 2500 Oe (FC) runs exist. The susceptibility vs. temperature for ZFC runs shows a peak at 37, 18 and 13 K for DyAgAl, HoAgAl and ErAgAl respectively.

The ac susceptibility (H = 2 Oe, f = 1 kHz) shows a cusp at 44, 24 and 15 K for DyAgAl, HoAgAl and ErAgAl respectively.

The electrical resistivity has been investigated in the temperature range 4.2-300 K with a current 100 mA. In the liquid helium (4.2 K) the resistivity values are large (2.69 μΩm for DyAgAl, 2.78 μΩm for HoAgAl and 3.39 μΩm for ErAgAl). The temperature dependence of the resistivity of ErAgAl shows local maximum at 12.5 K (3.4 μΩm) followed by minimum at 31 K (3.38 μΩm).

The thermomagnetic effects, cusps in ac and peaks in dc susceptibility indicate the spin glass-like state. We conclude that the disorder in nonmagnetic atoms sublattice of the $CeCu_2$ type structure causes the short mean path of the conduction electrons. The RKKY interactions are reduced to the closest neighbors. The Al and Ag atoms differ enough to disturb the symmetry in neighborhood of rare earths. In connection with reducing the range of the RKKY interactions it leads to concentrated spin glass state.

15:50 poster B-8

"Electron Spin Resonance in GdPdX compounds; X - p element"

Jerzy Jarosz, Ewa Talik

Institute of Physics, Silesian University, Uniwersytecka 4, Katowice 40007, Poland

Contact: jjarosz@us.edu.pl

It is well known that the relaxation processes in GdTX family; T – transition metal; depend very strongly on the contribution of the d states at the Fermi energy. However, when we fixed the d-metal it occurs that also different share of s and p electrons in valence band results in different bottleneck parameters observed in these compounds. In case of GdPdX compounds the bottleneck effect could even exceed that one, observed in $GdAl_2$ compound .

15:50 poster B-9

Crystal and electronic structure of GdPdX (X=Al, Si, Ga, Sn, Ge, In)

Ewa Talik[1], Joachim Kusz[1], Horst Böhm[2], Michał Matlak[1], Magdalena Skutecka[1], Monika Klimczak[1]

1. *University of Silesia, Katowice, Poland* **2.** *Johannes Gutenberg-Universität Mainz, Staudingerweg 9, Mainz 55128, Germany*

Contact: talik@us.edu.pl

GdPdX compounds (X = Al, Si, Ga, Ge, In, Sn) crystallized in the stable orthorhombic structures similar to GdRhAl. GdPdAl crystallized in TiNiSi type, GdPdSi in β - GdPdSi type, GdPdGe in β - GdPdGe type and GdPdGa, GdPdSn GdPdIn in Co_2Si type. The crystal structure of GdPdX strongly depends on the history of the sample. These samples were rapidly cooled and crystallized mostly in disordered crystal structures. The electronic structure measurements revealed the narrowing of the Pd 4d states in the compounds. Position of these states for all compounds is similar, about 4 eV below the Fermi level. However, the changes of full width at half maximum were clearly visible. A comparison between reduced resistivity and the resistivity resulting from the Bloch formula in the temperature range above the ordering point is performed. The agreement between theory and experiment is quite satisfactory. Some deviation is observed for the samples with indium and germanium.

15:50 poster B-10

Electrical resistivity and magnetic susceptibility of R_5Pd_2 (R=Tb,Dy,Ho,Er).

Monika Klimczak, Ewa Talik

University of Silesia, Katowice, Poland

Contact: monikaklimczak@yahoo.com

The intermetallics R_5Pd_2 (R = Tb, Dy, Ho, Er) crystallized in the cubic Dy_5Pd_2 - type structure [1]. Preliminary magnetic measurements showed that these compounds are antiferromagnetic [2]. The effective magnetic moments were enhanced in comparison with theoretical ones. Yakinthos et al. [2] explain this increase by the polarization of the conduction electrons by magnetic moment of rare earth.

The aim of this work is examination of the above mentioned compounds with variety physical methods. The samples were obtained by the induction melt in polycrystalline form. The sample with gadolinium crystallized in hexagonal Th_7Fe_3 – type of crystal structure.

The electrical resistivity measurements and ac magnetic susceptibility show a complex magnetic behaviour of these compounds. Several transitions are observed besides of the main peak in ac measurements. Moreover, an anomaly at about 270 K was observed for all compounds in ac and electrical resistivity temperature dependences.

1. M. L. Fornasini, A. Palenzona, J. Less Common Metals 38 (1974) 77-82.

2. J. K. Yakinthos, T. Anagnostopoulos, P. F. Ikonomou, J. Less Common Metals 51 (1977) 113-116.

15:50 poster B-11

Magnetic properties of Gd_7T_3 (T=Rh, Pd) single crystals

Monika Klimczak[1], Ewa Talik[1], Robert Troć[2], Joachim Kusz[1]

1. *University of Silesia, Katowice, Poland* **2.** *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland*

Contact: monikaklimczak@yahoo.com

Recently magnetic properties of the R_7Pd_3 intermetallic compounds were examined [1]. For La_7Pd_3 the electrical resistivity exhibits a tendency to saturation at higher temperatures and the superconducting transition at 6 K. Ce_7Pd_3 has an antiferromagnetic transition at 5 K, while Pr_7Pd_3 has such transition at only 1.7 K. Nd_7Pd_3 shows two characteristic temperature: first one 33 K related to ferromagnetic – antiferromagnetic transition and the second 38 K related to transition antiferromagnetic – paramagnetic. Sm_7Pd_3 exhibits two sharp peaks connected with antiferromagnetic – ferrimagnetic transition at 18.4 K and at 167 K connected with transition from ferrimagnetic state to paramagnetic. All measured properties are sensitive to the pressure. Magnetocaloric properties of Gd_7Pd_3 were investigated [2].

Canepa et al. [2] found the ferromagnetic transition for polycrystalline Gd_7Pd_3 at 323 K. This sample crystallized in the hexagonal Th_7Fe_3 type of crystal structure with space group $P6_3mc$. The addition of the second phase was less than 3%. The refrigerant capacity was estimated as 100 J/kg at applied

field 2 T and 380 J/kg at 5 T.

The electrical resistivity, lattice parameters temperature dependences, magnetization, magnetic susceptibility, thermopower and electronic structure were measured for single crystals Gd_7T_3. These compounds exhibit anomalous and anisotropic character. The ordering temperature for Gd_7Pd_3 single crystals is 331 K and is higher than obtained for polycrystalline sample [2]. Recently for polycrystalline sample Gd_7Rh_3 the giant magnetoresistivity was found. The aim of this work is to measure magnetic properties this compound along the principal directions.

1. H. Kadomatsu, K. Kuwano, K. Umeo, Y. Itoh, T. Tokunaga, J. Magn. Magn. Mater. 189 (1998) 335.

2. F. Canepa, M. Napoletano, S. Cirafici, Intermetallics 10 (2002) 731.

3. K. Sengupta, S. Rayaprol, E.V. Sampathkumaran, Europhysics Letters 69 (2005) 454.

15:50 poster B-12

THE ELECTRONIC STRUCTURE AND MAGNETIC PROPERTIES OF ADVANCED HEUSLER ALLOYS

Svetlana E. Kulkova[1,2], Gennagy E. Rudenskiy[1], Sergey S. Kulkov[2]

1. *Institute of Stregth Physics and Materials Science of SB RAS, Tomsk, Russian Federation* **2.** *Tomsk State University (TSU), Lenina, 36, Tomsk 634050, Russian Federation*

Contact: kulkova@ms.tsc.ru

Ferromagnetic alloys undergoing thermoelastic martensitic transformations have recently become very attractive owing to the possibility of inducing giant deformations by rotating martensitic domains with an externally applied magnetic field. It is expected that the ferromagnetic alloys with the Heusler structure may exhibit a strong magnetoelastic coupling resulting in potentially interesting magneto-mechanical properties. On other hand, half Heusler alloys were recently examined in the light of their half–metallicity and spintronic applications. The magnetic properties of alloys depend strongly on both the conduction electron concentration and chemical order. For spintronic applications, minimizing interfacial reactions and controlling the growth of Heusler alloys thin films are very important task. In this connection the investigation of structural and magnetic properties of systems incorporating Heusler alloys are desirable.

In present paper the electronic structure of several Mn-, Co- and Fe-based Heusler and half Heusler alloys is studied by means of the full-potential linearised augmented-plane-wave method. The structural trends and changes of magnetic properties of alloys with varying of one or two components are analyzed. We calculated electronic structure of Ni_2MnGa(001) thin film and it on a GaAs substrate using NiGa interlayer. The change of magnetic properties at surface and interface is analyzed. It is shown that the magnetic moment at surface Mn atoms slightly increases (about $0.3\mu_B$) whereas Ni atom has ~$0.3\text{-}0.4\mu_B$ in the surface and subsurface layers. GaAs substrate does not influence significantly the magnetic properties of Ni_2MnGa thin film.

This work was supported by Russian Foundation for Basic Research (grant N 05—02-16074).

15:50 poster B-13

Valence instability of europium in $EuCo_2Si_2$

Paweł Maślankiewicz, Jacek Szade

University of Silesia, August Chełkowski Institute of Physics, Department of Solid State Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: pmaslank@us.edu.pl

EuM_2X_2 intermetallics (where M = transition metal and X = Si, Ge or a mixture thereof) are well known for phenomena of intermediate or unstable valence of europium. Among these compounds, $EuCo_2Si_2$ was studied by Mössbauer spectroscopy [1] and X-ray absorption spectroscopy [2], which revealed an effective valence of europium close to +3. We have synthesised polycrystalline samples of $EuCo_2Si_2$ by multiple induction melting in a levitation crucible under a protective atmosphere of argon. A surplus of europium was necessary to compensate evaporation losses. As-cast samples were basically single-phase ($ThCr_2Si_2$-type orthorhombic structure, a = 3.9217(8) Å, c = 9.834(2) Å). Bulk methods such as magnetic susceptibility and comparison of lattice parameters with those of other $RECo_2Si_2$ (RE = rare earth) compounds suggest that most of europium in our $EuCo_2Si_2$ is trivalent, while X-ray photoelectron spectroscopy measurements performed on samples cleaned by argon ion etching in ultra-high vacuum indicate a divalent oxidation state of europium. This discrepancy can be attributed to local chemical changes induced by Ar^+ sputtering or to a difference between valence of bulk and surface europium. A weak ferromagnetic-like transition was detected at 74 K.

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[2] N.D. Samsonova, L.D. Finkel'shtein and E.M. Levin, Fiz. Tverd. Tela 24, 3711 (1982).

N.D. Samsonova, L.D. Finkel'shtein and E.M. Levin, Sov. Phys. - Solid State 24, 2116 (1982).

15:50 poster B-14

Structural and magnetic properties of $CoTi_{1-x}Mn_xSb$ solid solution.

Krystyna Mateja-Kaczmarska, Magdalena Wicher, Grażyna Chełkowska

Institute of Physics, Silesian University, Uniwersytecka 4, Katowice 40007, Poland

Contact: kaczmars@us.edu.pl

The series of the half-Heusler compounds $CoTi_{1-x}Mn_xSb$, starting with the Pauli paramagnetic semiconductor CoTiSb and ended by ferromagnetic metal CoMnSb, have been prepared. Polycrystalline samples were obtained by arc furnace melting and annealed at 700 C for one week. All samples crystallise with the MgAgAs regular structure. For composition with x > 0.6 superstructure lines, corresponding to a doubled cell parameter, appear. The concentration dependence of the lattice parameter does not follow Vegard's law. The ferromagnetic ground state appears in the system when 20% of Ti are replaced by Mn.

15:50 poster B-15

Low temperature specific heat and thermoelectric power of $UCu_3M_2Al_7$ alloys

Wojciech Suski[1,2], Krzysztof Gofryk[1], Alicja Hackemer[1], Konrad WOCHOWSKI[1]

1. *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland* **2.** *International Laboratory of High Magnetic Fields and Low Temperatures (ML), Gajowicka 95, Wrocław 53-421, Poland*

Contact: w.suski@int.pan.wroc.pl

Our previous investigations of the magnetic and electrical properties of the $UCu_3M_2Al_7$ alloys where M = Cr, Mn and Fe are being extended now to the examination of the specific heat and thermoelectric power. The specific heat has been measured in the temperature range 1.2 - 70 K in magnetic field H = 0 and 3T using a homemade and fully automatic calorimeter, whereas the thermoelectric power has been measured from 6 to 300 K emploing a differential method with copper as reference material. The specific heat vs. temperature plot exhibits only minute influence of magnetic field and no anomaly in the investigated temperature region. However, the coefficient of the electronic specific heat is enhanced amounting to about 200 $mJ/molK^2$ at high temperature and to about 400 $mJ/molK^2$ at low temperature. Strongly correlated electrons or crystallographic disorder are considered as a reason for these values. The thermoelectric power demonstrates moderate values in whole investigated temperature range.

15:50 poster B-16

UNSTABLE MIXING REGIONS IN CdMnHgTe AND ZnMnHgTe SOLID SOLUTIONS

Vitalij Deibuk, Sergey Ostapov

Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine

Contact: vdei@chnu.cv.ua

The development of the $Cd_{1-x-y}Mn_yHg_xTe$ and $Zn_{1-x-y}Mn_yHg_xTe$ solid solutions which has been proposed for infrared photoelectric devices with predetermined spectral characteristics has made significant progress as they are a narrow band gap materials with direct optical transition. To configure high-quality heterostructure films, lattice-matching growth is required. For these purposes it is important to clarify the relatively wide regions of instability and immiscibility, which restricts applications of these materials. In spite of the lattice constants being almost identical for the two limiting (binary) cases of CdHgTe (6.48Å for CdTe and 6.46Å for HgTe), the metallurgical interface is usually enriched with structural defects, in particular, mismatch dislocations, elastic deformations, gettered impurities, etc. This results in the significant changes of thermodynamics stability conditions thin quaternary films. This paper presents calculations of spinodal and binodal isotherms of $Cd_{1-x-y}Mn_yHg_xTe$ and $Zn_{1-x-y}Mn_yHg_xTe$ solid solutions and their pseudomorphic thin films on the basis of the delta-lattice parameter model. We used in this work ternary compound $Cd_{1-y}Zn_yTe$ and $Cd_{1-y}Mn_yTe$ as substrate for growth films. The novelty of the approach consists in the consideration of mismatch dislocations in elastic energy relaxation.

15:50 poster B-17

Structure studies of $Fe_{81}Nb_5B_{14}$ annealed alloys by Rietveld refinement

Małgorzata Karolus

University of Silesia, Institute of Material Science, Bankowa 12, Katowice 40-007, Poland

Contact: karolus@us.edu.pl

The amorphous $Fe_{81}Nb_5B_{14}$ alloys were obtained by melt spinning method as ribbons of 10 mm width and 25 μm thickness. For the creation of the nanocrystalline structure the alloys were annealed at the temperature range of 300 to 1025 K. The amorphous state was observed till temperature of 730 K. After annealing at 760 K the creation of the nanocrystalline solid solution based on α-Fe structure was observed. The full crystallization of α-Fe and borides (Fe_2B and Fe_3B) was observed after annealing at temperature of 1025 K.

The amorphous alloys were analysed by the Radial Distribution Functions in case of the coordination radii calculation.

The analysis has shown that the basic type of order is typical for α-Fe model structure.

Both, the amorphous and the nanocrystalline alloys were analysed by the Rietveld refinement. In case of the amorphous material the modificated model of the solid solution structure based on α-Fe was used. The determination of the lattice parameter of structure model was the result of the refinement (3.0491 Å to 2.9639 Å). In case of the nanocrystalline alloys on the X-ray patterns were observed diffraction lines typical for α-Fe (2.8748 Å – 2.8655 Å). The lattice parameter of α-Fe after crystallization was 2.8644 Å. The values of the received Rietveld refinement parameters were in the ranges of R_{wp} = 7 – 21 %, R_p = 5 – 16 %, R_{exp} = 5 - 20% and S = 1.09 – 1.58.

15:50 poster B-18

THE CRYSTAL STRUCTURE AND SOME MAGNETIC PROPERTIES OF $NdFe_8Ga_3C$ COMPOUND

Pavlo Y. Demchenko, Igor V. Oshchapovsky, Oksana I. Bodak, Yurij K. Gorelenko

Ivan Franko Lviv National University, Department of Inorganic Chemistry (LNU), Kyryla i Mefodiya str., 6, Lviv 79005, Ukraine

Contact: demchenko@franko.lviv.ua

The one of the main tasks of solid-state sciences is searching for a new group of intermetallic compounds with magnetic properties, based on which it is possible to make new magnetic materials for practical applications. In this communication we present the results of crystal structure determination and some magnetic properties for a new compound - $NdFe_8Ga_3C$.

The compound was synthesized by arc melting under an argon atmosphere in an arc furnace with a water-cooled copper bottom (Ti-getter) with following heat treatment at 870 K. X-ray diffraction pattern was obtained on a HZG-4a (FeKα radiation) automatic powder diffractometer. The crystal structure was solved and refined with the "WinCSD" program package [1] using Rietveld method. The magnetization versus applied field and temperature was measured using vibrating sample magnetometer.

The compound $NdFe_8Ga_3C$ crystallizes in tetragonal structure type $CeNi_{8.6}Si_{2.4}$ (space group $I4_1/amd$) with lattice parameters a=1.0279(1), c=0.66785(9) nm (R_I=10.07, R_P=4.39, R_{wP}=6.97) and atomic parameters: Nd (4b) 0 1/4 3/8 B_{iso}=0.0022(8)nm^2; Fe (32i) 0.2056(8) 0.1239(11) 0.0618(12) B_{iso}=0.0027(8)nm^2; Ga (4a) 0 3/4 1/8 B_{iso}=0.0154(8)nm^2; Ga (8c) 0 0 0 B_{iso}=0.0181(8)nm^2 and C (8d) 0 0 1/2 B_{iso}=0.02 nm2, G=0.5.

The magnetization at field 10 kOe is σ=104.7 A m^2/kg, the Curie temperature is T_C=389 K.

[1] L. Akselrud, Yu. Grin, V. Pecharsky, P. Zavalij, B. Baumgartner, E. Wolfel, in: Materials Science Forum, Proceedings of the Second European Powder Diffraction Conference, Enschede, The Netherlands, Use of the CSD program package for structure determination from powder data, Trans. Tech. Pub. Pt. 1 (1993) 335-340.

15:50 poster B-19

Application of soft magnetic amorphous alloy $Fe_{80}Nb_6B_{14}$ as electromagnetic shields

Grzegorz Haneczok[1], Ryszard Wroczyński[2], Piotr Kwapuliński[1], Artur Chrobak[3], Zbigniew Stokłosa[1], Józef Rasek[1]

1. *University of Silesia, Institute of Material Science, Bankowa 12, Katowice 40-007, Poland* **2.** *Wrocław University of Technology, Institute of Telecommunication and Acoustic, Wybrzeże St. Wyspiańskiego, Wrocław 50-370, Poland* **3.** *University of Silesia, Institute of Physics, Uniwersytecka 4, Katowice 40-007, Poland*

Contact: haneczok@us.edu.pl

Soft magnetic properties of amorphous alloys based on iron can be improved by a thermal annealing at temperatures closed to the crystallization temperature. This is usually attributed to formation of nanocrystalline grains (αFe, αFe(Si)) embedded into amorphous matrix. For the $Fe_{80}Nb_6B_{14}$ amorphous alloy the optimization effect was attributed to annealing out of free volume without formation of nanograins. Nb atoms cause a slowing down of diffusion processes which makes possible to obtain the relaxed amorphous phase. The optimization effect without formation of nanograins is very interesting because it can give very good soft magnetic material free of embrittlement.

The aim of the paper is to study the application of the $Fe_{80}Nb_6B_{14}$ amorphous alloy as electromagnetic shields. The strips of as received material (thickness d=25 μm) were annealed at 700 K/1h. This optimization annealing gives material with initial magnetic permeability about $3x10^4$ and coercive field less than 1 A/m. In order to study the shielding effectiveness single shields (25x25 cm^2) were made of two layers of optimized amorphous strips. Each layer was glued onto one side of double-sided scotch tape. Every shield consists of more than 50 strips forming a uniform soft magnetic plane with thickness d=50 μm.

For magnetic field (impedance<377Ω) in frequency range 0.2-10000 kHz for a single shield b decreases from 15 to 5 dB. For a quadruple shield (d=200 μm) b>20 dB in frequency range 0.2-10 kHz. The shielding effectiveness for electric field (impedance>377Ω) decreases from 50 to 20 dB (2-15 MHz) independently of shield thickness. The best result was obtained for electromagnetic field (far zone, 200-1000 MHz). In this case for a single shield in all frequency range the shielding effectiveness is better than 30 dB. For a triple (d=150 μm) and quadruple (d=200 μm) shields b>100 dB.

15:50 poster B-20

Soft magnetic properties of the $Fe_{72}Co_{10}Nb_6B_{12}$ amorphous alloy

Artur Chrobak[1], Grzegorz Haneczok[2], Piotr Kwapuliński[2], Dariusz Chrobak[2], Zbigniew Stokłosa[2], Józef Rasek[2]

1. *University of Silesia, Institute of Physics, Uniwersytecka 4, Katowice 40-007, Poland* **2.** *University of Silesia, Institute of Material Science, Bankowa 12, Katowice 40-007, Poland*

Contact: haneczok@us.edu.pl

It was shown [1] that soft magnetic properties of the $Fe_{86-x}Nb_xB_{14}$ (x=2-8) amorphous alloys can be improve by 1 h annealing at temperatures close to the crystallization temperature. Such an optimization annealing causes an increase of initial magnetic permeability μ (for the best alloy x=6, $\mu=3x\ 10^4$), and a reduction of coercive field (1-3 A/m). The observed effect was attributed to annealing out of free volume leading to formation of small iron clusters. An unfavorable effect of the high Nb content is a relatively low Curie temperature (390 K) which can be improved by a partial replacing of Fe by Co.

The aim of the paper is to study magnetic properties, crystallization and the optimization effect of the $Fe_{72}Co_{10}Nb_6B_{12}$ amorphous alloy for which the Curie temperature was determined as 515 K. Magnetic properties were studied by making use of: permeability measurements (Maxwell-Wien bridge, f=1 kHz, H=0.5 A/m), magnetic after-effect $\Delta\mu/\mu$ ($\Delta\mu=\mu(t_1=30s)-\mu(t_2=1800s)$, t_1, t_2 times after demagnetization), coercive field H_c (permalloy probe) and magnetization curves (fluxmeter).

It was shown that soft magnetic properties can be optimized by 1 h annealing at 675 K. For optimized samples $\mu=10^4$ and H_c=15 A/m. The crystallization was examined by applying measurements of magnetization (magnetic balance) with heating rates 3-10 K/min and derivation scanning calorymetry (5-20 K/min). The activation enthalpy of the nanocrystallization was determined as 3.8 eV.

[1] G.Haneczok, A.Chrobak, P.Kwapuliński, Z.Stokłosa, J.Rasek, N.Wójcik, Proceedings of International Conference on Soft Magnetic Materials, SMM'16, Düsseldorf September 2003, Verlag Stahleisen GmbH 2004, p.603.

15:50 poster B-21

Magnetic anisotropy of ultrathin cobalt in Au/Co/Si/Au structures with variable Si thickness.

Zbigniew Kurant[1], Maria Tekielak[2], Marek Schmidt[3], Feliks Stobiecki[3], Andrzej Maziewski[2]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *University of Białystok, Laboratory of Magnetism, 41 Lipowa street, Białystok, Poland* **3.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: kurantz@uwb.edu.pl

The influence of silicon layer on the magnetic anisotropy of ultrathin cobalt layer in Au/Co-wedge/Si-wedge/Au thin film structure was studied using the polar Kerr effect. The thickness gradients of Co and Si layers were perpendicular to each other. The sample was deposited by magnetron sputtering onto (100) silicon wafer covered with $(Ni_{80}Fe_{20}/Au)_4$ buffer layer. The buffer was chosen to improve the quality of the sample. Due to the negligible coupling between Ni-Fe and Co layers it was possible to extract the magnetooptical signal related to Co layer only.

Thin Co layers sandwiched between Au exhibit a transition from perpendicular anisotropy to in-plane anisotropy with increasing Co thickness. The addition of the Si layer 0-3 nm thick, systematically shifts the transition up to 0.2 nm in the Co thickness. The system does not show any significant changes in the magnetic properties for Si thickness greater than 3 nm. We discuss the changes of the magnetic anisotropy of Co layer as a result of Co-Si mixing at the Co/Si interface.

15:50 poster B-22

Surface magnetic anisotropy at a compensated ferromagnetic-antiferromagnetic interface

Mei-Feng Lai[1], Zung-Hang Wei[1], N. Usov[2], Ching-Ray Chang[1]

1. *National Taiwan University (NTU), Roosevelt Rd., Taipei 106-17, Taiwan* **2.** *Troitsk Institute for Innovation and Fusion Research, Troitsk 142190, Russian Federation*

Contact: d90222026@ntu.edu.tw

The exchange coupling is observed in many magnetic multilayers where ferromagnetic (FM) and antiferromagnetic (AFM) thin films are in atomic contact. This phenomenon is a consequence of a fundamental interaction between the AFM and FM spins at the interface. The nature of the phenomenon is still not understood completely. In our study we prove that certain periodic modulation of the AFM and FM spin structure near a compensated surface of AFM is energetically favorable. The calculations are based on Heisenberg Hamiltoni-

an for an AFM-FM bilayer. The amplitude of the modulation is shown to decrease exponentially into AFM and FM volumes as function of a coordinate perpendicular to the interface. Therefore, from a macroscopic point of view the AFM-FM exchange interaction can be considered as a surface magnetic anisotropy. It is shown that the interaction energy is proportional to the square of a scalar product of unit FM and AFM vectors. As a result, the surface magnetic anisotropy is invariant with respect to arbitrary rotation of the AFM-FM spin system as a whole. The surface anisotropy constant, K_s, is determined as a function of Heisenberg Hamiltonian parameters. Due to condition $K_s > 0$, the corresponding effective interaction makes the FM spins rotate perpendicular to the direction of the AFM spins at the interface.

15:50 poster B-23

GaN(0001) surface Fe atoms doped

Bronislaw A. Orlowski[1], Iwona A. Kowalik[1], Bogdan J. Kowalski[1], Nicolas Barrett[2], Izabella Grzegory[3], Sylwester Porowski[3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Laboratoire pour l'Utilisation du Rayonnement Electromagnetique (LURE), Bât.209D Centre Universitaire - B.P.34, Orsay 91898, France* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: orbro@ifpan.edu.pl

The valence band electronic structure study of GaN(0001)1x1 (N-side) clean surface sequentially covered by Fe atoms is presented. The experiment is devoted to the investigation of Fe doping of the surface and the surface region of the crystal volume at a very first stages of the Fe atoms deposition.

The bulk crystals were grown under high pressure conditions in the High Pressure Research Center, Polish Academy of Sciences, Warsaw, Poland. The wafers of thickness 0.1mm were obtained with a plane (0001) parallel to the plane of wafers. The photoemission study was performed with the application of the vacuum ultraviolet synchrotron radiation obtained from SUPER-ACO ring in LURE (Orsay, France) on the station SA73. The resonant photoemission study (Fano resonance) in the energy range corresponding to Fe 3p-3d transition was performed. The clean surface of the GaN crystal was prepared in UHV preparation chamber directly connected with the vacuum system of the analyzing chamber. The Argon ion bombardment treatment and annealing were used for preparation of clean GaN surface. First depositions lead to the appearance of the new peaks and shoulders corresponding to the Fe contribution to the GaN valence band electronic structure. With sequential Fe depositions the islands of Fe are created and sharp Fermi edge is created over the edge of the valence band. Heating of the sample leads to the diffusion of Fe atoms to the crystal volume.

15:50 poster B-24

Soft magnetic Fe-Co-Zr-W-B bulk glassy alloys

Piotr Pawlik

Institute of Physics, Częstochowa University of Technology, Al. Armii Krajowej 19, Częstochowa 42-200, Poland

Contact: pawlik@mim.pcz.czest.pl

Suction-cast rod samples of the diameters 1 and 2 mm and thin walled tubes of the outer diametes 2, 3 and 4 mm as well as the melt-spun ribbon samples of various thicknesses up to 350 μm of $Fe_{61}Co_{10+x}Zr_5W_{4-x}B_{20}$ (x=0, 2, 3) alloys, were investigated. X-ray diffraction and DSC analysis revealed fully amorphous structure of ribbon samples up to the thickness of 220 μm. Those results indicate good glass forming abilities of the alloys. The suction-casting process allows to produce the bulk amorphous samples by die-casting to the copper mould. X-ray diffractometry and Mössbauer spectroscopy revealed, that for the $Fe_{61}Co_{10}Zr_5W_4B_{20}$ alloy, the rod samples up to diameter of 2 mm and thin walled tubes up to 4 mm of outer diameter were also fully amorphous. Measurement of the magnetic properties for the rod and ribbon samples demonstrated relatively high saturation polarization $J_s = 0.8$ T and low coercivity $_JH_c \sim 4$ A/m. In the present paper, studies of W content influence on the magnetic properties and glass forming abilities are discussed. Decrease of the W contents results in the increase of the saturation polarization of the ultimate alloy due to the increase of the Co contents. However, drastic reduction of the glass forming abilities is also the consequence of these composition changes.

Work supported by Polish Scientific Research Committee (KBN) (projects no. 3T08A 046 27 and 3T08A028 25)

15:50 poster B-25

HgCdMnZnTe: growth and physical properties

Sergey Ostapov[1], Igor Gorbatyuk[1], Sergey Dremlyuzhenko[1], Vladimir Zhikharevich[1], Ilariy Rarenko[1], Ruslan M. Zaplitnyy[1], Igor M. Fodchuk[1], Vitalij Deibuk[1], Miroslav Koval'chuk[1], Nina Popenko[2], Igor Ivanchenko[2], A. Zhigalov[2], Sergey Karelin[2], Yaroslav Olikh[3], Mykola Tymochko[3]

1. *Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine* **2.** *A.Usikov Institute for Radiophysics and Electronics of the National Academy of Sciences, Kharkov, Ukraine* **3.** *Institute for Physics of Semiconductors of NAS of Ukraine, 45 Prospekt Nauki, Kyiv 03028, Ukraine*

Contact: sergey.ostapov@gmail.com

The monocrystals of a new narrow-gap solid solution $Hg_{1-x-y-z}Cd_xMn_yZn_zTe$ with the content of manganese and

zinc up to 5% are presented. X-ray and mechanical investigations of these crystals have been carried out and their basic electrophysical parameters such as the band-gap energy, the intrinsic carriers' concentration, the impurity state of samples, and the mobility of charge carriers have been determined. It is shown that a new material has the perfect crystal structure and high microhardness. The empirical formulas for calculations of band-gap energy and intrinsic carrier concentration of monocrystals like that are suggested.

The influence of ultrasonic loading on the galvanomagnetic effects in the $Hg_{1-x-y-z}Cd_xMn_yZn_zTe$ monocrystals have been investigated.

The obtained data allow announcing this material as an alternative one for infrared detectors for 3 - 5 μm and 8 - 14 μm spectral ranges.

15:50 poster B-26

PROCESSING AND PROPERTIES OF COMPOSITE MAGNETIC POWDERS CONTAINING Co NANOPARTICLES IN POLYMERIC MATRIX

Ewa Sówka[1], Marcin Leonowicz[1], Bartłomiej Andrzejewski2, Anatolii D. Pomogailo[3], Gulzhian I. Dzhardimalieva[3]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland* **3.** *Russian Academy of Sciences, Institute of Problems of Chemical Physics (IPCP RAS), Institutskii pr., 18, Chernogolovka 142432, Russian Federation*

Contact: ewasowka@inmat.pw.edu.pl

Polymeric matrix ferromagnetic nanocomposites, containing Co nanocrystallites were processed by an innovative fabrication method using frontal polymerisation of cobalt acrylamid complex (CoAAm), followed by further thermolysis. The products of thermolysis were in a form of irregular powder particles having broad range of size distribution, from 10 up to 300 mm. The powder particles contain nanocrystallites of Co having the mean 12 -15 nm, depending on the thermolysis temperature. The particles are randomly distributed and their size and agglomeration can be controlled by the processing variables.

The hysteresis loops recorded for materials processed at 873 and 1073 K, respectively proved that the coercivity depends on the processing temperature and very slightly on the measurement temperature. According to the commonly approved classification the coercivity of the composite material processed at temperatures 873 and 1073 K can be regarded as magnetically soft and hard, respectively.

15:50 poster B-27

Microstructures and magnetic properties of Cu-Co nanoparticles prepared by arc discharge

Zhiqing Yang[1], Caiyin You[2]

1. *Department of Physics, University of Antwerp, Antwerp, Belgium* **2.** *Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China*

Contact: zhiqing.yang@ua.ac.be

The investigations on ultra-fine particles of the immiscible system, such as Co-Cu and Co-Ag, are very challenging due to potential higher magnetoresistance and fundamental understanding how bulk properties transform to atomic ones. In the present study, immiscible Cu-Co nanoparticle was successfully prepared by arc-discharge technique in an atmosphere of a mixture of H2 (30%) and He (70%). Microstructures and magnetic propertied were investigated by using advanced transmission electron microscopy (TEM) and superconducting quantum interference device (SQUID). Most of the particles have sizes of 20~30 nm in diameters. High-resolution TEM observations reveal core-shell microstructures with coherent interfaces. Composition of the nano-particles was analyzed by nano-probe energy dispersive X-ray (EDX) analysis, revealing microstructure of cores of Cu-Co solutions covered by oxide shells. Co particles were occasionally observed. The magnetic properties have been analyzed by temperature dependence of magnetization, zero-field-cooled (ZFC) and field-cooled (FC) magnetization measurements. The loop shift in the hysteresis loop indicates the existence of the exchange bias between ferromagnetic and antiferromagnetic components at low temperatures. A block temperature about 180 K has been observed for as-deposited nanoparticles. For the annealed nanoparticles, the thermal magnetization at low temperatures is satisfied with Bloch's law.

15:50 poster B-28

Studies of a structural and magnetic properties of glass-coated nanocrystalline $Fe_{79}Hf_7B_{12}Si_2$ microwires

Arcady P. Zhukov[1], Carlos García[1], Julian Gonzalez[1], Valentina Zhukova[2], Rastislav Varga[3], Juan M. Blanco[4], Juan J. del Val[1]

1. *Basque Country University, Material Physics Dept., (UPV/EHU), Paseo Manuel de Lardizabal, 3, San Sebastian 20018, Spain* **2.** *Tamag Iberica S.L. (TAMAG), Parque Tecnológico de Miramón, Paseo Mikeletegi 56, 1ª Planta, San Sebastian 20009, Spain* **3.** *Institute of experimental Physics, Kosice (IEP SAS), Watsonova 47, Kosice 04353, Slovakia (Slovak Rep.)* **4.** *Dpto. Física Aplicada I, EUPDS (EUPDS), Plaza Europa, 1, San Sebastian 20018, Spain*

Contact: wupzhuka@sp.ehu.es

Soft nanocrystalline Fe-based alloys are excellent soft magnetic nanocomposite materials with the grain size of their primary crystallization products much smaller than their magnetic exchange length. Precursor materials are produced usually by rapid quenching with ribbon-shape with amorphous character. Recently, the Taylor-Ulitovsky technique has resulted very useful to produce glass-coating microwires with the metallic nucleus composition similar to that of the amorphous ribbons. In fact, in ref. [1] we reported on the coercivity behaviour of Finemet-type glass-coated microwires.

In the present work we deal with the fabrication of thin of $Fe_{79}Hf_7B_{12}Si_2$ (low Si content) glass-coated microwire with a nanocrystalline structure and structural and coercivity characterization of such samples which can be considered as a new family of these nanocrystalline materials. Pieces of 10 cm of this microwire were annealed (300-600 °C during 1 hour). The structural characteristics of the as-cast and annealed samples were determined, at room temperature, by X-ray Diffraction (XRD) technique. XRD measurements allow to obtain the evolution of the grain size (15-35 nm) and relative volume fraction (5-60%) of the nanograins as a function of the annealing temperature in the annealed samples. Coercive field (Hc) of the as-cast and annealed samples has been evaluated from the hysteresis loop of the samples obtained by a conventional induction method at 100 Hz. Thermal dependence Hc is quite similar to that reported in other nanocrystalline Fe-based alloys. It decreases from the as-cast state (relaxation process) showing a maximum at around 450 °C (pre-nucleation of nanograins) decreasing significantly between 500-600 °C (exchange coupling of the nanograins).

[1] V. Zhukova, A.F. Cobeño, A. Zhukov, J.M. Blanco, V. Larin and J. Gonzalez., Nanostructured Materials, 11 (1999)1319

15:50 poster B-29

Effect of various surface treatments on the properties of nitrogen-saturated austenite layers produced on N27T2JMNb steel

Tomasz Borowski, Tadeusz Wierzchoń, Jerzy Jeleńkowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: borowski.tomasz@wp.pl

Nitrogen-saturated austenite layers produced on practically carbon-free and chromium-free austenitic steel (metastable austenite) of the composition Fe-27Ni-2Ti-Mo-Al-Nb using glow discharge assisted nitriding. Prior to the nitriding, steel was subjected to surface treatments, such as grinding with a sand paper, burnishing, and cooling in liquid nitrogen, which result in martensite being formed in the surface layer. The paper compares the frictional wear resistance of the nitrided layers formed on steel with various structures: the initial structure after being subjected to grinding, a martensitic-austenitic structure formed by cooling in liquid nitrogen, and a structure subjected to cooling and then nitrided.

15:50 poster B-30

Thermal stability of carbon-encapsulated Fe-Nd-B nanoparticles

Michał Bystrzejewski[1], Stanisław Cudziło[2], Andrzej Huczko[1], Hubert Lange[1]

1. *Warsaw University, Faculty of Chemistry, Pasteura 1, Warszawa 02-093, Poland* **2.** *Military University of Technology (WAT), Kaliskiego 2, Warszawa 00-908, Poland*

Contact: mibys@chem.uw.edu.pl

Plasma methods can produce unique metastable materials, which are unavailable by conventional techniques. In particular, the magnetic materials (e.g., applied in information storage) have been of great interest and it is believed that ferromagnetic transition metal crystallites encapsulated in carbon shells might be used in this area. In such materials, the enclosed metal particles would retain their magnetic moments while being chemically and magnetically isolated from their neighbors.

Thermal stability of various magnetic nanomaterials is very essential, due to their prospective future applications. In this paper, thermal behaviour of the carbon-encapsulated Fe-Nd-B nanoparticles is presented. These nanostructures were produced by DC arcing of carbon anodes filled with $Nd_2Fe_{14}B$ material [1]. The TG-DTA curves were recorded in oxygen atmosphere. The thermal process was monitored by X-ray diffraction to follow the changes in the phase composition. The investigated samples have been thermally stable up to ca. 470-570 K, depending on their structure features.

Acknowledgment. This work was supported by the Committee for Scientific Research (KBN) through the Department of Chemistry, Warsaw University, under Grant No. 3 T08D 012 28.

[1] M. Bystrzejewski, A. Huczko, H. Lange, P. Baranowski, J. Kozubowski, M. Woźniak, M. Leonowicz, W. Kaszuwara, Solid State Phenomena, 2004, 99-100, 273-278.

15:50 poster B-31

X-ray analysis of ground NiAl-Cr_3C_2-Al_2O_3 composite powders with nanocrystalline NiAl phase

Grzegorz Dercz, Krystian Prusik, Tomasz Goryczka, Lucjan Pająk, Bolesław Formanek

University of Silesia, Institute of Material Science, Bankowa 12, Katowice 40-007, Poland

Contact: gdercz@op.pl

The composite powder with NiAl, Cr_3C_2 and Al_2O_3 phases

was prepared by mechanically activated SHS (Self-propagating High-temperature Synthesis) method. As-prepared powder was ground by high-energy attritorial mill up to 40 hours. The Rietveld method based on X-ray powder diffraction data was applied for the verification of qualitative and quantitative phase compositions of as-prepared and ground samples. Micrographs and diffraction patterns were obtained using TEM. The X-ray diffraction data were analyzed using Toraya procedure and Rietveld method. The Rietveld method was applied in the determination of phase abundance using Taylor and Matulis approach. The profile parameters of individual diffraction lines were determined using Toraya PRO-FIT procedure which applies Pearson VII function for the description of line profiles. The crystallite sizes and lattice distortions were estimated using Williamson-Hall method. From the detailed analysis of the breadth of diffraction lines for all phases it was stated that during milling the significant decrease of crystallite size is observed only for NiAl phase. It was stated that the crystallite size of this phase diminishes to nanoscale after 2 hours milling. Prolonged milling time to 10 hours results in further decrease of NiAl crystallite size to 36 nm. The crystallite size of Cr_3C_2 and Al_2O_3 phases is still above 100 nm.

15:50 poster B-32

The influence of thermal treatment on the properties of CdTe colloidal nano-solutions stabilized by thioglycolic acid

Peter Feychuk[1], Shcherbak Laryssa[1], Khalavka Yuriy[1], Bernhard Mingler[2], Gernot Friedbacher[3]

1. *Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine* **2.** *University of Vienna, Institute of Materials Physics, Vienna, Austria* **3.** *Vienna Technical University, 9, Getreidemarkt, Vienna 1040, Austria*

Contact: feychuk@chnucv.ua

CdTe nanoparticles are characterized by high luminescence quantum yield. In CdTe nanocrystals synthesized by metal-organic technologies this factor is near to 65% at room temperature. But the use of such materials is complicated by their luminescence instability at air. On the other side recently essential progress in CdTe nanocrystal growth in water solutions is observed. Surface modification procedures were developed as well as new properties' investigation methods. Luminescent CdTe nanoparticles stabilized by thiol compounds are used in light-emitting arrangements and photon structures and core-nucleus structures.

The investigation and optimization of semiconductor nanoparticles synthesis is an important problem. Preliminary research shows that CdTe nanoparticles solution thermal treatment improves their stability and particles size homogeneity. Prolonged thermal treatment at 100^0C results in edge absorption batochromic shift and amelioration of particles size uniformity owing to the Oswald ripening process.

The aim of this research was to compare the results of different thermal treating procedures in the 50 – 100^0C temperature range on the CdTe colloidal particles' size, stability and optical properties.

The solutions were prepared using a Cd^{2+}: Te^{2-}: RSH = 1:0.5:2.4 molar concentration ratio. During the synthesis the pH value was equal to 11.2. The CdTe nanoparticles' sizes were defined from absorption spectra as well as from AFM and TEM measurements.

The investigations show an essential improvement of the colloidal solutions stability. Thermal treatment at 50^0C allows to obtain nanoparticles with a uniform 7 nm size.

15:50 poster B-33

MAGNETOIMPEDANCE EFFECT AT HIGH FREQUENCY IN GLASS-COATED AMORPHOUS WIRES

Carlos García[1], Arcady P. Zhukov[1], Julian Gonzalez[1], Valentina Zhukova[2], Juan M. Blanco[3]

1. *Basque Country University, Material Physics Dept., (UPV/EHU), Paseo Manuel de Lardizabal, 3, San Sebastian 20018, Spain* **2.** *Tamag Iberica S.L. (TAMAG), Parque Tecnológico de Miramón, Paseo Mikeletegi 56, 1ª Planta, San Sebastian 20009, Spain* **3.** *Dpto. Física Aplicada I, EUPDS (EUPDS), Plaza Europa, 1, San Sebastian 20018, Spain*

Contact: wapgoesj@sc.ehu.es

The GMI effect attracts growing attention especially owing to the large sensitivity (up to 300%) to the DC magnetic field, when the high- frequency (100 kHz - 20 MHz)electrical current flows along the magnetic conductor. Such GMI effect is mostly observed in magnetic materials with large circular magnetic permeability associated to the vanishing magnetostriction. At such f the GMI effect in magnetic materials with considerable magnetostriction is almost negligible (of the order of few %). Recent progress in tailoring of magnetically soft thin wires enabled to enhance significantly the GMI ratio (up to about 600%) [1] in Co-rich compositions with vanishing magnetostriction, while Fe-rich amorphous microwires with rectangular hysteresis loop exhibit poor initial magnetic permeability and almost do not show GMI effect without special processing.

In this paper we report novel results on the GMI effect at high frequency region (10 MHz -500 MHz) in different compositions of amorphous microwires (Fe-rich, with positive magnetostriction, Co-rich with negative magnetostriction and Co-Fe-rich with vanishing magnetostriction) fabricated by the Taylor-Ulitovski method.

Increasing the frequency the GMI effect significantly in-

creases. For example, the shape of the Z(H) of Co-rich microwires with vanishing magnetostriction shows small hysteresis at low DC magnetic field and non-monotonic dependence with a maximum at certain DC magnetic field, H_m. Such H_m increases with increasing the frequency, f. A remarkable difference in the GMI effect with conventional amorphous wires can be attributed to the different magnetoelastic anisotropy of these wires, which should be ascribed to the differences in the fabrication technique resulting in different frequency dependence of the GMI effect.

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15:50 poster B-34

Growth and characterization of GaN and GaMnN layers obtained by Sublimation Sandwich Method

Michal Kaminski, Sławomir Podsiadło

Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland

Contact: michalk@konto.pl

Layers of gallium nitride and gallium nitride doped with manganese were obtained by Sandwich Sublimation Method (SSM). Experiments were carried out in tubular quartz reactors with induction heating at temperatures from $1100^{O}C$ to $1200^{O}C$. GaN layers were prapared from gallium nitride powder, which was the source of gallium. GaMnN layers were prepared from mixtures of powders of gallium nitride and manganese. In both cases, reagents were placed in a quartz boat on top of a graphite cylinder, which was the heating element. Layers were grown on GaN thin films obtained by MOCVD. The distance between powders and MOCVD substrate was about 5mm, and was indispensable for gallium and manganese transfer. The obtained GaN and GaMnN layers were characterized by optical microscope, X-ray studies and Raman studies.

Supported partially by KBN 3 T09B 056 28 and KBN-PBZ-044/P03/2001 from the State Committee for Scientific Research (Poland)

15:50 poster B-35

Nanometer Lamellar Microstructure and Mechanical Properties of gamma-TiAl+alpha2-Ti3Al Based Alloys

Timur Khismatullin

Institute for Metals Superplasticity Problems, Russian Academy of Science, Ufa, Russian Federation

Contact: Timur@imsp.da.ru

A decrease in lamellar spacing to nanometer thickness range (lambda~10 nm) is an effective way to increase strength properties of gamma-TiAl+alpha$_2$-Ti3Al based alloys. For that water quenching from the alpha phase field followed by low temperature aging is usually used. However, this method does not allow a large volume of the material to be obtained. In addition, low aging temperatures result in non-equilibrium microstructural condition at temperatures of potential application (700-800 C). To overcome these deficiencies, specially designed Ti-43Al-(Nb,Mo,B) and Ti-45Al-(Nb,Mo,B) alloys containing the elements with a low diffusive mobility (Nb and Mo) were used in the present work. Lamellar microstructure with a lamellar spacing lambda~10 nm was produced in bulk workpieces of the alloys using annealing in the alpha phase field followed by air cooling and aging. Mechanical tensile tests of flat specimens were carried out at T=20, 600-800 C and a strain rate 10-3 s-1. The tests revealed that high ultimate strength (sigma>900 MPa) was retained up to temperature as high as 800 C and reasonable ductility (elongation>1%) was obtained at room temperature in both alloys. Thus, using conventional heat treatment fine lamellar microstructures (with d<50 micrometers) having the nanometer lamellar spacing (lambda~10 nm) were successfully produced in bulk cast alloys Ti-43Al-(Nb,Mo,B) and Ti-45Al-(Nb,Mo,B) that allowed the mechanical properties to be considerably improved.

15:50 poster B-36

CHARACTERISATION OF POLYCRISTALLINE CuIn1-xGaxTe2 BULK FILMS

Bechiri Lakhdar

LCCM, Annaba university, Annaba 23000, Algeria

Contact: bechiri@hotmail.com

CHARACTERISATION OF POLYCRISTALLINE CuIn1-xGaxTe2 BULK FILMS

L. BECHIRI1, M. BENABDESLEM1, N. BENSLIM1, L. MAHDJOUBI1, R. MADELON2,

J.L. DOUALAN3 and G. NOUET4

1Laboratoire des Cristaux et des Couches Minces, Institut de Physique, Université de Annaba, BP. 12, 23200 Sidi Amar (Algérie). Fax: (00213 38-87-17-12). E-mail: bechiri@hotmail.com

2LERMAT, FRE 2149 CNRS, Institut des Sciences de la Matière et du Rayonnement ISMRA, 6 Boulevard du Maréchal Juin, 14050 Caen cedex (France)

3CYRIL, Institut des Sciences de la Matière et du Rayonnement ISMRA, 6 Boulevard du Maréchal Juin, 14050 Caen cedex (France)

4ESCTM - Laboratoire de Cristallographie des Matériaux, UMR 6508 CNRS, Institut des Sciences de la Matière et du Rayonnement, 6 Boulevard du Maréchal Juin, 14050 Caen cedex (France) Phone: 33 2 31 45 26 47, Fax: 33 2 31 45 26 60

ABSTRACT: Polycristalline CuIn1-xGaxTe2 bulk films were

synthesized by reacting, in stochiometric proportions, high purity Cu, In, Ga and Te in vacuum sealed quartz ampoule. The surface morphology, phase structure and composition of the bulk films were analyzed by scanning electron microscopy, X-ray diffraction and energy-dispersive X-ray analysis, respectively. Photoluminescence spectra were measured at temperatures from 7 to 200 K to characterize defects and the structural quality. The main peak as function of composition has been studied and a tentative assignement of defects to the peaks observed is provided.

Keywords: CuIn1-xGaxTe2 alloys– 1: Structure – 2: Photoluminescence – 3

15:50 poster B-37

Diffusion of Mn in gallium arsenide.

Rafał Jakieła[1,2], Adam Barcz[2,3], Elżbieta Wegner[1], Andrzej Zagojski[1]

1. *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **2.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **3.** *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland*

Contact: jakiela@ifpan.edu.pl

The aim of this work is to compare carrier concentration, diffusion coefficient and shape of the manganese atomic profile in GaAs layers treated under different annealing conditions. Diffusion was performed from implanted Mn as well as from external source of Mn. Manganese implanted GaAs, GaAs:Zn and GaAs:Te bulk samples were investigated. Implantation was performed at room temperature to a dose of $10^{16}/cm^2$ at an energy of 100 keV. The samples, protected with AlN layers, were annealed at $800^{O}C$ and $900^{O}C$ with RTA (rapid thermal annealing) method as well as at $900^{O}C$ and $1000^{O}C$ in sealed quartz ampoule.

After removing the AlN films the extent of diffusion of the implanted species was characterized using the SIMS (Secondary Ion Mass Spectrometry) technique.

The depth profiles of in-diffused manganese strongly indicate that the diffusion coefficient D is concentration-dependent. In case of quartz ampoule annealing of implanted samples the Mn diffusivity was found larger when GaAs was annealed with arsenic overpressure combined with the AlN cap than that annealed without cap. Over ten times shallower diffusion range in uncovered sample then that in covered with AlN is interpreted in terms of generation of additional vacancies in the Ga sub-lattice. Mn atoms incorporate in Ga sites lowering diffusion coefficient. In case of diffusion from external source into differently doped GaAs, the largest diffusion coefficient was found for GaAs:Zn. This result indicates highest Mn diffusivity in sample with low Fermi level, which provides lowest Ga vacancies concentration. Both results confirm an interstitial diffusion mechanism.

The Boltzmann-Matano analysis was employed to evaluate the concentration–dependent diffusion coefficient of Mn in GaAs.

15:50 poster B-38

Investigation of epitaxial $LaNiO_{3-x}$ thin films by High-Energy XPS

Sigitas Mickevicius[1], Sergej Grebinskij[1], Vladimir Bondarenka[1], Vengalis V. Bonifacas[1], Kristina Sliuziene[1], Bronislaw A. Orlowski[2], Wolfgang Drube[3]

1. *Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania* **2.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **3.** *Hamburger Synchrotronstrahlungslabor HASYLAB (HASYLAB), Notkestrasse 85, Hamburg D-22603, Germany*

Contact: sigism@uj.pfi.lt

In recent years certain perovskite-related metallic oxides, such as high-Tc superconductors, the compounds exhibiting giant magnetoresistance and conductive oxides, are intensively studied. The current interest in thin films of high temperature superconductors has made the problem of a suitable substrate an important point for epitaxy to be successfully. A number of Ln-containing (Ln = La, Pr and Nd) oxide crystals have been employed as the substrate material and many are under investigation now. There have been numerous studies on their morphology, structural, thermodynamic, magnetic, elastic properties and crystal growth.

The epitaxial $LaNiO_{3-x}$ films prepared on monocrystalline $NdGaO_3$ demonstrate the excellent in-plane orientation. It is known,[3] that the significant segregation of elements takes place for chemically synthesized $LaNiO_{3-x}$ samples. X-ray photoelectron spectroscopy (XPS) as the surface analysis method provides the direct information on the species concentration and their valence states. In this work we performed High-Energy XPS spectroscopy studies of the surface and volume composition of $LaNiO_{3-x}$ deposited on $NdGaO_3$ substrate by a reactive d.c. magnetron sputtering.

The photoemission data were obtained with the Tunable High-Energy X-ray Photoelectron Spectrometer at the X-ray wiggler beam line BW2 of synchrotron storage ring at HASYLAB. The angle dependent data were obtained by rotating the sample relative to the incoming beam and to the electron analyzer. Comparison of the spectra recorded at normal emission and grazing emission angle allows distinguishing surface and volume atoms contribution to the measured spectra.

The main core level photoemission spectra of La, Ni, O as well as valence band spectra were measured at different emis-

sion angles. The obtained results have been analysed in terms of surface-volume ratio: composition, valence states and the concentration of hydroxyl groups in the epitaxial $LaNiO_{3-x}$ films

15:50 poster B-39

Calculation of magnetostatic energy F_m of periodic domain structures formed near grain boundaries in plates of triaxial ferromagnets of silicon iron type

Sheiko L. M., Bagriytchuk A. S., Lyakishev B. Yu.

Zaporizhzhe National University (ZNU), Zhukovsky 66, Zaporizhzhe +380612, Ukraine

Magnetostatic energy F_m causes the essential contribution to the total balance of free energy F in ferromagnetic [1-3]. The role of energy F_m in formation of domain structure (DS) and magnetic properties of polycrystalline materials is especially great [3-5]. In the present work the results of computation by the micromagnetism theory of energy F_m and magnetostatic fields Hm of periodic DS, formed near GB in plates of polycrystalline ferromagnetics of silicon iron type (~3%Si) with a surface plane (011) in a demagnetized state (H^e=0) are discussed. The obtained values of F^*_m and H^*_m, in materials with enough considerable constant of a magnetocrystalline anisotropy (K_1 =10^3-10^6 J/m^3), are 6-8 times lower, than for before discussed values of F_m and H_m [2,3]. Character of studied states is the presence of areas with the inhomogeneous magnetization (divM_s ≠0) inside grains, instead of magnetization jumps (ΔM_n^{GB} =0) on GB surfaces. The main reason of their formation is the tendency to the energy Fm minimum, and the realization mechanism is deviations of M_s vectors inside domains from easy axes under the influence of magnetostatic fields H_m created by GB. Opportunities of use of the received results for the solution of problems which concern the development and application of magnetic materials and composites are discussed.

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15:50 poster B-40

Optimum potential barrier of single-domain particle in magnetic recording

Zung-Hang Wei[1], N. Usov[2], Ching-Ray Chang[1], Mei-Feng Lai[1]

1. *National Taiwan University (NTU), Roosevelt Rd., Taipei 106-17, Taiwan* **2.** *Troitsk Institute for Innovation and Fusion Research, Troitsk 142190, Russian Federation*

Contact: zhwei@phys.ntu.edu.tw

Superparamagnetic effect limits the achievable areal recording density. The stability ratio of the order of $U_{ef}/k_B T \sim 60$ is necessary to store information at the densities of the order of 100 Gbits/in^2 or higher. Here T is the absolute temperature and k_B is the Boltzmann's constant. For the stability ratio with such high value the thermal decay of single-domain particle magnetization is governed by the time constant $\tau \sim \exp(-U_{ef}/k_B T)$. Therefore, a pre-exponential factor and its temperature dependence are negligible. Thermal agitation also decreases the coercive force of a single-domain particle. Besides, it was reported that for switching times less than 1 ns or so, the magnetization reversal is controlled mainly by the gyro-magnetic precession. Actually, during fast switching there is not enough time for thermal energy to make an appreciable effect on the magnetization reversal. Therefore, it is sufficient to estimate the dimensionless ratio $H_c M_s/U_{ef}$, that can be used to characterize the recording properties of ferromagnetic media, based on usual micromagnetic expressions for the energy barrier and coercive force.

The aim of this study is to show the way to decrease this ratio for a single-domain particle itself. It is shown that this ratio depends on the shape of the energy barrier separating equivalent energy minima. The optimum shape of the barrier is obtained for particles with uniaxial and combined (both uniaxial and cubic) types of magnetic anisotropy. It is found that for the optimum shapes the ratio $H_c M_s/U_{ef}$ can be lowered down by a proportion of 20 - 40 % with respect to the usual case of Stoner-Wohlfarth particle.

15:50 poster B-41

Magnetostatic effect of the change in the shape of 180° domain walls near grain boundaries in plates of triaxial ferromagnets of the Fe-3% Si-alloy type

Sheiko L. M., Bagriytchuk A. S., Murashkinzeva T. N.

Zaporizhzhe National University (ZNU), Zhukovsky 66, Zaporizhzhe +380612, Ukraine

Grain boundaries (GBs) exert a substantial effect on the domain structure (DS) and magnetic properties of polycrystalline materials [1-3]. The role of such defects is especially large in the formation of structure and properties of nanomaterials and composites [4,5], in which the relative volumes of

grain boundaries and interphase boundaries can exceed 20%. In this work, we report the results of a theoretical investigation into the main features of the spatial distribution of spontaneous magnetization $M_s(r)$, the topology of magnetostatic fields $H^m(r)$ near GBs, and the results of calculations of the shape z(x) of 180° DWs in plates of triaxial ferromagnets such as silicon iron (~3% Si) with a surface plane (011) in a demagnetized state (at H^e=0). It is shown that in the states of thermodynamic equilibrium Bloch domain walls can bend as a consequence of deviations Δθ(x) of the magnetic moments of atoms m_a from the <100> easy axis under the effect of magnetic stray fields H^m generated by grain boundaries (the μ* - effect inside magnetic domains near grain boundaries). The magnitudes of the effects studied (the deviations Δθ(x) of the M_s vectors from the easy axes, the magnitudes of the flexures Δz^{bend}_s of domain walls, the extensions of the macroscopic zones Δx^{bend} characterized by inhomogeneous magnetization, as well as the maximum values of the strengths H^m_x of the magnetostatic fields H^m near grain boundaries) increase with decreasing magnetocrystalline anisotropy constant and increasing period of the domain structure 2D.

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15:50 poster B-42

Synthesis and Characterization of Gadolinium colloids in 2-methoxyethanol

Galo Cardenas[1], Guillermo Contreras[2], Olivia Godoy[1]

1. *University of Concepcion, Department of polymers, Chile, Chile* **2.** *University of Concepcion, Department of Inorganic and Analytical Chemistry, Chile, Chile*

Contact: gcardena@udec.cl

Gadolinium nanometric colloids were obtained in 2-methoxyethanol by CLD method. The colloids exhibit a low stability at room temperature. The UV spectrum show two bands at 227 and 278 nm, which are stable for several days. The solvent exhibit a band at 205 nm. The colloid concentration is approximate 6.45 x10^{-4} M.

The TEM studies reveal a wide average of particles size ranging from 20 to 30 nm. The particles look spherical and the electron diffraction shows the presence of Gd and Gd_2O_3. The EDX analysis gives the presence of Gd metal in the colloid. The colloidal particles exhibits a negative charge.

After solvent evaporation active white powders were obtained. The FTIR of the solid shows bands at 2183 cm^{-1} (ν CO), 1728 cm^{-1} (ν C=O), 1623 cm^{-1} (ν C=C) and 891 cm^{-1} (ν CH_2). The ν OCH_3 at 1260 cm^{-1} shows the presence of residual 2-methoxyethanol. The FAR-IR exhibit a very broad band at 384 cm^{-1} most likely due to metal-metal stretching mode.

The thermogravimetric studies reveal a first thermal decomposition at 105 ºC (water) and a second weight loss at 549 ºC (47%).

15:50 poster B-43

Magnetic studies of metallopolymeric nanocomposite produced by thermal decomposition of iron acrylate complex

Nataliya Nedelko[1], Anna Ślawska-Waniewska[1], Michal Kopcewicz[2], Marcin Leonowicz[3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: natal@ifpan.edu.pl

Magnetic metallopolymeric nanocomposites have recently been considered as an important materials because of their potential application, especially in medicine, biotechnology and ecology. In this work we report on the investigations of low temperature magnetic properties of a polymer matrix nanocomposite produced by thermal decomposition of Fe(III) acrylate complex ($[Fe_3O(CH_2{=}CHCOO)_6]OH{\cdot}3H_2O$) at 663 K. After such pyrolysis process a polyphase composite have been obtained, where the main product are magnetite nanocrystalites randomly distributed within the polymer matrix. The Mössbauer spectroscopy investigations, performed in the temperature range 80-295 K, show the superimposition of two contributions - a magnetically split component and a quadrupolar doublet. With increasing temperature the relative intensity of the doublet increases, indicating that it can largely be ascribed to isolated superparamagnetic particles. However, even at room temperature the magnetically split component is still observed pointing to the existence of large grains of particle agglomerates. The ZFC-FC magnetization curves exhibit irreversibility up to 300 K, but their shape is characteristic of system dominated by interparticle interactions rather than superparamagnetism. The magnetization curves display the coercivity up to 300 K and its decrease with temperature cannot be described by a simple thermally activated process. The strongest changes of several magnetic parameters (ZFC magnetization, coercivity, remanence) are observed below ~100 K. This can be a sign of a transition of isolated particles into the superparamagnetic state. Considering, however, that at low temperatures the exchange anisotropy appears (as evidenced by a displacement of the FC hysteresis loop) the other possible explanation is the freezing process of a non-collinear

spin structures either related to the disordered surface layer of particles or cluster-glass-like behaviour of particle agglomerates.

15:50 poster B-44

Magnetic Encapsulates: Nd-Fe-B@C and Fe@C for drug delivery systems.

Michal J. Wozniak[1], Michał Bystrzejewski[2], Andrzej Huczko[2], Waldemar Kaszuwara[1], Jan Kozubowski[1], Hubert Lange[2], Marcin Leonowicz[1], M. Zdrojek[3]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Warsaw University, Faculty of Chemistry, Pasteura 1, Warszawa 02-093, Poland* **3.** *Warsaw University of Technology, Faculty of Physics, Koszykowa 75, Warszawa 00-662, Poland*

Contact: wozmi@inmat.pw.edu.pl

Carbon nanostructures containing ferromagnetic material could find different application such as, drug delivery systems and medical contrast elements.

In the current work, magnetic encapsulates: Nd-Fe-B@C and Fe@C for drug delivery systems are described.

HRTEM investigation shows the product is composed of metal crystals encapsulated in carbon shells. The size of the particles is in the range of 10-100nm. XEDS spectra indicate the carbon encapsulates contain Fe and Fe-Nd crystals. MFM images show that nanoparticles demonstrate ferromagnetic behaviour.

After the acid treatment on the surface of encapsulates COOH groups should be present. These COOH groups can be easily substituted by drug molecules and targeting ligand molecules. The presence of the carbon shell protect magnetic nanocrystals from biodegradation when exposed to the biological systems. The encapsulates can be inject in the blood vessels and target with external magnetic field what will be discussed. The interaction between encapsulates and human living cells will be discussed as well.

15:50 poster B-45

Magnetic properties of anisotropic Nd-Fe-B resin bonded magnets

Marcin Dospial[1], Danuta D. Płusa[1], Urszula U. Kotlarczyk[1], Barbara B. Ślusarek[2], Tadeusz T. Mydlarz[3]

1. *Institute of Physics, Częstochowa University of Technology, Al. Armii Krajowej 19, Częstochowa 42-200, Poland* **2.** *Tele- & Radio-Technical Institute of Warsaw, Warszawa, Poland* **3.** *International Laboratory of High Magnetic Fields and Low Temperatures (ML), Gajowicka 95, Wrocław 53-421, Poland*

Contact: mdospial@wp.pl

The magnetic properties of an anisotropic, epoxy resin bonded magnet made from Magnequench powder of MQA-T type was investigated. The reversible magnetization processes and the magnetic parameters were examined by measurement of virgin magnetization curve, the major and minor hysteresis loops using a VSM mounted in a 14T electromagnet and the LakeShore VSM mounted in a 2T electromagnet. The coercivity and remanence versus the maximum magnetizing applied field were determined. As it results from the virgin magnetization curve shape and the field dependence of coercivity the magnetization reversal mechanism appeared to be the pinning one.

15:50 poster B-46

Vibration reduction and heat control in AC motors using ferrofluids

Namasivayam Murugesan, Jayachandran Nagarajan

Velammal engineering college, velammal nagar, ambattur redhills road, chennai 600066, India

Contact: shiv3086@yahoo.co.in

INTRODUCTION:

To reduce vibration and control heat in AC motors using ferrofluids.

DESCRIPTION:

Ferrofluid is a three component magnetic colloidal system consisting of subdomain magnetic particles, carrier liquid and surfacant. Ferrofluid behaves as a homogenous system even in an external Magnetic field. All the constituents of fluid range from 2-20 nm and fluid can offer good damping, sealing, heat transfer with reference to the applied field.

Basically Ferromagnetic materials are characterized by Curie temperature where Magnetization decreases as Temperature increases. Ferrofluid also has the same Curie temperature behavior. Fe3O4 based Ferrofluids have Curie temperature of 575 C (Ideal).

Major problem that occurs in an A.C. motor (I/P power) is due to heat gathered in poles. Ferrofluids, when placed on top every pole ensure good heat transfer. At an instant of high A.C. flux (peak voltage), intensive flux lines (reduce as moved away from pole) attract more fluid near the pole, and some are left behind. Heat conduction to the fluid takes place. Heating of particles causes the reduction in magnetization, so particle away from pole of comparatively higher magnetic moment gets attracted and particles with heat is moved away, causing Forced Heat Convection.

Convection of heat increases at high flux, low Curie temperature, high thermal conductivity, low viscosity, preferred volume/fraction of fluid.

Ferrofluids also play the role of acoustical mounts and soft vibrating materials rettored to acoustics. As these are held between rigid supports (outer cavity, platform and dynamic

parts) reduces vibration by offering damping. As 50 expansions and compressions per second frequency are carried without any tension and dynamic action is lubricated, vibration is reduced.

Nanosized magnetic particles move randomly in the cavity by placement of closed coils. Current can be reduced in it.

Vaporization in fluid is less, so greasing is offered better than wax

[ABSTRACT TRUNCATED TO 2000 LETTERS]

Tuesday, 6 September

Parallel Session
Tuesday afternoon, 6 September, 14:00
Main Building, room 219
Oksana I. Bodak presides

14:00 invited oral

Small Angle Neutron Scattering Investigations of Spin Disorder in Nanocomposite Soft Magnets

Oscar Moze[2], Carlo Vecchini[1], Andreas Michels[3], Kiyonori Suzuki[5], John Michael Cadogan[6], Klaus Pranzas[4], Joerg Weissmueller[7]

1. *INFM-S3 National Research Centre and Physics Dept, University of Modena and Reggio Emilia, Italy, Modena, Italy* **2.** *INFM-S3 National Research Centre and Physics Dept, University of Modena and Reggio Emilia, Italy, Modena, Italy* **3.** *Technische Physik, Universität Saarbrücken, Germany, Saarbrucken, Germany* **4.** *GKSS Research Centre, Geesthacht, Germany, Geestacht, Germany* **5.** *School of Physics and Materials Engineering, Monash University, Australia, Melbourne, Australia* **6.** *School of Physics, University of New South Wales, Australia, Sydney, Australia* **7.** *Forschungszentrum, Karlsruhe, Institut fur Nanotechnologie, Germany, Karlsruhe, Germany*

Contact: vecchini.carlo@unimore.it

The technique of small angle neutron scattering (SANS) furnishes unique information on the length scale and local magnetic anisotropy at the nanoscale in nanocomposite ferromagnets. Such information is not presently available using any other microscopic technique. The basic principles and results of the SANS technique will be presented with regard to a unique and unexpected observation of a dipole field controlled spin disorder in a nanocomposite ferromagnet, FeZrBCu, which consists of bcc Fe particles embedded in a magnetically active amorphous matrix.

14:45 oral

Formation and characterization of nanocrystalline binary oxides of yttrium and rare earths metals

Giora Kimmel[1], Jacob Zabicky[1], Elena Goncharov[1], Dmitry Mogilyanski[1], Arie Venkert[3], Yishai Bruckental[2], Yosef Yeshurun[2]

1. *Institutes for Applied Research, Ben Gurion University of the Negev, Beer-Sheva 84105, Israel* **2.** *Department of Physics, Bar-Ilan University, Ramet-Gan, Israel* **3.** *Physics Department, Nuclear Research Center Negev (NRCN), Beer-Sheva 84190, Israel*

Contact: kimmel@bgu.ac.il

Binary oxides of yttrium (Y) and rare earths (R) are used for their varied chemical and physical (e.g., optical and dielectric) properties. Coprecipitated xerogels, which are mixtures of hydroxides and oxides of Y and R, were fired in air at constant temperatures in the range from 100 to 1400ºC for 3h. The lowest temperature at which a pure oxide is obtained varies with composition. Whereas Y_2O_3 has always the Mn_2O_3 type cubic structure (cI80), pure R_2O_3 oxides have also other crystal structures. Therefore, formation of a single phase as a solid solution of yttria and a rare earth oxide is not certain, and is dependent on the xerogel composition and the temperature of annealing. A special case of dissimilarity is the attempt to alloy together Y(III) oxide with an R(IV) oxide. Solid solutions containing Y(III) and R(III) have chemical formula $(R_xY_{1-x})_2O_3$, and those containing Y(III) and R(IV) have formula $(R_xY_{1-x})_2O_{3+x}$. The latter compositions require a peculiar type of cubic structure, based on the Mn_2O_3 structure. The binary oxides of Y(III) and R(III) afford ideal solid solutions where the unit cell parameter obeys Vegard's law. In the case of Y(III)-R(IV) oxides, the alloys are not ideal binary solutions and the unit cell parameters are not a linear function of the atomic ratio x = R/(R+Y). The cell parameters usually are larger for alloys fired at lower temperatures. XRD line broadening analysis points to line broadening as being mainly due to small crystallite size whereas the contribution of microstrain to line broadening is relatively small. The XRD results are confirmed by TEM observations. The magnetic properties are strongly dependent on the particle size. Nanocrystalline Sm(III) oxide and the $SmYO_3$ solid solution are paramagnetic; however, superparamagnetism is also sometimes detected

15:15 oral

Synthesis and Properties of Iron Colloids and Fine Powders

Galo Cardenas[1], Yanko Moreno[2], Octavio Peña[3], Viviana Delgado[1]

1. *University of Concepcion, Department of polymers, Chile, Chile* **2.** *University of Concepcion, Department of Inorganic and Analytical Chemistry, Chile, Chile* **3.** *Institut de Chimie de Rennes, Universite de Rennes ., Rennes, France*

Contact: gcardena@udec.cl

The study of Fe nanoparticles, obtained by the Chemical Liquid Deposition Technique (CLD) [2], has great interest due to their magnetic and electronic properties [1]. Fe colloids were prepared by cocondensation of the metal at 77K with three different solvents.The particle size , determined by TEM, of the colloidal dispersions ranges from 3.94 nm to 6.70 nm. Electron diffraction exhibits the presence of FeO. Electrophoretic measurements such as colloid charge and zeta potential show the electrical charge of the colloids. The electrophoretic mobility reveals positive charge for Fe-2-methoxyethanol and Fe-2-propanol colloids being $\mu=3.42*10^{-9}$ and $3.87*10^{-9}$ m^2/V s, respectively. The colloids showed very high stability at room temperature, some of them over 6 months.

The UV-VIS for ethanol shows two adsorption bands at 201 and 320 nm, respectively which are coincident with theoretical spectrum calculated by Creighton [3].

The FTIR analysis of the solids shows two bands at 182 cm^{-1} to the ν Fe-Fe and other band at 480 cm^{-1} assigned to the ν Fe-O. The TGA was carried out between 25 to 550° C. The solids are very stable with a maximum weight loss of 40% near 240° C. The magnetic measurements indicate a histeresis magnetic moment vs applied field that correspond to a soft ferromagnetic system.

Acknowledgements

The authors would like to thank the financial support of Grant FONDECYT 1040456.

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Wednesday, 7 September

Parallel Session

Wednesday morning, 7 September, 9:00

Main Building, room 219

Andrzej Szytula presides

9:00 invited oral

Structure of iron-rich compounds in the Yb-Fe-Al and Yb-Fe-Ga systems.

Radovan Cerny

University of Geneva, 24 quai Ernest-Ansermet, Geneva 1211, Switzerland

Contact: Radovan.Cerny@cryst.unige.ch

Iron-rich rare-earth transition-metal alloys are of interest as constituents for permanent magnets. Ytterbium containing materials have not yet been studied in great detail because of problems with sample preparation. Our recent X-ray diffraction studies on single crystals [1,2] and powders [3] having compositional ratios close to Yb/(Fe,Al) or Yb/(Fe,Ga)=2/17 revealed four distinctly different structures, two having hexagonal symmetry ($LuFe_{9.5}$ and $TbCu_7$ type) at low and intermediate Al or Ga and Yb contents, respectively, and two having rhombohedral symmetry ($PrFe_7$(A) and $PrFe_7$(A+B) types) at high Al or Ga and Yb contents. All have a common hexagonal $TbCu_7$ type subcell and contain chains made up of Yb and $Fe(Al)_2$ or $Fe(Ga)_2$ dumbbells running along the c-axis that substitute for each other with various ordering schemes and concentrations. The subcell ratio c/a depends on both the concentration of the dumbbells, for which it generally increases, and the structure type (dumbbell order). We define an order parameter capable of describing the dependency of the subcell ratio c/a on the $Fe(Al)_2$ or $Fe(Ga)_2$ dumbbell ordering, irrespective of structure type, as the probability of finding two dumbbells within a chain that are separated by one Yb atom only.

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9:45 invited oral

Influences of Co on structural and magnetic properties of $R_3(Fe_{1-x}Co_x)_{29-y}M_y$ (R = rare earth metal, M = transition metal) intermetallic compounds

Orestis Kalogirou[1], Charalambos Sarafidis[1], Konstantinos G. Efthimiadis[1], Margaritis Gjoka[2]

1. *Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece* **2.** *Institute of Materials Science, NCSR "Demokritos", 153 10 Ag. Paraskevi, Attiki, Greece, Athens 153 10, Greece*

Contact: orestis@lab3.physics.auth.gr

The $Nd_3(Fe,Ti)_{29}$-type intermetallic compounds (3:29) crystallise in the A2/m space group and consist of 2:17R-type and 1:12-type segments in a ratio 1:1. The study of the effect of Co substitution on the structural and magnetic properties of $R_3(Fe_{1-x}Co_x)_{29-y}M_y$ (R = Y, Nd, Tb, Dy; M = Ti, V, Cr, Mn; $0\leq x\leq 1$; y=0.9-7.0) is presented. For the compounds with low Co content ($0\leq x\leq 0.4$) the unit cell volume is decreasing as the Co content increases. ^{57}Fe Mössbauer spectroscopy has shown that the Co atoms avoid those Fe sites shared with the Ti atoms, i.e., the dumb-bell sites. The Curie temperature values, T_C, increase monotonically with x. For the compounds with high Co content ($0.6\leq x\leq 1$) the situation is rather different. Starting with the 3:29 stoichiometry $[Tb_3(Fe_{1-x}Co_x)_{27.4}V_{1.6}$ and $Dy_3(Fe_{1-x}Co_x)_{27.8}V_{1.2}$; x= 0.6, 0.8, 1.0] a disordered variant of the hexagonal Th_2Ni_{17}-type structure is formed. In $Y_3(Fe_{1-x}Co_x)_{29-y}Cr_y$ the monoclinic 3:29 structure is retained if the amount of the stabilizing element is increased. The lattice parameters are changing in a constant manner with the increase of Co showing that the replacement of Co for Fe is a continuous procedure. The change in the monoclinic angle shows an interesting difference compared to the compounds with x<0.3; it increases with Co indicating that high Co concentration (x>0.3) is pushing the structure to its limit. The T_C values of the compounds are reduced; this should be attributed to the larger Cr content. The same behavior was observed in the case of Nd based products with $0.6\leq x\leq 1$. The T_C values present a considerable difference (more than 100 K for x=0.6). Preliminary studies show that in the $Nd_3(Fe_{1-x}Co_x)_{29-y}Cr_y$ samples the main phase is a rhombohedral Th_2Zn_{17}-type structure. In all cases the considerable effect of Co substitution on the magnetocrystalline anisotropy will be discussed.

Wednesday Poster Session
Wednesday afternoon, 7 September, 15:50

Thursday, 8 September

Parallel Session
Thursday morning, 8 September, 9:00
Main Building, room 219
Orestis Kalogirou presides

9:00 invited oral

Magnetoresistivity studies of intermetallic uranium compounds and their impact on the exhibited phenomena

Robert Troć

Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Wrocław 50-950, Poland

Contact: R.Troc@int.pan.wroc.pl

In recent years the occurrence of a large reduction of the electrical resistivity upon application of a magnetic field, called as a giant magnetoresistvity (GMR)effect,first of all observed in some magnetic-multilayer systems like Fe/Cr or Co/Cu, has attracted much attentions due to the possible applications.

It turned out that GMR is also possible in various uranium based compounds which crystaliize in layered-like structures. In the latter case one should drive a given antiferromagnetic uranium compound through a metamagnetic (MT) transition towards an induced ferromagnetic aligment.This is connected with a reorientation of the 5f-magnetic moment, which can be also the origin of GMR effects in multilayers. Thus this causes a drastic reduction of the electrical resistivity, which may exceed even that of multilayer systems.The high resistance state in bulk uranium antiferromagnets is attributed to the reduction of effective carrier concentration or change in the FS topology by arising the Fermi level gapping. The transverse magnetoresistance may give also the overall information on the Fermi surface of such uranium based systems (with open or closed orbits)allowing to distinquish a compensated from an uncompensated kind of metal. For some uranium materials the temperature dependence of MR gives evidence of an enormous scattering due to the spin fluctuations being responsible for their superconducting properties.

9:45 invited oral

Magnetocaloric effect in d-metal phosphides and arsenides

Daniel Fruchart[1], F. Allab[2], M. Balli[1], D. Gignoux[3], E.K. Hlil[1], A. Lebouc[2], Nataliya Skryabina[1,4], J. Toboła[5], P. Wolfers[1], R. Zach[5]

1. *Laboratoire de Cristallographie du CNRS (LC), BP 166, Grenoble cedex 9 ·38042, France* **2.** *LEG (ENSIEG), BP 46, St. Martin d'Heres 38402, France* **3.** *Laboratoire Louis Néel, CNRS (LLN), 25 Av. des Martyrs, Grenoble 38042, France* **4.** *Perm State University (PermSU), 15 Bukireva st., Perm 614990, Russian Federation* **5.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: fruchart@grenoble.cnrs.fr

We present a wide analysis of both the crystal structure peculiarities and the different aspects of magnetic properties, leading to better understand the many magneto-elastic effects exhibited by the MM'X series of transition metal phosphides and arsenides. Moreover, systematic neutron diffraction experiments have been performed thus allowing anticipate on the different contributions to entropy. Besides high magnetic field measurement namely under pressure were performed to quantify the most pertinent (T, P, H) phase diagrams. Finally, fair insights of the electronic polarisation instabilities were deduced from KKR-CPA band structure calculations. All the previous fundamental analyses lead to comfort the very interesting magneto-caloric contributions exhibited by the MM'X

series. Comparison is made with other high performance MCE systems.

Parallel Session
Thursday afternoon, 8 September, 14:00
Main Building, room 219
Wojciech Suski presides

14:00 invited oral

Half-metallic ferromagnetic ground state in CePdSb

Andrzej Ślebarski

University of Silesia, Institute of Physics, Uniwersytecka 4, Katowice 40-007, Poland

Contact: slebar@us.edu.pl

The CeMX compounds, where M is a transition metal, and X is an sp element, have a number of different types of ground state including magnetic, metallic, and insulating, and they exhibit anomalous physical properties, such as the Kondo effect, heavy-fermion behavior, valence fluctuations, etc. Recently, a Kondo-lattice state has been proposed for ferromagnetic CePdSb, which seems to be unusual. Many Kondo systems have an antiferromagnetic ground state due to the antiferromagnetic nature of the coupling of Ce 4f and the conduction electrons, however, the coexistence of ferromagnetism and Kondo behavior is an unexpected feature. In addition, our band structure calculations for CePdSb show a state very close to a half-metallic ferromagnets (HMF), having an energy gap for the partial density of states for one of the spin projections. On the base of Kondo-like HMF ground state properties of CePdSb we discuss its magnetic properties and electrical resistivity data. We also investigate the solid solutions $Ce_{1-x}La_xPdSb$ to determine the influence of alloying on the nature of the ground state accross the series, when trivalent magnetic Ce in CePdSb is replaced by La. We also report an unusual temperature dependence of the magnetic susceptibility, resistivity and specific heat for $Ce_{0.1}La_{0.9}PdSb$ compound, which exhibits the non-Fermi liquid-like behavior at the low temperatures.

14:45 oral

Magnetostructural phase transitions in ternary rare earth intermetallics

Andrzej Szytula, M. Duraj, Ł. Gondek E. Wawrzyńska,

Jagiellonian University, Institute of Physics (IF UJ), Reymonta 4, Kraków 30-059, Poland

Contact: szytula@if.uj.edu.pl

In this work properties of some compounds with the layered crystal structure are discussed. These compounds are in important alternative for delineating fundamental issues concerning transport in 2D magnetic materials. The SmMn2Ge2 crystallizes in the tetragonal ThCr2Si2-type crystal structure which is formed of the monoatomic layers along the c-axis with the following sequence Sm–Ge–Mn2–Ge. Magnetic data indicate complex magnetic properties. With decreasing temperature the change of the magnetic properties with the following sequence: antiferro- (between 385 and 341 K), ferro- (341–153 K), reentrant antiferro- (153–106.5 K) and reentrant ferro- (below 106.5 K) magnetic behavior is observed. Temperature dependence of the lattice parameters a and c shows the jump of these parameters equal $\Delta a \approx 0.2$ % at T1=106.5 K and T2=153 K and $\Delta c \approx 0.02$ % at T1 and ~0.06 % at T2. In these temperatures also the change of the electrical $\Delta\rho/\rho \approx 0.056$ at T1 and 0.05 at T2 and magnetoresistivity 3.2 % at T1 and 5.4 % at T2 is observed. It was suggested that due to the layered arrangement of the Mn layers in this compound an analogy to GMR effects in artificial magnetic multiplayer systems exists. This problem is discussed on the other isostructural compounds. Nd3Mn4Sn4 crystallize in the orthorhombic layered structures of Gd3Cu4Ge4. Magnetic and neutron diffraction data give that this compound is antiferromagnet with TN1=210 K. Below TN1 Mn moments form a collinear magnetic structure. Below

TN2=60 K the Nd moments also order and form collinear magnetic structure while the magnetic order of the Mn moments remains unchanged. The temperature dependence of lattice parameters a, b and c and unit cell volume V indicates a positive sign of the magnetostriction at TN2 and negative sing at TN1.

In TbRhSi the change of the magnetic structure is connected with the change of the lattice parameters.

15:15 oral

Transport properties in the pseudobinary Er_5Si_4 compound

Joao P. Araujo[1], Andre M. Pereira[1], Jose Teixeira[1], M. E. Braga[1], F Correia[1], R P. Pinto[1], J. B. Sousa[1], L. Morellon[2], C. Magen[2], P. A. Algarabel[2], Ricardo M. Ibarra[2]

1. *Departamento de Fisica and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal* **2.** *Universidad de Zaragoza-CSIC, Facultad de Ciencias, Pedro Cerbuna 12, Zaragoza 50009, Spain*

Er_5Si_4 belongs to the $R_5(Si_xGe_{1-x})_4$ family (R=rare earth), which has been intensively studied in recent years due to the appearance of a giant magnetocaloric effect. This effect is associated with a simultaneous 1st-order magnetostrutural transition which causes drastic changes on the crystal lattice, magnetic state and Fermi surface, inducing large entropy changes. However in Er_5Si_4 the two transitions are decoupled: the 1st-order structural transition occurs at T_S =190K, from the orthorrombic (O) to the monoclinic (M) phase below T_S, and the 2nd-order magnetic transition occurs at T_N =30K

from PM to AFM below T_N. We can therefore separately study the physical effects of each transformation.

Here we report detailed measurements of the thermopower S(T), electrical resistivity ρ(T) and magnetization on a polycrystal of Er_5Si_4 between 10 and 300 K. Particular attention is given to the corresponding behaviour near the structural (T_S) and magnetic (T_N) transitions.

The structural transition produces a large thermopower step (ΔS), associated with the change in crystal symmetry (O→M) and Fermi surface modification. As a consequence we have from S~-10 μVK^{-1} below T_S and S~-4 μVK^{-1} above T_S. The relative changes of the ρ(T) is however considerably smaller (Δρ/ρ~10%). Large thermal hysteresis (ΔT~10K) is also associated with the structural transition.

The magnetic transition T_N produces no discontinuities on S and ρ, but a pronounced decrease with temperature occurs in |S| and ρ within the AF-phase. This effect is analyzed in terms of electron-spin scattering within the Kasuya mean field treatment. At low temperatures ρ reaches its residual value imposed by electron impurity scattering, whereas S gets fairly small and changes from negative to positive (with dS/dT>0).Important spin fluctuation effects are observed in dρ/dT and dS/dT near T_N, extending in the PM phase up to ~$2T_N$.

Friday, 9 September

Parallel Session
Friday morning, 9 September, 9:00
Main Building, room 219
Carlo Vecchini presides

9:00 oral

Soft magnetic alloys with amorphous and ultrafine grain structure

Nataliya Skryabina

Perm State University (PermSU), 15 Bukireva st., Perm 614990, Russian Federation

Contact: magicflight@permonline.ru

We aim to review the main characteristics of the magnetic properties and the magnetic domain structure of different types of rapidly quenched alloys. Then, external parameters governing the stability of the microstructure will be considered for, those possibly influencing the relaxation processes. Correspondingly, hydrogen insertion in rapidly quenched alloys having ferromagnetic properties will be discussed as one of the effective tool acting on the microstructure. A comparative analysis between rapidly quenched and other soft magnet alloys exhibiting similar trends will be made based on the nature of alloyed elements, the chemical composition, the atomic size and chemical coordination, the type of structure, etc. We will conclude with a brief description of selected achievements and listing the perspectives.

9:30 oral

Low dimensional double molybdates and tungstates – strongly anisotropic magnetic materials

Eugene N. Khatsko

Institute for Low Temperature Physics, 47 Lenin ave., Kharkov 61103, Ukraine

Contact: khatsko@ilt.kharkov.ua

Magnetic properties of double rare earth molybdates and tungstates (general formulae - $MR(XO_4)_2$, where M = Cs, K, Rb, R = rare earth, and X = Mo, W) are of interest because these crystals display a number of fascinating peculiar its features associated with a low symmetry of the crystal lattice, a relatively small value of exchange, a considerable contribution of the dipole-dipole interaction of magnetic ions, the low dimensionality of a magnetic structure, and the competition between magnetic and electric interaction, which determines the energy spectrum of the magnetic ions and its features.

The layered structure and strong electron-phonon interaction, together with a degenerate or quasidegenerate electronic ground state, make these compounds structurally unstable; they typically exhibit several structural phase transition induced by weak external fields (temperature or magnetic).

It should be mentioned promising use these materials in engineering.

The magnetic susceptibility, the magnetization, and resonant investigations of this class of compounds are reported. The measurements were performed at temperatures between 0,5 and 100 K in magnetic fields up to 7 T along three principal magnetic axes. The magnetic properties are strongly anisotropic in all investigated temperature region, that is dictated by crystal structure and intricate character of magnetic interactions. The EPR investigation carried in wide range of frequencies (10-190 GHz) at 1.8-30 K allow to establish detailed characteristic and quantity of nonequivalent magnetic centers, including g-factors parameters and structure of magnetic ions energy spectrum.

Magnetic susceptibility measurements indicate diverse quantity and type of exchange interaction in different directions.

On the base of experimental data the magnetic structures of compounds are proposed. They demonstrate variety of structures – from simple collinear to noncollinear many sublatices ones.

10:00 oral

ATOMIC MIGRATION ON ORDERING IN BULK AND NANOSTRUCTURED FePt L10-ORDERED INTERMETALLIC COMPOUND SHOWING HIGH MAGNETIC ANISOTROPY.

Mirosław Kozłowski[1], Kinga Zapała[1], Marcin Bednarz[1], Veronique Pierron-Bohnes[2], Wolfgang Pfeiler[3], Marcus Rennhofer[3], Bogdan Sepioł[3], Gero Vogl[3], Rafal Kozubski

1. *Jagiellonian University, Institute of Physics (IF UJ), Reymonta 4, Kraków 30-059, Poland* **2.** *Institut de Physique et Chimie des Materiaux de Strasbourg, UMR7504, CNRS - ULP, 23, rue du Loess, BP 20, Strasbourg 67037, France* **3.** *University of Vienna, Institute of Materials Physics, Vienna, Austria*

Contact: mir-on@o2.pl

Polycrystalline bulk and epitaxially deposited multilayer (200A Pt/[20A 57FePt, 30A 56FePt] x10/MgO) samples of stoichiometric L10-ordered FePt intermetallic compound were examined for "order-order" kinetics (resistometry of bulk at 650 K < T < 1050 K and of the multilayer at 650 K < T < 800 K) and Fe diffusion (grazing incidence nuclear resonant scattering of X-rays (NRS) from multilayer at 773 K < T < 873 K.

Two time scales (fast and slow relaxation components) were observed in "order-order" relaxations. Strong and discontinuous change of chemical ordering dynamics was detected in bulk at T0 = 800 K, definitely away from the Curie point (735 K). The change affected, however, only the slow relaxation component, whose activation energy dropped from about (2,7 ± 0,1) eV above T0 (the value close to the activation energy for Fe* tracer diffusion in FePt measured traditionally at high temperatures) to (1,5 ± 0,3) eV below T0. The rate of the fast relaxation component showed very weak temperature dependence yielding the activation energy of about 0,3 eV. The analogous relaxations measured in the multilayer showed the activation energy of about 1,6 eV.

Dynamics of Fe in the multilayer was investigated with nuclear resonant scattering (NRS) of synchrotron radiation in grazing incindence geometry. A new method for investigating diffusion in very slow diffusing systems was used. The FePt multilayer samples were successively annealed at four temperatures between 773 K and 873 K followed by the reflectivity measurement at room temperature. The vanishing of the superstructure Bragg peaks was observed. From the intensity loss the activation energy for Fe diffusion in a FePt thin film was evaluated as equal to 1,7 eV.

It is concluded that diffusion mechanism in FePt changes at about 800 K – as directly evidenced by resistometry carried out in bulk over sufficiently wide temperature range.

10:30 oral

Magneto-optic lens using ferrofluids

Jayachandran Nagarajan, Namasivayam Murugesan, Shahil Kirupavathy, Devashankar Srinivasan

Velammal engineering college, velammal nagar, ambattur redhills road, chennai 600066, India

Contact: techosplash@yahoo.com

Ferrofluid is a three component magnetic colloid that behaves as a homogenous system even in applied magnetic field.γ-Fe_2O_3 water base ferrofluid (maghemite) has a good transmittance at a low magnetic volume fraction (less than 1%)it is possible to obtain good magneto-optic effect and transmittance.

Without any applied field ferrofluids are isotropic. But when a magnetic field is applied ferrofluids acquire an optical anisotropy two cases are possible. Faraday's rotation of plane polarized light when magnetic field is parallel to the light beam.

Anisotropy is linear in perpendicular configuration.If ferrofluid is placed in a cylinder which acts as an electromagnet producing lines of flux in direction of light, due to attraction of flux linear more fluid is attracted near cylinder wall producing a structure as shown.

This pattern acts as a lens (distance for light to travel varies instantly).And such a pattern can be varied depending upon the field of electromagnet and causing to produce auto focal lens. If ferrofluid added to sol-gel sample to produce sol-gel doped ferrofluid solid structure of ferrofluid with higher transmittance is obtained.

When sol-gel doped ferrofluid produced under zero magnetic field has good transmittance and residual birefrigerence ($\Delta n. \sim 8.8*10^{-7}$, which may be due to earth magnetic field. On eliminating the residual birefringence (equivalent to cello tapes) and on preparing in different shapes gives rise to constant focal length lens.

Uses:

· Transmittance varies with volume fraction for different wavelength and auto focusing can be obtained with wavelet filters.

· Can be auto focused easily to read VCD, DVD, MP3, MP5

· Can be used in focusing of laser with wavelet filter.

· Can be used to develop artificial human eye lens.

Symposium C

Shape memory materials for smart systems

Welcome

Recent progress in research and fabrication of shape memory materials has opened the opportunity to develop novel smart systems, which are capable of sensing and actuating and thus may respond to their thermal, mechanical and magnetic environment in a controllable way. Meeting the demands of smart systems requires the development of new multifunctional materials, detailed material characterization, the development of models of material behavior and technologies for implementation. The symposium emphasizes both the basic science and early engineering development in this rapidly evolving interdisciplinary field.

Special sessions are devoted to recent developments in the fields of conventional and ferromagnetic shape memory materials. In particular, ferromagnetic shape memory alloys experienced a widespread research boom in the last decade as they exhibit a unique combination of high energy densities, thermoelastic and ferromagnetic properties. One example are the Heusler ferromagnetic shape memory alloys Ni-Mn-Ga, which reveal in bulk single crystalline form, e.g., the giant magnetostrain effect, the inverse effect of strain-induced change of magnetization and a large magnetocaloric effect. It is expected that the multiple transducing effects being present in ferromagnetic shape memory alloy materials will enable various new solutions for actuation and sensing applications.

In order to stimulate the dialogue between basic research and engineering, further sessions focus on the modeling of material behavior and the engineering of practical devices and systems. For many applications, the design and simulation of three-dimensional structures is of prime importance. This requires the development of suitable models for predicting the thermo-mechanical, magneto-mechanical or the fully coupled thermo-magneto-mechanical response of the material. Numerical field simulations using, e.g., the finite element method are demanded. Further emphasis is on the combined response of shape memory materials with other functional materials taking into account the constraints of state-of-the-art technologies. New trends in the application of shape memory alloys will be presented ranging from the medical field to the maturing field of microsystems technology.

Welcome to this interdisciplinary event covering material science, mechanics and applications.

Organisers

- Manfred Kohl, Forschungszentrum Karlsruhe, IMT, Karlsruhe, Germany
- Volodymyr Chernenko, National Academy of Sciences of Ukraine, Kiev, Ukraine
- Etienne Patoor, ENSAM, CER de Metz, Metz, France
- Stefano Besseghini, CNR-IENI Sez. di Lecco, Lecco, Italy
- Bogdan Raniecki, Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland

Programme

Monday, 5 September

Shape Memory Materials 1
Monday afternoon, 5 September, 14:00
Main Building, room 315
Manfred Kohl, Shuichi Miyazaki presides

14:00 invited oral

Deformation instability and pattern evolution during phase transformation

Qingping Sun

Hong Kong Univ. of Science and Technology (HKUST), Clear Water Bay, Hong Kong Kowloon, Hong Kong

Contact: meqpsun@ust.hk

This talk reports the deformation instability and pattern evolution during stress-induced martensitic phase transformation in a superelastic NiTi polycrystalline shape memory alloy microtube. High-speed data and image acquisition techniques were used to investigate the dynamic as well as quasistatic events which took place during displacement controlled quasi-static tensile loading/unloading process of the tube. These events include dynamic formation, self-merging, topology transition, convoluted front motion and front instability of a macroscopic deformation band (domain). Nonlocal modeling and numerical simulations on the experimental phenomena are also reported briefly in the talk.

14:45 oral

Investigation on the Mechanism of the Stress-induced Martensitic Phase Transformation of Superelastic Shape Memory Alloys

Heinrich Kern[1], Paul Gümple[2], Stefan Gläser[3]

1. *Institute of Materials Technology, Technical University of Ilmenau, Gebäude "Werkstoffe 1", Ilmenau 98693, Germany* **2.** *Fachbereich Maschinenbau (MA), Fachhochschule Konstanz, Brauneggerstrasse 55, Konstanz 78405, Germany* **3.** *ETO MAGNETIC KG, Hardtring 8, Stockach 78333, Germany*

Contact: Kern@tu-ilmenau.de

Superelastic shape memory alloys (SMA) are able to change the phase when charged with an external mechanical tension. On this occasion a material, that shows e.g. an austenitic phase at room temperature will be changed into stress-induced martensitic phase. On the stress-strain-diagram this change of the lattice structure can be seen in a relatively wide ranged pseudo-plastic deformation at nearly constant force.

During the elongation process in the pseudo-elastic field a discontinuous change of the electrical resistance occurs. This change of electrical resistance takes place in a much more distinctive way as it has to be expected depending of the reduction of the cross sectional area of the specimen and its length. The reason of this change in resistance can be derived from the different specific resistances of the two lattices. Some conclusions concerning the mechanical processes during phase transformation can be derived from the resistance dependency on elongation velocity and elongation behaviour.

Experimental results and a differentiation of the resistance influenced processes during the elongation of a superelastic wire will be presented. In addition, some anomalies such as the dependency on velocity of the processes will be shown and discussed.

Furthermore the presented theories will serve as a basis of discussion

15:00 oral

Stress relaxation during superelastic behavior of TiNi shape memory alloy

Elzbieta A. Pieczyska, Wojciech K. Nowacki, Stefan P. Gadaj, Hisaaki Tobushi

The belt type specimens of TiNi shape memory alloy are subjected to the strain-controlled tension test at room temperature with the strain rate of 5x10-2 s-1.Thermomechanical aspects of martensitic and reverse transformations in TiNi SMA during tension test with stress relaxation sequences are investigated using a thermovision camera. Stress relaxation stages in loading and unloading branches are induced, respectively. The mechanical and the temperature data enable the study of nucleation and development of the phase transformations as well as analysis of the impact of the stress relaxation brakes on both the mechanical specimen behavior and the phase transformations progress. The uniform temperature distribution indicates homogeneity of the stress and the strain state along the specimen. The inhomogeneous temperature distribution on the specimen surface reflects immediately the origin and development of the new phase, due to the significant temperature variations between the parent phase and the new one. A monotonic stress drop is observed during the stress relaxation in both the course of loading and unloading, while, for the temperature-controlled test, in the unloading branch the stress increases under constant strain due to the recovery. Furthermore, on thermograms obtained from the loading branch at the end of relaxation brake, fine bands of deeper temperature occur starting from the grip area. They may manifest nucleation of the reverse transformation at the end of the relaxation brake, caused by temperature decrease due to the relaxation. After relaxation is completed, in both loading and unloading branches of loading, the phase transition processes develop, initiating however at substantially lower stresses and temperatures of the SMA sample.

Acknowledgments

This research has been carried out with the support of Polish Grant No. 4 T08A 060 24, and of the JSPS Grants: No. 13650104 (C), Post-doc PO4774 and Joint Research supported by JSPS and PAS: No. 6612.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

15:15 oral

The Ultrasound Influence on Pseudoelasticity in TiNi

Vasili V. Rubanik

Contact: ita@vitebsk.by

In the work one investigated the behavior of loop of hysteresis of alloy TiNi when realizing pseudoelasticity effect by ultrasound influence with a frequency of 22 kHz and amplitude of mechanical stresses up to 100 MPa. It is determined that simultaneous action of static loading and ultrasonic vibrations leads to a change of the hysteresis loop width. Depending on the conditions of ultrasound influence by loading it is possible both to increase and decrease stress of flow of a material. By unloading ultrasonic vibrations couse a growth of stress of flow, and narrow the hysteresis loop. Thus, the possibility of handling the width of pseudoelastic loop of hysteresis of TiNi due to excitement of ultrasonic vibrations is shown. The model explaining the observed effects due to force and heat constituents of ultrasound influence is offered.

Monday Poster Session

Posters should be on display from Monday to Wednesday
Monday afternoon, 5 September, 15:50

15:50 poster C-01

DMA characterization of shape memory alloys

Stefano Besseghini, Elena Villa

Istituto per l'Energetica e le Interfasi Consiglio Nazionale delle Ricerche Unità di Lecco (IENI Lecco), Corso Promessi Sposi, 29, Lecco 23900, Italy

Contact: s.besseghini@ieni.cnr.it

It is a know fact that the NiTi shape memory alloys have strong dependence of its special properties from the microstructure and its thermomechanical state. The DSC and mechanical test stress-strain vs temperature give many informations on the transformation temperature and on the

pseudoelastic effect but the DMA analyzer is able to investigate all the transition effects, because it is possible to study the transition temperature in both stressed and unstressed condition over a wide temperature range (-160C; 600 C). Moreover the dynamic experiments allow us to analyse the linear mechanical response and the viscoelastic properties in different ranges of strain and frequency. In this work we measure the response of NiTi material in all the phases: austenite, romboedral and martensite. We test the material to different frequencies and in temperature ramp dynamic experiments. The NiTi alloy chosen is a Ni50.2Ti in a ribbons shape of 11x0.5x50 mm size, after two different thermal treatment: 400 C 30‘ and 750 C 10’.

15:50 poster C-02

The effect of heat treatment on the transformation temperature in $Au_{52.5-x}$-$Cd_{47.5}$-Ag_x shape memory alloys

Yuki Matsuoka, Natsuko Kudo

Nara Women's University, Kita-Uoya Nishimachi, Nara 630-8506, Japan

Contact: matsuoka@cc.nara-wu.ac.jp

$Au_{52.5-x}$-$Cd_{47.5}$-Ag_x is one of noble metal based β -phase alloy, which undergoes a martensitic transformation on cooling. The Ag substitution for Au affects martensitic transformation temperature, crystal structure, and the orthorhombic distortion although it is expected that the substitution has no changes in atomic size, electron concentration.

The $Au_{52.5-x}$-$Cd_{47.5}$-Ag_x alloy have been studied and reported in which the Ag composition is below 41 at.%. The alloys have ¥beta-phase structure at room temperature and transform to martensite phase when they are cooled. But the alloy which have x > 41 at.% has not been studied because the transformation temperature go down to below 100 K.

In this work, $Au_{9.5}$-$Cd_{47.5}$-$Ag_{43.0}$ alloy was prepared by melting the component elements. The electrical resistivity was measured from room temperature to 100 K to determine the transformation temperature. The X-ray powder diffraction measurements and Rietveld analysis both at room temperature and at 77 K were performed on this alloy.

At this chemical composition, both of β-phase and α - phase exist at room temperature. Almost β - phase transformed to martensite phase, but α-phase doesn't transform to other phase on cooling. To avoid the coexistence of α - phase, the sample was annealed at 873 K and subsequently quenched into ice-water. The single phase sample of β-phase was obtained by this process. The electrical resistivity measurement and the X-ray powder diffraction measurements were performed again on the quenched sample. The martensitic transformation temperatures were different more than 50 K between these experiments. The difference of transformation temperatures is thought to be caused by the size effect of sample.

15:50 poster C-03

Fabrication of Ti-Ni alloys for proportional control

Tae-hyun Nam, Cheol-am Yu, Yinong Liu, Yun-Jung Lee

Gyeongsang National University (GSNU), 900 Gazwadong, Jinju 660-701, Korea, South

Contact: tahynam@gsnu.ac.kr

Ti-Ni alloys have been known to be very attractive for actuators because of their dual functional ability, i.e. sensing and operating in a single body. Especially, for applications to robotic devices, a linear relationship between the transformation strain and a physical quantity(usually temperature) together with a very small hysteresis is required. However, the transformation strain(ε) occurs steeply in a narrow temperature(T) range(usually within 3-5 K) in most of Ti-Ni based alloys, which makes difficult to control the position of actuators working according to changes in temperature. For an accurate position control responding to temperature changes, Ti-Ni alloys developing a gradual transformation strain(ε) over a wide temperature(T) range(i.e., low dε/dT) are desirable.

One method to obtain the gradual transformation strain over a wide temperature range is thought to let Ti-Ni alloy actuators have transformation temperatures varying continuously along a working direction. This can be accomplished by temperature gradient annealing method - annealing Ti-Ni alloys cold worked under a temperature gradient- because transformation temperatures of cold worked Ti-Ni alloys were known to depend largely on annealing temperatures.

An equiatomic Ti-Ni alloy was cold rolled by 25 %, and then annealed under a temperature gradient from 823 K to 466 K. Changes in TR (B2-R transformation start temperature) and Ms(R-B19’ transformation start temperature) by the temperature gradient annealing were found to be 17 K and 24 K. Stress-strain curves of specimens annealed under the temperature gradient did not show a stress-plateu suggesting a gradual elongation of specimens due to a gradient in transformation temperatures. Elongation vs. temperature curves showed very small dε/dT(less than 0.01 %/K) comparing to those obtained from specimens annealed at a specific temperature.

15:50 poster C-04

One- and Two-Step Phase Transformation in Ti-rich NiTi

Andersan S. Paula[1], Karimbi K. Mahesh[1], C.M.L. dos Santos[2], J.P.H.G. Canejo[1], F. M. Braz Fernandes[1]

1. *Universidade Nova de Lisboa / Faculdade de Ciencias e Tecnologia (FCT/UNL), Torre, Monte de Caparica 2829-514, Portugal* **2.** *IME, Pça Gen. Tibúrcio, Department of Mechanical and Material Engineering, Rio de Janeiro CEP 22290-27, Brazil*

Contact: kkm@fct.unl.pt

The study of the thermoelastic martensitic transformation in NiTi Shape Memory Alloys (SMA) has been mainly focused on the effect of various thermal and/or mechanical treatments and chemical composition on the transformation temperatures. To determine the transformation temperatures conventional techniques, such as, Differential Scanning Calorimetry (DSC), Electrical Resistivity (ER), Internal Friction (IF) and Thermoelectric power etc., are made use of. During the phase transformation various physical parameters of the material are modified and each technique senses different physical phenomena. Therefore, the phase transformation sequences shall be analyzed on the basis of at least 2 or 3 techniques in order to avoid singularities of a specific technique.

In the present study, four techniques viz., DSC, ER, dilatometry and X-Ray Diffraction (XRD) are employed to study the phase transformation in the strain annealed Ti-rich (Ti51.0at%-Ni) NiTi SMA. The objective of the study is to discuss the results of four techniques, principles involved in detection and mechanisms of the phase transformations. DSC being the most conventional technique to determine the phase transformation works on the principle of change in heat flow. A few researchers attempted to study the phase transformation using dilatometry, which is based on change in dimension. Present results show that B2<->R transformation is more clearly detected in dilatometry method than it is by DSC. This is further supported by XRD data, where the presence of peaks corresponding to the R-phase is clearly assigned to tests performed inside the temperature range of B2->R as given by DSC. ER is very sensitive to B2->R whereas B19'->R signal change is very much reduced. The observations are discussed highlighting on the mechanisms of phase transformation in NiTi SMAs.

15:50 poster C-05

SHAPE MEMORY EFFECT IN THE SOME Ti-Ta-Zr ALLOYS

Tengiz Peradze[1], Iuri Stamateli[1], Jan Cederstrom[2], Teimuraz Berikashvili[1], Aleksandr Razov[3], Kahaber Gorgadze[1]

1. *Georgian Technical University, Tbilisi, Georgia* **2.** *Stokholm, Skandinavian Memory Metals AB, Sweden* **3.** *Sankt-Peterburg University, Russian Federation*

Contact: mszgc@access.sanet.ge

The character of phase transformation due to the heating of alloys, the degree of recoverable strains reactive stresses, matrensite transformations (MT) dumping ability and pseudoelasticity of some Ti-Ta-Zr alloys were inverstigated and measured by differential calorimetry, internal friction measurements and evaluation of recoverable torsion strain.

The alloys speciments were quenched from different temperatures of β-phase (BCC) existence area and then subjected to additial different termo and mechanical actions necessury for the following measurements.

The degree of recoverable strains was measured on a self-assembled fram. The heating was performed by an electrical current through the specimens and by furnace. The initial deformation of the specimens was carred out by torsion.

For all the alloys the shape recovery was not less than 90-98% for initial strains up to 7-9%. The magnitudes of Ms and As for the alloys ranges in 250-400C interval.

So the shape memory effect is in the alloys may be conventionally named as a "high temperature" (respectively to TiNi).

The highest dumping ability was estimated in 44Ti-50Ta-6Zr alloy and that was equal to 23%. The values of the reactive stresses due to the reverse MT in the alloys was measured as 300-350 MPa. The dimension of pseudoelasticiti of the alloys (strain-stress cycled at a room temperature) comes up to 100% after 3-4 cycles for the initial strain ~3,5-4%.

Work was carry out by ISTC Project #G-499.

15:50 poster C-06

Thermal Dependence on Elasticity of TiNi Thin Films Prepared by Sputtering

Yutaka Sakurai, Fumio Takeda

Toyama National College of Technology, 13 Hongo, Toyama 939-8630, Japan

Contact: ysakurai@toyama-nct.ac.jp

This report describes the measurement of elastic coefficient and its thermal dependence for TiNi shape memory alloy films prepared by using a composition controlled sputtering technique. The sputtering technique was employed by which the diameter of ring shaped erosion region in a composite tar-

get is controlled by the magnetic field applied from the outside of the sputtering chamber, as reported by one of the present authors. The elasticity of the NiTi thin films was measured by using the vibrating reed method. The reed (cantilever) of the films was prepared by peeling from glass substrates, and shaped into 30mm length x 7mm width x 19micro-meter thick. The elasticity versus temperature curves were measured as changes in the first mechanical resonant frequency of the cantilever at each temperature. The displacement response to a mechanically excited vibration force of cantilever was detected by using an eddy current displacement meter, and the signal was inputted to PC via GPIB. The thermal elasticity curves obtained by this measurement have an infection point corresponding to the transition point estimated from electrical resistivity data. This measurement becomes difficult since the damping factor of the sample reed increases rapidly, and the displacement response extremely decreases, as the measuring temperature is exceeded above the transition point

15:50 poster C-07

Functional characteristation of SMA based devices for application in actuators

Luca Toia, Alberto Coda, Andrea Mangioni, Giorgio Vergani

SAES Getters S.p.A (SAES), Viale Italia 77, Lainate 20020, Italy

Contact: alberto_coda@saes-group.com

Actuation is one the most prosiming fields for industrial application of SMA based devices. Since most of the present industrial applications are related to medical devices, there is a lack of info and standardisation on the charactheristaion in actuation related applications.

This talk will give hints on the complete characterisation route, from the ingot to the finished wire in use condition, and on the influence of ingot purity on actaution related performances. Namely, focus will be given on the study of the actuation - de-actuation cycle, on the fatgue behaviour, and on a new concept of 100 % process and product control.

Specific focus will also be given on the definition of actuation paremters, and on the influence of these parameters (stroke, feeding current, load) on the fatigue behaviour.

15:50 poster C-08

Effect of Wheel Speed on Transformation Charcteristics in Rapidly Solidified Fe-Mn-Si Based SMA Ribbons

W. Y. Jang[1], Kwang K. Jee[2], J. W. Kang[1], W. S. Jo[1]

1. *Dept. of Metallurgical Engineering, Chosun University, #375 Seosuk-Dong, Dong-Gu, Gwangju 501-759, Korea, South* **2.** *Korea Institute of Science and Technology, Material Science and Engineering Division, Seoul, Korea, South*

Contact: wyjang@chosun.ac.kr

Effects of colling rate and annealing temperature on the microstructure and grain size have been investigated in Fe-Mn-Si based SMA Ribbons, and the correlation between those intrinsic factors and martensitic transformation characteristics has been discussed.

When Fe-Mn-Si based SMA ribbons were fabricated by RSP, grain size of the ribbon was refined up to 4 □ and the uniformity of grains were improved by increasing wheel speed i.e cooling rate. The volume fraction of thermal ε martensite was increased by increasing colling rate.

Refined grains of the melt-spin ribbon were partially grown by annealing above 300□×3min. By increasing annealing temperature, the volume fraction of thermal ε martensite was increased and that of α′martensite were also highly increased in the resultant ribbons. Ms temperature of annealed ribbon was higher than that of as-spun ribbon and transformation temperature hysteresis(Af-Ms) was decreased by increasing annealing temperature.

15:50 poster C-09

Effect of Betatizing Temperature on Transformationn Characteristics and Pseudoelasticity in an Unidirectionally Solidified Cu-Al-Ni Based Alloy

W. Y. Jang[1], K. H. Kim[1], Kwang K. Jee[2]

1. *Dept. of Metallurgical Engineering, Chosun University, #375 Seosuk-Dong, Dong-Gu, Gwangju 501-759, Korea, South* **2.** *Korea Institute of Science and Technology, Material Science and Engineering Division, Seoul, Korea, South*

Contact: wyjang@chosun.ac.kr

Cu-Al-Ni based alloy rod could be fabricated by heated-mold continuous casting, which shows unidirectional solidification structure in the [100] direction and the γ2 phase was precipitated in matrix along cast direction. When the rod was betatized at the elevated temperature above 750□, the γ2 phase is dissolved into the matrix so that Ms temperature was decreased. It could be also confirmed that both β1' and γ1' martensites coexited below - 20 □ by DSC measurement. The betatized rod showed pseudoelastic behavior but the stress-strain curves of compressive-deformed rods were not same

with betatizing temperature; the stress-strain curves of the specimen betatized at 700□ were linear but those of the rod betatized above 750 □ were non-linear.

15:50 poster C-10

Stabilization and Destabilization in Melt-spun Cu-Al-Ni Based SMA Ribbons

G.S. Yang[2], Kwang K. Jee[1], W. Y. Jang[3]

1. *Korea Institute of Science and Technology, Material Science and Engineering Division, Seoul, Korea, South* **2.** *Dept. of Materials Engineering, Chosun University, # 375 Seosuk-Dong, Dong-Gu, Gwangju 501-759, Korea, South* **3.** *Dept. of Metallurgical Engineering, Chosun University, #375 Seosuk-Dong, Dong-Gu, Gwangju 501-759, Korea, South*

In order to clarify the destabilization condition, the effect of heat treatments i.e. flash heating and aging on the microstructure and transformation characteristics of stabilized ribbons on account of chemical composition and wheel speed has been studied in Cu-Al-Ni based SMA melt-spun ribbons.

By flash heating above 750 □ × 5 sec, the parent phase was not retained at room temperature because of the disappearance of lattice defects introduced during melt spinning, while the grain size of the flash-heated ribbon became coarser with increasing flash-heating temperature.

When as-spun ribbon was aged at 600 □, remarkable change in microstructure was not observed with increasing aging time but transformation temperatures of the ribbon aged at 600 □ were lower than those in as-spun ribbon. With increasing aging temperature to 100 □ and 200 □, the volume fraction of retained parent phase was decreased, resulting in destabilization of parent phase. Microstructure of the ribbons aged at 100 □ and 200 □ were changed to columnar structure and Ms and As temperatures were risen with increasing aging temperature and time.

15:50 poster C-11

Ferromagnetic Shape Memory Microscanner System for Automotive Applications

Daniel Brugger[1], Manfred Kohl[1], U. Hollenbach[1], A. Kapp[2], C. Stiller[2]

1. *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **2.** *Universität Karlsruhe (TH), Institut für Mess- und Regelungstechnik, Engler-Bunte-Ring 21, Karlsruhe 76131, Germany*

Contact: daniel.brugger@imt.fzk.de

This paper reports on a microscanner system for scanning unknown environments with respect to angle and distance. The main components of the system are a ferromagnetic shape memory (FSMA) microactuator, a mirror for beam deflection, an integrated angle sensor, a pulsed laser diode and a time-of-flight measurement device for distance acquisition. The microactuator consists of a double-beam cantilever, which is fabricated by RF magnetron sputtering of a Ni-Mn-Ga thin film and subsequent photochemical micromachining. Depending on the temperature of the microactuator, either the ferromagnetic or the shape recovery forces dominate, which allows direct electrical actuation in two opposite directions with low biasing forces. The angle sensor is realized by integration of the microactuator on an optical bench including a beam splitter for partial reflection of the optical beam and a position sensing detector.

In the paper, the performance characteristics of the FSMA microactuator and angular sensor will be discussed first. The actuator can be operated with tunable frequencies, which can be categorized in an off-resonance regime below a thermal cut-off frequency of about 100 Hz and a broad resonance regime between 160 and 220 Hz. Furthermore, two optimum regimes of heating power exist, where a large net-force in both directions and thus a large actuation stroke is generated. The typical scanning angle and angular resolution are about 50 and 0.5, respectively. Further investigations are concerned with time-of-flight measurements and the two-dimensional scanning performance. The maximum scanning range is about 30 m for cooperative (reflecting) objects. The distance resolution is about 20 cm. The point clouds generated by the system are subject to further signal processing for segmentation, classification and tracking of objects. The small system size and associated small moving masses are particularly interesting for mobile applications such as vehicle environment sensing

15:50 poster C-12

The broadband magneto-electric energy conversion by the NiMnGa single crystal/piezoceramic system

Mykola M. Krupa[1], Volodymyr A. Chernenko[1], Victoria Karasiova[1], Stefano Besseghini[2]

1. *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **2.** *Istituto per l'Energetica e le Interfasi Consiglio Nazionale delle Ricerche Unità di Lecco (IENI Lecco), Corso Promessi Sposi, 29, Lecco 23900, Italy*

Contact: krupa@imag.kiev.ua

Measurements of the magnetostrictive characteristics of NiMnGa martensitic single crystal have been carried out under low alternating magnetic field in a range of 0 - 20 MHz.A longitudinal magnetostriction appeared to be 2-3 times higher than that for pure polycrystalline Ni and its amplitude increases at high dc magnetic field.

The transverse magnetostriction is found to be dependent on the orientation of the alternate magnetic field and crystallographic directions. This magnetostriction along two long crystallographic axes is essentially higher than that for pure

Ni in one-variant state of martensitic single crystal. Its magnitude inreases drastically in two-variant state.

The magnetic-to-electric conversion efficiency in the Ni-Mn-Ga/piezoceramic system was found 0.001-0.01 V/Oe in frequency range of 0 - 20 MHz.

15:50 poster C-13

PROPERTIES OF SPUTTER-DEPOSITED Ni-Mn-Ga THIN FILMS

Mykola M. Krupa[1], Volodymyr A. Chernenko[1], Manfred Kohl[2], Igor V. Lezhnenko[1], Makoto Ohtsuka[3], Yurii B. Skyrta[1]

1. *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **2.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **3.** *Tohoku University, Sendai, Japan*

Contact: krupa@imag.kiev.ua

The ferromagnetic thermoelastic martensites being sputter-deposited as thin films represent novel materials which can be incorporated in the ferromagnetic MEMS, microsensors etc.

Properties of such films are governed by many factors, among them sputter conditions, target composition and heat treatment are worth noting.

Here we study an influence of substrate and heat treatment on the film structure, formation of martensitic phase and ferromagnetic ordering of the submicron thin films sputter-deposited on alumina ceramic and glass substrates using targets of Ni49.5Mn28Ga22.5 and Ni52Mn24Ga24 (at.%).

R.f. magnetron deposition has been used to produce as-deposited Ni-Mn-Ga thin films with thickness ranging from 0.1 to 5 micron. The substrate temperature was kept about 320 K. Annealing of thin films was made in vacuum at 873K for 24h.(glass) and at 1273K for 1h. (alumina).

The structural characterization of films was made by X-ray diffraction and optical microscopy. The temperature dependencies of the electrical resistivity of films were measured in the interval of 290-430 K. On warming, as-deposited on glass films show a small reversible monotonous decrease of resistivity with the kink-like change in the vicinity of 340 K whose nature is not clear yet.The annealed films demonstrate a considerable increase of resistivity and anomalies associated with the martensitic transformation and Curie temperature typical for bulk Heusler alloys. The thermomagnetic curves measured by SQUID in the interval 250-400 K confirm a disordered magnetic state for as-deposited films and saturation magnetization values for annealed films typical for bulk alloy. The optical reflection spectra also show a difference for as-deposited and annealed films.

15:50 poster C-14

Ni-Mn-Ga and Ni-Mn-Ga-Ge Thin Films

Janusz Dubowik[1], Iwona Goscianska[2], Yuri Kudryavtsev[3]

1. *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland* **2.** *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **3.** *G.V. Kurdyumov Institute for Metal Physics National Academy of Sciences (IMP), Vernadsky Blvd. 36, Kyiv UA03680, Ukraine*

Contact: dubowik@ifmpan.poznan.pl

Depending on a deposition method and post-deposition annealing, the magnetic properties of Ni-Mn-Ga films can vary substantially. Even in the well annealed films the influence of the martensitic transformation -- the most characteristic feature of Ni--Mn--Ga -- on their magnetic properties is either very small or is substantial.

The aim of this contribution is to elucidate the origin of these contradictions having a large number of experimental results on the sputtered and flash--deposited Ni--Mn--Ga films of various compositions ($Ni_xMn_yGa_z$) and $Ni_{50}Mn_{25}Ga_{25-x}Ge_x$ films heat-treated at various temperatures (from 620 - 973 K). The magnetic properties were investigated with ferromagnetic resonance in a wide temperature range and are compared with electric resistivity data. We show that the anomalous magnetic behavior of the Ni-Mn-Ga films annealed at T<800 K results from their columnar microstructure (which determines the effective shape anisotropy of these nominally heterogeneous films) and changes in the magnetic anisotropy due to the martensitic transformation. The films annealed at the highest temperatures reveal the magnetic behavior typical of normal ferromagnetic films, in which the effect of the martensitic transformation is strongly reduced due to constraint of a substrate. Ge substitution of Ga in Ni-Mn-Ga-Ge films has a detrimental effect on the martensitic transformation.

15:50 poster C-15

Texture and Stress in Ni-Mn-Ga Thin Films Deposited on Alumina

Stephen Doyle[1], Manfred Kohl[2], Volodymyr A. Chernenko[3,4,6], Makoto Ohtsuka[5]

1. *Forschungszentrum Karlsruhe GmbH, Institute für Synchrotronstrahlung, ANKA, Hermann-von-Helmholtz-Platz 1, Karlsruhe 76344, Germany* **2.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **3.** *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **4.** *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **5.** *Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Sendai 980-8577, Japan* **6.** *Institute of Magnetism, NAS of Ukraine, 36-B Vernadsky str., Kiev 03142, Ukraine*

Contact: doyle@iss.fzk.de

Thin films of the Ni-Mn-Ga ferromagnetic martensites due to a strong coupling of the structural and magnetic degrees of freedom are promising materials to be incorporated in novel sensors, actuators and magnetic MEMS.

In general, because the properties of polycrystalline thin films depend strongly on their microstructure, an important research area is concerned with the control of this microstructure, in particular the texture and internal stresses which develop during deposition and post-deposition annealing processes.

In this work, we the X-ray diffraction facility ANKA-Diff at the ANKA synchrotron source in Karlsruhe to study texture and internal stresses in Ni-Mn-Ga thin films sputter deposited on alumina ceramic. Two series of films of two different compositions consisting of 6 specimens each were fabricated by the magnetron deposition at ambient temperature. Each series of films was designed as the single layers of 0.1; 0.2; 0.4; 0.6; 1.0 and 5.0 micron thickness attached to alumina. Films on substrates were vacuum annealed at 1073 K for 36ks. Diffraction measurements were made at 423K. An in-house made temperature controlled heating unit was designed for this purpose. The selected value of temperature ensured that the films were in the austenitic cubic state. The texture measurements were performed using 2Theta scans at fixed Phi and Chi angles which were varied by the 10 deg. step in a range of 0 to 90 deg.

A drastic change of mutual intensities of 220 and 400 peaks as a function of Chi was found while Phi dependence was much less pronounced. An analysis of the results leads to the conclusion about different degree of {110}-type in-plane texture for the films of different thickness. The thickness dependence of the lattice parameter evidences a different degree of internal elastic stresses developed by the films during cooling after annealing.

15:50 poster C-16

DEPENDENCE OF THE MAGNETIC ANISOTROPY ON THE THICKNESS AND ANNEALING OF SPUTTER-DEPOSITED Ni-Mn-Ga THIN FILMS

Mykola M. Krupa[1], Volodymyr A. Chernenko[1], Igor V. Lezhnenko[1], Makoto Ohtsuka[2], Galina V. Bondarkova[1], Yurii B. Skyrta[1], Manfred Kohl[3], Victor Lvov[4]

1. *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **2.** *Tohoku University, Sendai, Japan* **3.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **4.** *Taras Shevchenko University, Kiev 03022, Ukraine*

Contact: krupa@imag.kiev.ua

Magnetic characteristics of the martensitic Ni52Mn24Ga24 and Ni49.5Mn28Ga22.5 thin films prepared by the sputter-deposition on polycrystalline Al2O3 and glass substrates have been studied by ferromagnetic resonance and vibrating magnetometry.

As-deposited films turned out to be in paramagnetic state. Annealing at T=873 K or T=1273 K caused the films to be ferromagnetic with a weak in-plane anisotropy and strong, , perpendicular anisotropy, especially after annealing at T=1273 K. The magnetization of the films after annealing at 1273K is close to magnetization of pure nickel and decreases in two- three times for the films annealed at 873 K. The ferromagnetic resonance spectrum has a double structure and the width of the peaks becomes narrower with the increase of the temperature of annealing.

A perpendicular anisotropy takes place already in the annealed films of 0.1 microns thick. The width of the ferromagnetic resonance peak decreases with increasing film thickness while its position shifts toward the higher magnetic fields. It is found that the peak width for the annealed films on the glass substrate is narrower than that for the films on Al2O3 substrate. Moreover, the peak position for films on the Al2O3 is located at the lower magnetic fields than that for films on a glass. The aforementioned characteristics have been found to be dependent on the film composition as well.

15:50 poster C-17

MAGNETORESISTANCE OF Ni-Mn-Ga THIN FILMS IN THE VICINITY OF MAGNETO-STRUCTURAL TRANSFORMATION

Mykola M. Krupa[1], Volodymyr A. Chernenko[1], Igor V. Lezhnenko[1], Makoto Ohtsuka[2], Yurii B. Skyrta[1], Manfred Kohl[3]

1. *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **2.** *Tohoku University, Sendai, Japan* **3.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany*

Contact: krupa@imag.kiev.ua

The enhanced sensitivity of the martensitic transformation (MT) temperature on the magnetic field has been already reported for the Ni-Mn-Ga alloys transforming near Curie temperature. The manyfold increase of the value of resistivity anomaly at MT as a function increasing MT temperature in Ni-Mn-Ga alloys is also a common knowledge.

Aforementioned behavior gave us motivation to study a magnetic field influence on the resistivity anomaly near magneto-structural transformation exhibited by the Ni-Mn-Ga thin films.

Magnetron deposition and Ni52Mn24Ga24 and Ni49.5Mn28Ga22.5 targets have been used to produce as-deposited Ni-Mn-Ga thin films with the thickness ranging from 0.1 to 5 microns. The substrate temperature was kept about 320 K. Annealing of thin films was made in vacuum at 1273K for 1h. X-ray diffraction and optical microscopy were used for the structural characterization of the films. The temperature dependences of resistivity in the range of 293-373 K under in-plane and out-of-plane magnetic field up to 1.2 T were studied. A reversible hysteretic MT in the temperature interval of 350-370 K was observed. In the temperature region outside of this interval the magnetoresistance is small while in the temperature interval in which MT occurs the maximum of magnetoresistance is observed whose value reaches 6 % in magnetic field of 1.2T. An analysis of the electrical, structural and magnetic properties of investigated films offers the mechanism explaining anomalous behaviour of the magnetoresistance near MT in Ni-Mn-Ga thin films.

15:50 poster C-19

Effect of quenching on martensitic transformation course in non-stoichiometric NiMnGa alloy

Tomasz Goryczka, Marek Gigla, Henryk Morawiec

University of Silesia, Institute of Material Science, Bankowa 12, Katowice 40-007, Poland

Contact: goryczka@us.edu.pl

Since the time when a Ni-Mn-Ga alloy with composition near to the stoichiometric Heusler structure has been recognized as material revealing ferromagnetic shape memory effect, the number of experimental results and its practical application still increases. Especially, non-stoichiometric composition attracts its attention due to a course of martensitic transformation. Several types of martensite can be found such as non-modulated tetragonal martensite (T), five-layerd martensite (10M) as well as seven-layerd martensite (14M). Above the martensitic transformation, the parent phase undergoes ordering transformation from B2' to the Heusler's structure $L2_1$.

The purpose of presented work was to study the correlation between structure of the martensite type and ordering of the parent phase in the alloy with chemical composition: 51.2at%Ni, 27.6at%Mn and 21,1at% Ga. From the polycrystalline bulk material samples were cut and quenched after annealing at 700°C, 800°C and 900°C for one hour. Increasing of quenching temperature results in lowering the characteristic temperatures of the martensitic transformation and simultaneously broadens its thermal hysteresis. The parent phase, in sample quenched from 900°C, reveals the B2' structure whereas in sample quenched from 700°C - the Heusler's structure $L2_1$. The ordering transformation was confirmed by the results obtained from high temperature DSC measurement. On a heating curve broad thermal peak appeared in the range between 750 and 850°C.

Independently on the quenching temperature and the ordering of the parent phase the martensite shows the 10M structure type. However, in electron diffraction pattern additional effects of the diffusion electron scattering have been observed as a results of a short-range ordering caused by the Ni exceeding atoms.

15:50 poster C-20

Neutron study of the martensitic transformation in Ni-Fe-Ga alloys

Jon Gutierrez[1], Patricia Lázpita[1], Vasudeva Siruguri[1], Jose Manuel Barandiarán[1], Paul Henry[2], Volodymyr A. Chernenko[3], T. Kanomata[4]

1. *Facultad de Ciencia y Tecnología (UPV/EHU), P.Box 644, Bilbao 48080, Spain* **2.** *Institut Laue Langevin (ILL), 6 Rue Jules Horowitz, Grenoble Cedex 9, France* **3.** *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **4.** *Tohoku Gakuin University, Tagajo, Japan*

Contact: jon@we.lc.ehu.es

We present results of an extensive neutron study of the martensitic transformation in Shape Memory Alloys of nominal composition $Ni_{55-x}Fe_{19+x}Ga_{26}$ (x = 0, 1, 2 at.%) transforming close to room temperature. This study was performed at instrument D20 of the ILL, working at high resolution mode (λ =1.88 Å). Neutron experiments were performed by cooling and afterwards by heating the samples, so the temper-

ature of the structural transitions was also determined.

For each composition, a piece cut from the ingot was crushed to eliminate their strong texture, and the resulting powder was carefully annealed to recover from the defects induced by crushing. Magnetic characterisation before and after processing revealed no changes in the martensitic tra nsition and Curie temperatures.

For instance, the x=0 sample shows a martensitic transformation temperature T_M = 250 K and Curie temperature T_C = 289 K . Neutron measurements for this alloy were performed between 300 K (austenite phase) and 150 K (martensitic phase). At temperatures $T>T_C$ we have determined the coexistence of two phases: a $L2_1$ one with space group Fm3m and cell parameter a=5.7300 Å, and a γ fcc with cubic simmetry , space group Pm3m, with cell parameter a=3.5859 Å. This second pha se was also detected by magnetic measurements, giving rise to a second Curie temperature of about 363 K.

At temperatures $T< T_M$ the γ fcc phase remains unchanged, while the $L2_1$ phase transforms into a majority monoclinic seven-layer modulated s tr uct ure with parameters a= 4.0361 Å, b=2.8792 Å, c=28.135 Å and β =90.83°. Some contributions arising from a five-layer modulated structure have been also found.

Analysis of the structural results for all compositions, plus concordance between all st ruct ural and magnetic transitions elucidated from different experimental techniques are discussed.

15:50 poster C-21

Pre-martensitic phenomena in a near stoichiometric Ni2MnGa polycrystalline alloy

Jose Ignacio Perez-Landazabal[1], Vicente Sanchez-Alarcos, Vicente Recarte, Cristina Gomez-Polo, Volodymyr A. Chernenko[2,3,4]

1. *Universidad Publica de Navarra (UPNa), Campus de Arrosadia, Pamplona 31006, Spain* **2.** *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **3.** *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **4.** *Institute of Magnetism, NAS of Ukraine, 36-B Vernadsky str., Kiev 03142, Ukraine*

Contact: ipzlanda@unavarra.es

The Internal Friction (IF), the elastic modulus and the magnetic susceptibility of a near stoichiometric Ni2MnGa polycrystalline sample have been measured as a function of temperature from parent phase down to well below martensitic transformation temperature (MS) by Resonant ultrasound spectroscopy (RUS) and superconducting quantum interference device (SQUID) measurements. Two IF peaks and two minima in the elastic modulus have been observed, corresponding to the weakly 1^{ST} order transition from the cubic $L2_1$ parent phase to the intermediate phase (at T_1 = 220K) and to the Martensitic Transformation (at T_2 = 120K). The real part of the magnetic susceptibility shows a decrease and the imaginary part a peak at T_1.

15:50 poster C-22

Investigation of the Field-Induced Strain of Shape Memory Alloy in a Pulsed Magnetic Field

Takuo Sakon[1], Takashi Fukuda[2], Mitsuhiro Motokawa[3,4], Tomoyuki Kakeshita[2]

1. *Dept. Mechanical Enginerring, Fac. Engineering, Akita University (AKITAUNIV), Tegata Gakuenmachi 1-1, Akita City 010-8502, Japan* **2.** *Graduated School of Engineering, Osaka University (OSAKAUNIV), Osaka, Japan* **3.** *Institute for Materials Research, Tohoku University (IMR), Sendai 980-8577, Japan* **4.** *International institute for Advanced Studies (IIAS), Kizu, Kyoto, Japan*

Contact: sakon@ipc.akita-u.ac.jp

A system for the simultaneous measurement of magnetization and magnetic strain, which is designed to be used in a pulsed magnetic field, has been developed. In this system, a capacitor on a sample is used and its capacitance changes with the displacement of a sample due to the strain on the sample in a magnetic field. The most significant feature of this system is that magnetization and strain can be measured simultaneously. It is useful to compare the magnetization and magnetic strain (magnetostriction) with each other. Using this system, we have studied the precise magnetization and magnetic field-induced strain (MFIS) of the martensite metallic compound Fe-31.2%Pd (at.%) at temperatures down to 80 K in martensite phase, which is much lower than the martensitic transformation temperature T_M = 230 K. Large MFIS has been measured under a pulsed magnetic field with the time constant 6 ms, which corresponds to 80 Hz in frequency. It means that the MFIS occurs even in short-pulse magnetic fields.

15:50 poster C-23

Properties of martensitic Ni-Mn-Ga thin films deposited on Mo foils

Concepcio Seguí[1], Volodymyr A. Chernenko[2], Manfred Kohl[3], Makoto Ohtsuka[4], J Pons[1], Eduard Cesari[1]

1. *Departament de Fisica, Universitat de les Illes Balears (UIB), Ctra. de Valldemossa, km. 7.5, Palma de Mallorca E07122, Spain* **2.** *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **3.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **4.** *Institute for Materials Research, Tohoku University (IMR), Sendai 980-8577, Japan*

Contact: concepcio.segui@uib.es

Recently the Ni-Mn-Ga ferromagnetic martensites with unique magnetomechanical properties are subject of intense

development in polycrystalline thin film form, because they can be used as innovative component materials in nanosystems. Understanding of the fundamental relationship between their size, shape, structure and material properties and function is a crucial point to be clarified. This paper deals with the investigation of the structural (X-rays and electron diffraction), elastic (dynamical mechanical analysis, DMA), magnetic (SQUID) and magnetomechanical (beam bending tests) characteristics exhibited by submicron Ni-Mn-Ga thin films deposited and annealed on Mo foils of 5 and 10 micron thickness. Film series with two different compositions and thickness 0.1; 0.4 and 1 micron were prepared by r.f. magnetron sputtering. The crystal structure of martensitic phase in each film series is 10M or 14M, respectively. The magnetic characteristics are the typical for bulk Ni-Mn-Ga alloys. The temperature dependences of DMA signal performed in tensile mode have shown that composites of Ni-Mn-Ga thin film/Mo foil exhibit a hysteretic martensitic transformation accompanied by well pronounced step-like anomalies. The low-frequency elastic modulus is larger in austenite state than in martensitic one. Magnetoelastic measurements revealed a magnetic field-induced bending of bimetallic-type specimens with the estimated value of magnetostrain in Ni-Mn-Ga layer to be about 0.1 %.

15:50 poster C-24

Stress-temperature relationship in a Ni-Mn-Ga single crystal

Eduard Cesari[1], Volodymyr A. Chernenko[2], J Pons[1], K Ishikawa[3]

1. *Departament de Fisica, Universitat de les Illes Balears (UIB), Ctra. de Valldemossa, km. 7.5, Palma de Mallorca E07122, Spain* **2.** *Institute of Magnetism, N.A.S. of Ukraine, 36-b Vernadsky Blvd, Kyiv 252142, Ukraine* **3.** *Tohoku Gakuin University, Tagajo, Japan*

Contact: eduard.cesari@uib.es

Off-stoichiometric Ni-Mn-Ga Heusler alloys exhibit a large variety of martensitic structures as well as several accompanying phenomena, as precursor or pre-martensitic effects and inter-martensitic transformations (IMT). Both the ferromagnetic and the paramagnetic martensites tend to exhibit stress- and temperature- induced IMT. Three main martensitic phases are formed, namely 10M, 14M and 2M in the monoclinic coordinate system or, in cubic coordinates, designed as five-layer modulated tetragonal (c/a <1), seven-layer orthorhombic and non-modulated tetragonal (c/a>1), respectively.

The sequence of thermally and stress-induced intermartensitic transformations has been studied in a $Ni_{52.0}$-$Mn_{24.4}$-$Ga_{23.6}$ single crystal. Transmission electron microscopy through in situ cooling experiments has confirmed the thermally induced martensitic phases and their sequence. The stress-strain behavior under compression along the $<110>_P$ and $<100>_P$ crystallographic directions, at different temperatures, has also been studied for this compound and a stress-temperature phase diagram has been established.

15:50 poster C-25

TRANSFORMATION OF ULTRASOUND ON THE BORDER OF STRONGLY ANISOTROPIC FERROMAGNETIC CRYSTALS

Yuri A. Kuzavko[1], Dmitriy A. Kostiuk[1], Michael M. Karpuk[2], Vladimir G. Shavrov[3]

1. *Brest State Technical University, Brest 224017, Belarus* **2.** *Technical University of Koszalin, Raclawicka 15-17, Koszalin, Poland* **3.** *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation*

Contact: kuzavko@newmail.ru

Materials in which the reversible form and size control by magnetic field is possible, attracts much attention now. Highest effect is registered for $Ni_{2+x+y}Mn_{1-x}Ga_y$ alloys. Crystal size there can be changed up to 6% by magnetic field (the theoretical deformation limit at transformation from austenite to martensite). With different stechiometric composition the temperature of martensite transition T may be close to the room one and lower than the Curie point of material. Sometimes martensite phase transition (PT) is preceded by pre-martensite one (PM PT) with anomalies of speed and absorption of longitudinal and transverse acoustic waves (LA and TA). The transverse mode TA_2 is softened near the PM PT with the k = (ξ,ξ,0) wave vector at ξ =0.33 and e = (1 1 0) polarization vector. Its speed changes from 740 m/s (T=300 K) to 614 m/s (TPM=255 K) and 903 m/s (TM =215 K) at f=3.7 MHz frequency.

Acoustic anisotropy of a crystal reaches the value of A=28 at PM PT. Structural PT usually takes place in a Ni_2MnGa crystal, and after that the orientational PT can be realized at temperature decrease and in H||[001] field.

We examined LA and TA inclined fall in (100) plane, reflection from the crystall free border (110) and from its border with liquid or dielectric. Spread directions and amplitudes of reflected waves in (001) plane of a crystal were determined, and are not pure modes but quasi-longitudinal and quasi-transverse. The colossal acoustic anisotropy of a crystal in its PT area brings allows to control the angles of reflection and transformation of wave types by temperature and magnetic field. Starting from a critical fall angle of TA, the LA, appeared at reflection, turns into the accompanying surface oscillation, and close to PT point it reflects into the volume.

Authors are grateful to BRFFI and RFFI (F04R-080, 04-02-81058, 03-02-17443 grants) and Ministry of Education of Belarus (05-550 grant) for their financial support of carried

out research.

Tuesday, 6 September

Shape Memory Materials 2

Tuesday morning, 6 September, 9:00
Main Building, room 315
Bogdan Raniecki, Qingping Sun presides

9:00 oral

Ti-Ni, Ti-Ni-Pd and Ti-Ni-Cu SMA Sputter-deposited Thin Films and Their Microactuators

Shuichi Miyazaki

Institute of Materials Science, University of Tsukuba (UT), Tennoudai 1-1-1, Tsukuba 305-8573, Japan

Contact: miyazaki@ims.tsukuba.ac.jp

Ti-Ni based shape memory alloy (SMA) thin films including Ti-Ni, Ti-Ni-Pd, Ti-Ni-Cu and Ti-Ni-Zr have been developed utilizing the sputter-deposition method by the present author□s research group. The Ti-Ni is a standard material and other alloys have their own characteristics when compared with the Ti-Ni. The Ti-Ni-Pd and Ti-Ni-Zr are characterized by high transformation temperatures so that they can be expected to show quick response due to their higher cooling rate. The transformation temperature hysteresis of the Ti-Ni-Pd is narrower than that of the Ti-Ni, while the hysteresis of the Ti-Ni-Zr is wider than that of the Ti-Ni. Thus, the former alloy is more attractive as a microactuator material than the latter alloy. The transformation temperature hysteresis of the Ti-Ni-Cu is less than that of the Ti-Ni without decreasing the transformation temperatures, implying that the Ti-Ni-Cu is also an attractive actuator material for quick response. The transformation temperature hysteresis and the transformation strain of the R-phase of the Ti-Ni are less than one tenth of those of the martensitic transformation, thus the R-phase transformation can be attractive for high response microactuators. In the present presentation, the shape memory characteristics associated with the martensitic transformation of the Ti-Ni, Ti-Ni-Pd and Ti-Ni-Cu and those associated with the R-phase transformation of the Ti-Ni will be shown and the actuator characteristics of microactuators utilizing these thin films will be presented. These alloys can show the stable shape memory effect and superelasticity which are equivalent to those of bulk materials produced by conventional melting methods. Because of the unique microstructures consisting of non-equilibrium thin plate precipitates in the sputter-deposited thin films, they can sometimes show the stable properties even better than the bulk materials. Three types of high-speed diaphragm-type microactuators utilizing the TiNi-based

[ABSTRACT TRUNCATED TO 2000 LETTERS]

9:15 oral

Energy Storage and Absorption of TiNi Shape Memory Alloy under Various Thermomechanical Loading Conditions

Hisaaki Tobushi[1], Elzbieta A. Pieczyska[3,4], Stefan P. Gadaj2, W. Nowacki[2]

1. *Aichi Institute of Technology (AIT), 1247 Yachigusa, Takusa-cho, Toyota 470-0392, Japan* **2.** *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT PAN), Świętokrzyska 21, Warszawa 00-049, Poland* **3.** *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT), Swietokrzyska, Warszawa 00-049, Poland* **4.** *Department of Mechanical Engineering, AICHI Institute of Technology (AIT), 1247, Yachigusa-cho, Toyota 470-0392, Japan*

Contact: tobushi@

The enery storage and absorption characteristics in TiNi shape memory alloys were investigated experimentally based on the superelastic properties under various thermomechanical loading conditions. In the test, the influence of strain rate, cyclic loading and temperature-controlled condition on the characteristics of energy storage and absorption of the material was investigated. In the experiment under the temperature-uncontrolled condition, temperature on the surface of the material was observed and the influence of variation in temperature on the characteristics was clarified. The results obtained can be summarized as follows. (1) In the case of low strai rate, the stress plateaus appear on the stress-strain curves due to the martensitic tranformastion and the reverse transformation during loading and unloading. In the case of high strain rate, the slopes of the stress-strain curves in the phase-transformation regions during loading and unloading are steep. The energy storage per unit volume increases in proportion to temperature, but the dissipted work per unit volume depends slightly on temperature. In the case of low strai rate, the recoverable starin energy and dissipated work do not depend on both strain rate and the temperature-controlled condition. (2) In the case of high strain rate, the recoverable strain energy density decreases and dissipated work density increases in proportion to strain rate under the temperature-controlled condition, while the recoverable strain energy desity increases and dissiptaed work desity decreases under the temperature-uncontrolled condition. In the case of the temperature-uncontrolled condition, temperature varies significantly due to the martensitic transformation and therefore the characteristics of energy storage and absorption differ from these under the temperature-controlled condition.

9:30 oral

Composition gradients surrounding Ni_4Ti_3 precipitates in a Ni-rich NiTi alloy studied by EELS, EFTEM and EDX

Zhiqing Yang, Dominique Schryvers

Department of Physics, University of Antwerp, Antwerp, Belgium

Contact: zhiqing.yang@ua.ac.be

NiTi alloys with near-equiatomic composition can exhibit shape memory and superelastic properties resulting from a temperature or stress induced austenite-martensite phase transformation. The behaviour and characteristics of this transformation are strongly influenced by the presence of Ni4Ti3 precipitates in the B2 austenite matrix and which can be obtained by appropriate annealing procedures. The formation of Ni4Ti3 precipitates not only introduces a strain field in the surrounding matrix, it also affects the composition of the retained matrix since the precipitates are enriched in Ni with respect to the original material with a nominal composition of Ni51Ti49. Both the strain field and changes in compsition can effect the phase transformation behaviour. The strain field was recently studied by high-resoluton electron microscopy. In the present investigation, nanoprobe electron energy loss spectroscopy (EELS), Energy Filtered TEM (EFTEM) and energy dispersive X-ray (EDX) analysis are used to determine the presence of a possible variation in Ni concentration in close proximity of a Ni4Ti3 precipitate. A Ni depleted zone in the surrounding matrix up to 150 nm away from the matrix-precipitate interface was detected. The size and concentration change of the depletion zone perfectly compensate for the excess Ni in the precipitate. Moreover, since different precipitates are often found in nanoscale proximity, depletion zones from different precipitates can enforce one another in certain regions. These effects, next to the lattice strains
studied earlier, are very important with respect to the potential martensitic transformation and shape memory behaviour of the matrix.

9:45 oral

Development of a phalangeal prosthesis in NiTi

Stefano Besseghini[1], Villa Elena[1], Perina Matteo[2], Passaretti francesca[1]

1. *Istituto per l'Energetica e le Interfasi Consiglio Nazionale delle Ricerche Unità di Lecco (IENI Lecco), Corso Promessi Sposi, 29, Lecco 23900, Italy* **2.** *TECRES, Sommacampagna, Italy*

Contact: s.besseghini@ieni.cnr.it

This paper reports on the development of a metacarpal phalangeal prosthesis with an embedded NiTi pseudoelastic spring. The device was originally developed using a special steel spring. The use of a shape memory alloy was suggested by the observation of failures in the hinge structure of the prosthesis. In more detail it was frequently observed a permanent deformation of the stainles steel spring. In a large number of cases this was not due to the activation of the spring in the normal deformation range but by occasional overdeformation of the prosthesis.
A critical event in this respect is the installation surgical act itself.
Pseudoelastic NiTi withstands larger deformation without a permanent set.
Here we will not focus on the development of the prosthesis as a whole but on the characterization of the NiTi alloy used. One of the main difficulties encountered is related to the fatigue life of the springs which should ensure at least a 10^7 cycles life. Even if a dependence from the test temperature was expected to some extend the results confirmed that this parameter is of foremost importance.
Results obtained by differential calorimetric measurement before the cycling procedure and on passed (i.e. cycled more than 10^7 cycles) or broken components will be presented and discussed.

10:00 oral

SPIN TRANSISTOR IN STRUCTURES BASED ON SMART MATERIALS

Yuri A. Kuzavko

Brest State Technical University, Brest 224017, Belarus

Contact: kuzavko@newmail.ru

It is perspective to technologicaly use the spin of electron - a pure quantum quality with no macrocosm analogues. Spintrone technologies will allow quicker electronic devices. The main thing to be mastered is the control of spin currents and electrons flows. Not electric but magnetic fields are to be used for that, as electron can be represented as a micro magnet.
The idea of spintrone analogue of field transistor, firstly spoken by Datta and Das, based on controlling the spine polarization of charge carriers by spin-orbital interconnection with outer electric field, is still not realized. The same with the idea of of polarization transistor, formulated by Kuzavko and Shavrov, based on the bistable nature of the polarization change of the optical quantas, passed through the magnetic film in the area of its spin-reorientational transistor. Combining the easiness of charge control in semiconductors with use of electrone spin degree of freedom for the information storing and transmitting is very actual. It is reasonable to consider alternative theoretical models of spintrone and polarization devices, especially ones with use of high-temperature superconductors and ferromagnetic Heysler alloys with magneto-

controlled shape memory. Here the conditions of the spin current flow and in the structure of semiconductor-dielectric-Heysler ferromagnetic alloy are considered with taking into account the influence of temperature and external magnetic field. Transmitting the stresses of 10 HPa into the conducting film of the device from its substrate, made on base of spoken ferromagnetic, causes curving and new zones appearing for the charge carriers in semiconductor, with their spin polarization caused by spin-atomic interconnection in boundary regions of a spin transistor. The use of nanometric magnet-power microscopy for spin currents and stages registration is substantiated.

Author is grateful for BRFFI and RFFI (F04R-080, 04-02-81058, 03-02-17443 grants) for their support.

Ferromagnetic Shape Memory Alloys 1

Tuesday afternoon, 6 September, 14:00
Main Building, room 315
Vasiliy D. Buchelnikov, Volodymyr A. Chernenko presides

14:00 invited oral

Energy Evaluation for Twinning Plane Movement under Magnetic Field in Ferromagnetic Shape Memory Alloys

Tomoyuki Kakeshita, Takashi Fukuda

Graduated School of Engineering, Osaka University (OSAKAUNIV), Osaka, Japan

Contact: kakeshita@mat.eng.osaka-u.ac.jp

We have made an energy evaluation for the rearrangement of martensite variants (RMV) under a magnetic field in four types of ferromagnetic shape memory alloys: an Fe-31.2at.%Pd with so-called fct martensite, a Ni_2MnGa with the 10M martensite, a $Ni_{2.02}Mn_{1.09}Ga_{0.89}$ with the 14M one and a $Ni_{2.14}Mn_{0.92}Ga_{0.94}$ with 2M one. From magnetic field-induced strain measurements, magnetization measurements and in-situ optical microscope observations, we found that the RMV by a magnetic field occurs at any temperature for the Fe-31.2Pd and the 10M martensite, it occurs in a limited temperature range for the 14M martensite and does not occur at any temperature for the 2M martensite. The reason of such different behavior is quantitatively explained by evaluating two shear stresses: the magnetic shear stress acting across a twinning plane τ_{mag}, and the stress required for the twinning plane movement τ_{req}. The value of τ_{mag} is evaluated from the magnetocrystalline anisotropy constant and the twinning shear, where the former is obtained from magnetization measurements along the easy and hard axes and the latter from the lattice parameters. On the other hand, the value of τ_{req} is evaluated from tensile tests or compressive tests. By comparing the value of τ_{mag} and τ_{req} thus evaluated, we have confirmed that the following condition is universal regardless of temperature, field orientation and structure of the martensite phase: the value of τ_{mag} is larger than τ_{req} when the RMV occurs by magnetic field and vice versa.

14:45 oral

Phase Transitions in Novel Ferromagnetic Shape Memory Alloys Ni-Mn-Sn

Vladimir V. Khovailo[1], V V. Koledov[1], Vladimir G. Shavrov[1], Makoto Ohtsuka[2], Toshiyuki Takagi[2]

1. *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation* **2.** *Tohoku University, Sendai, Japan*

Contact: v-khovaylo@cplire.ru

Recently, it was found that for ferromagnetic Heusler alloys Ni_2MnSn, Ni_2MnIn, and Ni_2MnSb, the deviation from stoichiometry results in appearance of martensitic transformation [1]. For these systems, the martensitic transformation temperature decreases upon substitution of Mn for Sn, In or Sb; especially drastic decrease was observed in the NiMnIn system. The compositional dependence of the Curie temperature T_c of the austenite phase was found to be weak, while T_c of the martensite phase is strongly depended on the alloy composition and drastically decreases with decreasing In, Sn or Sb content in all the systems.

In this work we report on new experimental results obtained on two series of novel ferromagnetic shape memory alloy, $Ni_{50+x}Mn_{37-x}Sn_{13}$ and $Ni_{50+y}Mn_{39-y}Sn_{11}$. They were characterized by differential scanning calorimetry and low-field magnetization measurements. Our DSC measurements revealed that the martensitic transformation temperature T_m in the alloys studied shows a non-monotonous dependence on the Ni excess. Whereas an increase of T_m is observed in both the system as Ni excess increases from 1 at.% to 2 at.%, further substitution of Mn for Ni does not affect martensitic transformation temperature T_m. The low-field magnetic susceptibility measurements were used for determination of Curie temperature T_C of these alloys. The results obtained point to a complex magnetic properties of these materials. For instance, some of the alloys exhibit several anomalies on temperature dependence of susceptibility which gives ground to conclude that, contrary to NiMnGa alloys, in the studied Ni-Mn-Sn compositions Curie temperature of martensite is lower than Curie temperature of austenite.

This work was partially supported by Russian Foundation for Basic Research Grants No. 03-02-17443, 04-02-81058, and 03-02-39006.

[1] Y. Sutou et al., Appl. Phys. Lett. 85 (2004) 4358.

15:00 oral

Time-dependent phenomena and thermal phonons in the Ni–Mn–Ga magnetic shape-memory alloys

Victor Lvov, Nadia Glavatska

National Taras Shevchenko Kiev University, Department of Radiophysic (KNU), 64, Volodymyrska str., Kyiv, Ukraine

Contact: victorlvov@univ.kiev.ua

The isothermal time-dependent deformation of the ferromagnetic Ni–Mn–Ga martensite in the steady magnetic field or under the stationary mechanical loading is studied in both experimental and theoretical way. A concomitant time-evolution of twin structure is analyzed in order to clarify the physical mechanisms of time-dependent phenomena in the Ni–Mn–Ga martensites exhibiting a magnetic shape-memory. It was shown very recently (V. L'vov et al., Phys. Rev., B, 71, 024421 (2005)), that the time dependent magnetically-induced deformation is originated by the fluctuating mechanical stress. As the next step, the role of fluctuating strains in the wide scope of time-dependent effects is disclosed in the present report. The thermal phonons, which induce the fluctuating strain are considered. A simple relationship between the magnitude of fluctuating strain, temperature-dependent elastic modulus and twin width is derived. The temperature dependence of the fluctuating strain in vicinity of martensitic transformation is derived. A strong interdependence between the twin width and the effectiveness of the affect of thermal phonons on the twin structure is predicted theoretically and supported by the experimental data. The problem of reversibility of magnetically/stress-induced deformation is considered on the base of obtained experimental and theoretical results.

15:15 oral

Acoustical properties and acoustopseudoplastic effects in ferromagnetic shape memory alloy NiMnFeGa

Vladimir G. Shavrov[1], V V. Koledov[1], Yuri A. Kuzavko[2], Olga Y. Serdobolskaya[3], Evreniya I. Sidorova[3], Vladimir V. Khovailo[1], Vasiliy D. Buchelnikov[4], S V. Taskaev[4], Dmitriy A. Kostiuk[2]

1. *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation* **2.** *Brest State Technical University, Brest 224017, Belarus* **3.** *M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation* **4.** *Chelyabinsk State University (ChelSU), Br. Kashirinykh Str, 129, Chelyabinsk 454021, Russian Federation*

Contact: shavrov@mail.cplire.ru

Acoustical properties of Ni-Mn-Ga ferromagnetic shape memory alloys (FSMA) have been studied intensively in the recent years in connection with the martensitic and magnetic transitions phenomena which take place in these substances. Recently the effects of intense ultrasound upon the martensitic transitions in NiMnFeGa polycrystals [1] on the giant magnetic-field-induced strains due to martensitic domains boundaries movement in NiMnGa single crystals [2] have been observed. These effects may be of interest in view of possible applications of single and polycrystalline FSMA in sensor and actuator technology.

The aim of the present work is to study experimentally and theoretically the ultrasound velocity anomalies and acoustical emission accompanying the martensitic transition in polycrystalline $Ni_{2.14}Mn_{0.81}Fe_{0.05}Ga$ FSMA. Another purpose of the work is to investigate the effects of intense pulsed ultrasound on the martensitic transition and pseudoplastic deformation of the FSMA samples. The low field magnetic susceptibility and sample strain versus temperature curves were plotted depending on the regime of ultrasound pulses. The effect of ultrasound is found to narrow the hysteretic loop of martensitic transition. The partial martensitic transition accompanied by pseudoplastic sample strain due to ultrasound at constant temperature is observed. The optical in situ observations of martensitic domains structure evolution due to ultrasound action were performed.

This work is supported by RFBR (Grants 03-02-17443, 04-02-81058, 03-02-39006).

[1] V.Buchelnikov, I.Dikshtein, R.M.Grechishkin, et.al, JMMM, 272-276 (2004) 2025.

[2] B.W.Peterson, J.Feuchtwanger, J.M.Chambers, et.al. J. Appl. Phys. 95 (2004) 6963.

Ferromagnetic Shape Memory Alloys 2

Tuesday afternoon, 6 September, 15:50
Main Building, room 315
Laurent Hirsinger, Victor Lvov presides

15:50 oral

Martensitic transformation of single crystalline Ni_2MnGa under compressive stress

Jae-hoon Kim, Takashi Fukuda, Tomoyuki Kakeshita

Graduated School of Engineering, Osaka University (OSAKAUNIV), Osaka, Japan

Contact: kimjaehoon@mat.eng.osaka-u.ac.jp

Effect of uniaxial compressive stress on martensitic transformation has been investigated in a single crystalline Ni_2MnGa, which exhibits a parent to an intermediate (P→I) phase transformation at T_I (250K) and successively an intermediate to 10M (I→10M) phase transformation at T_M (200 K). We found that a new phase, which is referred to as X-phase, appears under a compressive stress applied along $[001]_P$ direction. That is, in a stress applying process, a suc-

cessive I→X→10M or P→X transformation appears depending on temperature. On the other hand, in a cooling process under a stress applied along $[001]_P$ direction, a successive P→X→I→10M or P→X→10M transformation appears depending on the strength of the stress. The I→X and the X→10M transformations are both first order with an apparent temperature hysteresis, but no hysteresis is observed for the P→X transformation. The X→10M and the P→X transformation temperatures increase as stress increases, but the I→X one decreases as stress increases. The increasing ratios against compressive stress, $dT/d\sigma$, are about 0.5 K/MPa, 0.3 K/MPa and −4.0 K/MPa for the X→10M, the P→X and the I→X transformations, respectively.

16:05 oral

MICROSTRUCTURE OF NiMnGa MAGNETIC SHAPE MEMORY ALLOYS IN THE AUSTENITE PHASE

Gursev Pirge[1,2], Sabri Altıntaş[2]

1. *Turkish Air Force Academy, Yesilyurt, Istanbul 34149, Turkey* **2.** *Bogazici University, Istanbul, Turkey*

Contact: g.pirge@hho.edu.tr

Magnetic shape memory (MSM) alloys are a class of actuator materials which produce strain via the magnetic field induced reorientation of twin variants in response to an applied magnetic field. MSM effect depends on the success of parameters like mobility of twin boundaries, magnetic properties of the alloy, proper specimen orientation and specimen shape.

Solidification behavior of these alloys has a strong effect on their microstructure, and hence on their mechanical and MSM properties. In order to produce single crystals showing a distinct martensite transformation and a strong MSM effect, it is necessary to obtain a uniform martensite in the entire sample. The martensite transformation temperature is very sensitive to the alloy composition. A single phase having a homogeneous composition is thus desirable in order to avoid having different components of the microstructure undergoing the martensite transformation at different temperatures, or not undergoing the transformation at all in the case of the eutectic structures and Mn-rich particles.

In this study, the microstructure, segregation tendency of the constituent elements and the effect of the composition gradients of NiMnGa alloys were investigated. For the current growth conditions, coarse cellular structures have been obtained which show significant solute segregation. Mn was found to segregate to the cell boundary, whereas Ga tended to segregate to the cell core. The results showed that as solidified, off-stoichiometric, alloys had three distinct microstructural features—a Heusler phase, an Mn Rich phase and a eutectic or eutectoid region. The latter could be removed by prolonged annealing at elevated temperatures, but that the coarse Mn-rich particles are much more difficult to remove. This phase is likely harmful to the mechanical properties of the alloy and should be eliminated in the future.

16:20 oral

Effect of grain size on amount of deformation-induced ε martensite and shape memory alloy in Fe-Mn Alloy

Kwang K. Jee

Korea Institute of Science and Technology, Material Science and Engineering Division, Seoul, Korea, South

Contact: kkjee@kist.re.kr

Effect of grain size on amount of deformation-induced ε martensite and shape memory alloy in Fe-Mn Alloy

K. K. Jee*, J. H. Han* and W. Y. Jang**

* Division of Materials, Korea Institute of Science and Technology, Seoul 136-791, Korea

** Division of Metallurgical and Materials Engineering, Chosun University, Gwangju 501-759, Korea

Abstract

In this work, a new method of measuring volume fraction of deformation-induced ε martensite for Fe-Mn alloys is proposed using endothermic heat on ε→γ transformation. The variation in the amount of ε induced by deformation with grain size is determined for an Fe-21Mn alloy, and its influence on shape memory effect is investigated.

ΔH/mole on reverse transformation for the alloy is obtained by comparing ΔH and the volume fraction of martensite. The latter is calculated from dilatometric work using the specific volume difference between γ and ε.

As grain size increases, the amount of ε martensite forming on cooling increases. However, with a decrease in grain size, more ε is induced by deformation, improving shape memory effect.

16:35 oral

Magnetocaloric effect in Ni2.19Mn0.81Ga Heusler alloy

Vasiliy D. Buchelnikov[1], A M. Aliev[2], A B. Batdalov[2], A M. Gamzatov[2], Rostislav M. Grechishkin[3], V V. Koledov[4], A V. Korolyov[5], N I. Kourov[5], V. G. Pushin[5], S V. Taskaev1, Vladimir V. Khovailo[6], Vladimir G. Shavrov[4]

1. *Chelyabinsk State University (ChelSU), Br. Kashirinykh Str, 129, Chelyabinsk 454021, Russian Federation* **2.** *Institute of Physics of DSC, RAS, Makhachkala 367003, Russian Federation* **3.** *Tver State University, Tver 170000, Russian Federation* **4.** *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation* **5.** *Russian Academy of Sciences, Ural Division, Institute of Metal Physics, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation* **6.** *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation*

Contact: buche@csu.ru

In Ni-Mn-Ga alloys coupled magnetostructural transition (MST) may occur. The giant magnetocaloric effect (MCE) observed during MST in Ni-Mn-Ga alloys is regarded promising for the development of economical and ecological refrigerants working near room temperature. In this work we study MCE during MST by two experimental methods. A direct MCE measurement is performed with the $Ni_{2.19}Mn_{0.81}Ga$ sample immersed into the calorimetric system. The largest entropy change at change of magnetic field from 0 to 26 kOe are equal about 9 J/kgK if temperature is increased and about 11 J/kgK if temperature is decreased. In the second method we are used the indirect measurements using the magnetization temperature dependences. From these dependences MCE values are deduced with the help of Maxwell relation. Most attention is concentrated on MCE behavior in the region of MST hysteresis. This is important, because, firstly, MCE has sharp peaks at the edges of MST hysteresis, and, secondly, it is the region for which the traditional indirect method of MCE measurement is least effective, because the thermodynamical relations are justified only for reversible 2nd order transitions. The experimental data are compared with each other and with the theoretical results which were obtained by the help of phenomenological theory based on the statistical physics and the molecular field theory. This work was supported RFBR (grants 03-02-17443, 04-02-81058, 03-02-39006).

16:50 oral

Martensitic transition in strong magnetic field and external stress in quaternary ferromagnetic shape memory alloy Ni-Mn-Fe-Ga

Victor V. Koledov[1], Rostislav M. Grechishkin[2], Vladimir V. Khovailo[1], Vladimir G. Shavrov[1], V. G. Pushin[3], A V. Korolyov[3], N I. Kourov[3], Evgeniy P. Krasnoperov, Alexandra A. Tulaikova[1], Vasiliy D. Buchelnikov[4], S V. Taskaev[4], Chengbao Jiang[5]

1. *Institute of Radio Engineering and Electronics RAS (IRE RAS), Mokhovaya, 11, Moscow 125009, Russian Federation* **2.** *Tver State University, Tver 170000, Russian Federation* **3.** *Russian Academy of Sciences, Ural Division, Institute of Metal Physics, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation* **4.** *Chelyabinsk State University (ChelSU), Br. Kashirinykh Str, 129, Chelyabinsk 454021, Russian Federation* **5.** *Beijing University of Aeronautics and Astronautics, Beijing, China*

Contact: victor_koledov@hotmail.com

Ferromagnetic shape memory alloys of the Ni-Mn-Ga family demonstrate a very spectacular illustration of magnetoelastic coupling. In these alloys the thermoelastic martensitic transition may occur in the ferromagnetic state. As a result giant magnetoinduced deformations up to several percents due to martensite domain boundaries movement [1] and magnetically controlled shape memory effect due to martensitic transition [2] can be observed in single- and polycrystals, respectively. The behavior of martensitic transition under the action of external field and stress is of extreme impotence in view of possible applications of polycrystalline alloys in sensor and actuator technology.

The aim of the present work is to study experimentally and theoretically the martensitic transition in polycrystalline $Ni_{2.14}Mn_{0.81}Fe_{0.05}Ga$ under the action of external uniaxial compressive stresses up to 40 MPa and magnetic fields up to 5 T. The measurements of low-field magnetic susceptibility near martensitic transition reveal that external stress tends to shift the martensitic transition hysteretic loop to a higher temperature region and to smoother the transition, widening the transition loop. Strong magnetic field up to 5 T shifts the hysteretic loop to higher temperatures without a pronounced deformation of the loop. In situ optical observations show the evolution of martensitic and ferromagnetic domains structure caused by the external stress. The theoretical and experimental results are in good agreement.

This work is supported by RFBR (Grants 03-02-17443, 04-02-81058, 03-02-39006).

[1] R.C. O'Handley, K.Ullakko, US Patent No 5958154, Sep.28.1999.

[2] A.N.Vasilev, A.V.Glebov, I.E.Dikshtein, et.al, Russian

Patent No 2221076, Jan.10.2004.

17:05 oral

A study of the magnetic resonance in a monocrystalline Ni50.47 Mn28.17Ga21.35

Bela D. Shanina[1], Valentin G. Gavriljuk[2], Sergej P. Kolesnik[1]

1. *Institute of Semiconductor Physics, National Academy of Sciences of Ukraine, Prospect Nauki,45, Kyiv 03028, Ukraine* **2.** *G.V. Kurdyumov Institute for Metal Physics National Academy of Sciences (IMP), Vernadsky Blvd. 36, Kyiv UA03680, Ukraine*

Contact: shanina_bela@rambler.ru

Non-stoichiometric alloy $Ni_{1-x-y}Mn_xGa_y$ with x = 0.2817, y = 0.2136 is studied using magnetic resonance spectroscopy: ferromagnetic resonance (FMR) and conduction electron spin resonance (CESR). Temperature dependence of the resonance field value H_{res}(T), the line width ΔH(T), and the integral intensity I(T) is measured across the wide temperature interval T= 4.2 K ÷ 570 K. Three phase transformations are found: paramagnetic↔ferromagnetic with Curie temperature of T_C = 360 K, austenite-to-martensite transformation (direct with M_s = 312 K and reverse with A_s = 313 K, and a transformation to a new state at T = 45 K. It is suggested that the low temperature transition proceeds through the spin glass state since the temperature dependence of the I(T) has the 'cusp'- like character. The angular dependence of the FMR signals was measured in the martensitic and austenitic states before and after martensite-austenite transition. Experimental data were used for determination of the magnetisation $4\pi I_s$ and anisotropy parameters K_1 = -0.77 kG^2 , K_2= 0.85 kG^2 for the martensitic state. The scattering of the values and disorientation of the magnetization directions over the sample are estimated using the analysis of the angular dependence of the line width. The temperature dependence of the resonance signals was investigated in the temperature range significantly higher than T_C where FMR is transformed to the conduction electron spin resonance. In the paramagnetic state (above T_C) the alloy reveals an intensive signal of CESR, which is an evidence for a high concentration of free electrons and correlates with the large magnetic-field-induced strain observed in the alloys of such composition. The temperature dependence of the skin layer is found from the sharp decay of the CESR signal with temperature, which is related to disappearing the large magnetic resistance after transformation to the paramagnetic state.

Wednesday, 7 September

Devices and System 1

Wednesday morning, 7 September, 9:00
Main Building, room 315
Concepcio Seguí, Stefano Besseghini presides

9:00 invited oral

Polymer-filled Superelastic Thin Film NiTi Tubes

Eckhard Quandt[1], Holger Rumpf[1], Christiane Zamponi[1], Christoph Bourauel[3], Dieter Drescher[2], Nikolai Botkin[1]

1. *center of advanced european studies and research (caesar), Ludwig-Erhard-Allee 2, Bonn 53175, Germany* **2.** *University of Duesseldorf, Polyclinic for Orthodontics, Düsseldorf 40225, Germany* **3.** *University of Bonn, Polyclinic for Orthodontics, Bonn 53117, Germany*

Contact: quandt@caesar.de

Superelastic shape memory materials are of special interest in medical applications due to the large obtainable strains, the constant stress level and their biocompatibility. Superelastic NiTi-polymer-composites have the potential to be used for novel applications in orthodontics and medical instrumentation as well as in certain areas of mechanical engineering. Especially, using NiTi thin films these composites have the potential to substantially reduce those forces compared to conventional NiTi wires and tubes. In orthodontic applications lowering the forces during archwire treatment is of special importance due to the risk of root resorption, which can be caused by the application of excessive forces. Furthermore, the use of superelastic materials or composites enables the application of constant forces independent of tooth movements during the therapy due to the superelastic plateau. Superelastic NiTi thin films have been fabricated by magnetron sputtering using extremely pure cast melted targets. Special heat treatments were performed for the adjustment of the superelastic properties and the transformation temperatures. A superelastic strain exceeding 6.5% at 400 MPa was obtained. In this paper the fabrication of superelastic NiTi thin walled tubes by magnetron sputtering their structural and thermomechanical properties are presented. Bending test results are compared to conventional wires in view of orthodontic applications.

Financial support of the German Federal Ministry of Education and Research (BMBF, Contract 03N4031A) is gratefully acknowledged.

9:45 oral

Two way shape memory training in NiTi shape memory alloys

Stefano Besseghini, Villa Elena, Passaretti francesca, Carcano Giordano

Istituto per l'Energetica e le Interfasi Consiglio Nazionale delle Ricerche Unità di Lecco (IENI Lecco), Corso Promessi Sposi, 29, Lecco 23900, Italy

Contact: s.besseghini@ieni.cnr.it

We report here on results obtained by training NiTi of differ-

ent compositions and submitted to different thermomechanical treatments in order to develop a device working exploiting the TWSME (two way shaoe memory effect). DSC measurements performed on the starting material allowed to assess the different response to the training procedure of the different materials used. Some evaluation on the stability on short term cycling of the material both in wire and strip shape was done.
The calorimetric evaluation was performed on the trained nad cycled material as weel. This allowed to asses the general effect on the microstructrure of the procedure performed. The results are here discussed at the light of the potential use of these training procedures in a real device.

10:00 oral

Design and Control of Multistep SMA Actuator

Yun-Jung Lee[1], HYUNG-MIN SON, JUN-BUM GU, Tae-hyun Nam[2]

1. *Kyungpook National University (KNU), Daegu 702-701, Korea, South* **2.** *Gyeongsang National University (GSNU), 900 Gazwadong, Jinju 660-701, Korea, South*

Contact: yjlee@ee.knu.ac.kr

In terms of positioning method, the conventional SMA actuators can be simply classified into two types; on-off type, continuous type. The on-off type SMA actuator has two controlled positions, i.e., fully elongated position and fully contracted position. An open loop control is used to actuate it in a way that electrical input power turns on or off. While the on-off type actuator has a disadvantage that it has only two controlled positions, its control system is very simple by virtue of sensor-less and on/off control scheme. For the continuous type SMA actuator, the close loop control is used to control it at an arbitrary position through a position-sensor feedback. The overall control system of the continuous type actuator has disadvantages which a bulky position sensor such as a potentiometer or an optical encoder is needed and the control method is usually complex to deal with SMA's inherent hysteresis with minor loops.
To compromise between control complexity and number of controlled positions, a novel multistep SMA actuator is proposed in this paper. The multistep SMA actuator has multiple controlled positions and it can be actuated by sensor-less and open loop control. A multistep SMA actuator can be constructed in a way that several electrical taps are connected on one SMA wire and each section between two taps has same length. The controlled positions vary stepwise according to the number of actuated sections. To control a multistep SMA actuator, a PWM(pulse width modulated) voltage with an appropriate duty ratio should be applied to designated sections in consideration of resistance value of the sections as well as a recommended current of the given SMA wire.
To verify effectiveness of the proposed concept of the multistep SMA actuator, experiments was performed using an actuator mechanism having one nitinol wire and a bias spring and an electronic control system with a micro-controller(TMS320F2812) and eight power transistors(D882-Y).

10:15 oral

An Isolation Bearing for Railway Bridges using Shape Memory Alloy Bars

Eunsoo Choi[1], Kyungtaek Yang[2], Cho Baik-Soon[3], Nam Tae-hyun[4]

1. *Korea Railroad Research Institute (KRRI), 360-1 Woulam-dong, Uiwang, Kyonggi 437-050, Korea, South* **2.** *Daelin College, Dept. of Mechatronics, Ahnyang, Kyonggi, Korea, South* **3.** *Inje Uisversity, Department of Civil Engineering, Kimhae, Korea, South* **4.** *Gyeoungsang National University, Department of Metal & Material Engineering, Jinjoo, Korea, South*

Contact: eunsoochoi@krri.re.kr

ABSTRACT: Conventional lead-rubber bearings may have a problem of instability and unrecovered deformation against a strong ground motion. Also, their lateral stiffness is too flexible to resist the braking forces in railway bridges. Therefore, it is hard for the bearings to be used for railway bridges.
To improve the problems, this study proposed a new concept of an isolation device in which shape memory alloy (SMA) bars are incorporated in an elastomeric bearing and a PTFE is laid between the top sole plate and the elastomeric bearing; the bearing is called ‘SMA-Frictional Rubber (SFR) bearing’. The characteristics of the proposed bearing such as; 1) the SMA bars contribute to propose more recentering force and the resistance in tension, 2) the PTFE increases the energy dissipation capacity during an earthquake and vehicle’s braking. The PTFE shows small frictional coefficient with slow movement such as thermal expansion and thus the developed force due to thermal expansion is not so high in the bearing.
The proposed bearing is tested with several loading frequencies. Also, conventional lead-rubber and elastomeric bearings are tested and their results are compared with the result of the SFR bearing. Based on the comparing, the advantages of the SFR bearings for railway bridges are discussed.

Devices and System 2

Wednesday afternoon, 7 September, 14:00
Main Building, room 315
Bernhard Winzek, Yun Luo presides

14:00 oral

Metallic Thin Film Composites with Shape Memory Alloys for Microswitches and Tactile Graphical Displays

Roman Vitushinsky[1], Fadila Khelfaoui[2], Sam Schmitz[1], Nikolai Botkin[1], Bernhard Winzek[1]

1. *center of advanced european studies and research (caesar), Ludwig-Erhard-Allee 2, Bonn 53175, Germany*
2. *Univ.Bonn, Bonn, Germany*

Contact: vitushinsky@caesar.de

According to the European recommendation 2000/78/EG, barrier-free access to electronic media has to be realized for visually impaired persons. While text files are presented by Braille cells or acoustically, the realization of graphic displays still requires huge technological expense and high costs. Further improvement is possible by shape memory alloy (SMA) actuators which provide the highest work output of the so-called smart materials. In particular, thin film SMA actuators are of special interest, since they provide much higher actuation frequencies than bulk actuators due to their high surface to volume ratio. In combination with other metallic films, SMA-composites show the two-way behavior right after deposition and annealing treatment. The work output upon the martensitic transformation is about ten times larger than the bimetallic effect. The corresponding actuators can be fabricated by cost effective parallel batch processing including magnetron sputtering and photolithographic structuring.

At caesar, a new mechanism to switch thin film actuators between two stable states has been developed.. Bistable membrane switches can be realized by snap-dome shaped metallic foils which are coated on opposite surfaces with Ti(Ni,Cu) and (Ti,Hf)Ni respectively whereby the narrow hysteresis of Ti(Ni,Cu) is within the broad one of (Ti,Hf)Ni. If both SMAs are martensitic or both are austenitic, they apply similar forces on the intermediate foil and the shape of the membrane remains constant. However, if one of them is austenitic and the other one martensitic, the austenitic one determines the curvature of the membrane. Switching is possible by two different heat pulses. Therefore, energy is only required to change the state of the membrane.

The new mechanism is described and first successful processing steps are presented. The mechanism allows the fabrication of microswitches and tactile displays. It won the second funding prize 2004 of the foundation ONCE, Spain.

14:15 oral

Ferromagnetic Shape Memory Microscanner with Large Deflection Angles

Daniel Brugger, Manfred Kohl, Berthold Krevet

Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany

Contact: daniel.brugger@imt.fzk.de

Ferromagnetic shape memory alloys (FSMAs) experienced a widespread research boom as they exhibit a unique combination of high energy densities, thermoelastic and ferromagnetic properties. Of particular interest have been the Heusler ferromagnetic shape memory alloys Ni-Mn-Ga, which in single crystalline state show the giant magnetostrain effect. Currently, thin film technology is being developed intensively in order to pave the way for applications in microsystems and nanotechnology. For engineering of novel devices, test systems need to be developed and investigated with respect to their spatially and time-resolved physical behaviour in order to clarify the underlying fully coupled thermo-magneto-mechanical material properties.

In the present work, a Ni-Mn-Ga microscanner is used as a test system. Previously, it has been demonstrated that this system allows the simultaneous actuation in two opposite directions by making use of both the ferromagnetic transition and martensitic transformation. The stationary and time-resolved motion of the microscanner is studied experimentally and coupled finite element simulations are performed to clarify the complex implications of the thermal, mechanical and magnetic material properties. The experiments reveal a complex driving power and frequency dependence of the scanning angle. Infrared microscopy investigations are in line with coupled finite element simulations of temperature profiles. Thus, cut-off frequencies are determined theoretically and experimentally. Typical cut-off and resonance frequencies are 60 and 150 Hz, respectively. Based on experimental data, the magnetic field and magnetic force distribution in the system is analyzed.

14:30 oral

A new application of superelastic SMAs in surgical instruments - hemostatic forceps with safe pressures

Yun Luo, Masaru Higa, Shintaro Amae, Tomoyuki Yambe, Toshiyuki Takagi

Tohoku University, Sendai, Japan

Contact: luo@tubero.tohoku.ac.jp

The authors have proposed a new application of superelastic SMAs in surgical instruments to enhance their safety. The constant stress property exhibited during the stress-induced

transition of superelastic SMA is used to control the clamping pressure of hemostatic forceps within a safe range on biological tissues. Conventional hemostatic forcepses have rigid structures and might apply pressure higher than enough on biological tissues. In contrast, with the new SMA combined forceps it is possible to apply sufficient but not excessive pressure for homeostasis or arrest of bleeding, and is therefore able to minimize the damage on tissues under clamping.
In this paper, the basic concept of this new application of superelastic SMAs will be described first. Then design issues including material selection, safe pressure range of general forceps, activation mechanism of induction of transformation by stress will be discussed. Basic designs will be given to match the different purpose and specification of hemostatic forceps. The validity of the proposed concept has been examined by the experimental results obtained from the developed prototypes.

14:45 oral

Magnetomechanical Properties of Polycrystalline Ni-Mn-Ga Thin Film Actuators

Anurag Agarwal[1,2], Manfred Kohl[2], Volodymyr A. Chernenko[3], M. Ohtsuka[4]

1. *Indian Institute Of Technology,Kharagpur (IIT), Kharagpur, India* **2.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany* **3.** *Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine* **4.** *Tohoku University, Sendai, Japan*

Contact: mailinganu@yahoo.co.in

A series of experiments have been performed on bending, tensile and bridge actuators of polycrystalline Ni-Mn-Ga thin films, to observe the effects of applied tensile load and varying magnetic field on the field-induced strain in martensitic state. Ni-Mn-Ga thin films of 10µm thickness have been deposited by magnetron sputtering. The actuators have been fabricated by photochemical micromachining and MEMS-based interconnection technology. An interesting enhancement of magnetostrain is noticed for both, tensile and bridge actuators. For tensile actuators, the maximum magnetostrain is evaluated to -2470 ppm at 0.5 T and a stress of 7 MPa, which is considerably larger than the ordinary magnetostriction of -240 ppm for a reference thin film of 1 µm thickness. A trend of decrease in magnetostrain is noticed for increasing load. The maximum magnetostrain for the bridge actuators is estimated to -710 ppm for a magnetic field of 0.5 T and a stress of 13 MPa. In this case, an increase of magnetostrain is observed for increasing load.

15:00 oral

NiMnGa Fibres for use in polymer composites

Dietrich Hinz, Nils Scheerbaum, Oliver Gutfleisch, Karl-Hartmut Müller, Ludwig Schultz

Leibniz-Institute for Solid State and Materials Research, P.O.Box 270116, Dresden D-01171, Germany

Contact: d.hinz@ifw-dresden.de

Bulk single crystals of Ni-Mn-Ga show the strongest magnetic shape memory (MSM) effect. Their use would be advantageous in application as actuators or as mechanical energy absorber. However, especially the high cost for the single crystals is a drawback. Therefore composites made of polymer and MSM particles may also be used for damping applications and as actuators. Ideally, the MSM particles should be, at the operating temperatures, in the martensitic state and single-crystalline when in the austenitic state. We used fibres of 51Ni-27Mn-22Ga (at.%), prepared by crucible melt extraction, as MSM material. The size of the fibres is about 60µm in diameter and 1cm in length. The structural and magnetic properties of the fibres are similar to those of bulk material with the same composition. SEM analysis reveals that the grain size of the melt-extracted fibres is about 5µm. The fibres were annealed at temperatures of 1000-1100C in order to achieve grain growth. After annealing, grain sizes in the order of the diameter of the fibres were observed. The annealing affects also the martensite start temperature. It increases from 30C to 45C. The Curie temperature, determined by DSC and susceptometer measurements to 98C, remains nearly unchanged. XRD analysis revealed that the cubic austenite changes to the tetragonal martensite (5M) with c/a=0.94. The martensite is highly twinned (determined by SEM) and SQUID measurements reveal a magnetisation process at low temperatures, which could be attributed to twin boundary motion. Additionally, the structural properties are strongly affected by a mechanical treatment (milling). The milled fibres no longer show a martensitic transition, unless they were annealed again at 600C. Here XRD analyses showed that milling leads to a higher fraction of the martensite phase compared to the non-milled state. First composites were prepared using different polymers applying various magnetic fields to align the MSM particles.

Wednesday Poster Session
Wednesday afternoon, 7 September, 15:50

Thursday, 8 September

Modeling and Simulation 1
Thursday morning, 8 September, 9:00
Main Building, room 315
Berthold Krevet, Stefan Seelecke presides

9:00 invited oral

Modeling and Finite element simulation

Ferdinando Auricchio

Dipartimento di Meccanica Strutturale, Universita' di Pavia, Via Ferrata 1, Pavia 27100, Italy

Contact: auricchio@unipv.it

An always increasing knowledge on material properties as well as a progressively more sophisticated production technology make Shape Memory Alloys (SMA)

extremely interesting for the industrial world.At the same time, SMA devices are typically characterized by complex multi-axial stress states as well as non-homogeneous and non-isothermal conditions both in space and time.

This aspect suggests the finite element method as a useful tool to help and improve application design and realization. With this aim, we focus on three-dimensional macroscopic thermo-mechanicalmodeling able to reproduce the most significant SMA features, such as pseudo elasticity and shape memory effect. Moreover, for the proposed models it is possible the development of a

time-discrete solution algorithm, which is effective and robust.

We verify the computational tool ability to simulate complex pointwise stress-strain histories as well as realistic thermo-mechanical boundary value problems.

As an example we may consider coronaric stents, which are small metal tubes, inserted into an artery at the site of a narrowing to act as an internal scaffolding or support to the blood vessel. Other simulations and details can be found on the web page

http://www.unipv.it/dms/auricchio/cofin_02/home.htm

9:45 oral

Finite element analysis of adaptative systems

Etienne Patoor, Bertrand PEULTIER, Tarak BEN ZINEB

Laboratoire de Physique et de mécanique des matériaux, UMR CNRS 7554 (LPMM), Ile du Saulcy, METZ 57045, France

Contact: patoor@lpmm.univ-metz.fr

Shape Memory Alloys are widely used in several fields such as aeronautics or biomedical applications due to their unique behaviors (superelasticity, constrain recovery, shape memory). These behaviors originate from a thermoelastic martensitic transformation responsible for the coupling between mechanical and thermal properties observed in these materials. Optimisation of applications dealing with SMA requires the determination of numerical simulation tools able to capture this coupling. This communication presents the implementation into a commercial FEM code of a SMA constitutive law developed from micromechanical considerations. This thermomechanical constitutive law is derived from a micromechanical approach in such a way to reach an analytical description adapted to structure analysis by finite element method. In order to preserve a good description of physical phenomena at the origin of SMA behaviours a two internal variables description is introduced. The first variable corresponds to the volume fraction of martensite and the second one is a tensorial one corresponding to the Mean Transformation Strain (MTS) defined over the RVE. This MTS is connected to the phase transformation induced by mechanical and/or thermal loading and also to the reorientation process observed in the martensitic state. This model describes in a unified way superelasticity, constrained recovery and shape memory effect. Simulations obtained are confronted with an experimental data base determined on NiTi alloys.

The implementation of this model in the Abaqus FE code is ensured via the subroutine Umat (User MATerial) where the thermomechanical tangent operators are calculated. This subroutine updates stresses and internal variables. The Newton-Raphson numerical method is adopted for the resolution of this problem. Application of this finite element analysis is presented on SMA structures.

10:00 oral

3D phenomenological thermodynamics of pseudoelasticity at finite strain, concepts

Andrzej Ziolkowski

Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT PAN), Świętokrzyska 21, Warszawa 00-049, Poland

Contact: aziolk@ippt.gov.pl

The familiar small strain thermodynamic 3D theory of isotropic pseudoelasticity proposed by Raniecki and Lexcellent is generalized to account for the geometrical effects. The Eckard-Mandel concept, of mobile isoclinic reference configurations, is used for multiplicative decomposition of total deformation gradient into elastic and phase transformation part and resulting from it additive decomposition of Eulerian strain rate tensor. As a set of state parameters entering macroscopic free energy function of SMA material in actual configuration logarithmic elastic strain, temperature and mass fraction of martensitic phase is used. There will be presented selected experimental results for support of adopted theoretical assumptions. In finite deformations theory we will adopt without a "proof" a number of concepts from small strains theory. For example, in small strains theory of pseudoelasticity it can be proved that parent phase reaches unstable absolute equilibrium states when thermodynamic driving force of phase transformations (p.t.) becomes zero. This gives merit for requiring that p.t. can start when thermodynamic driving

force of p.t becomes zero. The same condition is adopted in the finite deformation theory without formal support. The rate equations for SMA material formulated in the mobile Lagrangian configuration are translated to the actual configuration. They are relevant for the use of updated Lagrangian technique. Their combination with the nominal rate of stress tensor (by familiar bridging equation) and rate form of equilibrium equations leads to the fundamental set of partial differential equations of pseudoelasticty for the velocity field. Since SMA materials exhibit small elastic distortions but possibly large elastic dilatational changes, e.g. under dynamic loads. The postulated here elastic shear strain energy has the same functional form as the one used in small strains theory but upon substitution of small strain tensor with logarithmic strain

[ABSTRACT TRUNCATED TO 2000 LETTERS]

10:15 oral

The modeling of the deformation behavior of Cu-Al-Nb shape memory alloys containing primary particles

Jozef Lelatko, Henryk Morawiec

Silesian University of Technology, Department of Materials Science, Krasińskiego 8, Katowice 40-019, Poland

Contact: jlelatko@us.edu.pl

The deformation properties of a new group of the Cu-Al-Nb (up to about 8 wt.% Nb) base alloys exhibiting the shape memory effect at elevated temperature were the subject of this work. Niobium, which solubility in copper is low, exists in these alloys in a form of primary precipitates of intermetallic phases, mainly the Nb(Cu,Al)2 Laves phase. The carried out studies have shown that the primary precipitates of the intermetallic phases on the niobium base play a basic role in a simultaneous strengthening and increase in plasticity of these alloys.

Models elaborated for the metal-matrix composite materials were modified in order to enable the control of the deformation properties of shape memory alloys containing significant number of primary particles.

The modified model of the continuum mechanics approach based on the effective medium approximations and supplemented by the dislocation plasticity model enabled analysis of the deformation course of the Cu-Al-Nb type alloys. By comparing the simulated and experimental stress – strain curves for these alloys, the effect of particle size, volume fraction and their elastic properties on the deformation response are given. With this model it can be shown that the increase in the alloy hardening during deformation results from the presence of hard and elastic particles in the martensite matrix as well as dislocations formed at the partial accommodation of the residual stresses created during the alloy quenching.

Modeling and Simulation 2

Thursday morning, 8 September, 11:00
Main Building, room 315
Andrzej Ziolkowski, Etienne Patoor presides

11:00 oral

A Model for Ferromagnetic Shape Memory Thin film Actuators

Stefan Seelecke, Kwok-Lun Lee

North Carolina State University (NCSU), 3211 Broughton Hall, Raleigh 27695, United States

Contact: stefan_seelecke@ncsu.edu

The last decade has witnessed the discovery of materials combining shape memory behavior with ferromagnetic properties (FSMAs). These materials feature the so-called giant magnetostrain effect, which, in contrast to conventional magnetostriction is due motion of martensite twins. This effect has motivated the development of a new class of active materials transducers, which combine intrinsic sensing capabilities with superior actuation speed and improved efficiency when compared to conventional shape memory alloys.

Currently, thin film technology is being developed intensively in order to pave the way for applications in micro- and nanotechnology. As an example, Kohl et al., 2004a, recently proposed a novel actuation mechanism based on NiMnGa thin film technology, which makes use of both the ferromagnetic transition and the martensitic transformation allowing the realization of an almost perfect antagonism in a single component part. Possible applications in nanotechnology arise, e.g., by combination of smart NiMnGa actuators with scanning probe technologies.

The research presented in this paper aims at the development of a model that simulates the material behavior of the above device. It uses a model originally developed for conventional shape memory alloy behavior by Mueller, Achenbach, and Seelecke and couples it with an expression for the nonlinear temperature- and position-dependent effective magnetic force. Even though the model does not account for full coupling between SMA behavior and ferromagnetism yet, it shows the potential to predict the effect of different Curie temperatures on the device performance and will eventually allow optimizing material properties for a specific application.

11:15 oral

Coupled simulation of the thermo-magneto-mechanical properties of a Ni-Mn-Ga actuator

Berthold Krevet[1], Manfred Kohl[2]

1. *Forschungszentrum Karlsruhe (FZK), Postfach 3640, Karlsruhe D-76021, Germany* **2.** *Forschungszentrum Karlsruhe, IMT, Postfach 3640, Karlsruhe 76021, Germany*

Contact: berthold.krevet@imt.fzk.de

Ferromagnetic shape memory alloys (FSMA) are a new class of materials, which show a unique combination of thermoelastic and ferromagnetic properties. In order to understand the spatially and time-resolved physical behaviour of FSMA sensors and actuators, a fully coupled model for the thermo-magneto-mechanical material properties is needed.

Micromachined Ni-Mn-Ga cantilevers have been fabricated as a test platform for evaluating physical models of the different coupling effects. A simulation tool has been developed, which couples different kinds of finite element programs for calculation of hysteretic material behaviour under multi-field loading. A two-phase shape memory model including history effects is implemented in the mechanical solver. A magnetic solver has been developed, which is capable of calculating forces and their gradients acting on temperature-dependent magnetized magnetic shape memory material.

In this paper, we present calculated static and dynamic temperature distributions of a Ni-Mn-Ga double-beam cantilever under different heating conditions taking into account the effects of martensitic phase transformation. The results are used for a self-consistent mechanical deflection analysis this actuator in the inhomogeneous magnetic field of a permanent magnet, which is determined by the spatial distribution of ferromagnetic states. The influence of the shape memory and ferromagnetic effects on the actuator performance is discussed. Dynamic simulations are performed to understand the power-dependent frequency characteristics of the actuator.

11:30 oral

Modeling of rearrangement yield surface under biaxial magnetic field in Ni-Mn-Ga shape memory alloys

Laurent Hirsinger, Nicolas Creton

Institut FEMTO-ST, Dpt Applied Mechanics (FEMTO), 24 Chemin de l'Epitaphe, Besançon 25000, France

Contact: laurent.hirsinger@univ-fcomte.fr

In the search of new active materials, Ni-Mn-Ga ferromagnetic shape memory alloys have a prominent position thanks to their capability to rapidly develop a very important strain. This particular behavior is in fact due to variant structure rearrangement of twinned martensitic platelets (5M, 7M or TNM) at constant temperature. To predict this strain induced by an applied magnetic field and/or an applied stress, the authors have proposed a magneto-mechanical model based on phenomenology, thermodynamics of irreversible processes and changing of scale (or homogenization). More exactly, this model has been developed and validated for uniaxial magnetic field (in an fixed direction), but, for multiaxial stress in a single crystal with 5M and 7M twinned martensite.

In this paper, an extension of this model is proposed in order to predict the effect of biaxial magnetic field in single crystal. For sake of simplicity, no demagnetizing field is considered. A Representative Elementary Volume is classically introduced with different microstructural parameters, i.e. volume fractions of martensitic bands, magnetization rotations, width ratios of two consecutive magnetic domains. It must be noticed that these variables are linked to physical phenomenon, for example, the movements of 180 magnetic domains corresponds to the evolution of width ratios. On the contrary to what is done usually, the movements of magnetic domain walls between martensitic bands are dissociated. The driving force acting on twin boundaries is written. Some yield surfaces of rearrangement are deduced under applied biaxial magnetic field, showing the non influence of initial volume fraction of martensitic bands and the existence of directions (at ±45 in respect to c axis) in which the rearrangement under magnetic field is hard or impossible. These results are fully compatible with the experiments of Müllner et al describing the action of a rotating magnetic field.

11:45 oral

Prediction of the Martensite Transformation Temperatures of NiMnGa Magnetic Shape Memory Alloys Using Artificial Neural Networks

Murat Ermis[1], Gursev Pirge[1,2], Sabri Altıntaş[2]

1. *Turkish Air Force Academy, Yesilyurt, Istanbul 34149, Turkey* **2.** *Bogazici University, Istanbul, Turkey*

Contact: ermis@hho.edu.tr

Magnetic shape memory (MSM) alloys are a new class of actuator materials with high actuation frequency, energy density and strain. MSM effect occurs in alloys, which exhibit a martensite transformation and are ferromagnetic. It involves, under effect of magnetic field, a high strain achieved via reorientation of twinned martensite plates.

The presence of composition variations in the boules prepared by using the Bridgman technique is a major issue because of its effect on the transformation temperature and MSM effect is only possible in the martensite region. The major problem is that even a slight change in the alloy's composition causes drastic changes in the martensite transformation temperature. For boules with composition variations, both transformed and

untransformed regions will exist over some temperature range, degrading the performance of any actuator made from them.

Therefore it is crucial to be able to predict the martensite transformation temperature of any NiMnGa alloy. Artificial Neural Networks (ANN) with their learning and generalization ability may act as a suitable tool to predict the martensite transformation temperatures of NiMnGa alloys.

ANN learn the relationships between cause and effect by using a model-free approach. They are generally used when the problem cannot be explicitly described by an algorithm, a set of equations, or a set of rules and ANN approach has recently been applied in prediction due to its adequate performance in pattern recognition.

In this paper, for martensite transformation temperature prediction, the performance of a multi-layer perceptron has been studied. Next, a radial basis function has been employed for prediction. Training and validation stages of the approach are performed by using data sets from four separate analysis results and our chemical analysis results were used for testing.

Computational results show a possible relationship between alloy's composition and martensite transformation temperature.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

12:00 oral

Modeling the stress-induced transition behaviour of shape memory alloy using mixture theory

Takeshi Okuyama[1], Yun Luo[2], Masaru Higa[2], Toshiyuki Takagi[1], Minoru Matsumoto[1]

1. *Institute of Fluid Science, Tohoku University (IFS), Sendai, Japan* **2.** *Tohoku Unversity Biomedical Engineering Research Organization (TUBERO), Sendai, Japan*

Contact: okuyama@wert.ifs.tohoku.ac.jp

In this paper, the modeling of the mechanical constitutive relation during stress-induced transitions of shape memory alloys (SMAs) is discussed based on mixture theory. When the initial state of SMA material was a mixture of austenite parents and twinned martensite variants, external stresses for this material would induce both the reorientation from twinned martensite variants to detwinned martensite variants and the transformation from austenite parents to detwinned ones. Corresponding internal variables to describe these transitions are newly defined. This model enables the numerical prediction of the overall mechanical behavior of SMA materials in the temperature range from M_s to M_f. The approach of mixture theory; a series model is applied in the formulation of the constitutive behavior of SMAs with isostress conditions. Tensile tests have been carried out with Ti-Ni specimens in a temperature range including martensite transformation temperatures. Numerical simulation based on the proposed model accurately predicted the mechanical behavior of stress-induced transitions in the concerned temperature range.

Symposium D

Magnetoelectronics

Welcome

Symposium D is addressed to all scientists who do not only focus on the use of the electric charge of the electron but are also interested in the spin degree of freedom. This is of particular importance today as the conventional silicon-based electronics is now facing barriers of fundamental nature that impede further miniaturisation. In view of the ongoing research into new materials for application in magnetoelectronis, **Symposium D** is expected to bring together scientists who are active in this modern and exciting field of science and provide an opportunity to present recent developments and outline future research areas in the electronic and magnetic properties of a vast class of materials and nanomagnetic systems such as thin films, multilayers, intermetallic compounds, granular composites, fine-particle and molecular systems (including carbon and other nanotubes) and quantum dots. Therefore, **Sympo-sium D** is a multidisciplinary meeting, addressed to scientists interested in the development of spin elec-tronics from electrical engineering, materials sciences, solid state chemistry and physics, both theoretical and experimental.

Theoretical and experimental works to be presented at **Symposium D** should focus on the magnetic and transport phenomena in nanoscale structures and on heterostructures containing ferromag-netic species. There will be also place to discussmany physical phenomena connected with magnetic nanocontacts, materials showing GMR (giant magnetoresistance) or CMR (colossal mag-netoresistance), structures with TMR (tunnel magnetoresistance), spin injection and current-induced switching of magnetization and micro-scopic methods for characterization of magnetic nanos-tructures and their dynamics.

The symposium shall take a form of a discussion following of invited lectures, oral communications and poster presentations covering the topics:

- spin-dependent phenomena in nanostructured and layered systems,
- new magnetic materials: magnetic semiconductors, magnetic insulators, half-metals, and intermetallic compounds showing magnetoresistance effects,
- devices and device design for magnetoelectronics.

INVITED KEY-NOTE LECTURE:

- **A. Fert**, "Spin transport in semiconductors between a magnetic source and a drain", Unité Mixte de Physique CNRS/THALES, Orsay, France

INVITED LECTURES:

- **J. Barnaś**, "From Giant magnetoresistance to current-induced magnetic switching and excitations in magnetic structures", Institute of Physics UAM and Institute of Molecular Physics PAS, Poznań, Poland
- **V.K. Dugaev**, "Current-induced spin torque and the domain wall dynamics in magnetic nanowires", National Academy of Sciences of Ukraine, Chernovtsy, Ukraine
- **C. Felser**, "Properties of Co_2YZ Heusler compounds", Institute of Inorganic and Analytical Chemistry, University of Mainz, Germany
- **R. Ferreira**, "Magnetoelectronic devices: heads, memories and sensors", Instituto de Engenharia de Sistemas e Computadores Microsistemas & Nanotecnologia, Lisbon, Portugal
- **A. Hütten**, "Spin-injection devices with half-metallic Heusler alloys", Department of Physics, University of Bielefeld, Germany
- **A. Mycielski**, "Applications of the II-VI semimagnetic semiconductors", Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Ulrich K. Rößler**, "Magnetic structures and magnetization processes in layered systems with antiferromagnetic couplings" Institut für Metallische Werkstoffe, IFW Dresden, Germany

Organisers

- **Bogdan Idzikowski**, Institute of Molecular Physics, Polish Academy of Sciences, M. Smoluchowskiego 17, 60-179 Poznań, Poland, Phone/Fax: +48 61 86 95231/84524, Bogdan.Idzikowski@ifmpan.poznan.pl
- **Ulrich K. Rößler,** Institute for Theoretical Solid State Physics, Leibniz-Institute for Solid State and Materials Research Dresden, Helmholtzstr. 20, 01069 Dresden, Germany,Phone/Fax: +49 351 4659 542/537, U.Roessler@ifw-dresden.de
- **Michel Goiran,** Laboratoire National des Champs Magnétiques Pulsés, 143 Av. de Rangueil, 31432 Toulouse Cedex 4, France, Phone/Fax: +33 5 621728 63/16, goiran@lncmp.org
- **Witold Dobrowolski,** Institute of Physics, Polish Academy of Sciences, al. Lotników 32/46, 02-668 Warsaw, Poland, Phone/Fax: +48 22 843 7001/0926, dobro@ifpan.edu.pl

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Proceedings

The proceedings of the **Symposium D** will be published in a special issue of the **Journal of Alloys and Com-pounds**. We will accept only one manuscript submission from each presenting author. All manuscripts will be reviewed according to the standards of the **Journal of Alloys and Compounds**. The Editors will take care that each paper accepted for publication represents an original work that has not been published previously.

Instructions for authors are avail-able at: http://authors.elsevier.com/JournalDetail.html?PubID=522468&Precis=DESC

Please strictly obey the rules given in "Guide for Authors" on this www page. All articles should be submitted elec-tronically to the selected Symposium Organizers (**B. Idzikowski, M. Goiran**) or our senior Guest Editor **Professor Wojciech Suski** (suski@int.pan.wroc.pl) **before August 1, 2005**.

The length of invited and contributed papers should be 5 and 3 journal printed pages, respec-tively. Note that 3 double-spaced A4 page in the manuscript, including references, tables and figures (in the 1:1 printing scale), are approximately 1 final journal page. During the sympo-sium referee reports will be delivered to the corresponding authors. Final version of the manu-scripts must be submitted by the **end of September 2005**.

Acknowledgements

Scientific Network "New materials for magnetoelectronics"www.ifmpan.poznan.pl/MAG-EL-MAT

Programme

Monday, 5 September

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster D-1

Total energy and electronic structure of the ZnO nanotips: ab initio calculation

Ruslana M. Balabay, Helen Y. Chernonog

Contact: charly-15@mail.ru

Calculation has been performed within the local density approximation to the density functional formalism. The electron-ion interaction has been modeled by ab initio norm-conserving pseudopotentials Hartwigsen, Goedecker, Hutter. The ground state electronic and atomic structures and its energy are obtained by “dynamical simulated annealing” (the Car-Parinello method).

For the calculation a superlattice geometry was chosen. The atomic basis of the primitive tetragonal unit cell of the superlattice consisted of 13 atom, 7 of which were Zn atoms and 6 – were O atoms, which formed a isolate configuration.

Another atomic basis consisted of 11 atoms, 5 of which were Zn atoms and 6 were O atoms, which formed a defect configuration with 3 layers along the z-axis and the infinite plane along the x-axis. Were received total energy and cards of the valent electron density for 2 configurations.

15:50 poster D-2

Anomalous Hall Effect in IV-VI semimagnetic semiconductors

Beata B. Brodowska[1], Witold Dobrowolski[1], Monika Arciszewska[1], Evgen I. Slynko[3], Vitalii Dugaev[2,3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Department of Physics and CFIF, Instituto Superior Tecnico,, Av. Rovisco Pais, Lisbon 1049-001, Portugal* **3.** *NAS of Ukraine, Institute for Problems of Material Science, Chernitsi Department, 5 I.Vilde str., Chernivtsi 58001, Ukraine*

Contact: brodo@ifpan.edu.pl

There is a growing interest in the Anomalous Hall Effect (AHE) due to the importance of the spin polarization and spin-orbit interaction for transport properties of materials and structures of spin electronics. In ferromagnetic metals and in magnetic semiconductors where the AHE can be observed,

the transverse resistivity ρ_H contains a contribution proportional to magnetization M in addition to the usual Hall effect: $\rho_H = R_0 B + \mu_0 R_S M$, where B is the magnetic field, R_0 and R_S, respectively, are the normal and anomalous Hall coefficient, and μ_0 is the magnetic constant. The purpose of our work is to study the AHE in IV-VI narrow gap ferromagnetic semiconductors: $Sn_{1-x}Mn_xTe$, $Sn_{1-x-y}Mn_xEr_yTe$, $Sn_{1-x-y}Mn_xEu_yTe$. The IV-VI systems were chosen for several reasons. These materials crystallize in the simple NaCl structure. Their energetic structure parameters are well known. The Curie temperatures are in the range 10-25 K what allows to perform measurements in a wide temperature range below and under the Curie temperature.

The samples used in our experiments were grown by the modified Bridgman method, their chemical composition was measured by microprobe technique and by X-ray dispersive fluorescence analysis. The $Sn_{1-x}Mn_xTe$ samples had 7.5% and 12% of manganese, $Sn_{1-x-y}Mn_xEr_yTe$ had 5.8% and 6% of manganese, and about 0.05% of erbium,the $Sn_{1-x-y}Mn_xEu_yTe$ sample had 12.8% of manganese and 1.4% of europium. The Hall effect and electric conductivity measurements (up to 13 T in the temperature range 1.5-60 K) were performed using the standard DC technique. Magnetic properties were studied using Lake Shore 7229 Magnetometer/Susceptometer. Magnetization of those samples was measured at magnetic fields up to 9 T for the same temperatures as the /chi(T) dependence. As the result of the studies, both R_0 and R_S were evaluated and their dependence on temperature estimated.

15:50 poster D-3

Ferromagnetic (Eu,Gd)Te/PbTe semiconductor heterostructures

Piotr Dziawa, Sylwia Wrotek, Wiktor Domuchowski, K. Dybko, Leszek Kowalczyk, Elżbieta Łusakowska, Andrzej Mąkosa, A. Morawski, Victor Osinniy, Badri Taliashvili, Zbigniew Tkaczyk, Tadeusz Wosiński, Jerzy Wróbel, Tomasz Story

Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland

Contact: dziawa@ifpan.edu.pl

In n-(Eu,Gd)Te magnetic semiconductor alloys ferromagnetic ordering is driven by the RKKY interaction between magnetic moments of rare earth Eu^{2+} and Gd^{3+} ions. This material is expected to exhibit very high degree of electron spin polarization and serve as a ferromagnetic element of model all-semiconductor spintronic structures. In this work we discuss the growth and structural characterization of (Eu,Gd)Te/PbTe heterostructures and analyze their magnetic and electrical characteristics.

(Eu,Gd)Te/PbTe heterostructures were grown by molecular beam epitaxy on BaF_2 (111) substrates. In situ reflection high-energy electron diffraction as well as ex situ x-ray diffraction and atomic force microscopy characterization proved high structural quality of both PbTe and (Eu,Gd)Te epitaxial layers. For magnetic characterization the measurements of magnetic hysteresis loops and the temperature dependence of magnetization of (Eu,Gd)Te/PbTe layers were carried out with SQUID magnetometer. In various n-(Eu,Gd)Te/PbTe layers ferromagnetic transition was found with the Curie temperature T_C=11-15 K. The n-(Eu,Gd)Te/p-PbTe heterostructures were fabricated lithographically in the form of mesa structures with thick PbTe buffer layer serving as the bottom p-type electrode and the n-(Eu,Gd)Te top electrode with gold electrical contacts. For electrical characterization the I-V characteristics and the electrical conductance of the p-n heterostructures were examined as a function of temperature and magnetic field. The strongly non-linear I-V characteristics of such p-n heterojunctions were experimentally observed. In selected heterojunctions it was found that the junction electrical conductance is reduced (up to 30-60 %) below the Curie temperature. The external magnetic field of 600 Oe applied in layer plane also reduces the junction conductance (typically by 10%).

Work supported by KBN research project No. PBZ-KBN-044/P03/2001.

15:50 poster D-4

Negative magnetoresistance of a high-mobility 2D electron gas in a GaAs quantum well with AlAs/GaAs superlattice barriers in a nonlinear regime

Alexander K. Kalagin, Alexey A. Bykov, Askhat K. Bakarov, Alexander I. Toropov

Institute of Semiconductor Physics SB RAS, pr. Lavrentieva 13, Novosibirsk 630090, Russian Federation

Contact: kalagin@thermo.isp.nsc.ru

We study the magnetoresistance (MR) of the high-mobility 2D electron gas (2DEG) in a GaAs quantum well with AlAs/GaAs superlattice barriers [1] in linear and nonlinear regimes. The selectively doped structures under study were grown using molecular beam epitaxy (MBE) on (100) GaAs substrates. The width of the GaAs quantum well was 13 nm. It has been found that, as the measuring current I increases, the MR becomes negative in the range of classically strong magnetic fields. It has been shown that the observed negative MR is due to the transport of the 2DEG in the nonlinear regime. The current–voltage (I-V) characteristic for Hall bar devices becomes nonlinear as the magnetic field increases. In the presence of magnetic field, with an increase in I, the slope of the I-V characteristic becomes equal to the slope of the I-V characteristic in zero magnetic field. The I value above which the slope of the I-V characteristic becomes equal to one in zero magnetic field, is determined by the correlation length of the scattering potential [2]. We estimated the correlation length of

the scattering potential in our samples as 5–10 nm. This estimates coincides by order of magnitude with the distance between the GaAs quantum well and delta-doped layers in our structures and it agrees with the generally accepted estimates of the correlation length of the scattering potential in high-mobility structures with modulated doping. Our results are consistent with the theory of the magnetotransport of the high-mobility 2DEG with large filling factors in the nonlinear regime [2]. Thus, it has been shown that the observed negative MR in the nonlinear regime can be used to find the correlation length of scattering potential in high-mobility modulated doping structures.

This work was supported by the RFBR, project no. 04-02-16789.

[1] K.-J. Friedland, R. Hey, et al., Phys. Rev. Lett. 77, 4616 (1996).

[2] M. G. Vavilov and I. L. Aleiner, Phys. Rev. B 69, 035303 (2004).

15:50 poster D-5

Magnetization study of interlayer exchange in semiconductor EuS-PbS ferromagnetic wedge multilayers

Leszek Kowalczyk[1], Victor Osinniy[1], Marina Chernyshova[1], Piotr Dziawa[1], Tomasz Story[1], Coen J. Smits[2], Henk J. Swagten[2], Alexander Y. Sipatov[3], Valentin V. Volobuev[3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Eindhoven University of Technology, Department of Physics, Eindhoven, Netherlands* **3.** *National Technical University"Kharkov Polytechnical Institute", 21 Frunze str., Kharkov 61002, Ukraine*

Contact: kowal@ifpan.edu.pl

In EuS-PbS semiconductor multilayers grown epitaxially along [001] crystal direction on either PbS or KCl substrates the layers of EuS (a model Heisenberg ferromagnet) are antiferromagnetically coupled via diamagnetic PbS spacer layer. For the experimental analysis of this effect neutron diffraction and reflectivity as well as SQUID magnetometry methods were applied to study the spacer thickness and temperature dependence of the coupling mechanism. In this work we present the analysis of the interlayer coupling in EuS-PbS multilayer wedge structures grown on KCl (001) substrates with the wedges covering the PbS spacer layer thickness from 6 to 0.3 nm. The structural parameters of the wedges were examined by the x-ray diffraction analysis of the EuS-PbS superlattice period. We discuss the measurements of magnetic hysteresis loops of EuS-PbS structures performed by both SQUID (for small terminal parts of the wedge) and MOKE (magneto-optical analysis along the wedge) magnetometry. The strong changes of magnetic remanence, coercive field and saturation field are observed for EuS-PbS structures with the PbS spacer thinner than about 1.5 nm. To model the magnetic field and temperature dependence of the magnetization of these structures, the temperature dependent interlayer exchange, in-plane anisotropy and Zeeman energy contributions are considered.

Work supported by KBN research project PBZ-KBN-044/P03/2001.

15:50 poster D-6

Influence of electrodes on magnetoresistance signals in nanostructured permalloy rings

Mei-Feng Lai[1], Zung-Hang Wei[1], J.C. Wu[2], Ching-Ray Chang[1]

1. *National Taiwan University (NTU), Roosevelt Rd., Taipei 106-17, Taiwan* **2.** *National Changhua University of Education, Changhua, Taiwan*

Contact: d90222026@ntu.edu.tw

The rapid development of magnetic data storage and magnetoelectronic devices technology has prompted the study of magnetization reversal on nanostructured thin film devices. Among various patterned elements, the ring structure has become a promising candidate for the magnetic random access memory (MRAM). It is also a good system to study nucleation, propagation, and annihilation of domain walls. Recently, magnetoresistance (MR) behaviors of ring structures have attracted much attention and are used to investigate the corresponding magnetization processes. However, the influence of the electrodes on the MR signals has to be understood in the first step. In this investigation we study the MR of Permalloy rings and clarify the influence of electrodes, which short circuit some domain structures, on the MR signals.

We fabricated the ring-shaped Permalloy thin film with gold electrodes by a two-step standard electron beam lithography and thermal evaporation through a lift-off technique. Room temperature MR measurements are carried out with 1-mA dc sensing current and under an external magnetic field applied in the film plane longitudinal or transverse to the connecting axis of the electrodes.

The MR of Permalloy rings are investigated here by micromagnetic simulation and by experiment. Significant differences in the MR curves when the relative positions of the electrodes are different with respect to the field direction are observed and explained. In addition to the relative positions of the electrodes, their spanned angles also strongly influence the MR signals of the ring. As increasing of the spanned angle, the characteristic of the plateau in the MR curve corresponding to the flux-closure state diminishes, and the kink beside the plateau becomes more and more obvious. We can find that the simulation result and experimental result are in

very good agreement.

15:50 poster D-7

Structural and magnetic characterization of thin films prepared by ion beam deposition

Diana Leitao, Joao P. Araujo, J. B. Sousa, Isabel G. Trindade, Jose Teixeira, Rui A. Silva

Departamento de Fisica and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal

Polycrystalline magnetic thin films of NiFe and Co, with different thicknesses and underlayers were prepared by ion beam deposition. During film growth, a magnetic field was applied to the substrates to induce a uniaxial ansisotropy field. The film's structural phase and texture was characterized by x-ray diffraction, revealing a strong dependence on the thickness of the magnetic layers and/or type of underlayer. A MOKE set-up sensitive to both in-plane magnetization components was used to study the magnetization and reversal magnetization processes. These studies were complemented by MOKE domain imaging and magnetoresistance measurements. The MOKE imaging unit, using a CCD camera for Kerr effect domain visualization, provides direct evidence on the magnetization and reversal mechanisms, namely domain wall nucleation, domain wall propagation and magnetization rotation. The dependence of the electrical resistivity on the short-range spin disorder and on the angle between the magnetization and the electrical current (anisotropic magnetoresistance) provided further information on the reversal mechanisms. A correlation between the magnetic properties and the structural phase of the films is described.

15:50 poster D-8

Spin versus orbital Kondo effect in a set of electrostatically coupled quantum dots

Stanisław Lipiński, Damian Krychowski

Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland

Contact: lipinski@ifmpan.poznan.pl

The coherent transport through a set of N capacitively coupled quantum dots placed in a magnetic field is considered in the limit of infinite intradot and interdot interactions. The dots are attached to separate leads. The mean field slave boson approach and the equation of motion method are used. For the full spin-orbit degenerate case the low energy behavior is characterized by an SU(2N) symmetry with entangled spin and charge correlations and a phase shift Π/2N. Application of an external magnetic field gives rise to a crossover to charge Kondo state with SU(N) symmetry. The polarization of the transmitted current does depend on the magnetic field and the current becomes almost fully polarized for large fields. The calculations are also performed for finite interdot interaction. For small values, the spin Kondo effect (SU(2)) at each of the dots is weakened with increasing interdot interactions. For large values the SU(2N) Kondo effect with lower Kondo temperature than for infinite case is observed.

15:50 poster D-9

Self-consistent (CPA) theory of scattering at disordered interfaces in layered nanostructures

Victor F. Los

Institute of Magnetism, NAS of Ukraine, 36-B Vernadsky str., Kiev 03142, Ukraine

Contact: victorlos@mail.ru

A self-consistent theory of electron scattering at the real disordered interfaces in the layered nanostructures is developed. This theory generalizes the well known quantum mechanics results on the electron transmission through and reflection from the potential steps/wells/barriers for the case of the intermixed (alloy-like) interfaces. The closed analytical expressions for the specular and diffuse factors for electron reflection and transmission are obtained in the self-consistent (coherent potential) approach and plotted. It is particularly shown that the diffuse scattering results in electron scattering to the areas inaccessible for specular scattering at the perfect interfaces. Also, the specular transmission probability over a potential well exhibits the additional (to the conventional resonance states) oscillations caused by the real part of the effective scattering (at disordered interfaces) potential. The obtained results are believed to be important for interpreting the giant and tunneling magnetoresistance effects in the real layered nanostructures.

15:50 poster D-10

Spacer layer properties in antiferromagneticaly coupled Fe/Si_xFe_{1-x}

Tadeusz E. Luciński[1], Piotr Wandziuk[1], Janusz Baszyński[1], Feliks Stobiecki[1], Bogdan Szymański[1], Josef Zweck[2]

1. *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*
2. *Institut für Experimentelle und Angewandte Physik Universität Regensburg (Uni. Regensb), Universitätsstraße 31, Regensburg 93040, Germany*

Contact: lucinski@ifmpan.poznan.pl

Metal-semiconductor multilayers (Mls) are extensively studied because of their potential application in electronics. The main goal of our study was to examine whether the existence of the antiferromagnetic (AF) exchange coupling in Fe/Si_xFe_{1-x} (x=1, 0.66, 0.5) Mls is related to the appearance of the interfacial Fe silicides. The examined Mls were deposited in UHV by magnetron sputtering onto oxidized Si wafers for

different Fe layer thicknesses 0.3<d_{Fe}<4nm. The crystalline structure of our samples and their multilayer periodicity were examined using the high- and small-angle X-ray diffraction, supplemented with the transmission electron microscopy of the cross-section. We shoved that both magnetic and electronic properties of the AF coupled Fe/Si Mls are influenced by interfacial mixing between Fe and Si layers. The current-voltage (I-V) characteristics measured at room temperature, perpendicularly to the Ml planes allowed us to show the semiconducting character of nominally pure Si, $Si_{0.5}Fe_{0.5}$ and $Si_{0.66}Fe_{0.33}$ spacer layers. The tunnelling barrier height estimated for Fe(3nm)/Si(1.1nm) Mls was found to be 1eV. We showed that the application of interfacial Co (or Au) thin layers prevent the Fe(Si) interdiffusion into Si(Fe) layers leading to the absence of AF coupling.

In order to find out the influence of Ge on AF coupling in Fe/Si Mls, Si was partially substituted by Ge in the spacer layer. Magnetization measurements of the $[Fe(3nm)/Ge(d_{Ge})]_{15}$ Mls revealed no AF coupling up to d_{Ge}=3 nm, in contrast to Fe/Si system [1]. We found that addition of at least 0.5 nm of Ge, introduced either at the Fe/Si interface or in the center of Si spacer destroys the AF coupling.

[1] T. Luciński, M. Kopcewicz, A. Hütten, H. Brückl, S. Heitmann, T. Hempel, G. Reiss, J. Appl. Phys. 93 6501(2003).

15:50 poster D-11

Oscillation of tunneling current in CMR-manganite tunnel junctions

Yuansu Luo, Bernd Damaschke, Konrad Samwer

I. Phys. Institut, Universty Goettingten, Friedrich-Hund Platz 1, Goettingen 37077, Germany

Contact: yluo@gwdg.de

Manganite tunnel structures (1.5nm)/LCMO(25nm)/Al(5nm)/Ir(25nm)/LCMO(45nm)/Al_2O_3 were sputtered onto MgO(100) substrates and patterned in size of 50X50 mm2, where additional metallic layers Ir and Al were used to ensure a homogeneous current distribution over the junction area. Epitaxial growth of relevant layers was confirmed by x-ray structural analysis. Resistive measurements revealed a metal-isolating transition temperature of about 240K for the bottom electrode and about 80K for the upper electrode, respectively. A large low-field magnetoresistance ($\Delta R/R_{max}$) was found to be ~70 % at 10K, arising from spin dependent tunneling of two separated ferromagnetic LCMO layers. Near 80K, the magnetotransport behavior is dominant by colossal magnetoresistance effect ~80% (7 tesla). Surprisingly, at low bias voltage (< 0.2V), the tunneling current becomes oscillatory with increased magnetic field, showing resistive maxima at 0.5, 2.5 und 5.5 tesla, respectively. Fitting current-voltage (I-V) curves up to 1.4V provides an effective barrier height Φ_e~1.2eV and an effective width d_e~1,7nm which corresponds to the barrier layer of Al_2O_3. Fitting I-V curves within 0.25V, however, supplies Φ_e ~0.3V and d_e~4.0nm. This additional barrier with a relatively low height can be attributed to the interfacial LCMO.

Supported by DFG-project SA 337/9-1

15:50 poster D-12

Oxide Heterostructures Based on Fe_3O_4 Thin Films

Andrius Maneikis[1,2], Bonifacas Vengalis[1], Kristina Šliužienė[1], Vaclovas Lisauskas[1], Antanas Oginskis[1], Leonas Dapkus[1]

1. *Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania* **2.** *Vilnius Gediminas Technical University, Sauletekio al. 11, Vilnius LT-2040, Lithuania*

Contact: andrius@pfi.lt

Ferrimagnetic magnetite, Fe_3O_4, is a promising material for spin dependent electronics due to high Curie temperature (of 858 K) and high degree of spin polarized carriers at room temperature. Up to now, high quality Fe_3O_4 films have been grown heteroepitaxially on latticed-matched MgO, $SrTiO_3$ and Al_2O_3. However, for most applications such as tunneling magnetoresistance (TMR) devices there is great interest in various heterostructures composed of lattice-matched ferromagnetic, antiferromagnetic films, highly conducting underlayers and insulating barrier layers.

In this work, Fe_3O_4 thin films were grown on both lattice matched MgO or Al_2O_3 substrates and $La_{2/3}A_{1/3}MnO_3$ (A = Ca, Sr, Ce) underlayers by DC magnetron sputtering of metallic Fe target under a fixed Ar:O_2 (30:1) gas mixture pressure of about 5 Pa. The manganite films were prepared by the same method on both lattice-matched $SrTiO_3$ and $NdGaO_3$ buffered by highly conductive $LaNiO_3$ or RuO_2 layers. Crystalline structure of all the films and heterostructures were characterized by x-ray difraction (XRD) and reflected high-energy electron diffraction (RHEED). The electrical resistance, magnetoresistance and voltage versus current were measured for the films and multilayer structures in a wide range of temperatures and magnetic fields. Three point-probe method was applied to investigate the interface resistance and tunneling magnetoresistance of the heterostructures. Electrical properties of the interfaces were modelled to elucidate the effect of deposition conditions and possible oxygen diffusion at various interfaces.

15:50 poster D-13

Raman spectroscopy and magnetic properties of bulk ZnO:Co single crystal.

Goiran Michel[1], Marius Millot[2], Jesus Gonzalez[2], Z. Golacki[3], Jean Marc Broto[1], Harison Rakoto[1]

1. *Laboratoire National des Champs Magnétiques Pulsés, av. de Rangueil, Toulouse 31423, France* **2.** *Centro de Estudios de Semiconductores, Universidad de los Andes, Merida, Venezuela* **3.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland*

Contact: goiran@lncmp.org

We report on the investigation of structural and magnetic properties of the cobalt based diluted magnetic semiconductor with wurtzite structure: Zn1-xCoxO. Single crystals were grown by chemical vapour transport using chlorine as the transporting agent at a growth temperature of 900C. From the Raman measurements in the temperature range 10-300 K on samples with a Co concentration of 5%, we deduce the presence of small quantities of ferromagnetic hcp-Co and also the presence of anti-ferromagnetic CoO impurities. Theses results are in agreement with the behaviour of high magnetic field magnetisation measurements under pulsed fields. High frequency-high field ESR reveal clearly the large crystal field splitting of the energy levels of isolated Co ions as well as satellites lines which originate from Co clusters made of pairs or larger number of neighbouring magnetic ions.

15:50 poster D-14

Indirect tunneling between ferromagnetic electrodes

M. Misiorny[1], Józef Barnaś[1,2]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: barnas@ifmpan.poznan.pl

Transport of spin polarized electrons between ferromagnetic electrodes is considered theoretically in terms of the second-order perturbation theory. Both, direct and indirect tunneling is taken into account, and the considerations are limited to collinear (parallel and antiparallel) magnetic configurations. In the case of indirect tunneling, a tunneling electron interacts additionally via an exchange coupling with another quantum object in the barrier, like for instance a molecule. Discrete energy spectrum of the obstacle is taken into account. Tunnel conductance and tunnel magnetoresistance are calculated in the linear response regime. The results are discussed from the point of view of possible applications in spintronics devices.

15:50 poster D-15

Size effects in thin antiferromagnetic layers and "ferromagnet - nonmagnetic metal" multilayers

Alexander I. Morosov, Alexey A. Berzin, Alexander S. Sigov

Moscow State Institute of Radioengineering, Electronics, and Automation (Technical University) (MIREA), pr. Vernadskogo, 78, Moscow 119454, Russian Federation

Contact: morosov@mirea.ru

Peculiarities of spin-flop and spin-flip transitions in thin antiferromagnetic layers and “ferromagnet – nonmagnetic metal” multilayers were investigated by numerical modelling methods. In supposition that ferromagnetic layers in multilayer are magnetized homogeneously the behaviour of “ferromagnet – nonmagnetic metal” multilayer in external magnetic field is completely analogous to the behaviour of antiferromagnetic layer with parallel noncompensated surfaces.

The character of spin-flop transition in antiferromagnetic layer of nanometre scale thickness (“ferromagnet – nonmagnetic metal” multilayer with antiferromagnetically coupled ferromagnetic layers) strongly depends upon a number of atomic planes (ferromagnetic layers) parity. We have found that in the case of even number of atomic planes in the antiferromagnetic layer, the spin-flop phase transition of the first order takes place in the surface spin-flop transition field which is $2^{1/2}$ times smaller than the bulk one [1,2]. As a result the state with domain wall in the layer centre arises.

In the case of odd number of atomic planes in the antiferromagnetic layer the second order spin-flop phase transition occurs in the magnetic field higher than the bulk spin-flop one. The spin-flip transition in the layer with arbitrary atomic planes number parity takes place in the magnetic field lower than the bulk spin-flip one. The spin-flop and spin-flip phase transition field dependences upon number of atomic planes (ferromagnetic layers) were found.

This work is partially supported by CRDF – RF Ministry of Education and Science Award VZ-010-0.

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15:50 poster D-16

Fabrication and characterisation of arrays of sub-micrometric magnetic dots

Elena S. Olivetti[1,2], Paolo Allia[2], Edvige Celasco[1,2], Denis Perrone[1,2], Candido F. Pirri[1,2], Paola Tiberto[3]

1. *Materials and Microsystems Laboratory (Chilab), Lungo Piazza d'Armi 6, Chivasso 10034, Italy* **2.** *Dipartimento di Fisica, Politecnico di Torino, corso Duca degli Abruzzi, 24, Torino 10129, Italy* **3.** *Istituto Elettrotecnico Nazionale "Galileo Ferraris" (IEN), Strada delle Cacce, 91, Torino 10135, Italy*

Contact: elena.olivetti@polito.it

Ordered arrays of identical sub-micrometric elements of a magnetic metal are attractive both for fundamental studies and for technological applications. Their study allows one to clarify the effect of dimension shrinking on the overall magnetic response of the array. Moreover, ordered magnetic lattices are promising candidates in data storage technology for high-density magnetic recording media [1]. In this work, the fabrication of ordered arrays of magnetic dots through electron-beam lithography (EBL) will be discussed.

Arrays of dots with square and hexagonal symmetry have been realised in order to study the effect of array geometry and dot size on the magnetic properties. Three lattice spacing values (0.7, 1 and 1.3 μm) were examined. In all these cases, dipolar interaction is negligible and nearly independent reversal of the magnetisation is achieved. The dot diameter was reduced from 500 to 300 nm with decreasing the exposure dose. Patterns of 100x100 μm^2 area were produced on Si (100) wafers covered by a 250 nm layer of PMMA as electron-sensitive resist. The magnetic dots have been obtained after the deposition of a thin film and a lift-off process for pattern transfer [2]. Fe, Co and Ni films with thickness of 50 nm have been grown by e-beam and thermal evaporation under vacuum of the pure metals. The morphological characterisation, performed by Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM), revealed in most cases a good quality of the patterns. In addition, Magnetic Force Microscopy (MFM) has been exploited to investigate the magnetic domain structure of a single array element. Magnetisation curves were obtained at different temperatures using an alternating gradient magnetometer (AGM). The relation between magnetic response and geometrical properties of the array will be discussed.

[1] C. Haginoya et al. J. Appl. Phys 85 (1999), 8327

[2] J.I. Martin et al. J. Appl. Phys. 84 (1998), 411

15:50 poster D-17

Effect of interface roughness on the magnetotransport behaviour of NiO/Co/Cu/Co bottom spin valves

Paolo Allia[1], Alessandro Chiolerio[2,3], Marco Coisson[4], Elena S. Olivetti[2,3], Federica Celegato[1], Luca Martino[4], Candido F. Pirri[2,3], Denis Perrone[2,3], Paola Tiberto[4]

1. *INFM and Dipartimento di Fisica, Politecnico di Torino, Corso Duca degli Abruzzi, 29, Torino 10129, Italy* **2.** *Dipartimento di Fisica, Politecnico di Torino, corso Duca degli Abruzzi, 24, Torino 10129, Italy* **3.** *Materials and Microsystems Laboratory (Chilab), Lungo Piazza d'Armi 6, Chivasso 10034, Italy* **4.** *Istituto Elettrotecnico Nazionale "Galileo Ferraris" (IEN), Strada delle Cacce, 91, Torino 10135, Italy*

Contact: alessandro.chiolerio@polito.it

Antiferromagnetic (AF)/ferromagnetic (FM) exchange-coupled systems are widely used as active elements in devices based on giant magnetoresistance (GMR). The strict dependence of the AF exchange coupling on the spacer layer thickness requires fine control of deposition rate during film growth. Usually, sputtering deposition is exploited to prepare multilayers consisting of alternated ferromagnetic and non-magnetic (NM) metals.

In this work, thermal evaporation under vacuum has been checked as an alternative technique for multilayered films growth. Bottom spin valves of the family NiO/Co/Cu/Co with an AF layer of 100 and 140 nm and a 5/2/6 nm trilayer have been produced and characterised. The AF has been synthesized by thermal oxidation of Ni films and annealed in a magnetic field of 23 Oe.

Magnetotransport measurements were performed by the four points method in the temperature range 5–200 K, under a field up to 70 kOe. The observed GMR amplitude at T=5 K is about 2 ‰ for all the studied samples. In some samples, the GMR – an effect typically arising at FM-NM interfaces - is associated to a contribution from anisotropic magnetoresistance – a bulk effect originating within each FM layer.

Hysteresis loops were measured by Alternating Gradient Magnetometry under a field up to 20 kOe. The multilayer coercivity has been found to increase with the thickness of the AF layer.

AFM pictures show that surface roughness closely reproduces the AF underlayer's one [1], being about one order of magnitude larger than that of the metallic trilayer deposited directly on Si (1-3 nm). This value considerably exceeds the ones typically reported for sputter-deposited NiO [2] and is thought to be the cause of the very low observed GMR amplitude.

The role of interfacial roughness on the ratio between surface-

to-bulk magnetoresistance amplitudes is discussed.

[1] W.F. Egelhoff Jr. et al. Progr. Surf. Sci. 67 (2001) 355-364

[2] D.G. Hwang et al. JMMM 186 (1998) 265-276

15:50 poster D-18

Magnetic susceptibility of Si-Ge whiskers

I. P. Ostrovskii[2], Volodymyr M. Tsmots[1], Yurij Pavlovskii[1], Petro Litovchenko[3]

1. *State Pedagogical University, 24 Franko str, Drohobych 82100, Ukraine* **2.** *Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine* **3.** *Institute for Nuclear Research of NAS of Ukraine (INR), 47 Nauky Prospect, Kyiv 03028, Ukraine*

Contact: iostrov@polynet.lviv.ua

In present paper magnetic susceptibility (MS) of Si-Ge solid solution whiskers with 0,1-50 mkm in diameter was studied. Submicron whiskers are quasi-cylindrical crystals, while whiskers with d > 1 mkm are needle-like ones.

Si-Ge whiskers were grown by chemical transport reaction method in a sealed bromide tube. MS of whiskers was measured by Faradey method in magnetic fields (0,3-4,0) kQe at room temperature.

Size dependence of MS behaviour was found in the whiskers. This peculiarity has different nature for submicon and needle-like crystals. The main contribution in size effect for needle-like crystals makes presence of paramagnetic centers (mainly Pt impurity). Size effect revealed in submicron whiskers is caused by change of lattice MS, i.e., change in crystalline structure of the whiskers.

15:50 poster D-19

Experimental evidence of the spin-dependence of electrons reflections in magnetic multilayers

Bertrand Raquet[1], Etienne Snoeck[2], Christophe Gatel[2], Raphael Serra[2], Jean-Claude Ousset[2], Jean-Baptiste Moussy3, Alexandre Bataille[3], M. Pannetier[3], M. Gautier-Soyer[3], Jean Marc Broto[1]

1. *Laboratoire National des Champs Magnétiques Pulsés, av. de Rangueil, Toulouse 31423, France* **2.** *CEMES-CNRS (CEMES-CNRS), 29, rue Jeanne Marvig, Toulouse F31055, France* **3.** *CEA-Saclay, Bat 522, Gif-sur-Yvette 91191, France*

Contact: bertrand.raquet@lncmp.org

The giant magnetoresistance (GMR) of multilayers in the current in plane (CIP) geometry can be understood by considering the spin-dependent scattering effects within the magnetic layers and at the non magnetic/magnetic interfaces. In this paper we experimentally put on view the magnetoresistance effect created only by the spin-dependent reflection of electrons at the interfaces. Two epitaxial systems were studied, consisting of two ferrimagnetic insulators layers at low temperature ($CoFe_2O_4$ and Fe_3O_4) separated by a non magnetic metallic layer (M= Au or Pt). The transport properties indicate that conduction of the $CoFe_2O_4$/M/Fe_3O_4 trilayers take place within the thin metallic layer. An optimal magnetoresistance up to 5 % at 10 K associated to the switching from parallel to anti-parallel configuration of the magnetization of the two ferrite layers has been obtained. It is associated to the spin-dependent interfacial scattering contribution of the CIP-GMR.

An augmentation of the GMR is observed when decreasing the metallic layer thickness or increasing the oxide layers width suggesting this GMR is essentially an interfacial phenomenon. Moreover, ion implantation experiments were performed on such trilayers: at low doses (N^+ ions at 150 keV for doses $<10^{13}$ ion/cm^2) improves the GMR properties of the Au-based trilayers while for higher doses the magnetoresistance drastically vanishes. In such low dose ranges the damages induced in the MLs is very low (~ 5. 10^{-2} eV/at) and mostly located at interfaces. Our experiments suggest that the effect of low doses ion implantation is a slight smoothing of the interfaces which confirms the interfacial nature of the GMR.

The Pt-based system shows an additional AMR which is not evidenced in the Au-based trilayer. We suggest that this AMR comes from the polarization of the Pt layer by the ferrimagnetic oxides.

15:50 poster D-20

Effect of strain in LSMO epitaxial films with different crystallographic orientation

Umberto Scotti di Uccio[1,2], Bruce Davidson[3], Roberto Di Capua[2], Fabio Miletto Granozio[2], Giampiero Pepe[2,4], Paolo Perna[1], Antonio Ruotolo[2], Marco Salluzzo[2]

1. *Università di Cassino, Dip. di Meccanica, Strutture, Ambiente e Territorio (DiMSAT), Via Bi Biasio 43, Cassino 03043, Italy* **2.** *INFM - Coherentia, Via Cinthia, Napoli 80125, Italy* **3.** *TASC-INFM National Laboratory (TASC), S.S. 14, Km 163.5 in Area Science Park, Basovizza (Trieste) 34012, Italy* **4.** *University of Naples "Federico II", Department of Physical Sciences, Napoli, Italy*

Contact: scotti@unicas.it

The work that we present has been devoted to the study of high quality perovskitic La-Sr-Mn-O (LSMO) thin films. More than two hundreds samples were deposited by RF magnetron sputtering in Ar + O_2 atmosphere. Under suitable conditions, the films have the same cation stoichiometry as the target (that is, $La_{0.7}Sr_{0.3}MnO_3$), as demonstrated by Rutherford backscattering. They are also smooth, as Scanning Tunnel measurements (STM) show.

We investigated various substrates, i.e. $SrTiO_3$ (STO),

$_3$ (LAO), $NdGaO_3$ (NGO), with both (001) and (110) crystallographic orientations. We always observed the cube-on-cube structure that is typical of epitaxial growth of perovskites on perovskites. However, the difference in the lattice matching with substrate leads to films that are subject to compressive stress on LAO, and tensile stress on STO and, to a less extent, on NGO. The strained structure has been investigated resorting to x ray reciprocal space mapping (RSM). Although previous studies already unveiled some details of (001) strained LSMO, the data regarding (110) films are new. The lattice deformation in this case is peculiar, because it is partially due to shear stress. This feature is particularly interesting in the LSMO manganite, because it is well known that the magnetic and the transport properties of this compound strongly depend on strain, due to the complex interplay between lattice, electronic and magnetic degrees of freedom. Here we report on the comparison between the hysteresis loops of LSMO grown on (001) and (110) STO. In both cases, the magnetic properties are strongly related to the stress. The stress also influences the transport properties. The resistivity of films grown on (110) STO is anisotropic, and it is slightly higher than that of films on (001) STO. The possible relations between magnetism, stress and transport properties are object of the discussion of the experimental results.

15:50 poster D-21

Formation of cooper islands on surface of thermoelectric semiconductors Cu_2Se under laser treatment

O. Yu. Semchuk[1], N.N. Krupa[3], M. Willander[2], M. Karlsteen[2], V. N. Semioshko[1]

1. *Institute of Surface Chemistry NAS of Ukraine, 17 General Naumov, Kyiv 03164, Ukraine* **2.** *Chalmers University of Technology, Göteborg 412 96, Sweden* **3.** *Institute of Magnetism, NAS of Ukraine, Kyiv, Ukraine*

Contact: inchem@mail.kar.net

The photoinduced electron drift should also occur in metals; however, because of the high absorption coefficient of metals and the high concentration of conduction electrons in them, its observation in metals is greatly complicated.

In this paper, variations in the reflection of light from then multilayer metal films are theoretically and experimentally studied upon their irradiation by intense nanosecond laser pulses. The irradiation results in the optical rotation of light reflected from nonmagnetic metal films due to photoinduced drift of electrons with polarized spins.

The results, presented in this paper showed that nonmagnetic films in multilayer magnetic films could be magnetizations with polarized spins.

15:50 poster D-22

Magnetoresistance and magnetization processes in the helimagnetic compound $DyMn_6Ge_6$ and in related alloys $Dy(Mn_6Ge_6)_{1-x}(Fe_6Al_6)_x$

Peter Kerschl[1], Nadezda V. Kozlova[1], Zbigniew Śniadecki[2], Konstantin Nenkov[1], Ulrich K. Rößler[1], Bogdan Idzikowski[1,2], Karl-Hartmut Müller[1], Ludwig Schultz[1]

1. *Leibniz-Institute for Solid State and Materials Research, P.O.Box 270116, Dresden D-01171, Germany* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: sniadecki@ifmpan.poznan.pl

Hexagonal $DyMn_6Ge_6$ with $HfFe_6Ge_6$ structure is a prototypical rare-earth transition-metal compound with complex magnetic couplings. Rare-earth and transition metal ions order simultaneously in a ferrimagnetic helix with locally antiparallel moments of the sublattices at a high transition temperature (T_C = 423 K). This layered magnetic system is a suitable experimental model for studies on the relation between magnetization processes and magnetoresistance in complex metallic alloys. Field and temperature-dependent spin-reorientation in this compound cause anomalies of the electrical resistivity and sizeable magnetoresistance effects.

We report results from measurements of the magnetoresistance and magnetization in static and pulsed fields up to 50 T for polycrystalline samples of pure and diluted $Dy(Mn_6Ge_6)_{1-x}(Fe_6Al_6)_x$. The samples were prepared by melt-spinning and subsequent annealing. Annealed pure and low-doped samples $x<0.05$ have the $HfFe_6Ge_6$-structure. At room-temperature, the pure compound shows negative magnetoresistance MR(H) = (R(H) - R(0)) / R(0), that reaches about 7% at 4 T and displays a marked change of slope at the helix-fan-transition near 5 T. Below 100 K, positive magnetoresistance is found with a non-monotonic dependence MR(H) in high fields above 20 T. The anomalous decrease of MR(H) is explained by a metamagnetic behaviour related to a transition from the ferrimagnetic fan into a spin-flop state. For low substitutions of MnGe by FeAl (x = 0.01) an increase of the specific resistivity and reduced magnetic ordering-temperatures are found. Near the helix-magnetic ordering temperature, an anomaly of the resistivity occurs indicating a strong influence of magnetic impurity-scattering on electronic transport. As-quenched and more strongly alloyed samples show different crystalline phases, derived from the hexagonal $TbCu_7$-type structure, but have similar magnetoresistive behaviour.

This work is supported by BMBF project: 03SC5DRE.

15:50 poster D-23

Study on Ferromagnetism of GaMnN

Saki Sonoda[2], Tanaka Isao[1], Hidekazu Ikeno[1], Tomoyuki Yamamoto[3], Fumiyasu Oba[1], Koichi Kindo[4,6], Hidenobu Hori[5], Tsutomu Araki[7], Yasushi Nanishi, Yoshiyuki Yamamoto[5], Ken-ichi Suga[6], Youichi Akasaka[2]

1. *Department of Material Science and Engineering, Kyoto University, Kyoto, Japan* **2.** *Department of System Innovation, Osaka University, Toyonaka, Japan* **3.** *Department of Material Science and Engineering, Waseda University, Tokyo, Japan* **4.** *The Institute for Solid State Physics, University of Tokyo, Kashiwa, Japan* **5.** *School of Material Science, Japan Advanced Institute of Science and Technology, Nomi, Japan* **6.** *KYOKUGEN, Osaka University, Toyonaka, Japan* **7.** *Department of Photonics, Ritsumeikan University, Kusatsu, Japan*

Contact: saki@nano.ee.es.osaka-u.ac.jp

We review crystallographic, spectroscopic, magnetic and electrical properties of Mn-doped GaN films showing ferromagnetic behaviour at room temperature. Up to 8.2% of Mn concentration, the films keep transpearency with yellowish colour even with thickness more than 1μm. No second phase is observed by RHEED and XRD measurements. It was found that majority of Mn atoms replace Ga atoms in wurtzite GaN by XAFS study. At room temperature, optical absorption spectra show clear structures implying Mn 3d levels stay in the mid-gap of GaMnN.

15:50 poster D-24

Interlayer coupling in Ni-Fe/Au/Co/Au multilayers

Feliks Stobiecki[1], Maciej Urbaniak[1], Maria Tekielak[2], Bogdan Szymański[1], Tadeusz E. Luciński[1], Marek Schmidt[1], Andrzej Maziewski[2]

1. *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland* **2.** *University of Białystok, Laboratory of Magnetism, 41 Lipowa street, Białystok, Poland*

Contact: stfeliks@ifmpan.poznan.pl

Previously we have demonstrated that sputter deposited $(Ni_{80}Fe_{20}/Au/Co/Au)_N$ multilayers (N-repetition number) exhibit easy-in-plane and out-of-plane magnetic anisotropy for permalloy and Co layers, respectively. Such structures are interesting because of possible applications as recording media or in magnetoresistive sensors with linear R(H) dependence. The perpendicular anisotropy of cobalt layers and the coupling between Co and Ni-Fe layers play a crucial role in both applications.

Two types of samples were studied: (i) sandwich structures with wedge shaped Au spacer and Co layers, (ii) multilayers with different thickness of Co ($0.2 < t_{Co} < 2$ nm), Au ($0.5 < t_{Au} < 3$ nm) layers and $5 < N < 15$. The sandwich structures were investigated using multiscale magneto-optical methods based on the polar Kerr effect, whereas multilayers with VSM and magnetoresitance measurements (in our samples magnetoresistance effect of the GMR-type is observed). We have found that the Co layers exhibit perpendicular anisotropy in thickness range of $0.3 < t_{Co} < 1.2$ nm. For the Au spacer layer thickness $t_{Au} > 1$ nm the interlayer coupling is weak and magnetization reversal of ferromagnetic layers is nearly independent. However, for multilayers exhibiting dense stripe domains structure (confirmed by MFM measurements) the ferromagnetic coupling strongly increases in the field range corresponding to the existence the domains structure.

15:50 poster D-25

GMR Spin Valve with alternating in-plane and out-of-plane magnetic anisotropy

Bogdan Szymański, Feliks Stobiecki, Maciej Urbaniak

Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland

Contact: szyman@ifmpan.poznan.pl

In this paper a new type of spin valve (SV) structure is proposed. It consists of ferromagnetic layers, with in-plane (F_1) and out-of-plane (F_2) anisotropy, separated by nonmagnetic spacer (S). When the magnetic field H is applied perpendicularly to the surface of the (F_1/S/F_2/S) SV it stabilizes the magnetization direction of the layers with perpendicular anisotropy and forces a rotation of the magnetization direction of the layers with easy plane anisotropy. This situation is reversed for the magnetic field applied parallel to the film surface. The magnetization reversal results in the giant magnetoresistance (GMR) characterized by the linear R(H) dependence in a wide range of magnetic field H.

To realize the idea of the SV we have chosen thin Co layers sandwiched between Au as layers with perpendicular anisotropy (F_2). Our measurements proved that Co layers possess a perpendicular anisotropy for $0.4<t_{Co}<1.5$ nm and in-plane anisotropy for $t_{Co}>1.8$ nm. We have found that for Co thickness $0.6<t_{Co}<0.8$ nm the effective perpendicular anisotropy is relatively strong. It does not change appreciably in the temperature range from 100 to 423K and is resistant to a low temperature annealing, i.e., $T_{anneal}<450$K. As ferromagnetic layers with in-plane anisotropy (F_1) we have applied Ni-Fe ($t_{NiFe}=2$ nm) or Co with thickness $t_{Co}>1.8$ nm. For both types of structures, i.e., $[NiFe_1/Au/Co_2/Au]_N$ and $[Co_1/Au/Co_2/Au]_N$ multilayers (N is number of repetition), deposited by the magnetron sputtering, the GMR effect of up to 8% at RT was observed. Moreover, due to the considerable simplification, the $Co_1/Au/Co_2/Au$ structures are preferred for applications (only two materials are required).

15:50 poster D-26

Thermopower in nano specular spin valves

Andre M. Pereira[1], Joao Ventura[1], Jose Teixeira[1], M. E. Braga[1], R P. Pinto[1], J. B. Sousa[1], Paulo Freitas[2,3], A. Veloso[2]

1. *Departamento de Fisica and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal* **2.** *INESC-MN, Lisbon, Portugal* **3.** *Instituto Superior Técnico, Lisboa 1049-001, Portugal*

Contact: joventur@fc.up.pt

A spin valve (SV) is a magnetic nanostructure constituted by two ferromagnetic (FM) layers separated by a thin non-magnetic (NM) metallic spacer. The magnetization of one of the FM layers is fixed by an underlying antiferromagnetic (AFM) layer, while that of the other FM layer rotates when a small magnetic field is applied. Relatively high magnetoresistance (MR) values can be achieved with such SV design, but new ways to enhance MR are being researched to increase bit density in hard drives. One such way is the fabrication of a nano-oxide layer (NOL) just above and below the free and pinned layers, respectively [1]. Such NOL spin valves can more than double the MR ratio of simpler stacks, because of specular reflection of electrons at the FM/NOL interfaces. However, the microscopic physics of electron specular reflection is still incomplete. We recently showed that the NOL is formed by magnetic oxides with a PM-AFM transition below room temperature that can strongly affect transport properties, particularly MR [2]. To further study the effects of the NOL on the magneto-transport of SVs we measured thermopower (S) and electrical resistance (R) of Ion Beam Deposited MnIr/CoFe/NOL/CoFe/Cu/CoFe/NOL specular SVs in the 320-20 K temperature (T) range. We show that at room temperature the oxides constituting the NOL are in the paramagnetic phase and that at T_N~150K a magnetic transition to the AFM phase occurs. A linear S(T)-dependence extrapolating to the origin is observed above T_N, as characteristic of well behaved metals in the paramagnetic phase. The magnetic transition at T_N produces a pronounced change in the S(T) behaviour, leading to a quadratic temperature dependence at low temperatures, here related to the excitation of spin waves and their effect on the Seebeek coefficient. These features will be correlated with the observed peculiar MR(T) behaviour [2]. [1] A.Veloso et al. Appl. Phys. Lett. 77,1020(2000).[2] J.Ventura et al. J. Appl. Phys. 93,7690(2003).

15:50 poster D-27

Transport properties of a single level quantum dot in external magnetic field

Malgorzata Wawrzyniak[1], Martin Gmitra[1], Józef Barnaś[1,2]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: lauraphy@spin.amu.edu.pl

Theoretical analysis of resonant electronic transport through a non-interacting single level quantum dot attached to ferromagnetic leads has been carried out. An external magnetic field is applied to the system, which leads to spin splitting of the dot level. Magnetic moments of the leads and the external magnetic field are assumed to be noncollinear.

However, all of them are in a common plane. Transport characteristics are calculated using the nonequlibrium Green function technique, and the Green functions have been calculated via the equation of motion method. Dependence of the transport characteristics on the system parameters are discussed from the fundamental and application points of view.

15:50 poster D-28

Electron Transport Through Nanoscopic Spin Valves

R. Świrkowicz[1], M. Wilczyński[1], Józef Barnaś[2,3]

1. *Warsaw University of Technology, Faculty of Physics, Koszykowa 75, Warszawa 00-662, Poland* **2.** *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **3.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

A nanoscopic junction consisting of a quantum dot attached to ferromagnetic electrodes can act as a spin valve. Tunnelling current flowing through the system strongly depends on the relative orientation of magnetic moments in the leads. Usually the conductance is maximal for collinear alignment of the magnetic moments and is partly suppressed in canted configurations.

Spin-polarized transport through a quantum dot strongly coupled to ferromagnetic electrodes with non-collinear magnetic moments is analysed theoretically in the frame of the non-equilibrium Green's function formalism. Due to strong coupling of the dot to ferromagnetic electrodes the effective exchange field B_{ex} created by the electrodes leads to spin splitting of the dot level [1,2]. Magnitude of the field depends on the coupling strengths and on the intra-dot correlation parameter U. We have studied the interplay of spin-dependent transport and exchange field in a strongly correlated quantum dot spin valve. Both, linear and non-linear-response regimes

have been analysed. We have found that the effective field significantly influences the linear conductance for large values of U, which is a non-monotonic function of the angle between magnetic moments in the leads [2]. A monotonic dependence is obtained for the system with weak correlations, in which the spin-splitting is reduced. I-V characteristics and TMR are strongly influenced by the presence of exchange field. The low-bias Coulomb blockade can be partly reduced in parallel or nearly parallel configuration of magnetic moments by the spin splitting of the dot level. As a result a significant increase in the low bias TMR can be observed. On the other hand, for gate voltages for which the dot level is very close to the Fermi level in the leads, the spin-splitting induced by exchange field lowers the current leading to reduction of low-bias TMR.

[1] M. Braun et al., Phys. Rev. B 70, 195345 (2004).

[2] J. Fransson, cond-mat/0502288v1 (2005)

15:50 poster D-29

Torque due to spin-polarized current in ferromagnetic single-electron transistors with non-collinear magnetizations

Justyna Wiśniewska[1], Małgorzata Kowalik[1], Józef Barnaś[1,2]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: justyw@amu.edu.pl

Transport of spin-polarized electrons in a ferromagnetic single-electron transistor (FM SET) is analyzed theoretically in the sequential tunneling regime [1]. Two external electrodes and the central part (island) of the device under consideration are ferromagnetic, with the corresponding magnetizations being generally non-collinear. The regimes of fast and slow spin relaxations on the island are considered. Transport characteristics like conductance, tunnel magnetoresistance (TMR), and differential conductance have been calculated for different values of the bias and gate voltages. It is shown that the transport characteristics vary significantly with the angle between magnetizations [2]. We have also calculated torque due to spin transfer from conduction electrons to localized magnetic moments. The torque is calculated from the spin current absorbed by the island [3].

[1] Single Charge Tunneling, Vol. 294 of NATO Advanced Study Institute, Series B, edited by H. Grabert and M. H. Devoret (Plenum Press, New York, 1992).

[2] J. Wiśniewska, I. Weymann and J. Barnaś, Materials Science (Poland) 22, 461 (2004), and references therein.

[3] J. Barnaś, A. Fert, M. Gmitra, I. Weymann and V. Dugaev, cond-mat/0501570

15:50 poster D-30

Giant planar Hall effect in ferromagnetic (Ga,Mn)As layers

Tadeusz Wosiński[1], Andrzej Mąkosa[1], Janusz Sadowski[1,2], Oleh Pelya[1], Tadeusz Figielski[1], F. Terki[3], C. Hermandez[3], S. Charar[3]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Institut für Experimentelle und Angewandte Physik Universität Regensburg (Uni. Regensb), Universitätsstraße 31, Regensburg 93040, Germany* **3.** *Groupe d'Etude des Semiconducteurs CC074, Universite Montpellier II, Montpellier, France*

Contact: wosin@ifpan.edu.pl

Ferromagnetic semiconductors based on III-V compounds, combining semiconductor properties with magnetism, offer also new possibilities for investigation of interesting physical phenomena including the anomalous Hall effect, the so-called planar Hall effect as well as the anisotropic magnetoresistance.

In this paper we report on our recent investigation of the planar Hall effect in 20 nm thick layers of ferromagnetic semiconductor $Ga_{0.94}Mn_{0.06}As$ grown by means of low-temperature molecular beam epitaxy on semi-insulating (001)-oriented GaAs substrate. The structures have been subjected to post-growth low-temperature annealing performed under As capping in order to achieve high hole concentration exceeding 10^{21} cm^{-3} and high Curie temperature of 135 K. Magnetotransport measurements on lithographically patterned Hall bars of 150 μm width have been carried out at liquid helium temperatures for various orientations of the magnetic field swept up to 1 kOe. The investigated layers exhibit a giant magnitude of the planar Hall effect, which is several orders of magnitude greater than previously found in metallic ferromagnets. It results from the combined effects of strong spin-orbit interaction in the valence band of the zinc blende crystal structure and the large spin polarization of holes in (Ga,Mn)As [1]. When sweeping the magnetic field the effect varies non-monotonously alternating its sign. A characteristic feature of our results has been the appearance of a single or double hysteresis loops in the planar Hall effect data, depending on the magnetic field orientation and the sweeping range of the magnetic field. The results are discussed taking into account the magnetic anisotropy of the (Ga,Mn)As epitaxial layers grown under biaxial compressive strain.

[1] H.X. Tang, R.K. Kawakami, D.D. Awschalom and M.L. Roukes, Phys. Rev. Lett. 90, 107201 (2003)

[ABSTRACT TRUNCATED TO 2000 LETTERS]

15:50 poster D-31

Magneto-conductance through nanoconstriction in ferromagnetic (Ga,Mn)As film

Oleh Pelya[1], Tadeusz Wosiński[1], Tadeusz Figielski[1], Andrzej Mąkosa[1], Andrzej Morawski[1], Janusz Sadowski[1], Witold Dobrowolski[1], Rita Szymczak[1], Jerzy Wróbel[1], Attila L. Tóth[2]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland*
2. *Hungarian Academy of Sciences, Research Institute for Technical Physics and Materials Science, P.O.Box 49, Budapest H-1525, Hungary*

Contact: wosin@ifpan.edu.pl

Semiconductor-based magnetic materials offer new possibilities for the integrating electronic and magnetoelectronic devices providing a basis for future spin electronics.

In the present study we fabricated and investigated a simple magnetoresistive nanodevice formed by a narrow constriction in the epitaxial film of a ferromagnetic semiconductor (Ga,Mn)As. We performed experiments on 50 nm thick film of $Ga_{0.99}Mn_{0.01}As$ grown by the low-temperature molecular beam epitaxy on semi-insulating GaAs substrate. Magnetic properties of the film, which exhibits metallic-type hole conductivity, were measured by means of SQUID magnetometer showing the Curie temperature of 50 K. We fabricated constrictions of submicron width in the film by a method of the electron-beam-lithography patterning and oxygen ion implantation [1]. Individual devices containing the constriction with lithographic width of 400 nm and supplied with two Ohmic contacts were subjected to magnetotransport measurements at temperatures down to 1.5 K and the magnetic field up to 13 T. At the lowest temperatures both the constricted devices and the non-constricted reference samples exhibit a large positive magneto-conductance, which can be described by the suppression of weak localization of holes by the external magnetic field. Characteristic features revealed in the magneto-conductance of the constricted devices are abrupt jumps of an enhanced conductance that appear when the sweeping magnetic field crossed the regions of the coercive field of the film. We interpret these features as resulting from the erasing of weak localization by a magnetic domain wall nucleated in the constriction.

[1] T. Figielski, T. Wosiński, A. Morawski, O. Pelya, J. Sadowski, A.L. Tóth, and J. Jagielski, Phys. Stat. Sol. (a) 195, 228 (2003).

15:50 poster D-32

Room-Temperature Ferromagnetism in Co-doped ZnO Bulk Induced by Hydrogenation

Wang Yi, Sun Lei, Kang Jin-feng, Zhang Xing, Han Ru-qi

Peking University, Peking University P.R. Chian, beijing 100871, China

Contact: wangyi@ime.pku.edu.cn

The room-temperature ferromagnetism properties of Co-doped zinc oxide have been reported in this paper. The $Zn_{1-x}Co_xO$ ($0.02<x<0.1$) specimens were synthesized by the solid state reaction method with the powders of Co_2O_3 and ZnO. The precursor Co_2O_3 and ZnO powders were mixed and milled for 6 hours. The mixed powder was calcined at temperatures ranging from 600□ to 1000□ for 12 hours, and was cooled at ambient temperature. The powder was then milled, made block and sintered for 6 hours. The resulting $Zn_{1-x}Co_xO$ specimens were paramagnetic at room temperature. The specimens were then hydrogenated at 600□ for 3 hours and 800□ for 3 hours. The room-temperature ferromagnetism (RTFM) was found in the hydrogenated samples, and the hysteresis loops of the hydrogenated Co-doped ZnO samples were measured at 100 K and 300 K by using the superconducting quantum interference device (SQUID) magnetometer (Quantum Design). The coercive field was about 300 Oe for the room-temperature measurement, and the saturation magnetization of the hydrogenated sample was about 0.045 μ_B/Co. The samples were further measured by using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and high-resolution transmission electron microscopy (HRTEM). The results suggested the observed RTFM in the Co-doped ZnO was partly due to the existence of the cobalt nanoparticles in the hydrogenated samples.

15:50 poster D-33

Structural and Magnetic Properties of Cr/Gd Multilayers Deposited on Sapphire and MgO Substrates

Alexei Petroutchik, Lech T. Baczewski, Roman Minikayev, Wojciech Paszkowicz, Justyna Kaźmierczak, Anna Ślawska-Waniewska

Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland

Contact: petruc@ifpan.edu.pl

The Cr/Gd magnetic multilayers of the following configuration were deposited on 20 nm thick Mo buffer using Molecular Beam Epitaxy UHV technique: substrate/Mo/Cr_x/Gd_{30} where x=10, 20 and 30 A. Two different types of substrates were used - MgO and Al_2O_3. The growth mechanism and formation of Cr/Gd interface were investigated in situ by

RHEED. Bilayer (Cr+Gd) thickness and interface roughness were determined from X-Ray reflectometry curves by fitting procedure using Simulreflec software based on Parrat algorithm. The temperature and field magnetization dependencies M(H) and M(T) were measured by SQUID and high sensitivity VSM magnetometer at the temperature range from 5K to 250K.

It was shown that the roughness of the Cr/Gd interface strongly depends on the type of the substrate -the samples grown on the Al_2O_3 substrate were smoother then those grown on MgO. A strong influence of substrate temperature on Cr/Gd interface formation was observed - samples grown at room temperature were much smoother then samples grown at 250 C. The strong in-plane anisotropy has been observed from the hysteresis loops analysis. Furthermore, the magnitude of coercivity field was found to increase with increasing of Cr layer thickness. Curie temperatute dependence on Cr layer thickness was also investigated.

15:50 poster D-34

Spin dependent tunneling through a quantum dot attached to ferromagnetic electrodes with non-collinear magnetizations

Józef Barnaś[1,2], Malgorzata Wawrzyniak[1], Martin Gmitra[1]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: barnas@amu.edu.pl

Resonant tunneling through an interacting single-level quantum dot in an external magnetic field and coupled to ferromagnetic electrodes with non-collinear magnetizations has been analyzed theoretically. The non-equilibrium Green function technique based on the equation of motion method has been applied in order to calculate the angular dependence of the tunnel magnetoresistance and the average spin components on the dot. The Green functions have been calculated in the Hartree-Fock approximation. The calculations are restricted to the weak coupling regime.

15:50 poster D-35

Spin-valve effect in double-barrier systems with noncollinearly polarized magnetic barriers: linear response regime

Józef Barnaś[1,2], M. Mucha-Kruczyński[1]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: barnas@amu.edu.pl

Electron tunneling through a one-dimensional double-barrier structure with ferromagnetic barriers and nonmagnetic external and central electrodes has been considered theoretically in the linear response regime. Magnetic moments of the barriers are assumed to be generally non-collinear, but oriented in a common plane. Transmission coefficients, tunnel conductance and tunnel magnetoresistance are calculated numerically as a function of the angle between barrier magnetizations.

Tuesday, 6 September

Applications and devices

Tuesday morning, 6 September, 9:00
Main Building, room 219
Bogdan Idzikowski presides

9:00 invited oral

Magnetoelectronic devices: heads, memories and sensors

Paulo Freitas, Ricardo Ferreira

Instituto de Engenharia de Sistemas e Computadores Microsistemas & Nanotecnologia, Rua Alves Redol, nº 9, Lisbon 1000-029, Portugal

Contact: rferreira@inesc-mn.pt

Recent advances in magnetoelectronic devices are discussed including read heads, non volatile memories and sensors. For read heads, low RA AlOx and MgO barriers are compared, and junction structure discussed. High frequency (GHz) noise characteristics are discussed. Alternative CPP spin valve stacks are reviewed, in particular structures with current confined paths. For memory applications, thermally assisted MRAMS and spin transfer driven MRAM are discussed. Particular MTJ stacks used in both applications are reviewed. Finally, for sensor applications, it is shown that with new MgO barriers, highly sensitive field detectors can be fabricated with noise levels reaching 10-11 T/sqr(Hz).

9:30 invited oral

Spin-electronic devices with half-metallic Heusler alloys

Andreas Hütten

University of Bielefeld, Department of Physics, Bielefeld D-33615, Germany

As a consequence of the growing theoretically predictions of 100% spin polarized half- and full-Heusler compounds over the past 6 years, Heusler alloys are among the most promising materials class for future magnetoelectronic and spintronic applications. We have integrated Co_2MnSi as a representative of the full-Heusler compound family as one magnetic electrode into technological relevant magnetic tunnel junctions.

The resulting tunnel magnetoresistance at 20 K was determined to be 95% corresponding to a Co_2MnSi spin polarization of 66% in combination with an AlO_x barrier thickness of 1.8 nm. For magnetic tunnel junctions prepared with an initially larger Al layer prior to oxidation the tunnel magnetoresistance at 20 K increases to about 108% associated with a Co_2MnSi spin polarization of 72% clearly proofing that Co_2MnSi is already superior to 3d-based magnetic elements or their alloys. The corresponding room temperature values of the tunnel magnetoresistance are 33% and 41%, respectively. Structural and magnetic properties of the Co_2MnSi / AlO_x - barrier interface have been studied with X-ray diffraction, electron and X-ray absorption spectroscopy and X-ray magnetic circular dichroism and it is shown that the ferromagnetic order of Mn and Co spins at this interface is only induced in optimally annealed Co_2MnSi layer. The underlying atomic ordering mechanism responsible for achieving about its theoretical magnetic moment could be assigned to the elimination of Co-Si antisite defects whereas the reduction of Co-Mn antisite defects results in large tunnel magnetoresistance. The presence of a step like tunnel barrier which is already created during plasma oxidation while preparing the AlO_x tunnel barrier has been identified as the current limitation to achieve larger tunnel magnetoresistance and hence larger spin polarization and is a direct consequence of the oxygen affinity of the Co_2MnSi - Heusler elements Mn and Si.

10:00 oral

Towards room temperature polymeric spin valve

Sayani Majumdar[1,2], H S. Majumdar[1], R Laiho[2], R Osterbacka[1]

1. *Abo Akademi University, Department of Physics, Porthansgatan 3, Turku 20500, Finland* **2.** *Wihuri Physical Laboratory, University of Turku (UTU), Vesilinnantie 5, Turku 20014, Finland*

Contact: sayani.majumdar@utu.fi

The ability to manipulate electronic spin in organic molecular materials offers a new and promising way towards spin electronics, both from fundamental and technological points of view. This is mainly due to the unquestionable advantage of weak spin-orbit and hyperfine coupling in organic molecules, leading to the possibility of preserving spin-coherence over times and distances much longer than in conventional metals or semiconductors. The search of new materials suitable for injecting and transferring carriers with a preferential spin orientation is very important for the development of spintronics. We have prepared spin-valve devices with half-metallic La0.67Sr0.33MnO3 (LSMO) and Co as the two ferromagnetic electrodes and an organic polymer poly(3-hexylthiophene) (P3HT) as the spacer material. The devices exhibit significant changes in resistance as the angle between the magnetic moments in the two electrodes is varied, demonstrating that electrons can traverse the molecular barrier while remaining spin polarized, even at higher temperatures. We have also observed a strong bias and temperature dependence of the magnetoresistance suggesting that spin polarized transport is hindered by localized states in the molecular barrier.

Layered structures

Tuesday afternoon, 6 September, 15:50
Main Building, room 219
Albert Fert presides

15:50 invited oral

Magnetic structures and magnetization processes in layered systems with antiferromagnetic couplings

Ulrich K. Rößler[1], A. N. Bogdanov[1,2]

1. *Institut fuer Metallische Werkstoffe, IFW, Dresden, Germany* **2.** *Donetsk Institute for Physics and Technology, Donetsk 83114, Ukraine*

Contact: u.roessler@ifw-dresden.de

Antiferromagnetic superlattices, layered systems with antiferromagnetic interlayer couplings between different ferromagnetic layers, or ferromagnetic layers pinned to crystalline or synthetic antiferromagnets are typical structures in magnetoelectronic devices.

They can be considered as realizations of artificial antiferromagnetic or ferrimagnetic multi-sublattice structures. Owing to the close competition between intrinsic and induced magnetic anisotropies with interlayer exchange and external fields, these structures display a wide variety of magnetic states. From a basic micromagnetic point of view, we review our understanding of the related magnetization processes.

In particular, we show that most of these systems are characterized by sequences of first-order magnetic transitions in external fields. The importance of the concomitant domain-processes is explained.

We discuss the appearance of spin-flop phases and canted phases in antiferromagnetically coupled bilayers and multilayers, and in multilayer systems

pinned through exchange-bias mechanisms. The role of chiral symmetry breaking and biaxial anisotropy in layered systems is discussed within a general phenomenological theory for surface-induced magnetic interactions.

16:20 oral

Magnetic stripe domains in Fe/Fe-N multilayers

Wojciech Szuszkiewicz[1], Krzysztof Fronc[1], Bernard Hennion[2], Frederic Ott[2], Marta Aleszkiewicz[1], Marina Chernyshova[1], Leszek Kowalczyk[1]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Laboratoire Leon Brillouin, CEA-CNRS, CE Saclay (LLB), Gif-sur-Yvette 91191, France*

Contact: szusz@ifpan.edu.pl

The Fe-N system presents a variety of phases, with the crystal structure and the magnetic properties evolving with nitrogen concentrations. In particular, some of these phases do not show ferromagnetism at room temperature (e.g., the Curie temperature is between 4 and 60 K for Fe2N phase). Thus, one can expect an interesting temperature dependence of Fe/Fe-N system with a limited N content.

Fe/Fe-N multilayers have been grown at the Institute of Physics of the Polish Academy of Sciences in Warsaw. Several Fe(4 nm)/Fe-N(0.8-1.5 nm) samples were deposited on (001)-oriented GaAs substrates at room temperature by a sputtering technique. Neutron diffraction measurements and spin-polarized neutron reflectometry demonstrated a presence of magnetic stripe domains in investigated multialyers, with a period varying with the Fe-N layer thickness, typically between 180 and 240 nm. The magnetic stripe domain structure on the surface of these samples was also directly demonstrated by the magnetic force microscope (MFM).

The goal of the present paper was to analyze the domain structure above mentioned as a function of an external magnetic field and the sample orientation. The neutron scattering data were completed by the results of Kerr magnetometry, performed at room temperature.

16:35 oral

Impedance, magnetoimpedance and magnetization of CoFeZr nanoclusters embedded into alumina matrix

Alexander K. Fedotov[1], Saad Anis[2], I. A. Svito[1], Julia A. Fedotova[1], Bogdan V. Andrievsky[3], Yury E. Kalinin[4], Alexy A. Patryn[3], Vera V. Fedotova[5], Victoria Malyutina-Bronskaya[1], Alexander V. Mazanik[1], Alexander V. Sitnikov[4], Maria I. Tarasik[1]

1. *Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus* **2.** *2Al-Balqa Applied University, Salt, Jordan* **3.** *Technical University of Koszalin, Raclawicka 15-17, Koszalin, Poland* **4.** *Voronezh State Technical University, Voronezh, Russian Federation* **5.** *The Institute of Solid State & Semiconductor Physics, Belarus NAS (ISSSP), P. Brovka, Minsk 220072, Belarus*

Contact: fedotov@bsu.by

The 3 to 5 mcm thick films with the fraction x of CoFeZr alloy between 30 and 65 at.% in alumina matrix were sputtered on single substrate from the compound target in the chamber evacuated either with pure Ar or Ar-O gas mixture.

It was also revealed by Mössbauer spectroscopy and magnetization measurements that phase composition and magnetic state of metallic component strongly depends on gas mixture in the sputtering chamber: metallic nanoclusters in the samples sputtered in pure Ar displayed magnetic properties whereas at sputtering in Ar-O gas mixture they were superparamagnetic. We connect this difference with the influence of oxide precipitates formed on metallic nanoparticles due to the presence of oxygen in gas mixture in the sputtering chamber.

Real part of impedance versus temperature R(T) exhibited exponential laws characteristic for hopping mechanisms. It was also exhibited negative sign of R(B) with squared-like magnetic field dependence that can be ascribed to tunneling of carriers by localized states in alumina between metallic nanoparicles. For the samples with the metallic alloy concentration 40 at.% < x < 55 at.% R does not depend significantly on frequency f in the temperature range where it shows activational temperature dependences (as for DC resistance). For the samples with x < 40 at.% the R(f) ~ f in -s extent with s ~ 2 for temperatures less than 120 K and activational temperature dependences only for f < 400 kHz. For the samples over the percolative threshold (x > 55 at.%) the R(f) ~ f in -s extent dependence is disturbed although exponential character of impedance is remained.

16:50 oral

Quantitative magneto-optical analysis of out-of-plane magnetic structures in twinned $YBa_2Cu_3O_{7-x}/La_{1-x}Sr_xMnO_3$ bilayers

Laura Gozzelino[1], Danilo Botta[1], Roberto Gerbaldo[1], Gianluca Ghigo[1], Francesco Laviano[1], Bruno Minetti[1], Piotr Przyslupski[2], A Tsarev[2], Andrzej Wisniewski[2], Enrica Mezzetti[1]

1. *Dipartimento di Fisica, Politecnico di Torino, Italy, corso Duca degli Abruzzi, 24, Torino 10129, Italy* **2.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland*

Contact: laura.gozzelino@polito.it

Magneto-optical characterisations of $YBa_2Cu_3O_{7-x}$ films grown on top of $La_{1-x}Sr_xMnO_3$ layers are presented. This analysis is focused on the study of the magnetic interaction exerted on vortices in $YBa_2Cu_3O_{7-x}$ films by the manganite magnetic structures. The twin boundaries in the $LaAlO_3$ substrate induce splitting of the manganite magnetic domains with in-plane magnetization that are separated by out-of-plane magnetic structures, pinned at twin boundaries [1]. The vortices nucleated by the external magnetic field interact with the underlying out-of-plane magnetic moments depending on their local structure and magnetic polarization. By the inversion of the Biot-Savart law, the effects of the underlying magnetic structure on local current densities in $YBa_2Cu_3O_{7-x}$ films are evaluated. Different blocking mechanisms (sink or reservoir) are found for current parallel to twin-boundaries, whereas for current perpendicular to the out-of-plane magnetic structures either blocking or channelling effect is achieved. These channelling/blocking phenomena are fundamental for magnetic-quanta control in hybrid superconducting/ferromagnetic devices for spintronics.

Work partially supported by the Programme of Scientific and Technological Co-operation between the Italian Republic and the Republic of Poland 2004-2006. The Italian members of the working group also acknowledge the contribution of the MIUR-PRIN project n. 2004037901.

[1] F. Laviano, L. Gozzelino, E. Mezzetti, P. Przyslupski, A. Tsarev, and A. Wisniewski Appl. Phys. Lett. 86, 152501 (2005).

Wednesday, 7 September

Spin transport

Wednesday afternoon, 7 September, 14:00
Main Building, room 219
Wojciech Suski presides

14:00 invited oral

Spin transport in semiconductors between magnetic source and drain

Albert Fert[1], M. Elsen[1], J-M. George[1], H. Jaffrès[1], R. Mattana[1], F. Nguyen Van Dau[1], B. Lépine[2], A. Guivarch[2], G. Jezequel[2]

1. *Unité Mixte de Physique CNRS/Thales associée à l'Université Paris-Sud, Domaine de Corbeville, Orsay cedex 91404, France* **2.** *Unité PALMS, Université Rennes I, Rennes F34042, France*

Contact: albert.fert@free.fr

The structure including a semiconductor channel between spin-polarized metallic source and drain is at the basis of several concepts of spin transistor. The operation of this type of device requires a spin polarized current be injected into the semiconductor and the resulting spin accumulation be transformed into a significant electrical output signal (say that $\Delta I/I$ must be of the order of unity if ΔI is the current change between two magnetic configurations of the device). For spin injection, a large enough and spin dependent interface resistance (tunnel junction for example) must be introduced between the source (and the drain) and the semiconductor. On the other hand, as we will show, a significant electrical signal (say $\Delta I/I =\sim 1$) can be obtained only if the interface resistances are not too high, which gives a well defined window for the choice of the interface resistance. Having a distance between source and drain shorter than the spin diffusion length is necessary but not at al sufficient.

An illustration of the above conditions is given by our experiments of spin injection into GaAs, particularly by what we find for the variation of the output signal as a function of the tunnel resistances at the source/GaAs and GaAs/drain interfaces.

14:30 invited oral

From Giant magnetoresistance to current-induced magnetic switching and excitations in magnetic structures

Józef Barnaś[1,3], Vitalii Dugaev[2], Albert Fert[4], Martin Gmitra[1], Herbert W. Kunert[5], Malgorzata Wawrzyniak[1], I. Weymann[1]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Instituto Superior Técnico, Lisboa 1049-001, Portugal* **3.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland* **4.** *Unité Mixte de Physique CNRS-Thales, Domaine de Corbeville, Orsay 91404, France* **5.** *University of Pretoria, Department of Physics, University str., Pretoria 0084, South Africa*

Contact: barnas@amu.edu.pl

Giant magnetoresistance (GMR) effect in magnetic multilayers is a consequence of spin dependent scattering and spin dependent electronic structure. The effect describes the way in which magnetic structure of a system modifies its transport characteristics. An effect associated with GMR is the current-induced magnetic switching between different magnetic configurations (eg between parallel and antiparallel ones). This effect, in turn, describes the influence of electric current on magnetic configuration of the system. The magnetic switching may take place when electric current exceeds a certain critical value, and is due to absorption by the magnetic layer of a spin current component normal to the magnetization, which gives rise to spin-transfer torque.

The macroscopic description of current-perpendicular-to-plane (CPP) GMR has been recently extended to describe also the current induced magnetic switching (CIMS). Such a description is based on the classical spin diffusion equations for the distribution functions and on the relevant boundary conditions for the longitudinal and transverse components of the spin current in the situation of quasi-interfacial absorption of the transverse components in a magnetic layer. The torque is then expressed in terms of the usual parameters derived from the CPP-GMR experiments and additionally by the spin-mixing interfacial conductance. The spin transfer torque gives rise to magnetic switching phenomena and also may lead to transition to stationary precessional modes, which can occur when system parameters obey certain conditions.

15:00 oral

Electromigration-driven resistance switching in magnetic and non-magnetic tunnel junctions

Joao Ventura[1], Jose Teixeira[1], Andre M. Pereira[1], Joao P. Araujo[1], J. B. Sousa[1], Y Liu[2], Z Zhang[2], Paulo Freitas[2,3]

1. *Departamento de Fisica and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal* **2.** *INESC-MN, Lisbon, Portugal* **3.** *Instituto Superior Técnico, Lisboa 1049-001, Portugal*

Contact: joventur@fc.up.pt

Tunnel junctions (TJ) consisting of two ferromagnetic (pinned and free) layers separated by an insulator are strong candidates for MRAMs. Due to spin dependent tunneling one is able to obtain two resistance (R) states corresponding to parallel or antiparallel FM-layer magnetizations. Recently, reversible R-changes induced by an electrical current were found in thin TJs [1] and attributed to electromigration (EM) in nanoconstrictions in the insulating barrier [2]; we thus obtain Current Induced Switching (CIS). Here we report a study on the transport properties (R, MR and CIS) of thin magnetic MnIr/CoFe/AlO$_x$/CoFe and non-magnetic MnIr/CoFe/Ta/AlO$_x$/Ta/CoFe tunnel junctions. CIS at room temperature amounts to a 60% resistance change, and this effect is discussed in terms of nanostructural rearrangements of metallic ions at the FM/insulator and NM/insulator interfaces. Interestingly, the current direction for which R-switching occurs in non-magnetic TJs is opposite to that of magnetic ones. This will be related to the dominance of different EM contributions (direct/wind forces) in CoFe and Ta layers. After switching (in magnetic TJs) some ions return to their initial sites when the electrical current is reduced. This relaxation is thermally assisted and two energy barriers emerge in the R-evolution over time. The physical origin of these effects is discussed, including ion EM between different types of sites near the metal/insulator interfaces. In contrast, no relaxation is observed in non-magnetic TJs, indicating that migrated Ta ions are buried inside stable local energy minima. In both types of tunnel junctions the CIS magnitude decreases with decreasing temperature, as expected for a temperature assisted process. A three resistance state magnetic TJ device is demonstrated, using a suitable combination of MR (magnetic) and CIS (structural) switching.

[1] Y. Liu et al., Appl. Phys. Lett. 82, 2871 (2003). [2] A. Deac et al., J. Appl. Phys. 95, 6792 (2004).

Wednesday Poster Session
Wednesday afternoon, 7 September, 15:50

Thursday, 8 September

Materials for magnetoelectronics
Thursday morning, 8 September, 11:00
Main Building, room 219
Michel Goiran presides

11:00 invited oral

Properties of Co_2YZ Heusler compounds

Claudia Felser, Gehard H. Fecher, Sabine Wurmehl, Benjamin Balke

Johannes Gutenberg-Universität Mainz, Staudingerweg 9, Mainz 55128, Germany

Contact: felser@mail.uni-mainz.de

Half-metallic ferromagnets like the full Heusler compounds with formula Co_2YZ are supposed to show an integer value for the total magnetic moment. SCF calculations reveal, however, in certain cases of X = Co based compounds, non-integer values in contrast to experiments. Co_2YSi with Y = Mn, Fe are of particular interest because of there high T_C.

Co_2FeSi crystallizes in a ordered L_21 structure as proofed by X-ray diffraction. Mößbauer spectroscopy exhibits a single site for the Fe atom indicating a high local order in the samples.

Magnetic hysterisis was measured by SQUID up to 775K revealing a magnetic moment of $6\mu_B$. The magnetic moment is in agreement with the Slater-Pauling rule.The Curie temperat-

ure was estimated from a VEK versus temperature plot and measured with DTA to be higher than 1000K. XMCD spectra were taken to determine the partial magnetic moments of Co and Fe. SCF calculations revealed the measured magnetic moment and a halfmetallic ferromagnetic character if using an appropriate lattice parameter.

11:30 oral

Room Temperature Ferromagnetic Properties of Transition Metal Implanted $Al_{0.35}Ga_{0.65}N$

Jeremy A. Raley[1], Yung Kee Yeo[1], Robert L. Hengehold[1], Mee-Yi Ryu[1,2], Todd D. Steiner[3]

1. *Air Force Institute of Technology (AFIT), 2950 Hobson Way, Wright-Patterson Air Force Base, OH 45433, United States* **2.** *Kangwon National University, Chuncheon, Korea, South* **3.** *American Systems Corporation, Dumfries, VA 22026, United States*

Contact: jraley@afit.edu

A 1 μm thick film of MBE-grown $Al_{0.35}Ga_{0.65}N$ was implanted with Cr, Mn, and Ni at room temperature with an energy of 200 keV to a dose of 5 x 10^{16} cm^{-2} for Cr and Mn and a dose of 3 x 10^{16} cm^{-2} for Ni. The magnetic properties of these samples were measured using superconducting quantum interference device (SQUID) magnetometry and the implantation damage recovery was evaluated using cathodoluminescence (CL). Each of these samples shows evidence of ferromagnetism when annealed under the proper conditions, and can be a viable dilute magnetic semiconductor whose ferromagnetism persists to room temperature. Thus, these samples can be good candidates for spintronic device applications.

The Cr-implanted $Al_{0.35}Ga_{0.65}N$ annealed at an optimum anneal temperature of 775 °C shows the most convincing evidence of ferromagnetism persisting to room temperature with a coercive field (H_C) of 249 Oe and a remanent field (B_R) greater than 20% of the saturation magnetization (M_S = 6 x 10^{-5} emu) for measurements performed at 300 K. Field-cooled (FC) and zero-field-cooled (ZFC) magnetization measurements confirm ferromagnetic properties of this sample, and the Curie temperature to be estimated at around 350 K. The optimal anneal temperature was also confirmed through CL measurements.

$Al_{0.35}Ga_{0.65}N$ implanted with Mn has an H_C of 199 Oe and a B_R of 7 x 10^{-6} emu at room temperature when annealed under optimal conditions. Although temperature dependent magnetization measurements show a mild spin-glass phase, the ferromagnetic phase is clearly dominant.

Implanting $Al_{0.35}Ga_{0.65}N$ with Ni produces clear ferromagnetic hysteresis at temperatures up to 350 K. However, these samples show variability of M_S with respect to annealing temperature, which inhibits determination of optimal annealing conditions. FC—ZFC magnetization separation suggests the true presence of ferromagnetism in this material despite the variability in M_S.

11:45 oral

Field induced spin transitions and large magnetoresistance in the quasi-one-dimensional magnet $Ca_3Co_2O_6$

Koji Yamada[1], Jiaolian Luo[1], Zentarou Honda[1], Hiroko Katori[2]

1. *Saitama University, 255 Shimo-ookubo, Sakuraku, Saitama 338-8570, Japan* **2.** *Institute of Physical and Chemical Research (RIKEN), 2-1 Hirosawa, Wako-Shi, Saitama 351-0198, Japan*

Contact: yamasan@fms.saitama-u.ac.jp

Magnetic and magnetoresistance effect of needle shaped crystal and slightly Co rich $Ca_3Co_2O_6$ was investigated in a temperature range between 2 K and 100 K and in a DC magnetic field range less than 7 T and in pulsed fields less than 20 T. We found 1/3 ms and 2/3 ms plateaus of the magnetization for the full magnetization ms and large negative and positive magnetoresistance peaks of 10% at the same magnetic fields, respectively.

12:00 oral

Magnetism of CaAs and CaP half-metals

Oksana Volnianska, Piotr Boguslawski

Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland

Contact: volnian@ifpan.edu.pl

A considerable effort is currently devoted to the optimization of ferromagnetic III-V and II-VI semiconductors containing transition metal atoms, such as (Ga,Mn)As. The prospect of spintronic applications generates a search for new magnetic materials. In particular, Geshi et al. [1] predicted that CaAs and CaP are ferromagnetic (FM) compounds. This result is unexpected since in this case FM occurs in compounds that do not contain magnetic atoms, and thus Ca pnictides represent a new class of magnetic materials.

We analyzed the origin of magnetism of CaP and CaAs, as well as in MgP and MgAs. The calculations are based on the local spin density approximation, and the code developed in Ref. [2]. We find that both CaP and CaAs at equilibrium are half-metals with one free hole in the valence band per unit cell, and a full spin polarization of the hole gas. The paramagnetic (PM) phase of both crystals is higher in energy by about 30 meV/cell. In contrast, both MgP and MgAs are not magnetic.

By studying CaAs in a wide range of the lattice constants we show that its spin polarization is due to the spin polarization of the As atoms stemming from the Hund's rule. With the de-

creasing lattice constant an abrupt suppression of FM occurs. The effect is explained by observing that the kinetic energy of the fully spin-polarized FM free hole gas is higher than that of the unpolarized PM case. The energy difference between these two cases increases with the increasing hole concentration. Thus, the FM-to-PM transition corresponds to the crossover from low-density regime where exchange-correlation effects induce the FM state to the high-density regime where the kinetic energy dominates and stabilizes the PM state. This analysis holds for CaP as well. Similarly, the lack of FM in MgP and MgAs is due to their small equilibrium lattice constants.

The work is supported by grant PBZ-KBN-044/P03/2001

[1] M. Geshi et al, cond-mat. 0402641 (2004).

[2] http://www.pwscf.org

GMR and CIMS

Thursday afternoon, 8 September, 15:50
Main Building, room 219
Ulrich K. Rößler presides

15:50 invited oral

Current-induced spin torque and the domain wall dynamics in magnetic nanowires

Vitalii Dugaev[1,2], Vitor R. Vieira[1], Pedro D. Sacramento[1], Józef Barnaś[3,4], Miguel A. Araujo[5], Jamal Berakdar[6]

1. *Department of Physics and CFIF, Instituto Superior Tecnico,, Av. Rovisco Pais, Lisbon 1049-001, Portugal* **2.** *NAS of Ukraine, Institute for Problems of Material Science, Chernitsi Department, 5 I.Vilde str., Chernivtsi 58001, Ukraine* **3.** *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **4.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland* **5.** *Departamento de Fisica, Universidade de Evora, Evora P-7000, Portugal* **6.** *Max Planck Institute of Microstructure Physics (MPI), Weinberg 2, Halle 06120, Germany*

Contact: vdugaev@cfif.ist.utl.pt

We discuss recent theoretical results on the problem of current-induced spin torque in magnetic nanowires. Our main focus is on sharp domain walls, for which the domain-wall width is of the same order or smaller than the Fermi wavelength of electrons. Such a case is more suitable for magnetic semiconductors than metals.

We present the results of our calculations of the spin and spin current densities related to different modes of the scattering states. The accumulated transverse components of the spin density and the spin current oscillate in the vicinity of the wall and they essentially affect its dynamics whereas the longitudinal part of the spin current is responsible for another component of the spin torque, which creates a force for the current-induced motion of the domain wall along the nanowire.

We also analyze the dynamics of the sharp domain wall using the standard Landau-Lifshits-Gilbert formalism and the two-component spin torque calculated for this model. The model with a large constant of easy-plane anisotropy allows to simplify essentially the analysis of the domain wall motion, and our main calculations are performed for this particular model. We show that the domain wall changes its shape depending on the wall velocity, and we calculate this velocity as a function of the electric current. Within the model considered, there exists a critical current for the instability of magnetic system with respect to the spontaneous motion of the domain wall.

16:20 oral

Spin dynamics due to spin-transfer in magnetic spin valves

Martin Gmitra[1,2], Denis Horvath[2], Malgorzata Wawrzyniak1, Józef Barnaś[1,3]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *Department of Theoretical Physics and Astrophysics, Park Angelinum 9, Kosice 040 01, Slovakia (Slovak Rep.)* **3.** *Polish Academy of Sciences, Institute of Molecular Physics, Mariana Smoluchowskiego 17, Poznań 60-179, Poland*

Contact: gmitra@kosice.upjs.sk

The current-induced dynamics of magnetic moments in a spin valve structure is studied theoretically using a macroscopic model of spin transfer torque and numerical solution of the Landau-Lifshitz-Gilbert equation. The latter equation is generalized by including the torque due to spin transfer, which has been determined from the classical spin diffusion theory and macroscopic boundary conditions for the longitudinal and transverse components of the spin current.

The description includes parameters which can be taken from experiments on CPP-GMR, like bulk and interfacial spin asymmetry parameters,spin-diffusion lengths, bulk and interface resistances, and others. Some of the experimentally observed features , for instance the normal and inverse switching, hysteretic switching of the magnetic layer, as well as oscillatory magnetization modes can be qualitatively reproduced by the description.

16:35 oral

Giant magnetoresistance in p-n junction composed of betaFeSi$_2$ - Si

Koji Yamada, Hirohisa Ogawa, Zentarou Honda, Kiyoshi Miyake

Saitama University, 255 Shimo-ookubo, Sakuraku, Saitama 338-8570, Japan

Contact: yamasan@fms.saitama-u.ac.jp

We fabricated a p-n junction of p(beta FeSi$_2$)-n(Si), so called "environmental semiconductor". Large MR of 30% in 30T at RT was obtained for a well fabricated sample. We found the optimum annealing condition of the p-n junction for the largest MR was at T=840^{o}C, 2 hours for the deposited Fe on n-Si. Magneto-photo-transistor will be obtainable by the device in the future.

16:50 oral

Magnetostriction in combination with GMR/ TMR-structures

Stefani Dokupil, Maik T. Bootsmann, Markus Löhndorf, Eckhard Quandt

center of advanced european studies and research (caesar), Ludwig-Erhard-Allee 2, Bonn 53175, Germany

Contact: dokupil@caesar.de

Common GMR (giant magnetoresistance) /TMR (tunnel magnetoresistance) structures that are used e.g. in the field of data storage as read heads or MRAM (magnetic random access memory) devices consist of non-magnetostrictive materials in order to avoid any artefacts by external mechanical influences. GMR/TMR elements show a characteristic symmetrical behaviour in change of electrical resistance if an external magnetic field is applied. By replacing the non-magnetostrictive sensing layer by magnetostrictive material and using the inverse magnetostrictive effect such devices become sensitive towards mechanical strain and can be introduced as strain sensors [1, 2]. GMR and TMR structures have been developed consisting of one positive and one negative magnetostrictive sensing layer. This leads to a new characteristic resistance behaviour that depends on the alignment state of the magnetization of the two sensing layers, which is influenced by the applied external field and the level of mechanical stress. In correlation to the magneto electrical results to visualize magnetic domains of the sensing layer material under mechanical strain MOKE (magneto optical Kerr-effect) measurements will be presented. Additionally, the size dependency on these GMR/TMR structures will be discussed.

[1] Löhndorf, M.; Duenas-Lockwood, T.; Tewes, M.; Quandt; E., Rührig, R.; Wecker, J.: Highly sensitive strain sensors based on magnetic tunneling junctions (MTJs). Appl. Phys. Lett., 81 (2002), 313 – 315

[2] Löhndorf, M.; Dokupil, S.; Wecker, J.; Rührig, R.; Quandt; E.: Characterization of magnetic tunnel junctions (MTJ) with magnetostrictive free layer materials. J. Magn. Magn. Mater., 272–276 (2004), 2023–2024

17:05 oral

Low-temperature magnetic softening in sputtered FePtAg granular multilayers

Marco Coisson[1], Franca Albertini[2], Paolo Allia[3], Francesca Casoli[2], Federica Celegato[3], Simone Fabbrici[2], Paola Tiberto[1], Franco Vinai[1]

1. *Istituto Elettrotecnico Nazionale "Galileo Ferraris" (IEN), Strada delle Cacce, 91, Torino 10135, Italy* **2.** *IMEM- CNR (IMEM), Parma, Italy* **3.** *INFM and Dipartimento di Fisica, Politecnico di Torino, Corso Duca degli Abruzzi, 29, Torino 10129, Italy*

Contact: coisson@ien.it

Multilayered granular FePtAg films have been prepared by rf sputtering on a MgO (100) substrate covered by a 20 nm Ag undelayer. The basic unit of each multilayer is a FePt block (nominal thickness: 3.55 nm, atomic composition $Fe_{53}Pt_{47}$), topped with a Ag layer (nominal thickness: 5 nm). The basic unit is repeated 5 times. The substrate temperature was kept at 550 C during the whole deposition process. The as-deposited multilayers were annealed in situ at 550 C for 85 minutes in order to develop a tetragonal FePt phase with perpendicular anisotropy.

Magnetisation loop measurements perpendicular and parallel to the film plane have been performed in the 5-300 K temperature range by alternating gradient magnetometry under a field up to 18 kOe. Magnetoresistance (MR) measurements have been performed in the 2-300 K temperature interval by a standard four-contact method under a field up to 70 kOe. The magnetic field and the electrical current were applied parallel to each other and to the film plane. Magnetic measurements show that a substantial fraction of the magnetisation is spontaneously directed out of plane at all temperatures. At room temperature, saturation of MR is not achieved up to 70 kOe, while below 50 K a clear saturating trend is observed around 50 kOe. Above 10 K, the MR displays the usual negative behaviour with increasing field, and can be interpreted in terms of field-induced alignment of the magnetic regions governing spin-dependent electron scattering. A sharp downward jump of the zero-field electrical resistance has been observed on reducing T below 10 K. Such a jump is associated with the onset of a pronounced dip which opens at low fields in the R(H) curves, and is ascribed to anisotropic magnetoresistance. These results are explained in terms of partial magnetic softening of the alloy, related to competition between perpendicular and shape anisotropy, resulting in a local magnetisa-

tion which can be easily oriented by the applied field.

Friday, 9 September

Magnetic semiconductors

Friday morning, 9 September, 11:00
Main Building, room 219
Witold D. Dobrowolski presides

11:00 invited oral

Applications of the II-VI semimagnetic semiconductors

Andrzej Mycielski[1], Leszek Kowalczyk[1], Robert R. Gałązka[1], Roman Sobolewski[2], R. Rey-de-Castro[3], D. Wang[2], A. Verevkin[2], A. Burger[4], Małgorzata Sowińska[5], M. Groza[4], Paul Siffert[6], Andrzej J. Szadkowski[1], Barbara Witkowska[1], W. Kaliszek[1]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *University of Rochester, ECE Department, Rochester, NY 14627, United States* **3.** *Argonne National Laboratory (ANL), 9700 South Cass Avenue, Argonne, IL 60439, United States* **4.** *Fisk University, 1000 Seventeenth Avenue North, Nashville, TN 37208-305, United States* **5.** *EURORAD C.T.T., 23, Rue du Loess - BP 20, Strasbourg F-67037, France* **6.** *European Materials Research Society - Headquarters, 23, Rue du Loess - BP 20, Strasbourg F-67037, France*

Contact: mycie@ifpan.edu.pl

Selected possible applications of the (Cd,Mn)Te, a classical wide-gap semimagnetic semiconductor, will be described. Both the semimagnetic character and the other physical properties of this material give rise to its wide applications.

As a semimagnetic semiconductor - (Cd,Mn)Te offers "giant Faraday rotation" (of the direction of polarization in the strong static magnetic field) for the light with the energy of photons a little below the energy of free exciton. This effect, while being the strongest at low temperatures, is also significant at the room temperature. (Cd,Mn)Te-based "Faraday rotators" can be used in the optical telecomunications as "optical isolators" between the source of radiation (a strong laser) and the rest of the optical transmission line to avoid the parasitic light reflected back to the laser and disturbing its operation.

In the (Cd,Mn)Te crystals the Faraday effect is extremally fast. The results of the experiments, showing that the time of the respons of the (Cd,Mn)Te crystals to the pulsed magnetic field is in the range 300 ÷ 500 femtoseconds, will be presented, as well as the possible applications of this super-fast Faraday effect.

As a wide-gap semiconductor with high stopping power for the X and gamma radiation - the (Cd,Mn)Te can be applied to the construction of the radiation detectors. The required parameters: high resistivity (~ 10^{10} Ωcm) and high value of the μτ (mobility-life time product) (3 × 10^{-3} cm^2/V) have been already achieved and the results will be described.

11:30 oral

Structure and related properties of Si:Mn annealed under enhanced hydrostatic pressure

Andrzej Misiuk[2], Artem Shalimov[1], Jadwiga Bak-Misiuk[1], Barbara Surma[2,3], M. Prujszczyk[2], Jacek Jagielski[3,4]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland* **3.** *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **4.** *Andrzej Sołtan Institute for Nuclear Studies (IPJ), Świerk, Świerk 05-400, Poland*

Contact: misiuk@ite.waw.pl

Ferromagnetic ordering in silicon implanted with Mn+ ions, Si:Mn, has been reported recently; this ordering is evidently related to the structure of Mn-enriched near surface layer of implanted material [1]. As it has been stated earlier [2], not only temperature, HT, but also hydrostatic pressure, HP, applied at processing of implanted silicon affect its properties.

The effect of treatment at HT up to 1270 K under HP up to 1.1 GPa for time, t, up to 10 h on the structure and related properties of Si:Mn was investigated in present work by X-Ray, photoluminescence, and related methods.

Si:Mn was prepared by Mn+ implantation into 001 oriented oxygen-containing Si (Fz and Cz-grown) at 160 keV to a dose 1x10(16)(cm-2); temperature of the Si substrate, T, at implantation was kept at 340 K (AMn:Si)and at 610 K (BMn:Si).

Structural properties of Mn:Si are distinctly dependent on the implantation and treatment parameters. The structure of AMn:Si (T = 340 K) treated under HP at up to 1270 K for up to 5 h remains to be strongly disturbed; no magnetic ordering has been detected in AMn:Si if treated at 1070 K - 1.1 GPa for 1 h. Contrary, an annealing of this sample at 1070 K under atmospheric pressure produces well ordered structure. The BMn:Si samples prepared at T = 610 K and treated at atmospheric pressure as well as under HP were of improved crystalographic perfection; still in treated BSi:Mn some dislocations were detected.

The effects of T, HT, HP, and t on the structure and related properties of Si:Mn will be discussed.

1. M.Bolduc, C.Awo-Affouda, A.Stollenwerk, M.B.Huang, G.G.Ramos, G.Agnello, V.P.LaBella, Phys.Rev.B 71 (2005)033302.

2. A.Misiuk, A.Barcz, J.Ratajczak, L.Bryja, J.Mater.Sci.: Ma-

ter.in Electronics, 14 (2003) 295.

11:45 oral

Magnetic and Transport Characteristics of Ge(MnFe)

Heiko Braak, D. E. Bürgler, D. Rata, R. R. Gareev, M. Luysberg, M. Boese, P. A. Grünberg, C. M. Schneider

Research Centre Jülich, Institute of Solid State Research (IFF), Jülich, Germany

Contact: h.braak@fz-juelich.de

New materials for spintronic devices we considered to the class of diluted magnetic semiconductors. Recently Ge-based magnetic semiconductors attracted special attention due to their straight-forward integration into semiconductor technology.

Here we report on $Ge_{100-(x+y)}(Mnx,Fey)$ films showing ferromagnetism above RT. We show the magnetic and transport characterisation for a variety of (x,y) combinations.

The samples are grown by thermal evaporation on GaAs(001) at T=520K in a sequentional deposition scheme with a total thickness of about 100nm. In the first series the samples are annealed for one hour at elevated temperatures (T=730K).

The magnetic characterisation is done by a commercial SQUID magnetometer. The temperature dependence of the remanent magnetisation after zero field cooling shows a curve composed of two magnetic contributions with different phase transition temperatures. Thus it appears that the samples consist at least of two different magnetic phases. Transmission electron microscopy (TEM) pictures give evidence for clusters with a diameter of the order of 20 nm. Changing the final annealing conditions to 520K during 16 hours in our second series we find a reduction of the formation of clusters as evidenced by TEM. From the magnetisation measurement we can identify a high temperature phase associated with the clusters.

The van der Pauw scheme is applied to rectangular samples in order to determine the normal and anomal Hall coefficients. The temperature dependence of the transport properties gives a hint that the low temperature phase is connected to the formation of a genuine ferromagnetic semiconductor phase with a Curie-Temperature of T_c ~200K.

12:00 oral

Structural stability and formation of MnAs inclusions in (Ga,Mn)As

Pawel Jakubas, Piotr Boguslawski

Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland

Contact: bogus@ifpan.edu.pl

(Ga,Mn)As is one of the most intensively investigated magnetic semiconductors due to its high Curie temperature and compatibility with the GaAs technology. While annealing at about 200 C results in a substantial improvement of its properties [1], annealing at higher tempera-tures leads to the formation of inclusions. Depending on the annealing conditions, inclusions consist in smaller zinc-blende Mn-rich (Ga,Mn)As nanoclusters, or larger clusters of MnAs in the NiAs phase [2].

To understand structural stability of (Ga,Mn)As we performed first principles calculations using the local spin density approximation, and we generalized the definition of the mixing enthalpy to the case of alloys with a varying crystalline structure. We find that the stability of the zinc blende phase persists up to about x=0.75, while for higher Mn contents the equilib-rium structure is the NiAs phase. Moreover, due to the lattice mismatch, cubic GaMnAs is intrinsically unstable with respect to the segregation into pure end compounds. This suggests that formation of MnAs inclusions occurs in two steps. First, intrinsic instability results in fluctuations of the alloy composition, i.e., formation of Mn-rich clusters. In the second step, the clusters undergo a structural transformation from the metastable zinc blende to the stable NiAs structure.

Finally, we extend the analysis to (Cd,Mn)Te. The phase diagrams of both (Ga,Mn)As and (Cd,Mn)Te are similar, exhibiting a transition from the cubic to the NiAs phase with the increasing Mn content. For both systems we discuss magnetic ordering (which is ferromagnetic for (Ga,Mn)As, and antiferromagnetic for (Cd,Mn)Te), and its contribution to the structural stability.

The work has been supported by grant PBZ-KBN-044/P03/2001.

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Symposium E

Hybrid Polymer Systems of Industrial Importance

Welcome

Hybrid polymeric materials have been studied intensively during last years mainly due to their unique properties needed for more sophisticated or high-tech applications. Designing of the architecture of hybrid systems is the modern method to create novel polymeric materials, specifically applied as binders for advanced coatings or adhesives as well as composites, membranes and biomaterials. The polymers which are recently preferred as components of modern hybrid polymer systems are: silicones, polyesters, polyurethanes, polyacrylics and fluoropolymers since they provide unique combination of properties and often create possibility to achieve synergistic effect. Good examples of such systems can be IPNs involving silicones and fluoropolymers, silicones and polyurethanes, polyurethanes and polyacrylates as well as hybrid systems of specific particle architecture e.g. core-shell dispersions. Separate group of hybrids are organic-inorganic hybrid systems where the inorganic component has been entrapped in organic matrix or vice versa. Recently, great attention has been paid to hybrid polymer systems containing nanoparticles e.g. functionalized silica nanospheres which enables to achieve excellent material properties at low level of nanoparticle content.

The main aims of the Symposium are connected with the following aspects:

- promotion of strong and efficient international co-operation in the field of hybrid polymeric materials within European Research Area,
- successful commercialization of advanced hybrid polymer systems technologies, in particular in SMEs,
- contribution to environment protection through minimum use of natural resources due to development of novel hybrid polymer systems based on existing starting materials.

Organisers

Symposium Chairman

Prof. J. Kijeński, Industrial Chemistry Research Institute, Warsaw, Poland

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- Dr. B. Ameduri, Laboratory of Macromolecular Chemistry Ecole Nat. Sup. Chimie de Montpellier, France
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- Dr. M. Zielecka Industrial Chemistry Research Institute / Centre of Competence POLMATIN, Warsaw, Poland

Acknowledgements

Symposium is supported by Centre of Competence for Advanced Polymeric Materials of Industrial Importance POLMATIN (European Commission Project No.: GMA1-2002-72029).

The Centre of Competence POLMATIN, is situated in the structure of Industrial Chemistry Research Institute and recognized by Ministry of Science and Information Society Technologies as the Centre of Excellence.

Programme

Monday, 5 September

Monday Parallel Symposia

Monday afternoon, 5 September, 14:00

Main Building, room 213

Stanislaw Slomkowski presides

14:05 invited oral

Modified main groups metal oxides as potential active fillers for polymers

Jacek Kijenski

Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland

Contact: jacek.kijenski@ichp.pl

Main-group metal oxides play an important role not only as industrial catalysts and catalyst supports but also as highly efficient adsorbents. They are also successfully used as fillers of polymeric materials and polymer-matrix composites. Their catalytic activity may, however, result in changes in the polymer networks. In particular, selective interactions of the metal oxides with electron donors or acceptors can result in the formation of superbases or superacids reactive enough to remove protons from polymeric structures or to protonize inactive hydrocarbon moieties. To increase the efficiency of polymeric materials used as active fillers, they can be suitably modified by selecting the parameters of their preparation. As a result of progress in nanotechnology, the hybrid organic-inorganic nanocomposites were lately developed. The metal oxides can be there generated by a sol-gel process simultaneously with the polymerization of respective monomers. The structure and properties of the nanocomposites are determined by the properties of their components.

14:50 oral

Study of nano-plastics containing fillers of different types

Michael Ioelovich[1], Oleg L. Figovsky[2]

1. *Israel Research Center Polymate, Kibbutz Industrial Zone, Migdal Ha'Emek 23100, Israel* **2.** *Israel Research Center Polymate, Kibbutz Industrial Zone, Migdal Ha'Emek 23100, Israel*

Contact: polymate@borfig.com

In contrast with inorganic, organic fillers can contain various functional groups allowing them bonded with an organic polymer that leads to strength rising of the plastics. Moreover, organic fillers have low density, hardness and abrasibility and increased stability to settling in liquid systems, such as paints, varnishes, coatings, etc. Besides, organic fillers made from natural polymers are biodegradable. Nano-powders show excellent extender properties for emulsion paints and coatings contained mineral pigments. Introduction a low amount of the nano-extenders into SAR-based paint compositions permits replacing a significant amount of TiO2-pigment without changing in paint opacity value. However, due to lower specific weight and better compatibility with organic polymer binder nano-cellulose is more effective extender than nano-clay. So, the organic nano-filler imparts to plastics some peculiar properties, such as light-weight, increased strength, reduced hot-conductivity, selective water permeation, barrier against oxygen transmission and raised biodegradability. Moreover, this nano-filler improves properties of polymer based emulsion paints and coatings. Wide application of the organic nano-fillers can create new nano-materials having unique characteristics and properties.

15:10 invited oral

On thermal analysis of hybrid polymer systems

Zbigniew Dobkowski

Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland

Contact: zbigniew.dobkowski@ichp.pl

Recently, hybrid polymer systems have attracted a great deal of attention as materials that combine different features of their components with a synergistic improvement of their properties. Thermal properties of such systems are often needed for many applications, and the differential scanning calorimetry (DSC) and thermogravimetry (TG) methods are used for this purpose. Literature data revealed that characteristic temperatures (such as glass transition, melting, and decomposition temperatures) and mass residues, as well as activation energies of decomposition, are usually determined. The TG and DSC methods were also applied for investigations of the polysiloxane-polytetrafluoroethylene (SIL-PTFE) hybrid system of a semi-IPN nature. Perkin Elmer DSC-7 and TGA-7 instruments were used. The thermal stability of tested materials has been compared using 1/Tdi vs the heating rate plots (Tdi is the temperature of initial mass loss). The activation energies of decomposition (Ead) have also been determined using the Kissinger (KI) and Ozawa (OZ) methods, respectively. Both methods are found to be consistent, since the Ead(KI) value is represented by one point on the plot of Ead(OZ) vs conversion degree. For the SIL-PTFE system it has been found that the thermal stability of SIL-PTFE material is increasing with the increase of the content of PTFE component, according to the following sequence: SIL < SIL-PTFE20 < SIL-PTFE50 < PTFE. It has been found from DSC data that the optimum physical properties of the SIL-PTFE system as antigraffiti coatings are observed for the highest content of entrapped PTFE macromolecules in the crosslinked SIL cages. In general, it can be concluded that the TA techniques are useful for characterization of hybrid polymer systems.

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster E-1

Theoretical investigation of the surface roughness effect on the contact angle hysteresis which occurs at the water adsorption/desorption in/from a hydrophobized mesoporous silica gel matrix

Claudiu Valentin Suciu[1], Kazuhiko Yaguchi[2]

1. *Fukuoka Institute of Technology (FIT), 3-30-1 Wajiro-Higashi, Higashi-ku, Fukuoka 811-0295, Japan* **2.** *Fuji Silysia Chemical Ltd., 1846-2 Kozoji-cho, Kasugai-shi, AICHI 487-0013, Japan*

Contact: suciu@fit.ac.jp

Our research derives from the study of a novel principle of mechanical energy loss, called surface dissipation, induced by the dynamic contact angle hysteresis which occurs when a lyophobic liquid is forced to penetrate and then naturally exudes from a mesoporous matrix. Here, the mesoporous matrix is from silica gel modified by linear chains of alkyldimethylchlorosilanes and water is the associated liquid. This new kind of dissipation could be attractive for many applications, e.g., oil-free and environment-friendly absorbers, but the subject remains almost unexplored in the scientific literature. Though the energy loss can be explained by the contact angle hysteresis, its mechanism is not fully understood; it seems to be mainly induced by the surface roughness and its chemical heterogeneity. In this work, silica gel micro-grains are supposed to be obtained by the aggregation of nano-grains, which produces rough mesopores of variable radius. Considering the amorphous silica gel as a distorted crystal of β-

tridymite, the minimum diameter of the nano-grains reaches about 1nm. At the boundary of two silica gel nano-grains the maximum angular coating range is determined by the steric hindrance that occurs when the upper parts of the bonded molecules come into contact. Modification of the contact angle and surface energy during the water adsorption/desorption in/from the hydrophobized mesoporous silica gel matrix is evaluated in the nanometer range by employing a generalized Wenzel-Cassie model. Since the contact angle hysteresis has a dissipative effect, the energy loss occurs as the difference of the surface energy in advancing and receding motion of the interface. One finds the optimum diameter of the nanoparticles which maximizes the dissipated energy. Such results are useful for the appropriate design of ultrahydrophobic surfaces in general, and for the optimal design of silica gel grains with mesoporous architecture destined to environment-friendly dampers.

15:50 poster E-2

Vesicular Polymeric Films as High-Efficiency Active Media for the Random Lasers

Vasil P. Yashchuk[1], Eugen O. Tikhonov[2], Olga A. Prygodiuk[1], Vadym V. Koreniuk[1], Liudmyla P. Paskal[3]

1. *Department of Physics, Taras Shevchenko Kyiv University, 6 Gloushkov avenue, Kyiv 252022, Ukraine* **2.** *Institute of Physics, National Academy of Sciences, Prospekt Nauki 46, Kyiv 03650, Ukraine* **3.** *Kyiv National Taras Shevchenko University, Department of Chemistry, Volodymirskaya Str. 64, Kyiv 01033, Ukraine*

Contact: yavasil@ukr.net

Random lasers are new class of coherent light sources which inherent feature is positive feedback formation due to multiple light scattering in the active medium. Lasing threshold decreases under growth of the scattering efficiency. In the case of known random lasers multiple scattering is achieved by high concentration of fine dispersed particles (10^8-10^{11}cm^{-3}) embedded into the active medium (solid or liquid solution of dye) or by dispersity of the medium itself (semiconductor powder). The scattering efficiency in the suspension depends on relative refractive index of the scattering centers, their effective size and concentration. But as the refractive index of polymeric matrix is high the relative refractive index of the highly refractive materials (rutile, diamond) is $n_{rel}>1.6$. Moreover the homogeneity of particle distribution in the medium is difficult to be achieved that influences the random laser parameters. The close value of the relative refractive index ($n_{rel}=1.5$) can be obtained using gas vesicles in the active medium.

In the work we investigated the lasing threshold and the lasing spectra (pumped by Q-switched YAG:Nd^{3+}laser) parameters of the random laser based on films of solid polymeric dye (R6G, concentration 10^{-3}M) solution with N_2 vesicles as scattering centers. The vesicles parameters were varied by the intensity and duration of UV irradiation. The investigations were carried out depending on the vesicles concentration, films thickness (15-30mkm) and the polymer composition.

According to obtained results all the films possess the ability for lasing with lower lasing threshold and narrower lasing spectra when compared to suspension and dispersed media. The further decreasing of the lasing threshold can be predicted under optimisation of dye concentration and vesicles concentration and size. The obtained results allow asserting that investigated polymer films can be perspective to produce compact random laser.

15:50 poster E-3

Applications of quantitative image analysis to the description of the morphology of hybrid polyurethane with modified boehmite

Piotr Zapart, Joanna Ryszkowska, Magdalena Jurczyk-Kowalska

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: zapart@go2.pl

Study of structure property relationships in segmented polyurethanes (PUR) and its composites has acquired importance due to the broad range of PUR applications. This paper presents the application of quantitative image analysis to the description of morphological properties of modified boehmite and hybrid polyurethane. This analysis was used inter alia in the description of internal structure images and surface layer of materials as well as in description of their fractures. Fracture images of boemithe, and cut surface of polyurethane were obtained using high-resolution electron microscopy technique and atomic force microscopy. Quantitative analysis of the mechanical change images of fracture structure obtained with that technique allowed us to explain the mechanism of the changes and the thermal properties of polyurethane composites, as well as allowed us to determine relationships between structure characteristics and properties of examined materials.

Stereology methods were also found to be useful in comparative analysis of polyurethane composites structure and structure of boehmite and products of its modification. Ingredients used for PUR synthesis were: poly(ethylene adipate) (PEA) mol.wt 2000, 4,4'diphenylmethane diisocyanate (MDI), glycol (G1), glycerine (G2). The filler was added to PUR matrix in 3,5% wt. Synthesis was performed in one-shot process. Polyurethanes with PEA:MDI:G1:G2 molar ratio 6:9:2:1 were synthesized. The curing reaction was performed in the temperature of 120ºC for 16 hours.

The mechanical and thermal properties of polyurethane

boehmite composites were investigated.

The author thank the Prof Z.Florjańczyk and Mrs A. Wolak for supplying us modified boemite.

This research was financed by Polish State Committee for Scientific Research (as research projects PBZ-KBN-095/T08/2003 and 3T08A07428).

15:50 poster E-4

Metal oxide hybrid UV-curable painting for anti-static film

Shu-Mei Chang, Chung-Nan Li, Chun-Fa Chen

Natinal Taipei University of Technology (NTUT), 1, Sec. 3, Chung-Hsiao E. Rd.,, Taipei 106, Taiwan

Contact: f10914@ntut.edu.tw

Applying the modified nano-composite particles into UV curable acrylic monomer or resin to achieve higher surface conductivity film on flexible plastic surface. The nano conductivity metal oxides are studied The modified colloidal metal oxides are prepared from through liquid phase acrylization. The film properties are depended on the compatibility of acrylates/nano-composite hybrid, the selection of proper acrylic resin and the curing factor. Curing by a UV light system is a suitable fabrication for plastic substrates due to it takes shorter exposed time and less heat deformation occurred then the multi-step thermal baking. The hybrid films will be investigated by FTIR, DSC, DPA, GPC, AFM, particle size measurement, hardness measurement.

15:50 poster E-5

Applications of quantitative image analysis to the description of the morphology of hybrid polyurea-nitryle-urethanes

Joanna Ryszkowska, Ewelina A. Zawadzak, Ida Dulińska, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: idad@meil.pw.edu.pl

This paper presents the application of quantitative image analysis to the description of morphological properties hybrid polyurea-nitryle-urethanes (PUNU). Properties of polyurethanes are determined by type of polyols, isocyanates and chain extending agents. The type of used components; their molar ratio and synthesis condition affect the length of macromolecules and degree of cross-linking. These features determine properties such as: tensile strength, chemical and thermal resistance.

Cast segmented polyurea-nitryle-urethanes were obtained from: poly(ethylene adipate) (PEA) mol.wt 2000, 4,4'diphenylmethane diisocyanate (MDI) and dicyandiamide(DCDA). DCDA is crystalline powders with diameter of particles higher than 0,05um. Polyurethanes with PEA:MDI:DCDA molar ratio 3:4:1 were synthesized. The mixtures of PEA and DCDA were dehydrated for 2h at temperatures from 140 to 170±5C under vigorous stirring. Synthesis was performed in one-shot process. The polymers were cured for 16h at temperature from 110 to 190±5C. The samples were conditioned for two weeks at room temperature. This paper also describes application of quantitative image analysis to the description of particles diameter and domain structure of hybrid polyurea-nitryle-urethane.

Cut surface of polyurethane was obtained using high resolution electron microscopy technique and atomic force microscopy. The mechanical and thermal properties of polyurea-nitryle-urethane were investigated.

Quantitative analysis of the images of fracture structure allowed us to explain the changes of mechanical and thermal properties of examined materials.

This research was financed by Polish State Committee for Scientific Research as research projects 3T08A07428

15:50 poster E-6

Novel conjugated nanomaterials based on pyrimidine structures - synthesis and physicochemical profile

Joanna Cabaj[1], Jadwiga, Maria Sołoducho[1,2], Antoni Chyla[1], Krzysztof Idzik[1]

1. *Wrocław University of Technology, Department of Chemistry, Wyb. Wyspianskiego 27, Wrocław 50-370, Poland* **2.** *Wrocław University of Technology, Wybrzeże Wyspiańskiego, Wrocław 50-370, Poland*

Contact: joannacabaj@wp.pl

A great deal of research around the world has been focused on developing novel materials composed of conjugated, self - assembled and saturated units. Conjugated polymers such as polypyrroles, polyphenylenes, polythiophenes have been extensively studied, and many important electrical and optical properties have been discovered [1]. Consequently, there is an apparent attention in the preparation and purification of these materials.

Our considerable interest in precursors of conducting polymers chemistry stimulated us to obtain several, novel symmetric oligoheterocycles. According to continuation previous effects [2], here is reported on the synthesis and certain physicochemical profile of symmetric bis-thiophene or bis-ethylenedioxythiophene derivatives based on pyrimidine.

An interesting results have been obtained by starting from chalcones. Obviously, in our case, after reaction of chalcones with proper amidines we isolated bis(halogenephenyl)pyrimidines. Applying variation of standard Stille procedure for the synthesis C-C coupling products,

in the next step bis(phenyl)pyrimidine derivatives were condensed with 2-(trimethylthin)thiophene to get the final compound – 2-alkyl-4,6-bis[4-(2-thiophene)]pyrimidine. Similar results have been achieved by performing the coupling reaction between 2-trimethylthin-3,4-ethylenedioxythiophene with bis(halogenophenyl)pyrimidines.

As it comes from aggregation experiments some pyrimidine derivatives give stable and good quality Langmuir and Langmuir-Blodgett films (in binary systems with docosanoic acid and octadecylthiophene) of desired composition and their electrical properties are currently under investigation.

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15:50 poster E-7

A new sensing nanostructured material for nitroaromatics detection

Xavier Le Guével

Commiseriat à l'Energie Atomique (CEA), CEA Le Ripault ,BP16, MONTS 37260, France

Contact: xavier.le-guevel@cea.fr

With the increased use of explosives such as nitroaromatics in terrorist attacks the development of efficient, portable and low-cost explosion detection devices has become an urgent worldwide necessity. For 10 years, many studies have been performed on the detection of nitroaromatics compounds using chemical gas sensors as highly sensitive and selective detectors of various explosives [1,2].

A new promising material based on nanostructured high-porosity silica coating has been developed and tested regarding chemical sensing of nitroaromatics. The silica nanoparticles contained in the sensing coating are prepared in solution (so-called sol) using the ammonia-catalysed hydrolysis of tetraethylorthosilicate (TEOS) [3].

High- porosity values of the deposited films were evaluated using BET specific surface area measurement (450m2/g). The hydroxyl surface coverage of nano-sized silica particles can be adjusted depending on solvent selected for dispersion (water or ethanol) or by further exposition to catalytic vapour-curing (ammonia hardening or hydrophobic curing). The type of particle surface species coverage was highlighted to direct impact onto structural properties of the films and therefore onto detection performances.

Evaluation of the detection performances of silica-based coatings when used as sensitive coating for quartz crystal microbalance (QCM) sensors or as fluorescent sensor when grafted with a fluorescent molecule show remarkable results. Among the different silica coatings, we have selected a fluorescent film showing high sensibility (@0.1ppm and @1ppm), excellent reversibility (80% of fluorescent intensity), robustness, good selectivity regarding V.O.C interfering compounds (Volatil Organic Compounds).

Moreover, an hydrophilic silica thin film used as a detecting material shows a good sensibility whatever the operating humidity conditions (from 0 to 80%RH).

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15:50 poster E-8

Thermal stability of hybrid polyurethane and modified boehmite

Joanna Ryszkowska[1], Magdalena Jurczyk-Kowalska[1], Bartlomiej J. Wasniewski[1], Andrzej Wolak[2], Zbigniew Florjanczyk[2], Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland*

Contact: bwasniewski@inmat.pw.edu.pl

Polyurethanes (PUR) are materials that show high strength, elasticity, significant elongation, and high chemical resistance among others. These properties are result of a difference in their chemical structure. Considering this properties PUR are used in coal mining industry very often. In this application polyurethanes have not enough thermal stability. One of the methods to increase this factor is using nanofillers. Addition of small quantity particles to polyurethane can change its properties and create new class materials. The most common nanocomposites are composed of polymers and organically modified boehmite.

In this work, the influence of boehmite variable additives on polyurethane nanocomposite properties and structure were investigated.

Components used for PUR synthesis were: poly(ethylene adipate) (PEA) mol.wt 2000, 4,4'diphenylmethane diisocyanate (MDI), glycol (G1), glycerine (G2). Polyurethanes with PEA:MDI:G1:G2 molar ratio 6:9:2:1 were synthesized. As fillers were used: unmodified boehmite and boehmite modified tributhylphosphate. The fillers were added to PUR matrix in 0,5; 1,5; 3,5%wt. For incorporation of boehmite in polyurethane was adopted one-shot, in – situ polymerization process. The curing reaction was performed in the temperature of 120ºC for 16 hours.

In this work mechanical and physical properties of obtained materials were investigated. Mechanical properties were stud-

ied using Instron 1115 tensile machine. Abrasive wear were studied using Schoppera-Schlobacha machine. Thermal properties of neat PUR and PUR/boehmite composites were studied using DSC (Q1000 TA Instruments) and TGA (Q 500 TA Instruments). Morphologies of PUR and nanocomposites were examined using atomic force microscope (AFM).

This research was financed by the Polish State Committee for Scientific Research (PBZ-KBN-095/T08/2003)

15:50 poster E-9

Investigation of metal polymer nanocomposites

Igoris Prosyčevas[2], Judita Puišo[1,2], Algimantas Juraitis[2], Asta Guobienė[1,2], Sigitas S. Tamulevičius[1,2], Ramūnas Naujokaitis[1]

1. *Kaunas University of Technology, Department of Physics, Studentu 56, Kaunas LT-3031, Lithuania* **2.** *Kaunas University of Technology, Institute of Physical Electronics, Savanoriu 271, Kaunas LT-3009, Lithuania*

Contact: judita.puiso@ktu.lt

Nanocomposites materials consisting of metallic nanoparticles incorporated into polymers have been paid immense attention in today's research for their potential applications in the fields of catalysis bioengineering, photonics and electronics. Silver particles have also been used to detect DNR and RNR. They also demonstrate great affinity sulfur in living organics, but biological properties of silver are still not understood properly. Processes of nucleation and shaping of nanostructures on surface on polymer substrates were investigated. Ag thin films (thickness 1-20 nm) were deposited on PMMA-PET structures by e-beam evaporation in vacuum. PMMA thin layers were produced from 5% PMMA solutions by dip coating. XRD and AFM were employed to study structural and morphological changes of the nanocomposites. Surface composition of these nanocompoites was investigated by XPS. Changes of optical were registered by UV-VIS and IR measurements. The relative intensity of (111) peak in XRD patter is significantly higher than that for Ag powder diffraction, suggesting a preferred orientation of some Ag grains starting at very early growth stages (~ 10 nm). The resistivity was measured as a function of silver layer thickness. The surface plasmon resonance absorption peak is observed at 420 nm. The blue shift in the peak position is associated with a reduction of particle size.

15:50 poster E-10

Evaluation of degradation degree of dental hybrid composite used as permanent fillings

Joanna E. Siejka - Kulczyk, Malgorzata Lewandowska, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: jsiejka@inmat.pw.edu.pl

The aim of this study was to evaluate the degradation process of dental hybrid composites used as permanent fillings. Bis – GMA resin was used as polymer matrix. Ceramic glasses with various chemical compositions and particle sizes as well as nanosilica were used as composite fillers. The specimens were stored in water for 168 h and in 0,1M NaOH for 6, 24 and for 168h. Degradation was evaluated by Scanning Electron Microscopy (SEM) microstructure observation and mechanical testing, such as flexural strength, microhardness. The results indicate that the chemical compositions of fillers influence significantly the degradation process.

15:50 poster E-11

Properties of hybrid polyurea-nitryle-urethanes

Joanna Ryszkowska, Magdalena Jurczyk-Kowalska, Ewelina A. Zawadzak, Monika M. Bil, Marcin Jędrzejczyk

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: mjedrzej@meil.pw.edu.pl

Polyurea-nitryle-urethanes (PUNU) are block copolymers containing soft and hard segments. Soft and hard segments of these polymers differ in polarity. At temperatures below 120^{o}C polar hard segments typically do not mix homogeneously with less polar soft segments. As a consequence hard and soft domains are formed. Properties of PUNU such as: tensile strength, hardness, abrasive wear, chemical and thermal resistance are determined by parameters of synthesis process. Hard segments of hybrid polyurea-nitryle-urethanes are obtained with dicyandiamide (DCDA). DCDA is crystalline powder with diameter of particles higher than 0,05um. Dicyandiamide contains two amino groups and highly polar cyanimine group. The strong polar hard segments influence PUNU properties, especially high thermal stability. This polymers oxygen index is 36% (without antioxidants).

The aim of this paper is to characterize the hybrid polyurethanes, synthesized by different dehydrating and curing temperatures. Cast segmented polyurea-nitryle-urethanes were obtained from: poly(ethylene adipate) (PEA) mol.wt 2000, 4,4'diphenylmethane diisocyanate (MDI) and dicyandiamide.

Polyurethanes with PEA:MDI:DCDA molar ratio 3:4:1 were synthesized. The mixtures of PEA and DCDA were dehydrated for 2h at temperature from 140 to 170+/-5°C under vigorous stirring. Synthesis was performed in one-shot process. The polymers were cured for 16h at temperature from 110 to 190+/-5°C. The samples were conditioned for two weeks at room temperature.

The mechanical (tensile strength, ultimate elongation, hardness, abrasive wear) and thermal (glass transition temperature, DSC thermograms, thermal stability – TGA, dynamical mechanical thermal analysis - DMTA) properties of hybrid polyurea-nitryle-urethanes were investigated. Images of the structure were obtained using HRSEM and AFM.

15:50 poster E-12

Nanocomposite binders for powder coatings

Jolanta Rosinska[1], Michał Kędzierski[2], Zbigniew Bończa-Tomaszewski[3]

1. *Instytut Chemii Przemysłowej (IChP), Rydygiera 8, Warszawa 01-793, Poland* **2.** *Instytut Chemii Przemysłowej (IChP), Rydygiera 8, Warszawa 01-793, Poland* **3.** *Instytut Chemii Przemysłowej (IChP), Rydygiera 8, Warszawa 01-793, Poland*

Contact: jolanta.rosinska@ichp.pl

Development of powder coating technology takes place in many segments of industrial manufacturing, new and better products are being developed to meet the needs of a demanding marketplace. There are the following trends in powder technology:

- lower cure temperature products
- enhanced surface flexibility
- enhanced application properties (flowing) and workability
- enhanced UV durability.

Thermosetting carboxylic polyesters and UV-curable solid unsaturated polyesters were synthesized.

Carboxylic polyesters were obtained from: neopentyl glycol, ethylene glycol, terephthalic acid, isophthalic acid, trimelitic anhydride.

Unsaturated polyesters were obtained from: neopentyl glycol, 1,6-hexanediol, terephthalic acid, maleic anhydride.

All polyesters were modified with nano-filler - montmorillonite (1-2 wt.%) in order to improve processing and application properties.

Conclusions:

1. All polyesters modified with nano-filler were more resistant from agglutinating during storage in comparsion to unmodified polyesters.

2. There was no difference in melting point, glass temperature, melt viscosity and curing time between modified and unmodified carboxylic polyesters.

3. Introducing of nano-filler to unsaturated polyesters resulted in increased glass temperature and melt viscosity with unchanged curing time and melting point.

15:50 poster E-13

Comparative study of different solvents on preparative Temperature Rising Elution Fractionation of HDPE

Nattawood Samoth, Supawan Tantayanon, Varawut Tangpasuthadol

Functional Polymer & Petrochemistry Research Laboratory, Department of Chemistry, Faculty of Science, Chulalongkorn University, Payathai road, Bangkok 10330, Thailand

Contact: mottomoth@hotmail.com

It has been known that Temperature Rising Elution Fractionation(TREF) is the most effective technique for investigating the microstructure of semicrystalline polymer especially polyolefins, such as PE and PP. Moreover, TREF was also applied for determining the degree of branching which affected on their mechanical properties. In the present work, the influence of medium polarity on physical properties of fractionated polymers was investigated. The commercial HDPE resins were used in this study. It was found that using mixed solvent with higher polarity can enhance the crystallization rate of polymers while the physical properties of the fractionated polymers obtained from both ones, using non-polar solvent and mixed solvent were not different significantly.

Tuesday, 6 September

Tuesday Parallel Symposia

Tuesday morning, 6 September, 9:00
Main Building, room 213
Roberta Bongiovanni presides

9:00 invited oral

Doped Polymeric Systems based on sol gel technology, their optical properties and potential industrial applications

Renata Reisfeld

Polish Academy of Sciences, High Pressure Research Center (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: Renata@vms.huji.ac.il

Sol gel technology is based on hydrolysis followed by polycondensation of variety of precoursors.

The process starts with solution and terminates with solid materials that may have a required shape of the bulks or alternatively thin films maybe formed on glass quartz, semiconductors or metals.

As an example prepared in our laboratory are systems such as silica, titania, zirconia, silica glymo (3-glycidopropyxy trimetoxy silane) zirconia silica-polyurethane (ZSUR) (1), (2).

During the formation of the polymeric systems a large number of active additives were included resulting in new optical materials:

Tunable lasers in the visible range (3), Sensors (4), Active waveguides (5), Photochromic materials (6), Electrochromic materials (7), Semiconductor quantum dots (8), Solar materials (9).

We shall present the scheme of sol gel formation incorporating the new laser dyes, emission and laser dyes threshold of the tunable lasers, and preparation of quantum dots and rods as well as their optical and electronic properties of our materials.

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[ABSTRACT TRUNCATED TO 2000 LETTERS]

9:30 oral

Experimental investigation of the dynamic contact angle hysteresis past a solid surface from hydrophobized silica gel

Claudiu Valentin Suciu[1], Kazuhiko Yaguchi[2]

1. *Fukuoka Institute of Technology (FIT), 3-30-1 Wajiro-Higashi, Higashi-ku, Fukuoka 811-0295, Japan* **2.** *Fuji Silysia Chemical Ltd., 1846-2 Kozoji-cho, Kasugai-shi, AICHI 487-0013, Japan*

Contact: suciu@fit.ac.jp

Motivation for such research derives from the investigation of a novel principle of mechanical energy dissipation, called surface dissipation, and its attached machine element, named a colloidal damper. Similar to a hydraulic damper, this absorber has a cylinder-piston structure, but oil is replaced by a colloid consisting of a mesoporous matrix and a lyophobic liquid. Here, the mesoporous matrix is from silica gel modified by linear chains of $ClSi(CH_3)_2C_mH_{2m+1}$, $Cl_2Si(CH_3)C_mH_{2m+1}$ and $Cl_3SiC_mF_{2m+1}$ (m = 1-18); water is the associated liquid. Such absorber, being oil-free and employing a mixture of water and silica gel, might be considered as an environment-friendly application of nanotechnology in the field of mechanical engineering. Energy loss can be explained by the dynamic contact angle hysteresis in advancing and receding motion of the interface. Although such hysteresis is not fully understood, it seems to be induced by surface roughness, surface chemical heterogeneity, and a variety of nonequilibrium phenomena, such as dissolution, swelling, and surface reorientation of the functional groups. In this work, the architecture of the used silica gel as well as the structure of the grafted coating is briefly discussed. Test rig and the measurement technique of the dynamic contact angle hysteresis are described. From experimental data one calculates the dissipated energy versus length of the grafted molecule on the silica gel surface. Influence of the interface movement frequency, pre-pressurization and maximum applied pressure on the dynamic contact angle hysteresis and dissipated energy is found for silica gels with different hydrophobic coatings. One finds the optimum length of the grafted molecules which maximizes the dissipated energy of the colloidal damper. Such results are generally useful for the appropriate design of ultrahydrophobic surfaces.

9:50 oral

Preparation of Natural rubber/Polypyrrole/Clay Composites by Chemical Oxidative Polymerization

Rungaroon Saelim, Supawan Tantayanon

Functional Polymer & Petrochemistry Research Laboratory, Department of Chemistry, Faculty of Science, Chulalongkorn University, Payathai road, Bangkok 10330, Thailand

Contact: Rungaroon.S@Student.chula.ac.th

Conductive rubbers nanocomposites may be used instead of metallic conductors because the soft materials have the obvious advantage of flexibility and ability to absorb mechanical shock. Both soft and rigid materials have the following advantages over metals: ease of shaping, low density, wide range of electrical conductivity, and corrosion resistance. Conductive rubber nanocomposites has been investigated in this research, natural rubber/polypyrrole/montmorillonite composites (NR/PPy/MMT) were prepared by chemical oxidative polymerization of pyrrole in a suspension of montmorillonite (MMT) and natural rubber latex using ferric chloride as oxidizing agent. For finding the suitable conductive rubber composites their conductivity properties were evaluated. The

conductivity of NR/PPy/5%MMT composite prepared by both 5%MMT and natural rubber latex were mixed together prier to adding the monomer before mixing with an oxidant was 6 Scm^{-1} at weight ratio of natural rubber/pyrrole about 0.8. In addition, the conductivity of NR/PPy composite at the same condition was 5 Scm^{-1}. It was a slightly lower than NR/PPy/MMT. It concluded that the intercalation of conductive rubber into clay layers may result in the formation of hybrid materials with good electrical conductivity. The thermal stability of these composites were also investigated. The first significant weight loss in MMT and PPy occurs already at temperature between 50-100^{o}C because of their hydroscopic nature. The peak degradation temperature of the NR/PPy/MMT composites are seen at 324 and 467^{o}C, corresponding to the decomposition of PPy and NR.

10:10 invited oral

Silicone-containing polymer matrices. The effect of surface layer structure on antisoiling properties

Maria Zielecka[1], Elżbieta Bujnowska[1], Robert Nowakowski[2]

1. *Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland* **2.** *Institute of Physical Chemistry, Polish Academy of Science, Kasprzaka 44/52, Warsaw 01-224, Poland*

Contact: maria.zielecka@ichp.pl

The objective of this work is to examine the effect of surface layer structure on antisoiling properties. Fluoropolymers and acrylic resins were applied as organic component of the polymer matrices. The surface properties of protective coatings received from silicone-containing protective matrices were studied based on wettability (dynamic contact angle and surface free energy), atomic force microscopy [AFM], x-ray electron microscopy [ESCA-XPS]. Their morphology, roughness and structural regularity will be discussed to show the effect of organic component on the surface properties. The application properties of such coatings materials were tested based on antisoilig tests, water vapour permeability, water absorption and weathering resistance (freezing, salt and uv resistance).Very good correlation of application properties of these coating materials and their surface properties such as high dynamic contact angle, low surface free energy, surface morphology was observed and will be discussed.

Tuesday Parallel Symposia

Tuesday afternoon, 6 September, 14:00
Main Building, room 213
Janusz Kozakiewicz presides

14:00 invited oral

Hybrid polymeric materials for medical applications

Stanislaw Slomkowski, Teresa M. Basinska, Beata Miksa, Ewelina Przerwa

Polish Academy of Sciences, Center of Molecular and Macromolecular Studies (CMMS-PAS), Sienkiewicza 112, Łódź 90-363, Poland

Contact: staslomk@bilbo.cbmm.lodz.pl

Materials used for medical applications, in particular for medical diagnostics devices, often should have a set of properties difficult to obtain for polymers, metals or ceramics alone. However, in many instances the required properties could be achieved for combinations of materials belonging to the mentioned above classes into polymer-metal or polymer-ceramic hybrids. During presentation we will report on fabrication of hybrid materials based on polymeric nano- and microparticles, the materials developed for using them for production of biosensors. A set of fundamental properties of materials for construction of detectors of biosensors includes: (i) the ability to immobilize chosen bioreceptors (e.g., enzymes or antibodies) on selected surfaces in a way allowing retention of their bioactivity, (ii) suitability for transmission of electrical, optical or other signals accompanying interaction of receptors with an analyte, (iii) an easy, simple and inexpensive production. In our laboratory we developed methods for controlled synthesis of polymeric particles with diameters in a range from ca 100 nm to few micrometers - particles with interfacial layer containing reactive hydroxyl, carboxyl or aldehyde groups suitable for covalent immobilization of proteins and oligonucleotides. Syntheses of these particles and methods suitable for immobilization of biomacomolecules onto them will be presented. Convenient and simple methods for physical adsorption and covalent immobilization of particles onto glass, quartz, mica, and onto other substrates have been developed. Special attention was concentrated on fabrication of hybrid polymer nanoparticle – inorganic substrates with controlled morphology of particle monolayers. In the lecture we will describe investigated models of biosensors constructed on the basis of the mentioned above hybrid materials and discuss other potential applications of these materials for fabrication of diagnostic devices.

14:30 invited oral

Binary polymer brushes as a versatile tool to create stimuli responsive coatings

Petra Uhlmann[1], Leonid Ionov[1], Nikolay Houbenov[1], Mirko Nitschke[1], Karina Grundke[1], Sergiy Minko[2], Manfred Stamm

1. *Institute of Polymer Research, Dresden 01069, Germany*
2. *Clarkson University, Potsdam NY, United States*

Contact: uhlmannp@ipfdd.de

Heterogeneous binary polymer brushes consist of an assembly of polymer chains of two incompatible polymers that are attached by one end to the surface with sufficient grafting density and have been investigated experimentally only for a short time. Those brushes can be used in the form of ultrathin polymeric layers as a versatile tool for surface engineering to tune physico-chemical surface characteristics as wettability, surface charge, chemical composition or morphology, and furthermore to create switchable and responsive surface properties1. For the fabrication of these layers "grafting from“ (as radical polymerization at the interface) and „grafting to“ (as tethering of the polymer chains from solution) methods were developed and investigated in detail.1

The amplification of the amplitude of switching (from superhydrophilic to ultrahydrophobic) by creating hierarchically structured surfaces2 will be explained as well as the creation of switchable surface structures by environmental responsive lithography3. Additionally examples will be given for the transfer of the concept of binary polymer brushes to particles, which may also have interesting perspectives for an application in the field of coatings.

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15:00 invited oral

Organic-inorganic Nanohybrid Materials for coatings.

Oleg L. Figovsky, Leonid Shapovalov

Israel Research Center Polymate, Kibbutz Industrial Zone, Migdal Ha'Emek 23100, Israel

Contact: figovsky@netvision.net.il

The high-dispersed inorganic phase can be obtained in the polymer matrix. In according with such idea we use epoxy, cyclocarbonate and methacrylic oligomers for creation of nano-structured organic-inorganic hybrids. To obtain this several families of curing agents aryl- and alkyl-silanes dendroaminosilane hardeners were synthesized on the base of aminosilanes and tetraethylortosilicate (TEOS)or tetraethyortotitanate (TEOT). Using as interpenetrating polymer networks (IPN) principle in production of composite materials provides a unique possibility to regulate their both micro-and nanostructured properties. Coatings have improved wear resistance and photo-stability. The key advantage of the technology is based on the development of a class of additives, which is adaptable to all kinds of coatings. Main advantages were received for hybrid nonisocyanate polyurethanes.

Tuesday Parallel Symposia

Tuesday afternoon, 6 September, 15:50
Main Building, room 213
Renata Reisfeld presides

15:50 invited oral

Fluorinated hybrid networks through photopolymerisation processes

Roberta Bongiovanni, Aldo Priola, Giulio Malucelli, Marco Sangermano

Politecnico di Torino - Dipartimento di Scienze dei Materiali e Ingegneria Chimica, C.so Duca degli Abruzzi 24, Torino 10129, Italy

Contact: roberta.bongiovanni@polito.it

The use of fluorinated structures in polymeric networks is very attractive due to their outstanding properties, connected to the presence of fluorine, such as chemical and thermal stability, weathering resistance, low surface tension, optical and electrical behaviour. In the last years we have been investigating the homopolymerisation and copolymerisation of fluorinated monomers through the UV curing technique, obtaining interesting materials. A wide range of polymeric networks have been prepared, changing systematically the structures of the monomers and their functionality: it has been showed how not only the fluorine content, but also the structural characteristics of the polymeric matrix and the distribution of the fluorine atoms along the polymeric chains play an important role in the final performances of the materials.

By homopolymerization of fluorinated oligomers, networks exhibiting special morphology and bulk properties have been prepared: in particular when perfluoropolyether structures are present, a biphasic nanostructure is formed.

When the fluorinated products are copolymerised in very low amount (less than 2%w/w) with hydrogenated oligomers by a photoinitiated curing process, a selective migration of the fluorinated monomers to the film surface is observed. Interestingly, the surface composition has been found dependent on the type of the fluorinated chain, its molecular weight, the identity of the end group and the length and the nature of the spacer between the reactive group and the perfluorinated chain. Also the process conditions are relevant to tailor the

surfaces of the cured networks.According to the surface enrichment in fluorine, a modification of the surface properties of the UV cured films is achieved while the bulk properties remain unchanged: first the materials become highly hydrophobic and oleophobic, then an improvement of the solvent resistance, antigraffiti properties, lower friction are observed.

16:20 invited oral

"Hybrid" silicones. An overview on polysilalkylene or silarylene siloxanes fluorinated or not.

Francine Guida-Pietrasanta

Laboratoire de Chimie Macromoléculaire - UMR-CNRS 5076 - Ecole Nationale Supérieure de Chimie de Montpellier (ENSCM), 8 rue de l'Ecole Normale, Montpellier 34296, France

Contact: fguida@enscm.fr

Because of the increasing need for high-temperature resistant materials, like elastomers for applications in advanced technological areas (aerospace, defense, computer applications), a vast amount of research was developed in the area of inorganic-organic hybrid polymers. Organosiloxane polymers are one of the most important class of such materials due to their outstanding properties like thermal stability, low glass transition temperature, unusual surface properties, excellent gas permeability. But the classical polysiloxanes present the drawback of depolymerization in certain conditions (acid or base medium or high temperature). So, in order to try and avoid this possible depolymerization, a new class of polysiloxanes has been developed, mainly since the 1970s: the so-called "hybrid silicones", that are polysilalkylene siloxanes, fluorinated or not and polysilarylene siloxanes also fluorinated or not.

The elastomers prepared by crosslinking of these polymers present a very high thermal stability and a rather low temperature flexibility, and depending on the nature of their backbones or of their side chains, they may exhibit an excellent resistance to solvents or fuels.

This overview on Polysilalkylene or silarylene siloxanes fluorinated or not, so-called "Hybrid" silicones will present:

- the main synthetic routes to obtain these polymers.

*Synthesis of alpha,omega-bis silanol monomers (prepared either via an organometallic route or via hydrosilylation of alpha,omega-dienes) followed by polycondensation of these bis silanols

* Polyhydrosilylation of alpha,omega-dienes by alpha,omega-dihydrooligosiloxanes

- the properties of the different polymers.

16:50 oral

X-ray diffuse scattering investigation of PTFE surfaces

Christoforos Gravalidis, Stergios Logothetidis

Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece

Contact: cgrava@physics.auth.gr

The surface properties of PTFE are essential for many applications. Especially, the morphology of the surface is critical in the interaction with other solids or liquids. Thus, X-Rays electromagnetic radiation is appropriate in the study of the topography in short length scales, for example in nanoscale. We present an X-Ray Diffuse Scattering XDS technique which can determine, except for the surface roughness, other issues concerning morphology like distribution of in-plane fluctuations and fractal dimension on the surface. The scattering intensity is calculated under Distorted-Wave Born Approximation (DWBA) and depend on quantities related both to 1^{st} and 2^{nd} order surface statistics like, surface roughness σ, lateral correlation length ξ and "Hurst coefficient" or "roughness exponent" h ($0<h<1$). There are two different scan types for XDS: rocking scan and off-specular reflectivity using the appropriate modification in the intensity. The samples were prepared by spraying a PTFE dispersion directly on preheated Si wafer, at three different spraying times. Afterwards the samples were annealed into inert atmosphere at 340 ^{o} C. The aim of this work is to study the effect of spray duration on the surface morphology. Firstly, the amorphization induced due to the annealing was observed by XRD. The analysis of the XDS rocking scans gave that the mass density is increased with the spraying time and the roughness is almost the same. However, the surface morphology described also by the distribution of the vertical fluctuations, through the correlation length ξ, showed that the narrow distribution is realized at middle, while broad one at long or short spraying times. Furthermore, the surface exponent h, calculated by XDS off-specular scan, is constant and low, meaning that the low smoothness of the surface is not affected by the spraying time.

Wednesday, 7 September

Wednesday Parallel Session

Wednesday morning, 7 September, 9:00

Main Building, room 213

Francine Guida-Pietrasanta presides

9:00 invited oral

Silicone-containing hybrid polymer dispersions designed for coating applications

Janusz Kozakiewicz, Maria Zielecka, Anita Koncka-Foland, Elżbieta Bujnowska, Anna Szulc

Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland

Contact: janusz.kozakiewicz@ichp.pl

Silicone-containing hybrid polymer systems have been attracting attention of researchers for last couple of years since silicones can provide a set of unique properties to the hybrid material, including: improved low temperature elasticity, heat resistance, specific surface properties and excellent water resistance combined with gas and water vapour permeability. Novelty materials of this kind have been introduced to the market and are now being used widely in the industrial applications, including coatings. The macro-scale properties of hybrid materials that represent combination of organic polymers and silicones can be tailored to the demands of particular application through designing of the specific micro- or even nano-scale architecture of the hybrid system. In this paper, the results of studies on novel aqueous silicone-containing hybrid polymer dispersions of specific architecture designed for coatings will be presented. Dispersions were synthesized either via polymerization of acrylic monomers in aqueous polysiloxaneurethane anionic dispersions prepared from the mixture of polysiloxanediol, saturated polyol and unsaturated polyol and cycloaliphatic diisocyanate or via polymerization of silicone monomers in commercially available acrylic polymer dispersions The effect of structural parameters of the hybrid polymers which constituted dispersion particles on the properties of dispersions, films and coatings was studied and it was found that they significantly influenced by the share and kind of silicone component. More detailed studies of surface properties of the coatings using wettability determinations (DCA, SFE), ESCA, and AFM techniques revealed self-assembling of the surface layers depending on the chemical composition of the polymers constituting the hybrid system.

9:30 oral

Hybrid and other nanoparticles for vinyl chloride polymerisation nucleation

Maria Obloj-Muzaj[2], Maria Zielecka[1], Agnieszka Abramowicz, Anna Szulc[1], Wojciech Domanowski[1]

1. *Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland* **2.** *Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland*

Contact: Maria.Obloj-Muzaj@ichp.pl

Suspension polymerisation of vinyl chloride has been carried out on various types of nanoparticles chosen from clays, silica (pure or functionalized), silicone polymer or hybrid core/shell ones. Nanofillers used for VC polymerisation nucleation significantly influence the structure and shape of PVC grain: from typical "cauliflower" in case of control sample and in the presence of silica to "cakes" in case of functionalized silica, "sea-shells" with silicone polymer use or "balls" - unusual for suspension PVC - in the presence of core/shell particles. The last ones look just as emulsion PVC although were prepared in suspension polymerisation process.

It has been found using SEM and TEM methods that distribution of nanofiller in polymer was rather uniform. Montmorillonite looks to be intercalated and partially exfoliated in PVC grain. Silica nanospheres look to be able to change the shape under VC polymerisation conditions and to form sharp-edged structures. Some ordered regions showing mesoporosity were also found suggesting the formation of more ordered structures. These regions are visible especially in the samples nucleated with silica.

There are strong interactions between functionalized or hybrid nanofillers with suspension stabilisers and nanofillers are often captured in stabilisers' net. The presence of nanofillers in PVC grain as well as the change of PVC grain shape caused by them significantly influences the final properties of PVC and its processability.

9:50 oral

Hybrid uv-curved coatings containing montmorillonites

Roberta Bongiovanni, Daniele Mazza, Aldo Priola, Silvia Ronchetti, Elisa A. Turcato

Politecnico di Torino - Dipartimento di Scienze dei Materiali e Ingegneria Chimica, C.so Duca degli Abruzzi 24, Torino 10129, Italy

Contact: roberta.bongiovanni@polito.it

This work describes the preparation and characterisation of new coatings based on UV-curable resins intercalated into phyllosilicates.

The UV-curing process was employed to produce such materials in form of films, as it is a fast and environmentally friendly technique.

Two types of montmorillonites were used: a montmorillonite in the sodium form (Cloisite® Na^+ CEC = 92.6 meq/100 g) and an organophilic montmorillonite (Cloisite® 30B CEC = 90 meq/100 g, where the functionalisation is made with a dihydroxyethyl octadecyl methyl ammonium chloride). The fillers were dispersed in 3,4-epoxycyclohexylmethyl-3',4'-epoxycyclohexilcarboxylate, (CE) or in a waterborne polyurethane acrylate resin (Ucecoat 6558 by UCB) at a maximum concentration of 5%

w/w, with the aid of an ultrasonic bath. The mixtures containing the oligomer together with the clay were then added of the proper photoinitiator at 2-4% w/w. They were exposed to UV light and films 200 micron thick were obtained. Before and after the photopolymerization, the hybrid systems were investigated by XRD analysis. The system made of CE and Cloisite® 30B showed the expected intercalation with an increase of the interlamellar spacing d001 from 18.5Å of the powder to 34.6Å in the CE oligomer. Similar results were obtained with the Cloisite Na^+ in the acrylic system: the interlamellar spacing d001 moved from 12.1 Å of the powder to 18.6 Å in the oligomer.A further confirmation of the intercalation was obtained by TEM analysis.

After the UV-curing no further change in intercalation occurred. The presence of the organophilic montmorillonite affected the polymerisation process of the epoxy oligomers which react via a cationic mechanism. The decrease of the conversion rate in the cationic polymerisation of CE was due to the basicity of the quaternary ammonium salts impurities in the clay: they reduced the activity of the photoinitiator acid fragments produced under irradiation.

10:10 oral

Dual curing photopolymerization in coating compositions

Zbigniew Bończa-Tomaszewski, Anna Bańkowska

Instytut Chemii Przemysłowej (IChP), ul. Rydygiera 8, Warszawa 01-793, Poland

Contact: zbigniew.boncza-tomaszewski@ichp.pl

The aim of the paper is to report a dual-cure powder coatings hybrid system that combine the benefits of free radical polymerization and photo-cationic polymerization and overcome their limitations. The coating is based on solids epoxy polyester and solid urethanized acrylate. The final coatings properties are achived by the combination of free radical and cationic polymerization in the presence of hydroxyl-ketone initiator and photo acid generated initiator. Single source UV-light irradiation was employed to produce either single stage or dual-stage hybrid polymerization. The cure film are more flexible, adhere better to the substance and does not have a pin hole compare to 100% free radical or cationic system. Also, the "dark cure" mechanism provides a mean to cure the film in areas not fully exposed to the light.

Wednesday Parallel Symposia

Wednesday afternoon, 7 September, 14:00
Main Building, room 213
Maria Zielecka presides

14:00 invited oral

Advanced polyoxymethylene-based nanostructured materials

Krzysztof Pielichowski, Agnieszka Leszczynska

Cracow University of Technology, Department of Chemistry and Technology of Polymers, ul. Warszawska 24, Kraków 31-155, Poland

Contact: kpielich@usk.pk.edu.pl

Polymeric nanostructured materials differ from conventional composites in that the mixing of phases occurs over a much smaller length scale in comparison to the micrometer length scale of conventional composites, resulting in to nanocomposite materials with properties substantially different from those of the parent end members. The properties of the matrix, the distribution and properties of the filler as well as the nature of their interface control the behaviour of a typical composite material. Thus, the nanoparticles often strongly influence the properties of the composites at very low volume fractions. This is mainly due to their small interparticle distances and the conversion of a large fraction of the polymer matrix near their surfaces into an interphase of different properties as well as to the consequent change in morphology. As a result, the desired properties are usually reached at low filler volume fraction, which allows the nanocomposites to retain the macroscopic homogeneity and low density of the polymer. One of the most important engineering polymers with a unique set of mechanical, thermal, chemical and electrical properties is polyoxymethylene (POM). Among various POM systems with high impact resistance, blends of POM and (nano)segmented thermoplastic polyurethanes (TPUs) are considered as very promising since TPUs, as modifiers, offer a wide range of properties resulting from their segmented structure. Furthermore, they show partial miscibility with POM based on their ability to form hydrogen bonds between urethane group in polyurethane macrochain and ether linkage in polyoxymethylene backbone. To the other group of POM-based materials that has recently attracted considerable attention belong nanocomposites with layered silicates; both classes of nanostructured materials will be discussed.

14:30 oral

New higher added value polymeric materials made of poly(ethylene terephthalate)-contained fabrics waste and polyamide 6

Regina Jeziorska

Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warszawa 01-793, Poland

Contact: regia.jeziorska@ichp.pl

Post-production PET-contained fabrics waste (PET) and poly-

amide 6 (PA) were blended with maleic anhydride grafted linear low density polyethylene (LLDPE-g-MAH) as a reactive modifier. The reactions of LLDPE-g-MAH with blend components were studied by Fourier Transformation Infra Red Spectroscopy (FTIR), solubility behavior of the products in formic acid and rheological measurements. Investigated blends were prepared in a co-rotating twin-screw extruder and characterized by differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). The static tensile properties, and impact strength of the blends were tested. The results indicate that the reactions of modifier with free carboxyl end groups of PA and polyesters run, at least in part, towards the formation of PA-co-modifier–co-PET copolymers acting as the effective compatibilizers for these blends.

14:50 oral

Random Laser with Increased Efficiency

Vasil P. Yashchuk[1], Eugen O. Tikhonov[2], Olga A. Prygodiuk[1], Volodymyr I. Bezrodny[2]

1. *Department of Physics, Taras Shevchenko Kyiv University, 6 Gloushkov avenue, Kyiv 252022, Ukraine* **2.** *Institute of Physics, National Academy of Sciences, Prospekt Nauki 46, Kyiv 03650, Ukraine*

Contact: yavasil@ukr.net

The random laser is the strongly scattering active medium itself where the inter particle distance is about of light wavelength. The lasers do not demand any cavity use so they are of great interest due to the construction simplicity being useful for their producing and operation. But the random lasers have not been adopted yet because of low efficiency and high threshold of the pump intensity. In this work the method of these parameters refinement is proposed.

Lasing in the random laser is supplied by two non-resonant methods of the feedback formation: multiple scattering within the sample volume and diffusive reflection from the sample surfaces. The second feedback type is more effective and can be attained by sample thinning down to the excited region size and covering of the back sample surface with highly refracted powder. When the first condition is accomplished the sample is through depth excited and essential part of the radiated light achieves the back sample surface. Then highly refracting coating of the back sample side completely returns the radiated light to the excited region that is equal to the excited region extension. We produced and investigated the random laser on solid polymer solution of R6G with embedded particles of SiO_2 (the mean diameter of the particles is ~0.5-1mm) in concentration $10^{11}cm^{-3}$. The back side of the sample was covered with fine dispersed powder CeO_2.

The energetic characteristics of the produced random laser were obtained to be higher that ones of the usual random laser and to depend on the laser effective thickness. The highest possible laser efficiency is at the sample thickness conforming to the excited region depth d_{act}.The steady lasing is at any thickness but the least lasing threshold was achieved at the thickness ~$3*d_{act}$. Further improving of lasing parameters can be achieved by use of micro lenses conjugated with the active medium.

Wednesday Poster Session

Wednesday afternoon, 7 September, 15:50

Symposium F

Interfacial Processes and Properties of Advanced Materials

Welcome

The aim of the symposium is to bring together top experts on interfaces in order to theoretically and experimentally explore the connection between the atomic structure, the chemical composition, the diffusion effect, growth dynamics and other interfacial phenomena with the materials properties. The theoretical aspect will deal with the geometrical models, which are nowadays used for the description of interfaces and interfacial phenomena. It will also encompass modeling of the atomic structure by empirical potentials and ab initio techniques. The materials to be addressed include metals, semiconductors, and ceramics whose industrial development needs a strong support from fundamental approaches and in which interfacial phenomena play a critical role (polycrystalline semiconductors, epitaxial films of oxides, superconductors and semiconductors). Experimental work will report on the structural information, segregation, grain growth, and interface formation in various materials. Mechanical, optical, and electronic properties will be covered. The ultimate objective is to establish a robust knowledge of the role of interfaces on the properties of key materials. The combined approach of theoretical and experimental expertise of the key speakers will shed a new insight on device fabrication, which is becoming critically dependent on the interfacial behaviours.
The topics will include but will not be exclusive of:
Theory and modelling

- Structure analysis
- Physical and chemical behaviour of interfaces
- Atomic and electronic structure of interfaces
- Martensitic transformation

Semiconductors

- Hetero-epitaxial growth of highly mismatched materials
- Stress, strain and relaxation in small lattice mismatched III-V epitaxial systems

Oxides

- Metal-oxide interfaces
- Si-oxide interfaces
- Interfaces in perovskite epitaxial heterostructures

Devices

- Capacitors and CMOS transistors
- Varistors

Scientific Committee:

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- **Dr. J.-L. Maurice**, Unité Mixte de Physique, CNRS/ Thales, Orsay, France
- **Dr. P. Bristowe**, Department of Materials Science & Metallurgy, University of Cambridge, Cambridge, United Kingdom
- **Prof. Ph. Komninou**, Department of Physics, Aristotle University of Thessaloniki, Thessaloniki, Greece

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Programme

Monday, 5 September

Interfaces and dislocations in compound semiconductors 1
Monday afternoon, 5 September, 14:00
Main Building, room 231
Jean-Luc Maurice presides

14:00 invited oral

Ab initio based multiscale simulations of dislocations in GaN

Joerg Neugebauer, Liverios Lymperakis

Max-Planck-Institut für Eisenforschung , Department of Computational Materials Design, Max-Planck-Str. 1, Düsseldorf D-40237, Germany

Contact: neugebauer@fhi-berlin.mpg.de

A major challenge in fabricating/optimizing group-III nitride based devices is the huge dislocation density commonly ob-

served in this materials system. A thorough understanding of the structure and particularly of the electrical activity of dislocations is thus crucial. However, so far even an agreement whether dislocations in GaN are electrically active or not is lacking. From a theoretical point of view a major challenge in describing dislocations is the large range of different length scales: While the core structure is rather localized the surrounding strain field is long range. Combining elements of density functional theory (DFT), empirical potentials, and continuum elastic theory we were able to describe edge dislocations consisting of a few 10^5 atoms with ab initio accuracy. Using this approach we were able to (i) identify a hitherto not considered dislocation structure in GaN[1], (ii) demonstrate that the huge strain field around edge dislocations in GaN may cause deep gap states independent of the specific core structure, and (iii) understand the role of defects (vacancies) on the structure and electrical activity of dislocations. Based on these results the effect dislocations have on the optical properties of epitaxial films grown at various conditions and by different techniques.

[1] L. Lymperakis et al., Phys. Rev. Lett. 93, 196401 (2004).

14:30 oral

Ab-initio tight-binding study of the core structures of the c edge dislocation in wurtzite GaN

Imad Belabbas[1,2], Mohamed Belkhir[1], Jun Chen[3], Antoine Bere[2,4], Gerard Nouet[2]

1. *Laboratoire de Physique Theorique, Groupe de Physique du Solide, Universite A.Mira, BEJAIA, Algeria* **2.** *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **3.** *Laboratoire de Recherche sur les Proprietes des Materiaux Nouveaux (LRPMN), Damigny, France* **4.** *Laboratoire de Physique et de Chimie de l'Environnement, Universite de Ouagadougou, Ouagadougou, Burkina Faso*

Contact: imad.belabbas@ensicaen.fr

GaN layers grown by means of heteroepitaxial techniques contain a huge amount of threading dislocations with typical density of $10^{10}cm^{-2}$. The majority of these dislocations are (a) edge and the remaining ones are both (c) screw and mixed (a+c). A great improvement in layers quality has been achieved with ELO (Epitaxial Lateral Overgrowth), by lowering this dislocation density with several orders, laser diodes with lifetime reaching l0.000h has been realized. However, it's well known that in the ELO technique, dislocations have the tendency to bend and then some of them can change their character for screw to edge and vice-versa. In this work, we explore the issue of what could be the core structure of a c screw dislocation (b = <0001>) when it bends and changes its character to edge (b=<0001>, l=<11-20>).

In the framework of self-consistent tight-binding methods (SCC-DFTB), the core geometries of four configurations of the c edge dislocation are presented and their structures analyzed in terms of bonds angles and lengths. The dislocations are modeled in pseudo hydrogen terminated clusters of about 720 atoms, periodic along the dislocation line direction <1-210>. The equilibrium core configurations were obtained by relaxing the clusters in a procedure based on a conjugate gradient algorithm. The two core configurations without wrong bonds display a 4/8 atoms rings like structure while those with wrong bonds display a 5/8/5 atoms ring like one.

14:45 oral

Partial dislocation core distribution in GaN using high resolution transmission electron microscopy

Joseph Kioseoglou, George P. Dimitrakopulos, Philomela Komninou, Thomas Kehagias, Theodoros Karakostas

Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece

Contact: sifisl@auth.gr

High resolution transmission electron microscopy (HRTEM) observations of 1/6 <20-23> edge partial dislocations delineating the I_1 basal stacking fault in wurtzite GaN are evaluated by the use of geometric phase analysis and image simulations. The I_1 stacking fault is introduced by a shear following a vacancy or interstitial disc precipitation and it may be bounded by an edge or mixed type partial dislocation. The geometric phase analysis of the HRTEM images is used in order to extract the strain field of the partial dislocations. Atomic models of these partials have been obtained previously from anistotropic elasticity and molecular dynamics calculations using a modified Stillinger-Weber type empirical interatomic potential. The core radius is determined on the experimental micrographs using the dislocation distribution tensor, which presents non-zero values only at the dislocation core, and it is found in agreement with the empirical potential calculations. Image simulations of the relaxed atomic models are employed for identification of the core structures. Strain and dislocation distribution components measured on the simulated images are compared with the relative components of the experimental micrographs.

15:00 oral

The atomic configuration of tilt grain boundaries around <0001> in GaN

Jun Chen[2], Pierre Ruterana[1], Gerard Nouet[1]

1. *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **2.** *Laboratoire de Recherche sur les Proprietes des Materiaux Nouveaux (LRPMN), Damigny, France*

Contact: gerard.nouet@ensicaen.fr

III-V nitride semiconductors grown on different substrates contain a very high density of threading dislocations. Some of them can form low-angle grain boundaries, and for the most important disorientation high-angle grain boundaries. For specific rotation angles, coincidence orientations are obtained and be given in the concept of coincidence site lattice (CSL). The atomic structure of these coincidence orientations is periodic and based on a limited number of structural units. For the coincidence tilt grain boundaries around [0001] axis in wurtzite structure, the shape of the unit cell is a lozenge and the two diagonals corresponding to the largest density of coincidence sites can be chosen to describe them. Since some experimental grain boundaries are not described by the edge or the shortest diagonal of the CSL unit cell, we present the atomic structure of the second diagonal. These diagonal boundaries contain the same structural units then the edge boundaries. Their atomic structures are determined in the range from 0^{o} to 60^{o}, and compared to the atomic structures of the edge.

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster F-1

Scanning Tunneling Microscopoy and surface Simulation of c-GaN(001)-4x1 Ga Tetramer Reconstruction

Hamad A. AL-Brithen[1,2], Muhammed B. Haider[2], Nancy Sandler[2], Arthur R. Smith[2], Pablo Ordejon[2,3]

1. *King Saud University (KSU), P.O. Box 2455, Riyadh 11451, Saudi Arabia* **2.** *Ohio University (OU), Dept. of Physics & Astronomy, Athens, OH 45701, United States* **3.** *Institut de Ciencia de Materials (ICMAB) - CSIC (ICMAB), Campus de la UAB, Barcelona 080193, Spain*

Contact: brithen@ksu.edu.sa

Scanning tunneling microscopy images of c-GaN(001)-4×1 acquired at both positive and negative sample biases show that the surface consists of "tetramer" rows aligned along [110] with a spacing along [1-10] of 12.8 Å, or 4-atomic spacings. Dual-bias imaging indicates a shift of the corrugation maximum position by 180^{o} as the sample bias changes sign. Simulated images of the surface reconstruction based on the 4×1 linear tetramer model by Neugabauer et al.,[(1)] confirm the STM data, showing the occupied states peak at the middle of the tetramer and the unoccupied states peak between tetramers. Scanning tunneling spectroscopy also shows the semi-insulating nature of the 4×1 reconstruction, having a surface gap of 1.3 ± 0.1 eV, as also predicted by theory. Higher Ga coverage on the surface shows a variety of Ga-adatom reconstruction.

[(1)] Neugebauer et al., Phys. Rev. Lett. 80, 3097(1998)

15:50 poster F-2

SURFACE PROPERTIES OF CARBONIC THIN FILMS PREPARED BY ELECTROCHEMICAL DEPOSITION

Aurel V. Andrei[1], Fulger Manuela[1], Diaconu Constantin[1], Andrei V. Elisabeta[2]

1. *INSTITUTE FOR NUCLEAR RESEARCH (ICN), CAMPULUI, NO.1, PITESTI 115400, Romania* **2.** *National Institute for Research and development in Microtechnologies (IMT-Buchares), P.O.Box 38-160, Bucharest 023573, Romania*

Contact: ANDVIC12@YAHOO.COM

Thin films of diamond-like (DLC) and graphitic materials are characterized by low thermal expansion coefficients, high thermal and electrical conductivities and chemical inertness. DLC, in addition, exhibits extreme hardness.

A technique of carbon film synthesis based on electrochemical process was used. A solution of acetylene in liquid ammonia was employed as the electrolyte; films were deposited on the various substrates.

We report the characterization by XPS and XAES of the diamond vs graphite nature of amorphous carbon films deposited; the outstanding problem is the determination of the ratio of carbon atoms with diamond-like sp3 tetrahedral bonding to carbon atoms with carbon graphitic sp2 trigonal boding.

The electochemical behaviour of the thin films is studied by Electrochemical Impedance Spectroscopy.

The electrochemical processes of electrodeposition are analysed, to understand the relationship between properties of the deposited carbonic thin films (actual film microstructure) and the deposition experiment parameters.

15:50 poster F-3

Energy and electronic structure of gallium and nitrogen interstitials in GaN Tilt Boundaries

Bere Antoine[1,2], Belabbas Imad[1], Gerard Nouet[1], Pierre Ruterana[1], Jean Koulidiati[2], Chen Jun[3]

1. *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **2.** *Laboratoire de Physique et de Chimie de l'Environnement, Universite de Ouagadougou, Ouagadougou, Burkina Faso* **3.** *Laboratoire Universitaire de Recherche d'Alençon, IUT d'Alençon (L), Damigny 61250, France*

Contact: antoine.bere@ensicaen.fr

Defects containing point defects, impurities, and intentionally doping in GaN, are the subject of numerous experimental and theoretical investigations. It is well known that their presence can reduce the performance of optoelectronic devices due to a multiplicity of parasitic luminescence which they are respons-

ible.

While it is now well known that yellow luminescence (YL) around 2.2 – 2.3 eV observed in undoped GaN layers results from a transition between band states and deep levels, the origin of the defects that are associated with this YL is still under debate.

Cathodoluminescence studies have recently shown that the YL is not uniform in the crystal and therefore can be associated with the presence of extended defects like dislocations and grain boundaries. Then, several atomistic models of extended defects may be considered in order to explore the ones that give rise to the parasitic YL.

In this work, molecular static atomistic simulations using a Self-Consistent-Charge Dentity-Functional-Tight-Binding (SCC-DFTB) approach will be applied to study the core structure of tilt boundaries in interaction with gallium and nitrogen interstitials. The geometry of the nearest neighbours, the formation energies and the electronic states associated to these defects will be discussed in comparison with those in GaN bulk and dislocations.

15:50 poster F-5

The size effect in strength and fracture toughness of deformation origin submicro- and nanomaterials.

Katya M. Borysovska[1], Viktor N. Slyunyayev[1], Yuriy M. Podrezov[1], Zbigniew Pakiela[2], Krzysztof J. Kurzydlowski[2]

1. *Institute for Problems of Materials Science, National Academy of Sciences of Ukraine, Krzizanovsky, Kyiv 38(044), Ukraine* **2.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: podrezov@ipms.kiev.ua

The effect of dislocation wall availability and of dislocation wall-crack tip distance on the crack tip dislocation emission is simulated. The detailed analysis of the system with an account of the image forces is carried out.

The calculation results allow the influence of dislocation-wall-shielding effect on crack tip stress intensity factor as well as on total energy of the system to be evaluated.

It was shown that dislocation wall results in redistribution of dislocations ahead of crack tip. This gives rise to decrease of crack tip stress intensity factor and total energy of the system.

15:50 poster F-6

STUDY OF p-n JUNCTIONS BASED ON LANTHANUM MANGANITES

Renata Butkute[1], Bonifacas Vengalis[1,2], Jelena Devenson[1], Fiodoras Anisimovas[1], Antanas Oginskis[1], Alexandra M. Rosa[3]

1. *Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania* **2.** *Vilnius Gediminas Technical University, Sauletekio al. 11, Vilnius LT-2040, Lithuania* **3.** *University of Lisbon, Lisbon 1749-016, Portugal*

Contact: renata@pfi.lt

In this work, we present the results of growth and investigation of p-n diode structures based on hole-doped lanthanum manganite films. High crystalline quality p-type $La_{2/3}A_{1/3}MnO_3$ (A = Ba, Ca and Ce) thin films were grown heteroepitaxially by pulsed laser deposition and magnetron sputtering onto conductive n-type $SrTiO_3$:Nb (STON) substrates. X-ray diffraction measurements demonstrated perfect crystalline structure of the prepared LBMO and LCaMO films, while traces of CeO_2 as a secondary phase have been indicated for the LCeMO films. Resistance versus temperature curves measured for the films with current in-plane geometry were compared to zero field temperature-dependent junction resistance of the p-n structures. The current-voltage characteristics of all the structures exhibited a rectifying behaviour similar to p-n diode with the characteristic barrier voltage in a case of forward bias increasing gradually from about 0.25 V at 300 K to 0.8 V at 78 K. Special attention was focussed on a comparative study of nonlinear current-voltage characteristics, magnetoresistance and photoconductivity of the prepared diode structures. Various heterostructures were annealed at different temperatures under various oxygen pressure conditions. The observed thermal instability of the LCaMO/STON and LCeMO/STON interfaces was associated with formation of high resistance region (tunnel barrier) due to a diffusion of oxygen ions under strong internal electrical field and possible phase separation in the manganite films at the interface.

15:50 poster F-7

Intrinsic Ferroelectric Hysteresis Behaviors for Heterostructures

Khian-Hooi Chew[1], Yoshihiro Ishibashi[2], Franklin G. Shin[1]

1. *The Hong Kong Polytechnic University (HK, PolyU), Hung Hom, Hong Kong, Hong Kong* **2.** *Aichi Shukutoku University, Nagakute-cho 480-1197, Japan*

Contact: apkhchew@polyu.edu.hk

Intrinsic ferroelectric hysteresis behaviors for a heterostructure of interface between two different ferroelectrics of the

second-order transition are studied using the Landau-Ginzburg theory. We show that the existence of interfacial coupling could lead to a new polarization reversal mechanism. A detailed description of the polarization reversal mechanism of the heterostructure is discussed based on the hysteresis loops and the spatial variation of polarizations under various electric field strengths.

15:50 poster F-8

Influence of enhanced temperature and pressure on structural transformations in pre-annealed Cz-Si

Andrzej Misiuk[1], Jerzy Ciosek[1], Jadwiga Bak-Misiuk[2], Barbara Surma[1,3], Jacek Ratajczak[1], Artem Shalimov[2], Victor G. Zavodinsky[4], Andrzej Kudła[1]

1. *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland* **2.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **3.** *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **4.** *Institute of Materials Science, Tikhookeanskaya 153, Khabarovsk 680042, Russian Federation*

Contact: jciosek@ite.waw.pl

Processing of Czochralski silicon (Cz-Si, semiconductor in microelectronics and optoelectronics) at high temperature (HT) leads to clustering of oxygen always present in Cz-Si (oxygen content, c_o, can reach > 1.1 x10^{18} cm^{-3}), with a creation of SiO_{2-x} precipitates exhibiting tensile stress on the matrix. Oxygen precipitation, the stress mentioned and so the defect structure of Cz-Si are affected by hydrostatic pressure (HP) exerted by inert medium at HT. The effect of HT - HP treatment (typically for 5 h) at 1230 /1400 K under HP up to 1.2 GPa on structural transformations in Cz-Si was investigated by microhardness, photoluminescence (PL), ellipsometric, and related measurements.

To create nucleation centres for precipitation (NC's), Cz-Si was subjected to one step pre-annealing at 720 K (samples A, resulting c_o = 1 x10^{18} cm^{-3}) or to 4 steps pre-annealing at up to 1000 K (samples B, c_o = 3.6x10^{17} cm^{-3}).

Pre-annealing of Cz-Si at 720 K for up to 20 h affects its microhardness (H) only slightly while the B samples are less hard (H equals to 15.9 and 14.4 GPa, respectively). The HT - HP treatment of A samples results in decreased c_o (after the treatments at 1230 K under 0.01 GPa and 1.2 GPa, c_o equals to 7.3x10^{17} cm^{-3} and 5.8x10^{17} cm^{-3}, respectively). The treatment of B samples at 1230/1400K under HP results in increased H (up to 16.6 GPa) while the density of dislocations (evidenced by the presence of PL lines at 0.81 eV and 0.87 eV) decreases with HP in the case of treatment at 1230 K but increases after the treatments at 1400 K.

Structural transformations in treated Cz-Si are related to the effect of HP on the creation and stability of NC's as well as on diffusivity of oxygen and of Si interstitial.

15:50 poster F-9

Atomic ordering in GeSn and SiSn advanced semiconductor alloys

Vitalij Deibuk, Yuriy G. Korolyuk

Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine

Contact: vdei@chnu.cv.ua

The IV-IV semiconductor alloys have recently emerged as a possible candidate for the creation of non-polar, miscibility gaps semiconductor, with carrier mobility higher then those III-V and II-VI compounds. The GeSn and SiSn alloys is an important example of this semiconductor alloy, and understanding its phenomena is increasingly valuable as alloys become used in electronic devices today. Part of this understanding is the material's atomic crystal structure, which is then the basis for the understanding other properties, such as the electronic band structure, defects, and surface growth morphologies.

In this work we report the theoretical study of atomic ordering in GeSn and SiSn semiconductor alloys. Our investigations were based on NVT molecular dynamic method with three-particle Tersoff's potential of interactions with mirror-boundary conditions.

We calculated and analyzed the enthalpy of formation of ZB(zinc-blende)-ordered and RH(rhombohedral)-ordered $Ge_{0.5}Sn_{0.5}$ and $Si_{0.5}Sn_{0.5}$ alloys. The bond-length of Ge-Ge, Ge-Sn, Si-Si, Si-Sn and Sn-Sn bonds is calculated by using the radial-distribution functions. Also it was investigated the enthalpy of disordered $Ge_{0.5}Sn_{0.5}$ and $Si_{0.5}Sn_{0.5}$ alloys and compared with the ordered one and study of spontaneous atomic ordering. Our results are in a good agreement with recent experimental data.

15:50 poster F-10

Structural properties of quaternary InAlGaN MQWs grown by plasma-assisted MBE

George P. Dimitrakopulos[1], Philomela Komninou[1], Thomas Kehagias[1], Joseph Kioseoglou[1], Alexandros Georgakilas[3], Gerard Nouet[2], Theodoros Karakostas[1]

1. *Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece* **2.** *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **3.** *Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology – Hellas and University of Crete, Department of Physics, P.O. Box 1527, 71110 Heraklion, Crete, Greece, P.O. Box 1527, Heraklion 71110, Greece*

Contact: gdim@auth.gr

Quaternary $In_xAl_yGa_{1-x-y}N$ multiple quantum wells (MQWs) with nominal composition x = 0.085, y = 0.285, and GaN spacers are characterized by high resolution transmission electron microscopy (HRTEM), geometric phase analysis (GPA), and energy dispersive x-ray nano-analysis in a scanning transmission electron microscope (STEM). The MQWs were grown by rf plasma MBE using metal-rich conditions and a relatively low growth temperature. Specimen preparation was performed by tripod polishing in order to minimize artifacts due to excessive ion milling. The material exhibits sharp well-defined interfaces. Indium clustering is not observed; the onset of clustering occurs only after prolonged observation under the electron beam. The GPA indicates that the quantum wells are lattice-matched to the GaN spacers. The chemical concentration profiles are obtained by deconvolution of EDX scans and confirm the chemical sharpness of the interfaces.

15:50 poster F-11

Electrical properties of nanocrystalline $HfTiO_4$ gate insulator

Jaroslaw Domaradzki[1], Danuta Kaczmarek[1], Agnieszka Borkowska[1], Marek Wolcyrz[2], Bogdan Paszkiewicz[1]

1. *Wroclaw University of Technology, The Faculty of Microsystem Electronics and Photonic, Wrocław 50-372, Poland* **2.** *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland*

Contact: jaroslaw.domaradzki@pwr.wroc.pl

Microelectronics technology needs the use of a stable dielectric thin films for novel metal-oxide-semiconductor (MOS) applications. So far, as a gate insulators the typical dioxides (MO_2), where M = Si, Ti, Ru, Hf, have been used. Due to the fabrication of MOS devices, the structures should be characterized by low equivalent oxide thickness (EOT), bellow 1 nm. Such requirement led the need of preparation of either thin film dielectrics with large band offset (HfO_2) or very high permittivity (TiO_2). In the present work the results of electrical properties of mixed $Hf_xTi_{1-x}O_2$ solid solution thin films have been presented. The $HfTiO_4$ thin films, were grown on monocrystalline (100) oriented silicon substrates by the low pressure hot target reactive sputtering from the Ti:Hf (46.7:53.3) mosaic target. Appropriate conditions of deposition process assured almost pseudoepitaxial conditions of the layer growth what is testified by the stoichiometric composition of prepared thin films by the XPS investigations. The microstructure of the thin films was verified by X-ray and AFM examinations. The stable $HfTiO_4$ phase with the grain size in the range of few tenths of nanometers and the homogenous distribution of crystallites appeared over the whole sample surface of the thin films being investigated.

Electrical properties of prepared MOS structures were examined by means of C-V and I-V characterization. It have been shown, that the mixture of high-k TiO_2 and wide band gap HfO_2 dielectrics, gives the reasonable solution for fabrication of the thin films of insulator with intermediate properties.

15:50 poster F-12

Laser Composite Surfacing of Stainless Steel with SiC

Jyotsna Dutta Majumdar[1], B Ramesh Chandra[1], A. K. Nath[2], Indranil Manna[1]

1. *Indian Institute Of Technology,Kharagpur (IIT), Kharagpur, India* **2.** *Centre for Advanced Technology (CAT), Industrial CO_2 Laser Centre, Indore 452013, India*

Contact: jyotsna@metal.iitkgp.ernet.in

Stainless steels are widely used in chemical and petrochemical industries for its superior corrosion resistance and superior strength. However, failure of materials due to wear and erosion are often encountered in practical applications. Laser composite surfacing is a recent surface engineering approach that aims to improve the wear resistance by reinforcing ceramic powders in molten metallic surface with a high power laser. In the present study, laser composite surfacing of AISI 304 stainless steel with SiC has been carried out by predeposition of SiC powders (thickness of 100 mm) on metallic substrate and subsequently, melting it using a 2 kW continuous wave CO_2 laser. The main process variables for this study were laser power, scan speed, particle chemistry and powder deposition rate. Following laser processing, a detailed microstructural investigation was undertaken (using optical and scanning electron microscope) to study the influence of laser parameters on the thickness of the composite layer, volume fraction of the particles and its distribution with depth. The

average microhardness of the surface layer and its variation with depth was measured using a Vickers microhardness tester with 25 g applied load and correlated with laser parameters. The wear resistance of the composite surfaced Al were evaluated using a pin-on-disc wear-testing machine with 1 kg applied load against diamond indenter. The kinetics of wear was evaluated by measuring the weight loss due to wear at regular interval. Finally, the corrosion behavior of composite surfaced layer has been compared with that of as-received one in a 3.56 wt.% NaCl solution using a standard potentiodynamic polarization study. A superior microhardness and wear resistance was achieved in laser composite surfaced 304 stainless steel with negligible deterioration of corrosion property.

15:50 poster F-13

Quantum-chemical simulation of electrically active complexes at silicon grain boundaries

Alexander K. Fedotov[1], Anis Saad[2], Alexander L. Pushkarchuk[1], Alexander V. Mazanik[1], Kuten A. Semen[1]

1. *Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus* **2.** *2Al-Balqa Applied University, Salt, Jordan*

Contact: fedotov@bsu.by

The electrical activity or inactivity of contaminant-related deep centers at silicon grain boundaries (GBs) is very often connected with contaminating O- and/or C-atoms. In this work we analyse the tendencies in atomic and electronic structure of O- or/and C-containing complexes when contaminants were incorporated into Si tilt GB either separately or simultaneously. In the study we used a MM and MO LCAO method in PM3 approximation.

We have studied GBs containing SiOm or SinC complexes where m and n were between 1 and 4. It was shown that only incorporation of O-atoms between Si-atoms can form electrically active complexes with "chain"-like or "ring"-like configurations for m = 1-2 and SiO3 or SiO4 configurations for m = 3-4. C-containing electrically active complexes consisted of Si2C, Si3C and Si4C configurations, depending on number n of C-atoms inserted into 5- or 7-fold interstitial positions at GB "core". Both O- and C-containing electrically active complexes at GB "core" were donor-like giving energy levels that were shifted with increasing number of contaminating atoms from EC to EC – 0.6 eV for O-incorporation and from EC – 0.536 eV to EC – 0.043 eV for C-incorporation.

The 2nd stage of simulation consisted of incorporation of C-atoms into SiOm complexes. This resulted in formation of Si-O-C-Si chains and shifting of donor-like levels generated by SiOm configurations back to the bottom of conduction band EC with the increasing of number of incorporated C-atoms. Therefore, simultaneous incorporation of O- and C-atoms into GB "core" in some configurations of atoms can result in compensation of electrical activity of O-containing complexes in SiOm configurations at the GB "core".

15:50 poster F-14

X-ray high-resolution diffraction study of GaInAs and GaAsP grown by a modified Czochralski method

Jerzy Gronkowski, Grzegorz Kowalski, Tomasz Słupiński

Warsaw University, Institute of Experimental Physics (IEP UW), Hoza 69, Warszawa 00-681, Poland

Contact: Jerzy.Gronkowski@fuw.edu.pl

Growth of bulk ternary crystals based on $A^{III}B^{V}$ compounds with chemical compositions given by $A^{III}_{x}B^{III}_{1-x}C^{V}$ or $A^{III}B^{V}_{x}C^{V}_{1-x}$ is a difficult task due to the thermodynamical properties of their solutions. The basic new idea used for an improvement of the homogeneity of the bulk samples is to control their growth by a supersaturation of the liquid solution while maintaining a constant temperature, and not by cooling as for binary compounds.

We report preliminary results of x-ray investigations of $Ga_{1-x}In_{x}As$ and $GaAs_{1-x}P_{x}$ single crystals of sufficient parameter homogeneity for subsequent applications [1-3]. Our samples were studied using several methods: (i) high-resolution diffractometry which allowed to obtain both rocking curves and reciprocal-space maps of the diffracted and diffusely scattered intensity, (ii) diffraction plane-wave topography in the reflection mode. The components of alloy were added to the solution in the crucible prior to the growth. Due to component segregation, the homogeneity of ingots had to be characterised in different length scales by the above mentioned x-ray methods.

This work was supported by the State Committee of Scientific Research (grant No. 3T11B)39 26).

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15:50 poster F-15

X-ray diffuse scattering study of defects in α-sapphire

Jerzy Gronkowski, Elżbieta Zielińska-Rohozińska, Janusz Borowski

Warsaw University, Institute of Experimental Physics (IEP UW), Hoza 69, Warszawa 00-681, Poland

Contact: Jerzy.Gronkowski@fuw.edu.pl

Sapphire single crystals have been widely studied in the past few decades because this material is used on a large scale as substrate for the epitaxial technique, e.g. in the laser and light

emitting diode fabrication. Various dislocations with different Burgers vectors were studied by x-ray topography [1-3]. Theoretical and experimental results of x-ray topographical investigations of perfect dislocations were reported in [4]. In the present study 006 reflection $CuK_{\alpha 1}$ maps from both sides of α-Al_2O_3 substrate were taken in the triple-axis mode. The obtained isointensity contour shapes revealed features resembling those from dislocation loops in other materials. In order to identify the defects the isocontours were simulated using the double-force tensor approach [5] applied to hexagonal crystals [6, 7].

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15:50 poster F-16

INTERFACIAL REACTIONS OF TANTALUM THIN LAYER ON HIGH STEEL SUBSTRATE

Yazid Hadjar, Rachid Halimi

Université de Constantine, Laboratoire des Couches Minces et Interfaces, Route de Ain El-Bey, Constantine 25000, Algeria

Contact: hadjar_yazid@yahoo.fr

Interfacial reactions between a tantalum thin layer (4 µm thickness) and a high steel substrate (containing 1% of carbon) on vacuum annealing from 600 to 1100C were investigated by X- ray diffraction (XRD), secondary ion mass spectroscopy (SIMS), Auger electron spectroscopy (AES) and scanning electron microscopy (SEM). The product phases identified by X-ray diffraction are Ta_2C and TaC in a temperature range 800-900C. At T≥ 900C no more unreacted tantalum is detected and the stable phase TaC grows at the expense of the binary carbide Ta_2C. SIMS and AES analyses show that the adapted thermal annealing allows the diffusion of the carbon atoms from high steel substrate towards the thin layer of tantalum, and causes thereafter the transformation of this last into tantalum carbide. It is found, that the interfacial reaction of coating with steel substrate start at 600C, and the formed carbides grow from steel substrate to the free surface of coatings. Observation of surface and cross section morphology by SEM shows a good adhesion of formed coating layer. Finally, we discuss the mechanisms that may account for the considerable performance of these coatings.

15:50 poster F-17

A theoretical analysis of the electronic properties of tin dioxide surface

Weronika Izydorczyk[2], Boguslawa Adamowicz[1], Marcin Miczek[1], Krzysztof Waczynski[2], Jerzy Uljanow[2]

1. *Silesian University of Technology, Institute of Physics, Department of Applied Physics, Krzywoustego 2, Gliwice 44-100, Poland* **2.** *Silesian University of Technology, Institute of Electronics, Akademicka 16, Gliwice 44-100, Poland*

Contact: Weronika.Izydorczyk@polsl.pl

Tin dioxide is one of the most studied semiconductor metal oxides because of its various applications in gas sensors, solar cells, optical devices, transparent electrodes etc. due to interesting optical, electronic and thermal properties of SnO_2. In most of these structures the surface electronic status of SnO_2 layers plays crucial role. However, the standard models of electrical effects in SnO_2 layers are usually simplified and neglect the contribution of surface states to charge transport phenomena [1].

The aim of this work is a rigorous theoretical analysis of the influence of surface states on the conductance of SnO_2 layers with a thickness larger than the Debye length. In the calculations different continuous and discrete surface state distributions in the energy gap were assumed. From the one-dimensional numerical solution of the Poisson equation, the detailed in-depth profiles of the potential barrier V(x) and carrier concentrations n(x) and p(x) were obtained. Then, the values of surface potential, surface charge, Fermi level position and layer conductance were determined. These magnitudes were calculated for different bulk doping and carrier mobility. In addition, the surface fixed charge representing adsorbed ions or surface delta-doping was introduced to modify the band bending and layer conductance. Furthermore, the influence of temperature on near-surface region and conductance with assumption of non-mobile bulk donors was studied. The temperature dependence of the effective density of states, carriers mobility and concentration of donors was included into account for temperature dependent modeling. The results of theoretical analysis were compared with experimental data obtained from measurements of the SnO_2 sensor response to the changes of NO_2 concentration in synthetic air.

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15:50 poster F-18

Study of partial dislocations in wurtzite GaN using gradient elasticity

Joseph Kioseoglou[1], George P. Dimitrakopulos[1], Philomela Komninou[1], Iason Konstantopoulos[2], Elias C. Aifantis[2,3], Theodoros Karakostas[1]

1. *Dept. of Physics, Aristotle University of Thessaloniki, 54 124 Thessaloniki, Greece, Thessaloniki 54 124, Greece* **2.** *General Department of Physics and Mathematics, Polytechnic School, Aristotle University of Thessaloniki, GR-54124 Thessaloniki, Greece, Thessaloniki GR-54124, Greece* **3.** *Michigan Technological University, Houghton, MI 49931, USA, Houghton MI 49931, United States*

Contact: karakost@auth.gr

The gradient elasticity analytical solution for the dislocation energy can eliminate the core region singularities of elasticity theory. Such an approach is employed here in connection to previous empirical potential calculations of 1/6 <20-23> partial dislocations delineating the I1 intrinsic basal stacking fault in wurtzite GaN. Twelve stable configurations obtained for each of the two GaN polarities, their core radii, and their energies are considered. The gradient coefficients and the dislocations' parameters are evaluated and compared with those obtained with elasticity calculations. The gradient solution is found to interpret accurately the dislocation energies calculated by the empirical potential within the core region.

15:50 poster F-19

Study of interfacial processes between tungsten thin layer and steel substrate

Fares Khalfallah, Yazid Hadjar, Rachid Halimi

Laboratoire des Couches Minces et Interfaces, Route de Ain El-Bey, Constantine 25000, Algeria

Contact: fa_fares@yahoo.fr

Material parts that are required to perform under severe environmental conditions often have to display high hardness and wear, erosion and corrosion resistance. An effective and economical way to meet these requirements is by creating surface coatings that possess the needed properties. Carbides, particularly of the transition metals, have a number of valuable properties, which make them the most promising materials for use in various new fields of technology. Tungsten carbide layers deposited by various techniques appear suitable candidates to be used as resistant and protective coatings.

In this work, we have formed thin hard coatings of tungsten carbides. The samples are thin layers of Tungsten deposited by cathodic sputtering on two types of steel (containing 1 and 0,7% wt. carbon) substrates held at temperature of 500C during 30 min. The samples (W layer + substrate) were submitted to thermal annealing in vacuum, at various temperatures (500-1000C) and during different times.

The formation of tungsten carbides, the evolution of the microstructure and the morphology of the surface of samples were followed by X-rays diffraction (XRD), scanning electron microscopy (SEM). The measurements of micro-hardness were carried out by Vickers tests.

Thermal annealing produces reactions and some other structural changes in the tungsten layers which depend on the carbon content in substrate. It is established that the formation of tungsten carbides occurs above annealing at 800C. The reaction is more rapid on substrate containing 1%C. The monocarbide WC forms after annealing of samples at 1000C. The hardness of coatings was found to be correlated to their composition. The hardness of coatings increases continuously from 400 to 1200 Kg/mm^2 as the temperature of annealing increases from 500 to 900C. The samples rich on carbon show higher hardness.

15:50 poster F-20

Nanocrystalline nickel surface layers produced electrochemically and their corrosion resistance

Magdalena Kowalewska, Maria Trzaska

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: mk@inmat.pw.edu.pl

Nickel surface layers were produced by the method of electro-chemical reduction, using a basic bath of the Watts type and a basic bath modified with D1 organic substances. The process was carried out at a constant cathodic current density equal to $3A/dm^2$. The effect of the organic component on the topography, structure and morphology of the layers was determined. Examinations of corrosion resistance of produced layers were carried out. It has been found that by using the Watts bath modified with an organic additive, we can reduce the surface roughness of the electro-plated nickel layers, refine their structure for nanometric scale of grain size and change the shape of their grain boundaries. It was determined that the nanocrystalline nickel layers exhibit smaller corrosive resistance than the nickel layers about the microcrystalline structure.

15:50 poster F-21

THE PHOTON PRESSURE EFFECT AND CHANGE OF MAGNETIC-OPTIC CHARACTERISTICS OF MULTILAYERED NANOFILMS

Mykola M. Krupa, Yurii B. Skyrta

Institute of Magnetism NASU (IMag), Vernadsky 36-b, Kyiv 03142, Ukraine

Contact: krupa@imag.kiev.ua

The results of the research of dependence of magnetic-optic characteristics multilayered nanofilms Bi-SiC-TbFe-SiC, Ti-SiC-TbFe-SiC, SiC-TbFe-SiC and Tb-Au-Fe on the power and direction of fall of the nanosecond pulses of laser radiation on film structure are presented. It is revealed that the value of Kerr and Faraday angle in films Bi-SiC-TbFe-SiC, Ti-SiC-TbFe-Si and Tb-Au-Fe changes differently. Character and value of these changes depend not only on the intensity and duration of laser pulses, but also on the structure of a film as well as on the direction photon flux fall on the film structure and the direction of an external magnetic field.

The analysis of the results allows show that the obtained changes of the magnetic-optic characteristics are connected with the contribution of nonequilibrium electrons with a high spin polarization. Under the action of pulses of the laser these electrons, due to photon pressure, go from a magnetic layer into a nonmagnetic layer of the film structure and magnetize them locally, which causes an additional turn of the plane of polarization of the light and change its magnetic-optic characteristics.

Experimental dependencies on the intensity and duration of pulses, on the direction of fall of the nanosecond pulses of laser radiation on the film structure and the direction of an external magnetic field will be well coordinated with the described physical model, which stresses the necessity of taking into account the effect of photon pressure of light while investigating the interaction of laser radiation with multilayered nanofilms structures.

15:50 poster F-23

Evaluation of average domain size and microstrain in silicides by the Williamson-Hall method

Dmitry Mogilyanski[1], Joushua Pelleg[2], E Elish[2]

1. *Institutes for Applied Research, Ben Gurion University of the Negev, Beer-Sheva 84105, Israel* **2.** *Department of Materials Engineering, Ben Gurion University of the Negev, Beer-Sheva 84105, Israel*

Contact: mogily@bgu.ac.il

The structure of chemical vapor deposited (CVD) WSi2 in a multilayered configuration of Si/SiO2/poly-Si/WSi2/poly-Si was analyzed by X-ray diffraction (XRD). Individual layers of this multilayered structure were deposited sequentially on separate wafers and the changes occurring in WSi2 at each stage of the fabrication process were determined by XRD in the as deposited and annealed conditions. XRD line broadening was used to construct the Williamson-Hall (WH) plots. As a first approach to get preparatory information reflections from all the planes observed by the XRD analysis were used to evaluate by the WH technique the mean domain size and the microstrain, respectively. Next, planes of crystallographic families of WSi2 were applied for two cases, a partially and fully crystallized multilayered films, to construct WH plots. When the film is well annealed with small or no anisotropy using planes of a family and all the plains to evaluate the strain and the domain size will provide almost the same values. This technique is used for the first time to illustrate that the WH method can be applied for simultaneous grain size and microstrain evaluation for silicide films also.

15:50 poster F-24

Effect of annealing treatment to improve TiC coating surface properties

Lazhar Mohammedi[1], Rachid Gheriani[1], Rachid Halimi[2]

1. *Laboratory of Materials Physic. University of Ouargla (LMP), OUargla, Ouargla 30000, Algeria* **2.** *Université de Constantine, Laboratoire des Couches Minces et Interfaces, Laboratoire des Couches Minces et Interfaces, Route de Ain El-Bey, Constantine 25000, Algeria*

Contact: mohammedi_lazhar@yahoo.fr

The improvement of physical and mechnical surface proprerties takes a good place today. In the present work we prepared TiC coating by indirect method, using magnetron sputtering. The titanium is deposited on high carbon steel substrates (X210Cr12), and followed by annealing in vaccum from 400C to 1000C to transform titanium to titanium carbide coating. The formation of TiC coating and the interdiffusion of C, Fe and Ti between the coating and substrate were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Auger electron spectroscopy (AES) and hardness measurements. XRD reveals the formation of TiC at 700C. SEM and AES show the interdiffusion of C, Fe and Ti at high temperatures. It was noted that the formation of TiC coating is consistent with the high value of hardness which is larger than the bulk value.

Key words: thin films, hard coatings, titanium carbide, interdiffusion, titanium

15:50 poster F-25

Oscillation phenomenon of transition temperatures in Fe/V/Fe superlattice

Hossein Moradi

Ferdowsi University of Mashhad, Azadi Square, Mashhad, Iran

Contact: moradi@ferdowsi.um.ac.ir

Many interesting physical properties of multilayer consisting of ferromagnetic films separated by nonmagnetic metallic spacers arise because of the coupling between the ferromagnetic layers. This coupling is mediated by the conduction electron in the spacer layers and oscillates with spacer thickness. The coupled system will have either ferromagnetic or staggered order depending on the sign of the interlayer coup-

ling. The transition temperatures of these systems exhibit an oscillatory dependence on the spacer thickness and have a period of oscillation of half the period of the interlayer exchange coupling [1]. If there are fluctuations in the interlayer coupling caused by variations in the spacer thickness then each plane will experience a random magnetic field caused by combination of the strong correlation in the neighboring planes and the random exchange. This led to a theory, which predicted that the ordering temperature of multilayers thin films, may be less than the Curie temperature of an isolated plane [2].

In this paper, Curie temperature of an Ising plane in the presence of a random field is calculated in the mean field theory for a two dimensional lattice. The expressions for the susceptibility are used to obtain the transition temperature of coupled planes. It is shown that the results are fitted to experimental results and estimate the second AFM coupling peak in Fe/V/Fe superlattice. This indicates that the unusual oscillation behavior of the transition temperature as a function of spacer layer thickness is closely related to the interlayer exchange coupling in magnetic multilayers.

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15:50 poster F-26

Formation of Aluminum Silicate Film on Steel in Granite-Copper-Dry steam Reaction System

Yongcheng Jin[1], Inna R. Korablova[2], Yoshihiko Oke[2], Nakamichi Yamasaki[2]

1. *Graduate School of Engineering, Tohoku University, Sendai, Japan* **2.** *Graduate School of Environmental Studies, Tohoku University, Sendai, Japan*

Contact: yosshy@mail.kankyo.tohoku.ac.jp

We have observed that a highly protective amorphous aluminum silicate layer on steel surface was formed after granite-dry steam interaction in the presence of copper plate near critical point. In this process, the mass transfer of aluminum and silicon species from the granite to the steel surface takes place after copper is transferred to the granite surface. The rate of this process is accelerated by the presence of copper on the granite surface. In previous studies, we have found that the mass transfer of copper occurred due to copper-dry steam interaction in the presence of O_2. Finally, formed Cu_2O was transferred through dry steam as $Cu_2O{\bullet}nH_2O$

At present, we determined the effect of transferred copper species on the weight loss of granite in dry steam with different ratio O_2 and N_2. It was found also that transferred copper species at lower O_2 level promote the weight loss of granite. Four types of experiments were conducted at low O_2 ratio in different density steam: 1) steel-steam, 2) steel-Cu-steam, 3) steel-granite-steam, 4) steel-granite-Cu-steam. It was established that an aluminum silicate film formed on steel surface only at the steel-granite-Cu-dry steam interaction. The formed film was characterized by X-ray photoelectron spectroscopy(XPS), and scanning electron microscopy (SEM) equipped with an energy dispersive X-ray (EDX).

15:50 poster F-27

Transport properties of the two-dimensional electron gas in GaN/AlGaN heterostructures grown by ammonia molecular-beam epitaxy

A J. Nikitin, A G. Pogosov, A V. Budantsev, R A. Lavrov, V V. Preobrazhenskii, V G. Mansurov, K S. Zhuravlev

Institute of Semiconductor Physics SB RAS, Lavrentjeva 13, Novosibirsk 630090, Russian Federation

Contact: aj-nik@yandex.ru

Electron transport properties and weak localization of the two-dimensional electron gas (2DEG) in AlGaN/GaN heterostructures were experimentally investigated. Samples with the $Al_{0.35}GaN_{0.65}$/GaN heterojunction were grown by ammonia molecular-beam epitaxy (MBE) on sapphire substrates under nitrogen-rich conditions. Electron concentration and mobility in 2DEG under the temperature 4.2 K were $n_s = 1.7x10^{13}$ cm^{-2} and $\mu = 2\ 000$ cm^2/Vs, respectively.

At the temperature of 4.2 K the $\delta\sigma(B)$ experimental dependence the $\delta\sigma(B)$ is described quite well by the quantum correction to the 2D system conductivity in the approximation of the effective length of phase coherence l_φ^*: $\delta\sigma(B)=(\alpha e^2/2\pi^2\hbar)[\Psi(1/2+\hbar/2Bel_\varphi^{*2})-\Psi(1/2+\hbar/2Bel^2-2\ln(l_\varphi^*/l)]$, where l is the transport free path, α is the system geometry parameter, $\Psi(x)$ is digamma function.

However, at the temperature of 1.8 K it is impossible to approximate the experimental dependence $\delta\sigma(B)$ in the measured magnetic field range by the above dependence with only one adjustable parameter l_φ^*. In weak magnetic fields, when $l_B=(h/2\pi eB)^{1/2} >> l_\varphi=(2D\tau_\varphi)^{1/2}$, the conductivity correction is $\sim l_\varphi^4/l_B^4$. When l_B is about l_φ, this correction is $\sim \ln(l_\varphi/l_B)$. So for two conductivity subbands the effective length of phase coherence is $l_\varphi^{*i}=((l_{\varphi1}^*)^4+(l_{\varphi2}^*)^4))^{1/4}$ in weak magnetic fields and $l_\varphi^{*ii}=(l_{\varphi1}^* l_{\varphi2}^*)^{1/2}$ in large magnetic fields.

The fact that $\delta\sigma(B)$ is well described by only one effective length at the high temperature can be explained by the inter subband scattering which results in averaging of the effective lengths.

The work is supported by RFBR (grants nos. 05-02-17259a, 05-02-16901-a)

15:50 poster F-28

Defect structure of silicon crystals implanted with nitrogen - a study of Si:N annealed under high hydrostatic pressure.

Kamila Orlinska[1], Adam Adikimenakis[2], Patrik Vagovic[3], Andrzej Misiuk[4]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Physics Department, FORTH/IESL and University of Crete, Greece, Greece* **3.** *Slovak Academy of Sciences, Institute of Electrical Engineering (IEE SAS), Dubravska cesta 9, Bratislava 841 04, Slovakia (Slovak Rep.)* **4.** *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland*

Contact: orlin@ifpan.edupl

Implantation - induced amorphisation near projected range of implanted ions (Rp) in implanted semiconductors is accompanied by changed volume and density of the host matrix and by swelling of the damaged area.

The formation of buried insulating layer inside Si wafers by oxygen implantation and subsequent annealing at high temperature is widely used in silicon-on-insulator (SIMOX) technology [1, 2]. Similar structures can be possibly prepared by implantation of nitrogen, so Si:N deserves investigation in this respect. Enhanced hydrostatic pressure of ambient gas at annealing of Cz-Si:N significantly influences its properties [3]. In this work we investigate the effect of high pressure (HP) – high temperature (HT) treatment on the defect structure of Si:N prepared by nitrogen implantation into Cz-Si, with nitrogen doses of $1x10^{17}$ and $1x10^{18}$ cm^{-2}.

After implantation, the samples were HT - HP treated at up to 1400 K under hydrostatic Ar pressure up to 1.1 GPa for 5 hours. The structural properties of Si:N, before and after the HP-HT treatment, were investigated in the double and triple axis configurations, using the high-resolution X-ray X'Pert Philips diffractometer. Enhanced hydrostatic pressure of ambient gas at annealing of Si:N results in the strain modification and/or in defect structure changes dependent on pressure and temperature during the treatment. In the HT - HP treated samples the changed strain and defect structure were detected by determination of the FWHM values and of diffuse scattering intensity. Explanation of the HT - HP effect on the microstructure of treated Cz-Si:N will be proposed.

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15:50 poster F-29

INVESTIGATION OF Si WIRE TRANSMISSION

I. P. Ostrovskii[1], Anatoly A. Druzhinin[1], Alla I. Klimovskaya[2], Evgenij G. Gule[2]

1. *Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine* **2.** *Institute of Semiconductor Physics, NAS of Ukraine, Kiev, Ukraine (ISP), 45 pr. Nauki, Kyiv 03028, Ukraine*

Contact: iostrov@polynet.lviv.ua

Si wires are prospective for size effect studies. Recently we have found the row of size effects in the wires. Among them there is decrease of lattice parameter in submicron Si wires as comparison with lattice parameter of silicon bulk. In addition visible light photoluminescence is observed in such structures similar with luminescence of porous silicon. Such peculiarities of the wires were explained by peculiarities of their structure. The investigation of crystal structure showed that the wires are heterostructures consisted of Si strained core and porous Si envelope.

In the present paper we study transmission spectra of Si wire array prepared on Si substrate.

The growth method consists in regular deposition of Au dots on Si substrate. Then Si substrate is loaded in a reactor of open vapour-transport system. Thermoprocessing of the reactor at 1100K provides the wires growth from Si-Au droplets according to VLS mechanism. The results of investigation of Si wire structure in AFM have shown that thickness of envelope is of about 5-20 nm depending on the wire diameter (500-5000 nm).

Transmission spectra of wires were measured by use of He-Ne laser with 632,8 nm light-wave length. The prominent performance of the spectra is their strong angle dependence, which we detect using goniometer system. The angle dependence of transmission would be explained by light diffraction on transversive dimensions of the wires. But we have performed the investigation using IR source of light and obtained the same angle figures. Therefore, the revealed peculiarities of transmission are connected with other reason.

Nanosilicon is known to be a crystal with strong birefringence. One can suppose that porous envelope of Si wires is corresponding for angle dependence of light transmission due to appearance of light birefringence. The peculiarities of angle figures depending on the wires geomentry are discussed.

15:50 poster F-30

Raman spectra of misfit layer semiconductor structure (PbS)0.59TiS2 under variation of laser power

Sergey V. Ovsyannikov[1], Vladimir V. Shchennikov[1], Andres Cantarero[2], Ana Cros[2], Alexander N. Titov[1]

1. *Institute of Metal Physics, Urals Division of Russian Academy of Sciences, Kovalevskaya 18, Yekaterinburg 620219, Russian Federation* **2.** *Materials Science Institute, Valencia University, Valencia, Spain*

Contact: sergey_v_o@imp.uran.ru

In the present work we studied the variation of Raman spectra in misfit layer semiconductor structure $(PbS)_{0.59}TiS_2$ under variation of laser power. The spectra were excited with 514.5 and 488.0 nm laser lines. At the low laser powers the spectra exhibited: (i) two softened (in comparison with TiS_2 crystal) phonons of TiS_2 host layers at 219 and 333 cm^{-1}; (ii) LO(Γ), 2LO and 3LO phonons from the incommensurate PbS layers at 203.5, 412 and 634 cm^{-1}, respectively; and (iii) a possible superposition mode of PbS and TiS_2 layers at 292 cm^{-1}. A process of the misfit's surface oxidation was investigated under increase of both laser power and time of exposition of the samples under the laser irradiation. The novel wave numbers were observed related to PbO, TiO_2, PbO_2, $PbSO_4$, "PbO*$PbSO_4$" complexes, and to other oxysulfates at ~ 140, 183, 254, 280, 364, 391, 434, 511, 605 cm^{-1}. A nature is discussed of a peak at 151 cm^{-1} appearing at high laser powers. In the low-frequency Raman spectra of the misfit (PbS)0.59TiS2 the peculiarities at 19, 73 and 95-96 cm-1 were observed associated with vibrations of the PbS layers.

The work was supported by the INTAS (Nr. 03-55-629) and RFBR (Gr. No. 04-02-16178).

15:50 poster F-31

Surface modification/oxidation of GaAs in electrolytes for cell-cultivating bio-sensing devices

Kazunari Ozasa[1,2], Shigeyuki NEMOTO[1], Masahiko HARA[1], Mizuo MAEDA[1]

1. *Institute of Physical and Chemical Research (RIKEN), 2-1 Hirosawa, Wako-Shi, Saitama 351-0198, Japan* **2.** *CREST-JST, 3-1-6-5 Shibuya, Tokyo 150-0002, Japan*

Contact: ozasa@riken.jp

The surface modification/oxidation of GaAs substrates in electrolytes has been investigated from the viewpoint of bio-sensing devices using AlGaAs/GaAs FET structures, where the cultivation of T2 cells directly on a bare gate-surface is intended for a high sensitivity to cell-activities such as adhesion or cell-cell interaction. The stability of GaAs surface (gate surface of FET) in DMEM solution (electrolyte for cell cultivation) is essential and must be elucidated in order to realize such cell-sensor devices, especially when gate-bias voltage is applied through the electrolyte.

When n+ GaAs(001) substrate was immersed in deionized pure water (37°C) for 24h under 17 μW VIS-light, the oxide layer of 70-80 nm in thickness was formed on the surface. For the immersion in the dark, however, the thickness was as small as 10-20 nm, indicating that the photo-excited electron-hole pairs cause the surface oxidation. Moreover, we have found that NaH2PO4 is effective to suppress the photo-induced oxidation of GaAs. The oxide thickness of 6-8 nm was obtained by 24h immersion in 0.8 mM NaH_2PO_4 solution, even under VIS-light. The suppression mechanism we consider is that the mid gap states of GaAs surface are modulated by H_2PO_4 fragments, and as a result, the surface-band bending prevents the photo-excited holes from diffusing to the surface.

The application of gate-bias voltage (Vgs) through DMEM solution enhances the degradation of bare-gate-FET performance. Based on the above findings, we have succeeded in suppressing the degradation adequately by shielding the gate area from VIS light, but not by NaH_2PO_4. The decrease in Ids (Vgs = 0.5 V) was 14% for 10h operation in the dark, while it was 98% under VIS light.

This study clearly shows that the cells can be cultivated on bare GaAs surfaces in DMEM solution (containing 0.8 mM NaH_2PO_4), and adequate FET performance is maintained in the dark for cell-activity measurements.

15:50 poster F-32

Weak ferromagnetism in CoO and exchange biasing of CoPt in core-shell nanoparticles

Aphrodite Tomou[1], Dimitrios Gournis[1], B Kooi[2], Ioannis Panagiotopoulos[1]

1. *Department of Materials Science and technology, University of Ioannina, Ioannina 45110, Greece* **2.** *Materials Science Centre, University of Groningen,, Nijenborgh 4, Groningen 9747 AG, Netherlands*

Contact: ipanagio@cc.uoi.gr

CoO and CoPt/CoO core-shell nanoparticles with size around 9 nm were prepared by solution phase chemical synthesis involving simultaneous reduction of cobalt acetate and platinum acetylacetonate. This method is known to lead to a narrow particle size distribution due to the use of stabilizing agents as oleic acid and oleyl amine creating an organic residue that surrounds and separates the nanoparticles. The microstructure has been studied by Transmission Electron Microscopy (TEM). The blocking temperature of the particles is considerably lower than the Néel temperature of CoO. Magnetic measurements show appearance of coercivity and significant exchange biasing (loop shift of Heb=1125 Oe) in the CoPt/

CoO composites at temperatures below 50 K, as a result of exchange coupling between CoO and CoPt. The antiferromagnetic CoO nanoparticles on the other hand, when field cooled to temperatures below 200K, show displacement of the magnetization vs field curves (along the magnetization axis) characteristic of weak ferromagnetism phenomena, that are attributed to the uncompensated surface magnetic moments. The temperature dependencies of the two phenomena are compared and discussed within recent models that stress on the correlation of unidirectional anisotropy with the pinned surface moments as opposed to the total uncompensated surface moment of CoO.

15:50 poster F-33

Simulation of grain boundaries resistance in two-phase thin films of lanthanum manganites

Vytautas Petrauskas, Evaldas E. Tornau

Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania

Contact: vytas@pfi.lt

We present the model describing temperature and magnetic field dependences of grain boundary (GB) resistivity and magnetoresistivity of 2-phase epitaxial and/or polycrystalline films of lanthanum manganites. Assuming film thickness d dependence on concentration of both phases, we calculate by mean field approximation the magnetization of the 2-phase system and its dependence on d. Introducing the concept of grains, we obtain the difference in neighboring grains magnetization and link it to the GB resistivity by the phenomenological formulae. Characteristic features of temperature dependences of resistivity of lanthanum manganite films (peak of resistivity maximum T_m, difference of T_m and Curie temperature T_c, high resistivity of ultrathin films and shift of T_m with increasing d to higher temperature) might be found studying thus obtained GB resistivity. Our calculations show that, in order to obtain the peak at T_m, both grain boundary layers have to be, at least, weakly ferromagnetic. Increase of the ferromagneticity of these layers, leads to decrease of GB resistivity, increase of T_m and decrease of the difference between T_m and T_c. Increase in the number of grain boundaries also leads to decrease of GB resistivity. Our model allows to describe the difference observed in temperature dependences of resistivity for very thin (strained) films and thicker films with the electric and magnetic properties of the bulk systems.

15:50 poster F-34

STRUCTURAL AND OPTICAL PROPERTIERS OF IRON SILICIDE FORMED BY ION BEAM MIXING

Ayache Rachid[1], A. Bouabellou[2], F. Eichhorn[3], E. Richter[3]

1. *University of Batna, Pharmacy Department, Batna 05000, Algeria* **2.** *Université de Constantine, Laboratoire des Couches Minces et Interfaces, Route de Ain El-Bey, Constantine 25000, Algeria* **3.** *Forschungszentrum Rossendorf (FZR), Dresden 01314, Germany*

Contact: r_ayache@excite.com

Fe films of 150 nm deposited onto P-type Si(111) substrates were irradiated at room temperature (RT) with 300 KeV Xe^+ ions with a dose of $5x10^{16}$ Xe^+/cm^2. After Xe-ion beam mixing, the samples were annealed in a N_2 atmosphere at 850 ˚C for different times. The obtained samples were analyzed by using Rutherford backscattering spectrometry (RBS), X-ray diffraction (XRD), Raman spectroscopy and Photoluminescence (PL) techniques. The experimental results show that the Xe irradiation at RT leads to the formation only of the ε -FeSi phase, and after thermal annealing the Fe layer is transformed to a mixture of ε -FeSi, A-$FeSi_2$ and β -$FeSi_2$ phases. The Raman signals for β -$FeSi_2$ are slightly shifted toward lower energies. A PL measurement at 12 K indicates a luminescence peak localized at the energy of about 0.81 eV corresponding to the band gap energy of β -$FeSi_2$ phase.

15:50 poster F-35

NANOMETRIC SEMICONDUCTOR PARTICLES IN MESOPOROUS SILICA: SYNTHESIS AND CHARACTERIZATION

Maria Luisa Saladino, Delia Chillura Martino, Eugenio Caponetti, Lucia Pedone

Università di Palermo (UNIPA), Viale delle Scienze, Pallermo 90128, Italy

Contact: saladinoluisa@libero.it

The synthesis and characterization of mesoporous silica (MCM41) containing semiconductor (CdS) nanoparticles is reported. MCM41 was synthesised using CTAB micelles as template [1]; its structure was determined by X-ray diffraction (XRD). CdS nanoparticles were synthesised using a water in oil microemulsion as reaction media [2]; their structure was determined by X-ray diffraction (XRD) and transmission electron microscopy.

The heptane containing CdS-amine capped nanoparticles was added to the porous silics; CdS nanoparticles spontaneously localized inside MCM41 pores giving the. The obtained hierarchical material was characterised using IR, UV-Vis, ATR, fluorescence spectroscopies and XRD and Small Angle Neut-

ron Scattering (SANS) techniques. The spectroscopic investigation gave the size, polidispersity, surface and morphological properties of nanoparticles. SANS and XRD investigation gave structural information such as mesoporous structure, pore size and nanoparticles localization.

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15:50 poster F-36

Surface morphology and IR optical properties modification of Co-based amorphous metallic alloys during initial stages of crystallization

Kateryna L. Vinnichenko[1], Mykola V. Vinnichenko[1,2], Leonid V. Poperenko[1]

1. *National Kyiv Taras Shevchenko University (NKTSU), Volodymyrska 64, Kyiv 01033, Ukraine* **2.** *Forschungszentrum Rossendorf (FZR), Dresden 01314, Germany*

Contact: kvin@univ.kiev.ua

The ribbons of $Co_{59}Fe_5Ni_{10}Si_{11}B_{15}$ (at.%) AMA, prepared by a rapid quenching using a melt spinning technique, were annealed in vacuum for 10 min at T_a =350, 400, 425, 450 and 475 C. The crystallization temperature is known from the literature to be around 490 C. The modification of a surface morphology of the AMA due to the treatment is investigated by atomic force microscopy (AFM). The AFM data were contrasted with a plan-view transmission electron microscopy (TEM) and Fourier-transform infrared spectroscopic ellipsometry (FTIR-SE) results. In the as-prepared state the ribbons have smooth surface with a root-mean square (RMS) roughness around 1 nm at the area of 10 μ m x 10 μ m. TEM shows the homogeneous amorphous microstructure and amorphous halo in electron diffraction. Annealing at 350 C leads to a formation of 5-13 nm high and 100-1000 nm wide isolated features at the ribbon surface. In the subsurface layer of the annealed ribbon TEM shows ellipsoidal nanocrystalline inclusions with a characteristic size within the ~15-80 nm range in the field of vision. Using the TEM data, the observed AFM surface features are interpreted as the crystalline nuclei which grow with annealing temperature. Because the features are larger at the ribbon surface than in the subsurface area, it evidences that the crystallization starts from the surface. The RMS values strongly increase at T_a =475 C and its dependence on the Ta can not be described by exponential laws. It points that classical nucleation and growth theory fails and the AMA crystallization can be described using only concomitant theory accounting for a system disorder [1]. The optical conductivity spectral behavior changes from non-Drude-like to Drude-like at T_a =475 C where the ribbon seems to have the highest content of crystallites. It points to the conduction mechanism change due to a film morphology modification.

[1] A.B. Pevtsov, et al, Phys Rev. B. 52 (1995) 955.

15:50 poster F-37

Studies of gas sensing, electrical and chemical properties of n-InP epitaxial surfaces

Katarzyna WIERZBOWSKA[2], Boguslawa Adamowicz[1], Marcin Miczek[1], L. Mazet[2], J. Brunet[2], A. Pauly[2], L. Bideux[2]

1. *Silesian University of Technology, Institute of Physics, Department of Applied Physics, Krzywoustego 2, Gliwice 44-100, Poland* **2.** *LASMEA, Universite Blaise Pascal, Clermont-Ferrand, France*

Contact: Katarzyna.WIERZBOWSKA@lasmea.univ-bpclermont.fr

InP-based multilayer structures are largely applied in fabrication of heterojunction transistors and optoelectronic devices. Recently, our experimental and theoretical studies of InP epitaxial layer behaviour in gas atmosphere showed the large sensitivity of the layer resistance and surface Fermi level position to adsorption of ionic species and, on the other hand, marked stability of the initial resistance in pure air versus time [1, 2]. These properties allowed the construction of a novel high-sensitivity InP based gas sensor for toxic gas detection working in the ppb range [1]. The growing interest in such kind of sensing structures, easily integrated with other III-V devices, requires further optimisation of their parameters. In this order, systematic research of sensor electronic and chemical properties, in particular focused on a surface and near-surface region are necessary.

In this paper, the resistance response of gas sensing n-InP epitaxial layer based structures was examined under oxidising gas (NO_2) exposition, at temperature of 80 and 100^0 C. The gas concentration varied in a very low range from 20 to 100 ppb. The stabilization of the layer initial resistance and evolution of sensor response due to cycles of adsorbed gases was systematically investigated. The experiments were carried out for different thickness of InP layers (in the submicron range) to reveal the influence of the depletion layer on the gas sensitivity. The experimental data were compared with the results of numerical analysis of the influence of surface states and near-surface region on the relative resistance changes upon ion adsorption. In addition, the spectroscopic studies, using XPS technique, of the chemical properties of InP surfaces before and after gas submission were performed.

[1] L. Talazac et al., Sensors and Actuators B 76 (2001) 258

[2] B. Adamowicz et al., Thin Solid Films 436 (2003) 101

15:50 poster F-38

Investigation of insulated buried layers obtained by ion implantation in AlGaAs with various Al concentration

Wojciech Wierzchowski[1], Krzysztof Wieteska[2], Walter Graeff[3], Grzegorz Gawlik[4], Andrzej Turos[1,5], Arndt Mucklich[6]

1. *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **2.** *Institute of Atomic Energy, Otwock-Świerk 05400, Poland* **3.** *Hamburger Synchrotronstrahlungslabor HASYLAB (HASYLAB), Notkestrasse 85, Hamburg D-22603, Germany* **4.** *Institute of Electronic Materials Technology (ITME), Warszawa 01919, Poland* **5.** *Soltan Institute for Nuclear Studies, Hoża 69, Warszawa 00-681, Poland* **6.** *Forschungszentrum Rossendorf (FZR), Dresden 01314, Germany*

Contact: wierzc_w@sp.itme.edu.pl

The important element of modern $Al_xGa_{1-x}As$ semiconductor lasers is an insulating buried layer introduced by selective implantation with He or H ions. The difficulty in obtaining of such layers is connected with controlling of strain and defects introduced by implantation, which may disturb the action of the laser. The strain may be however controlled in less complicated laterally homogenous structures or even single implanted layer.

The insulated buried layers obtained by the implantatation of 150 keV He ions to $Al_xGa_{1-x}As$ with various concentration of Al and GaAs were studied with different synchrotron diffraction methods exploring both white and monochromatic beam. The selected samples were studied with high resolution transmission electron microscopy. The implantation were performed in from room temperature, 80º C and 120º C. The doses were in the range from 2×10^{16} to 6×10^{16} cm^{-2}. The synchrotron experiment included taking local rocking curves using small 50 x 50 μm^2 probe beam. The rocking curves exhibited characteristic sequence of interference maxima and enabled the analysis of the strain profiles by fitting the theoretical rocking curves obtained by numerical integration of the Takagi-Taupin equations. The white beam synchrotron back reflection topography revealed a sequence of strain modulation fringes similar to the main interference maxima in the rocking curves. They also confirm the uniformity of applied dose and lack of extended crystallographic defects which could be caused by implantation. The characteristic feature of the evaluated profiles was the existence of the deformed region close to the surface which points that the deformation is mainly caused by the point defects produced by incident ions and the recoils. The other feature increasing with the temperature of implantation was the flattening of top part of the strain maximum corresponding to the insulating buried layer. This flattening was more distinct for

[ABSTRACT TRUNCATED TO 2000 LETTERS]

15:50 poster F-39

Discrete State Analysis for Elaboration of the Steering Model with Properties of Nanostructure

Mariusz A. Wojcik

AGH University of Science and Technology, Department of Biomaterials (AGH-UST), Al. Mickiewicza 30, Kraków 30-059, Poland

Contact: wojmar@uci.agh.edu.pl

Literature data shows that crystallisation processes of manufacturing of the various modification of aluminium oxides are not sufficiently recognised when considering the modelling and steering of different nanostructural forms. An explanation of a mechanism of a formation of characteristic not differentiated ceramic nanostructures such as peak, ridge or dendrites crystals having searched properties was presented in paper.

The inspection of the crystalline structure proofs that we usually have a contact with not smooth objects. The typical and analytical description does not reveal of the singularity structure. In relation to this the operation on the formalism leading to the description of such singularity structure can make same progress in the analysis of nanostructures as well as in steering of their construction. Author, assuming and considering conclusions resulting from Boltzman's equations and analysing suitable energetic functional, obtains the description of the structure, which can be constructed by superposition of the elementary crystalline movements passing on not differentiated trajectories. It response on a visible crystallographic structures.

Analysing of the energy functional a formal solution has been developed which shows that its extremes described not differentiated structures. Such approach, not known so far in materials engineering, explains the formation of the mentioned crystalline nanostructures and can bring a practical meaning in research works on nanostructures in the material engineering as well as in metallography or foundry.

15:50 poster F-40

First principles study of electronic structure of InN and AlN substitution atomic layers embedded in GaN

Abdelaziz Lakdja[1], Bachir Bouhafs[3,5], Pierre Ruterana[2,4]

1. *Modelling and Simulation in Materials Science Laboratory (MSMSL), University of Sidi Bel-Abbes, Sidi Bel-Abbes 22000, Algeria* **2.** *Laboratoire CRISMAT - UMR 6508, ISMRA et Universite de Caen, 6 Boulevard de Marechal JUIN, Caen 14050, France* **3.** *Abdus-Salam International Center for Theoretical Physics (ICTP), strada costiera, 11, Trieste 34014, Italy* **4.** *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **5.** *Modelling and Simulation in Materials Science Laboratory (MSMSL), University of Sidi Bel-Abbes, Sidi Bel-Abbes 22000, Algeria*

Contact: alakdja@univ-sba.dz

The electronic structures of an embedded layer in GaN are investigated by means of the first-principles full-potential linearized augmented plane waves method within the local density approximation (LDA). A supercell approach with periodic boundaries is used to study such systems. It is shown that InN and AlN substitution atomic layers embedded in GaN modifies the localization of electronic charge distribution in the valence band maximum (VBM) and in the conduction band minimum (CBM) of InN/GaN and AlN/GaN systems. We discuss also the change of the band gap which depends on the depth of the substitution layer in GaN.

15:50 poster F-42

Raman spectroscopy and morphology investigation of porous GaAs

Valeriy V. Kidalov[1], George A. Suckach[2], Lotfy Beji[3], Andrey S. Revenko[1], Artem D. Baida[1], Yuri Yatsenko[1]

1. *Berdyansk State University (BDPU), Shmidt, Berdyansk 71100, Ukraine* **2.** *Institute for Physics of Semiconductors of NAS of Ukraine, 45 Prospekt Nauki, Kyiv 03028, Ukraine* **3.** *Université de Sousse, Tunisia, Tunisia*

Contact: kid@bdpu.org

Porous GaAs attract attention in recent years as promising substrate for GaN epitaxy for the purpose of minimizing some negative effects due to large lattice mismatch and difference in thermal expansion coefficient of GaAs and GaN, that usually leads to high strain and high defects density in GaN/GaAs structures. First attempts of using porous GaAs as a substrate for GaN epitaxy was reported in [1,2].

Samples of porous GaAs was obtained by anodic electrolytic treatment of (111)B plane of n-GaAs with concentration of Si $2*10^{16}$ cm^{-3} [3]. With the aim to surface cleaning GaAs samples was preliminary treated in acetone, propanol, ethanol and washed in distillery water and after that samples was dried in flow of N_2. The back of samples was mapped by ohmic contact. Platinum plate was as a second electrode. Electrolytic etching process was carrying out in HF + ethanol solution (of differential concentration). After etcing GaAs samples was washed in deionized water and dry in N_2 flow.

From Raman spectroscopy and scanning electron microscopy investigation the size of GaAs nanocrystallites was determined, its value was estimated about 10-100 nm. Estimation of the size of nanocryslallites in por-GaAs both by Raman shift and scanning electron microscopy gives approximately the same values.

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Tuesday, 6 September

Interfaces and dislocations in compound semiconductors 2

Tuesday morning, 6 September, 9:00
Main Building, room 231
Joerg Neugebauer presides

9:15 oral

Structural analysis of the behaviour of the ultrathin AlN capping layer interface during the RE implantation and annealing of GaN for electroluminescence applications

Pierre Ruterana, Tomasz Wójtowicz, Florence Gloux, Katharina Lorenz, Eduardo Alves

SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France

Contact: florence.gloux@ensicaen.fr

Since the last few years due to many optoelectronic and photonic applications an increased interest in rare earth (RE) doped semiconductors has been observed. One of the effective techniques of RE incorporation in GaN is ion implantation, however it contributes to creation of a considerable amount of structural defects. Their negative impact can be removed by post implantation high temperature annealing which results in optical activation of rare earth ions. Unfortunately high temperature annealing leads to the decomposition of the GaN surface. In order to avoid this decomposition, a number of solutions have been proposed i.e. use of GaN proximity cap, annealing under high N2 overpressure (>10×1010

Pa), and recently implantation through a thin AlN cap layer epitaxially grown on top of GaN. By Rutherford backscattering and channeling (RBS/C), it was shown that the presence of the AlN layer allowed decreasing the implantation damage. It also provided an efficient way to protect the GaN surface for high temperature annealing.

In this work, we investigate the structure of a 10 nm AlN layer grown on GaN by MOCVD. The influence of RE implantation and subsequent high temperature annealing have been studied. Our aim is to understand the protective role of AlN cap during implantation process and high temperature annealing. Scanning electron microscopy (SEM) observations allow us to report on the morphological evolution of the surface. Structural analysis at the atomic scale of the layers and the interface between AlN and GaN is done by the use of high resolution transmission electron microscopy (HRTEM). It is shown that a number of defects like surface pinholes may contribute to the relaxation of the mismatch between AlN and GaN. These defects need to be avoided as their evolution during implantation and annealing leads to significant degradation of the layer.

9:30 oral

Biaxial strain and Poisson ratio in InN films grown heteroepitaxially on GaN(0001) by plasma assisted molecular beam epitaxy

Eleftherios Iliopoulos, Emmanouil Dimakis, Katerina Tsagaraki, Alexandros Georgakilas

Microelectronics Research Group, Institute of Electronic Structure and Laser, Foundation for Research and Technology-Hellas, and University of Crete, Physics Department (MRG), Heraklion 71110, Greece

Contact: iliopoul@physics.uoc.gr

InN (0001) films were grown heteroepitaxially on GaN/Al_2O_3 by radiofrequency plasma assisted molecular beam epitaxy (RF-MBE). The growth conditions and the layers' thicknesses were systematically varied resulting in different strain levels in the films. High resolution x-ray diffraction (HR-XRD) was employed to study their structural properties.

Films' lattice parameters were measured using the extended Bond method for the (0004) symmetric and (10-15) asymmetric diffractions. The c versus a relation exhibited a linear behavior which is typical for the case of biaxial strain present in the heteroepitaxial films. The biaxial strain is attributed to the differences between the in-plane lattice parameters and between the thermal expansion coefficients of GaN and InN.

The values of c- and a- lattice parameters varied from 5.706 Å to 5.686 Å and from 3.521 Å to 3.545 Å correspondingly. In the case of films with lower c/a ratio cracks or microcracks were observed, confirming the presence of tensile strain. From the data the value of Poisson's ratio for InN was determined equal to 0.42+-0.05.

9:45 oral

Properties of the CdTe/InSb Interface Studied by Optical and Surface Analytical Techniques

Zhe C. Feng

National Taiwan University (NTU), Roosevelt Rd., Taipei 106-17, Taiwan

Contact: zcfeng@cc.ee.ntu.edu.tw

The interface properties of CdTe/InSb grown by molecular beam epitaxy (MBE) and the In interdiffusion were studied by optical and surface techniques of photoluminescence (PL), Raman scattering, X-ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS). Correlationship between two types of investigations was established. We have the following results:

(1) The In interdiffusion was revealed directly by SIMS depth profile and XPS In 3d and 4d spectra, indicating that the In concentration in CdTe film grown with high substrate growth temperature Ts is much higher than that with low Ts. Higher Ts enhanced the In interdiffusion across the CdTe/InSb interface, leading to more indiums detected.

(2) PL spectra from two Ts samples are quite different. The low Ts one possesses strong free and acceptor-bound excitons, relatively weaker and sharp emissions in the deep level range of 1.4-1.5 eV, characteristic of high quality of single crystalline CdTe. The high Ts one has dull FE feature only and strong defect-related E and F lines and deep 1.44 eV broad band.

(3) We had varied the Ts between 170-285 C and obtain an optimum Ts of 185 C. It shows that there would exist an exact lattice match between CdTe and InSb at 185 C and that a(CdTe) > a(InSb) as T<185 C and a(CdTe) < a(InSb) as T>185 or 200 C. At higher Ts, the bigger lattice mismatch would lead to dislocations and In will diffuse more efficiently along these dislocation lines from InSb substrate into CdTe layer. The big amount of In leads to the strong signals of XPS spectra and SIMS profiles, and also to the strong In and defect-related PL and Raman features. Optimizing Ts and other growth conditions are efficient to depress the In interdiffusion during the II-VI/III-V growth.

10:00 oral

Interfacial diffusion and precipitation in rf magnetron sputtered Mn doped ZnO layers

Pierre Ruterana[1], Morad Abouzaid[1], C. Liu[2], Hadis Morkoc2

1. *SIFCOM, UMR6176, CNRS-ENSICAEN, 6 Bld Maréchal Juin, Caen 14050, France* **2.** *Virginia Commonwealth University (VCU), Department of Electrical Engineering, Richmond, United States*

Contact: pierre.ruterana@ensicaen.fr

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 well above room temperature by doping with transition metals, especially Mn and Co. Ferromagnetism was recently observed in insulating (Zn,Mn)O, n-type (Zn,Mn)O, and p-type (Zn,Mn)O. However, the absence of ferromagnetic ordering in (Zn,Mn)O was also reported. To develop a better understanding of the observed magnetic behavior, a detailed microstructural analysis of transition metal-doped ZnO thin films is necessary because the incorporation of transition metals may lead to structural disorder or the formation of transition metal-related micro-clusters in the films. The existence of such type of structural imperfections can impede the clarification of experimentally observed ferromagnetism in DMS materials. In this work, we have investigated samples made of a ZnO buffer layer and an Mn-doped ZnO film were deposited on sapphire at 650 C and 550 C, respectively. The as-deposited films were annealed at 850 ºC for 1 hour in air to improve the crystalline quality. The ferromagnetic Mn-doped ZnO films showed magnetization hysteresis at 5 and 300 K. This structural investigation shows that the Mn doped part of the layers contains a high density of round-shaped and elongated MnZn oxide precipitates, we report on the structural relationship between the precipitates and the ZnO matrix. Moreover, the interface between the undoped ZnO and the sapphire substrate exhibits Mn rich phase, meaning that there is a strong interdiffusion in these layers. We analyse the structure, composition and possible origin of these interfacial phases.

Gate oxide interfaces

Tuesday afternoon, 6 September, 14:00
Main Building, room 231
Philomela Komninou presides

14:00 invited oral

Interfaces between a polar high-k perovskite and silicon

Isabelle Devos[1], Pierre Boulenc[1,2]

1. *Institut d'Electronique de Microélectronique et de Nanotechnologie (IEMN), BP 60069 - Cité scientifique-avenue Poincaré, Villeneuve d'Ascq Cedex 59652, France* **2.** *STMicroelectronics, 850 rue Jean Monnet, Crolles Cedex 38926, France*

Contact: Isabelle.Devos@isen.fr

In the search of high-κ materials deposited on silicon surfaces, we lace ourselves in the perspective of epitaxy which is chosen to try to avoid interface states. Oxides with the perovskite structure are known as good candidates as they can be considered as constructed on a superposition of layers which are compatible with the silicon surface. The main problem is to know the atomistic details about the interface as several possibilities generally exist to realize it, with several bonding. We show here, taking the $LaAlO_3$ perovskite as prototype, that this atomistic description is important on several points. It has first to insure that there are no gap states at the interface. To construct such an interface, one may use i) the "electron counting model" which we show that it has sometimes to be extended for polar oxides ii) ab initio electronic structure computations. We present several interfaces which have been constructed for $LaAlO_3$ and their relaxed positions. Another important point to respect is the value of the conduction band offset. We demonstrate on computations in the density functional theory framework that the offset is greatly dependant on the interface (from -0.5eV to +3eV). From all these results, we deduce general rules about the high-κ materials interfaces with silicon.

14:30 oral

Band alignment at metal-gate/high-k/semiconductor interfaces

S. J. Wang[1], Y. F. Dong[2], Q. Li[2], Y. P. Feng[2], A. C. H. Huan[1]

1. *Institute of Materials Research and Engineering, Singapore, Singapore* **2.** *Department of Physics, National University of Singapore, Singapore, Singapore*

Contact: sj-wang@imre.a-star.edu.sg

The interfaces between metal, oxide and semiconductor are very critical for the application of metal/oxide thin films, including gate dielectrics and ferroelectric transistors. Much research has focused on understanding and exploiting the properties of novel metal/oxide/semiconductor interfaces to fulfill the stringent requirements for these and other applications. With the alternative high-k gate dielectrics are expected to re-

place current SiO_2 gate oxide for the continued scaling of metal-oxide-semiconductor field-effect transistors (MOSFET), there is an immense interest in replacing conventional poly-Si gate with metal gates because of the serious problems related to poly-Si gate depletion and high gate resistance. However, the possible atomic bonds of metal-metal or metal-oxygen at metal gate/oxide gate dielectric interface are quit different from conventional silicon-oxygen bond at poly-Si-SiO_2 interface. How these bonds affect the band alignment at the metal/high-k oxide interface is important issue for the implementation of this gate stack. In this presentation, we present the band alignment studies for Ni/ZrO_2(HfO_2)/Semiconductor interfaces by photoemission study and first-principle calculation. The Schottky-barrier heights for the Ni and ZrO_2 (HfO_2) interfaces have been determined by means of x-ray photoemission spectroscopy (XPS). Depending on the interface treatment, the band alignment could be tuned. First-principles calculations for model interfaces provide a microscopic explanation of such variation. The results show that the band alignment at metal/high-k/semiconductor could be engineered through the interface structure-control.

14:45 oral

Spectroscopic analysis of thin HfO2 and HfSiOx gate dielectric thin films exposed to NH3 anneal processing

Patrick S. Lysaght[1], Joel Barnett[1], Manuel Quevedo-Lopez[3], Paul D. Kirsch[2], Gennadi Bersuker[1], Byoung-Hun Lee[5], Joseph Woicik[4], Daniel Fischer[4]

1. *SEMATECH, Austin 78741-6499, United States* **2.** *IBM Assignee to SEMATECH, United States* **3.** *Texas Instruments Assignee to SEMATECH, United States* **4.** *Brookhaven National Laboratory, National Synchrotron Light Source (NSLS), Upton, NY 11973, United States* **5.** *IBM Assignee to SEMATECH, United States*

Contact: pat.lysaght@sematech.org

We have used small angle neutron scattering and synchrotron x-ray photoelectron spectroscopy to determine microstructure variations in HfO2 and Hf silicate, (HfO2)x (SiO2)1-x, x ~ 0.7, gate dielectric thin films deposited on Si (100) substrates. 3 nm thick films exposed to a post deposition anneal (PDA) of NH3/700ºC/60s exhibit a significant amount of N in the bulk film, quantified by nuclear reaction analysis, and corroborated by intense Hf-N peak in binding energy spectra. We report on the effectiveness of N to suppress the diffusivity and retard crystallization kinetics. Therefore, by comparing NH3 PDA with N2 PDA at 700ºC/60s, the influence of incorporated N has been decoupled from that of the thermal cycle alone. Ultra-thin nanophased materials have extremely high surface to volume ratio and the specific surface area dependence of crystallization has been addressed by characterizing 2 nm and 4 nm thick films exposed to each anneal processes. X-ray diffraction spectra indicate N exchanges for O in the crystal cell structure where Hf-N bonds will contribute local strain in tetragonal and monoclinic HfO2 [1]. The Hf-N bonds may also coexist in some volume fraction as an amorphous HfxOyNz composition. Additionally, Hf silicate samples with duplicate anneal processing has been spectroscopically analyzed and we report on the mechanistic pathways associated with phase separation and subsequent crystallization of end member oxide, HfO2, corresponding to the influence of N in the bulk films. Samples exposed to a rapid thermal anneal (RTA) process of N2/1000ºC/10s (full polycrystalline) have been analyzed for comparison [2-3].

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15:00 oral

The interface of atomic layer deposited Al_2O_3 on silicon

Kunyuan Gao[1], Florin Speck[1], Konstantin Emtsev[1], Thomas Seyller[1], Lothar Ley[1], Michael Oswald[2], Walter Hansch[2]

1. *Universität Erlangen-Nürnberg, Institut für Technische Physik II, Erwin-Rommel-Str. 1, Erlangen 91058, Germany* **2.** *Technische Universität München, Lehrstuhl für Technische Electronik, Arcisstrasse 21,, München 80333, Germany*

Contact: gao@physik.uni-erlangen.de

Al_2O_3 films 1.5nm to 20nm thick were deposited as alternative high k gate dielectric on hydrogen-terminated silicon by Atomic Layer Deposition (ALD) technique and characterized by Synchrotron X-ray Photoelectron Spectroscopy (SXPS), Fourier Transform Infrared (FTIR) absorption spectroscopy and admittance measurements. The SXPS results indicate that about 60% of Si-H bonds are preserved at the Al_2O_3/Si interface and this is confirmed by monitoring the Si-H stretching modes by FTIR spectroscopy in the Attenuated Total Reflection (ATR) mode both before and after ALD of Al_2O_3. The remaining 40% of Si-H bonds are replaced by Si-O bonds as verified by SXPS. In addition, a fraction of a monolayer of SiO_2 formed on top of the Al_2O_3 dielectric during deposition. The presence of OH groups at a level of 3% of the total oxygen content was detected throughout the Al_2O_3 layer through a chemically shifted O1s component in SXPS. Admittance measurements give a satisfactorily high dielectric constant of 9.12, but a relatively high density of interface traps between 10^{11} and $10^{12}cm^{-2}eV^{-1}$.

15:15 oral

Si-oxide and Si-oxynitride interfaces analysed by ultra-low energy SIMS

Michał Ćwil[1,2], Piotr Konarski[1], Tomasz Bieniek[3], Romuald B. Beck[3]

1. *Industrial Institute of Electronics (PIE), Dluga, Warszawa 00-241, Poland* **2.** *Warsaw University of Technology, Faculty of Physics, Koszykowa 75, Warszawa 00-662, Poland* **3.** *Warsaw University of Technology, Institute of Microelectronics & Optoelectronics (imio), ul. Koszykowa 75, Warszawa 00-662, Poland*

Contact: cwil@if.pw.edu.pl

Silicon oxides and oxynitrides are used for electronic application in wide scale. In the case of oxynitrides technology, the prime importance is to control the atomic concentration in thin SiO_xN_y layers. In the present paper we compare the depth profile analyses of oxides and oxynitrides using SIMS (Secondary Ion Mass Spectrometry) technique, which is ideally suited for nanostructure characterisation. A subnanometer depth resolution can be achieved with ultra-low energy (below 1 keV) ion beam due to atomic mixing reduction in sputtering process [1-2].

SiO_2 on Si samples were formed by thermal heating (800-1100^oC) of silicon wafers in oxygen flow. SiO_xN_y layers were obtained by nitrogen implantation followed by plasma oxidation process. The nitrogen plasma implantation was performed with NH_3 and pure N_2 plasma sources in 350^oC. Influence of r.f. power, used during implantation on the oxynitride layer properties, was studied. The SiO_xN_y and SiO_2 layers were 1, 2.5, 3.5, 4, 5 nm and 5, 15, 22, 50, 65, 85 nm thick respectively.

Ultra-low energy Ar^+ ion beam (880 eV) was used in SAJW-05 SIMS analyser with quadrupole spectrometer. Quantitative atomic concentrations of N and O were calculated basing on Si_2N^+ and Si_2O^+ secondary ion currents according to Berkum [3]. Sputtering rate was calibrated basing on ellipsometry measurements. The obtained layers were also characterised by X-ray Photoelectron Spectroscopy.

Charge build-up effects during positive secondary ion detection were observed dependent of the SiO_2 layer thickness. The SiO_xN_y/Si interface regions show differences related to the kind of plasma and the r.f. power of nitrogen implantation used.

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Interfaces and defects in electroceramics

Tuesday afternoon, 6 September, 15:50
Main Building, room 231
Kathrin Dorr presides

15:50 invited oral

Strain and charge modulation in multiferroic manganite - titanate film systems

Kathrin Dorr

Institut fuer Metallische Werkstoffe, IFW, Dresden, Germany

Contact: k.doerr@ifw-dresden.de

There are lattice-matching ferromagnetic and ferroelectric perovskites like manganites (La,A)MnO3 (A = Sr, Ca, Ce) and titanates Pb(Zr,Ti)O3 which are favourable for the growth of epitaxial multilayers and offer interesting perspectives for the investigation of magnetoelectric coupling phenomena (mainly mediated elastically). Magnetoelectric coupling means the magnetization of the studied system depends on electric field, or electric polarization can be induced by magnetic field. Perovskite manganites have been investigated for two general reasons during the last decade: The very high spin polarization of conduction electrons might be useful in spintronics devices. Further, the correlation of several electronic and lattice degrees of freedom creates various ground states being particularly sensitive to external parameters like magnetic or electric fields, light, but also hydrostatic pressure or biaxial lattice strain. The latter is present in films and offers interesting ways to control their properties.

In our work, epitaxial film systems of manganites and titanates (PbZr0.48Ti0.52O3 (PZT)) have been prepared by off-axis pulsed laser deposition (PLD). In bilayers of a manganite and a PZT layer both, (i) biaxial strain induced by the PZT inverse piezoelectric effect and (ii) charge density modulation in the manganite induced by electric field effect from surface charges at the interface have been investigated. Both effects can be studied in a field effect device structure where they induce hysteretical resistance modulations of distinguishable type. Larger and more uniform strain has been obtained using piezoelectric substrates which allow electrical control of the strain state of films deposited on top. This approach provides access to the modulation of magnetization behavior by electric field, i. e. a primary magnetoelectric response in a composite multiferroic.

[1] C. Thiele, K. Dorr et al., Sensors and Actuators A (in press); Appl. Phys. Lett. (subm.)

16:20 invited oral

Chemical bonding and electronic structure at hetero-interfaces: the case of perovskite oxides

Jean-Luc Maurice[1], Cécile Carretero[1], Karim Bouzehouane[1], Marie-José Casanove[2], Sabrina Guyard[1], Jean-Pierre Contour[1], Christian Colliex[3]

1. *Unité Mixte de Physique CNRS/Thales associée à l'Université Paris-Sud, Domaine de Corbeville, Orsay cedex 91404, France* **2.** *CEMES CNRS, BP 4347, Toulouse cedex 31055, France* **3.** *Laboratoire de Physique des Solides Université Paris-Sud (CNRS UMR8502), Orsay cedex 91405, France*

Contact: jean-luc.maurice@thalesgroup.com

The generic perovskite unit cell is a simple cube of formula ABO_3 where A and B are respectively a large and a small cation. Depending on the nature of cations, the substitutions on either cationic site, and the amount and possible order of oxygen vacancies, perovskite oxides span an extremely wide range of solid state electronic and magnetic properties. Associating them by epitaxy creates a large field of research towards applications in the microelectronics area, in a context where silicon-based technology faces major challenges.

In <100> directions, perovskites are stacks of {200} planes of alternate compositions AO and BO_2; there are thus two possibilities of stacking sequence at a {100} interface. Depending on the valency of the different cations in presence, these two may have dramatically different properties: at the interface between the insulators $LaAlO_3$ and $SrTiO_3$ for instance, high-mobility carriers were detected in the case of the -LaO-TiO_2- sequence while the sequence -AlO_2-SrO- was found insulating [1].

In order to reproduce the metallic -LaO-TiO_2- interface, we have grown $LaAlO_3$ onto TiO_2-terminated (001) $SrTiO_3$ using pulsed laser deposition; the samples obtained were conducting indeed. We have then observed this interface at the atomic level by aberration-corrected high-resolution transmission electron microscopy and electron energy loss spectroscopy. On the basis of our experimental observations, we discuss in this communication the origin of the measured electronic properties.

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16:35 oral

The influence of defects on the surface behavior of TiO_2

Kris J. Hameeuw, Giovanni Cantele, Domenico Ninno, Giuseppe Iadonisi

University of Naples "Federico II", Department of Physics, Piazzale Tecchio, 80, Napoli I-80125, Italy INFM - Coherentia, Via Cinthia, Napoli 80125, Italy

Contact: hameeuw@na.infn.it

Titanium dioxide is a semiconductor oxide with a wide range of applications. The material is being used as a catalyst, as a gas sensor, as a coating, in ceramics and in electric devices such as a varistor. Recently, TiO_2 has also been suggested to replace Si dioxide as a gate insulator in MOSFETs.

In all of these applications, the surface physics of TiO_2 plays a major role and is critically influenced by the occurrence of defects. Understanding the surface properties of TiO_2 at a fundamental level is considered to be crucial to improve the performance of TiO_2 based devices in many fields.

We carried out an exhaustive theoretical study of the technologically most important TiO_2 (110) surface, using ab initio techniques. First we will discuss the surface and electronic properties of the clean (110) surface. Next, we will present a detailed analysis of the influence of surface and subsurface defects. These defects consist of missing oxygen atoms in the crystalline structure, the so called oxygen vacancies. We will show that the presence of oxygen vacancies has a strong impact on both the structural and electronic properties. The position of the vacancy in the material alters the way it affects the surface behavior.

This thorough investigation contributes to a deeper understanding of the interaction of TiO_2 with other materials. As such, it is a crucial step to grasp the interfacial behavior of this semiconductor oxide. This will be illustrated with the results of a theoretical study of metal atoms on the TiO_2 surface that act as a catalyst.

16:50 oral

Electrical Transport Mechanism in VO_2 Thin Films

Alexander Axelevitch, Boris Gorenstein, Boris Sigalov, Gady Golan

Holon Academic Institute of Technology (HAIT), 52 Golomb St., Holon 58102, Israel

Contact: alex_a@hait.ac.il

Vanadium oxide (VO_2) is a material with “smart” properties. This material transforms from semiconductor to a metal state at the temperature of 67 ^{0}C. All its electrical and optical properties dramatically change while transformation. Current tech-

nologies are using such "smart" materials in order to develop optical switching devices, electro-optical sensors and displays. VO_2 thin film systems, deposited by various methods, are most often applied for these "smart" applications.

This paper presents the investigation and experimental results of the optical and electrical properties of vanadium oxide thin films deposited by vacuum thermal evaporation on various dielectric substrates. We have studied the electro-physical behavior of these films during their phase transition. It was shown that the electrical transport mechanism of the obtained vanadium oxide films differs in low and high electrical fields. In low electrical fields, conductivity is obtained by the Schottky transport mechanism, whereas in high electrical fields conductivity ranges from Ohmic mechanism for low fields, to Poole-Frenkel for higher fields. FTIR, NIR and visible optical characteristics of the obtained films, were studied as well.

17:05 oral

First principle calculation of the effect of La and Y impurities on the energy gap of α-Al2O3

Mohammad R. Benam

Physics Department,Payame-Noor University of mashhad, Mashhad, Iran, Boulvare Vakilabad,Shahid Sattary St,PAyam-Noor Univ, Physics Dept,, Mashhad 91735/433, Iran

Contact: mrz_benam@yahoo.com

We have performed a first principle calculation of the band structure and density of state of the α-Al2O3 and also have studied the effect of the La and Y impurities on the size of its band gap. The results show that these impurities decrease considerably the size of the energy gap.

Most of the calculations have been performed based on the Density Functional Theory (DFT) using the full potential Augmented Plane Wave + Local Orbital method (APW + L0), as implemented in the WIEN2K package.

Wednesday, 7 September

Metal-metal interfaces and interfacial modelling

Wednesday morning, 7 September, 9:00
Main Building, room 231
Paul D. Bristowe presides

9:00 oral

Promising high quality short period Fe/Fe-N multilayers deposited by the sputtering

Wojciech Szuszkiewicz[1], Krzysztof Fronc[1], Piotr Dłużewski[1], Roman Minikayev[1], Wojciech Paszkowicz[1], Bernard Hennion[2], Frederic Ott[2], Gregory Chaboussant[2]

1. *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **2.** *Laboratoire Leon Brillouin, CEA-CNRS, CE Saclay (LLB), Gif-sur-Yvette 91191, France*

Contact: szusz@ifpan.edu.pl

The properties of Fe/Fe-N based structures are a topic of intensive studies during at least ten last years. Up to now, the successful Fe/Fe-N multilayer growth has been reported by a few groups only and was mostly limited to the long-period structures. Moreover, available results suggested wide and rough Fe/Fe-N interfaces. Similar multilayers were recently deposited by the sputtering on GaAs substrates at the Institute of Physics of the Polish Academy of Sciences. The goal of the present paper was to demonstrate the successful growth of the short-period Fe/Fe-N multilayer, consisting of 22 repetitions of 40 Å thick Fe layer followed by 12 Å thick Fe-N layer. The high quality of the investigated structure was proved by X-ray reflectometry, neutron scattering, and transmission electron microscopy. In particular, our results demonstrated the presence of narrow (sharp) Fe/Fe-N interfaces, which are important for possible new applications.

9:15 oral

Laser assisted fabrication of Co on Ti for bio-implant application

Jyotsna Dutta Majumdar[1], S. M. Ganeshan[1], Indranil Manna[1], A. K. Nath[2]

1. *Indian Institute Of Technology,Kharagpur (IIT), Kharagpur, India* **2.** *Centre for Advanced Technology (CAT), Industrial CO_2 Laser Centre, Indore 452013, India*

Contact: jyotsna@metal.iitkgp.ernet.in

Laser assisted fabrication is a materials processing technique that utilizes high-power lasers to melt the material in the form of a powder or wire, controlled deposition of the molten metal in a layer-by layer fashion and subsequent solidification in the controlled atmosphere to induce a pre-determined shape of a component. The process has successfully been employed for the fabrication of metallic, ceramic and polymer materials. In the present study, attempts have been made to fabricate a Co layer on the surface of Ti substrate (with a compositionally graded interface) by laser assisted fabrication technique. Laser assisted fabrication was carried out by melting of Co powder (of 25 mm particle size) with a continuous wave CO_2

laser and subsequent deposition of molten metal on Ti substrate in a layer by layer fashion using Ar as shrouding environment to avoid oxidation. The process variables were applied power density, scan speed and number of layers. During the development of 1st layer, laser power and scan speeds were varied to develop a compositionally graded interface. After fabrication, a detailed microstructural study of the surface and cross section of the fabricated components was carried out using optical and scanning electron microscopy to understand the influence of laser parameters on microstructure of the surface and interface between the successive layers. X-ray diffraction study and energy dispersive spectroscopic analysis were conducted to see if non-equilibrium cooling associated with the process has caused formation of any new phase or segregation of elements in the microstructure. Finally, the characteristics of the fabricated component have been correlated with the process parameters to optimise the processing zone for the fabrication of 316L. The mechanical properties of the fabricated components have been evaluated using microhardness testing machine and correlated with the microstructure.

9:30 oral

A hybrid atomistic-continuum finite element modelling of locally disordered crystalline structure

Paweł Dłużewski[1], Piotr Traczykowski[1], Grzegorz Jurczak[1], Marcin Maździarz[1], S Nagao[2], Roman Nowak[2], Krzysztof J. Kurzydlowski[3]

1. *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT PAN), Świętokrzyska 21, Warszawa 00-049, Poland* **2.** *Nordic Hysitron Laboratory, Dept. Materials Sci. & Engg., Helsinki University of Technology (TKK/HUT), Vuorimiehentie 2A, Helsinki FIN-02015, Finland* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: pdluzew@ippt.gov.pl

Problem of locally disordered atomic structure is solved by using a hybrid formulation in which nonlinear elastic finite elements are linked with discrete atomic interaction elements. The continuum approach uses nonlinear hyperelasticity based upon the generalized strain while the atomistic approach employs the Tight-Binding Second-Moment Approximation potential to create new type of elements. The molecular interactions yielding from constitutive models of TB SMA were turned into interactions between nodes to solve a boundary value problem by means of finite element solver. Atomistic psudoelements are noting more than two-node atomic interaction. The application is used to deal with the problem of edge dislocation and its dissociation into two Schockley's dislocation in Cu crystals.

Three examples are shown. In the first, the whole considered crystal region has been discretized and solved by means of the nonlinear elastic FEs. In the second example, the same FE region has been discretized by means of the molecular lattice and solved by molecular dynamics and statics. Finally, in the third example the regions around the dislocations' cores have been replaced by discretized atomic structure and linked on the boundary with corresponding nodes of continuum FEs. In this way, a single boundary value problem with two different types of discretization of the crystal structure has been solved in this example. A transition on the continuum/atomic interface is assured by taking into account a crystallographic data in the finite element mesh preparation. In this example the regions of dislocation cores were modelled using molecular interaction mesh while the ordered lattice was discretized by FEs. The obtained MD-FE model has been applied to simulate the nanoindentation test on nanocrystalline copper in order to conclude on the singularities observed in P-h curves.

9:45 oral

Prediction of degree of localisation of misfit dislocation cores in intercrystalline interfaces based on interfacial adhesion

Witold Lojkowski

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: wl@unipress.waw.pl

When two crystals form an interface (or grain boundary if they are of the same kind) the local atomic arrangement along the interface is different than in the bulk. There are special orientations of the two crystals and of the interface itself, where the energy of the interface has a minimum as a function of crystallographic parameters characterizing the interface. We will call them LEORs: Low Energy Orientation Relationships. They are also frequently called special interfaces, favoured interfaces, low energy interfaces. In order to minimize energy, interfaces tend to acquire a structure where a large fraction of their area has a structure identical with that of an LEOR. The difference in misorientation angle is taken over by misfit dislocations. However, a challenge is to predict the LEORs as well as the degree of localization of the misfit dislocation cores.

In this paper we show that for that purpose one can use the ratio of the adhesion energy and elastic constants of the crystals. This ratio is expressed numerically as localization parameter p. The system is in a trade of situation, where minimizing misfit leads to decrease local energy in the interface, but increases the long range elastic fields energy. The value of the localization parameter permits to predict which tendency will win: to minimize elastic energy or to minimize misfit energy. It can also predict criteria for low energy interfaces.

10:00 oral

High resolution transmission electron microscopy study on the nano-scale twining of θ-NiMn precipitates in Fe-Ni-Mn maraging alloy

Syamak Hossein Nedjad[1], Mahmoud Nili Ahmadabadi[2], Tadashi Furuhara[3], Tadashi Maki[3]

1. *Sahand University of technology (SUT), Sahand New Town, Tabriz, Iran, Tabriz 51335-1996, Iran* **2.** *University of Tehran (UT), North-Kargar, Tehran 14395-731, Iran* **3.** *Kyoto University, Kyoto 606-8501, Japan*

Contact: hossein@sut.ac.ir

Precipitation of nanometer-sized f.c.t. θ-NiMn intermetallic compounds was found to cause age hardening in a Fe-10Ni-7Mn (wt. %) maraging alloy during isothermal aging at 773 K. Conventional and high resolution transmission electron microscopy (TEM) was used to study microstructural characteristics of the f.c.t. θ-NiMn precipitates. Bright-field TEM images showed twinning of both fine matrix and coarse grain boundary precipitates. High resolution lattice imaging of the precipitates illustrated a nano-scale twinning of the precipitates. An atomistic model, corresponding to the lattice images, was developed for twinning reaction indicating the {111} and <111> as twin planes and directions, respectively.

Keywords: Maraging: Precipitation: NiMn: Twin

Interfaces in nanostructured and amorphous thin-film systems

Wednesday afternoon, 7 September, 14:00
Main Building, room 231
Piotr Dłużewski presides

14:00 oral

INTERFACE KINETIC IN CRYSTAL GROWTH FROM SOLUTIONS

Horia Alexandru

University of Bucharest, Faculty of Physics, Bucharest-Magurele p.o.box mg-11, Bucharest 76900, Romania

Contact: horia@infim.ro

ADP crystal shows different kinetic mechanism of growth from solutions [1]. In "situ" macroscopic growth rates of prismatic and pyramidal faces were measured (+/- 0.01 mm accuracy) versus supersaturation (s). After seed regeneration, a transient regime of growth was found for the prismatic faces of ADP. Within a fresh grown layer, smaller than 1 mm, the growth efficiency decreases 3 to 6 times and it is 6 to 12 times higher than for the elementary dislocation [2]. Data compare well with interferometric measurements of Chernov et al [3]. Edge free energy on prismatic faces of ADP was estimated as 0.84 kT.

Macroscopic growth kinetic (at s <1%) of the pyramidal faces of ADP, measured in a long run, show also the decreasing of the growth efficiency (data not published yet). But this time, the effect has to be associated with the divergence of several dislocations from pyramidal, towards the prismatic faces. However, a surprising macroscopic effect was found. The "switching" of efficiency was found around s = 0.5%. A similar effect was found by laser interferometry in ref. [4], but at higher supersaturations s □ 3% when the dominating center of growth change the Burger's vector from m=3 to m=2, or so. The microscopic parameter L/ma = 160-200, we have associated with the switching effect, is larger than the data from ref. [4]. Some other data related to KDP kinetic shall be presented.

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14:15 oral

Deposition of nanostructured thallium sulfide layers on organic and inorganic surfaces in a solution of higher polythionic acid

Ingrida Bruzaite[1], Valentinas Snitka[2], Vitalijus Janickis[1]

1. *Kaunas University of Technology, Department of Chemistry, Studentu 56, Kaunas 3000, Lithuania* **2.** *Kaunas University of Technology, Research Center for Microsystems and Nanotechnology (RCMN KTU), Studentu 65 - 203, Kaunas 3031, Lithuania*

Contact: ingrida.bruzaite@ktu.lt

Materials with three-dimensionally periodic structures have attracting considerable attention over the last decade. These materials have the potential for use as photonic materials, advanced catalysts, sensors. Thallium sulfides electrical conductivity changes with exposure to light, therefore making this compound useful in photocells and sensors.Thalium sulfide layers with different composition on the surface of polyethylene were obtained when this polymer firstly have been sulfured in a solution of higher polythionic acid, H2S33O6, and then treated with alkaline solution of thallium(I) sulfate.

In this work we investigate the growth of nano-structured thalium sulfide thin films on the surface of organic (polyethylene), inorganic (LiNbO3) and matrix (silica-polystyrene latex) substrates. Silica films containing three dimensionally (3D) ordered polystyrene latex (PSL) nano-beads were prepared by dip coating method. Silica sol-gel films derived from tetraethylorthosilicate (TEOS) containing polystyrene latex beads with diameter 50 nm and 200 nm

and were used for the nano-ordered thallium sulfide dots deposition. The stoichiometric composition of thallium sulfide layers varies between Tl0.3S and Tl2.6S in dependence on the conditions of the formation of the layers of TlxSy. A phase composition of the layers obtained was confirmed by the method of X-ray photoelectron spectroscopy. Spectra of Tl 4f7/2, O 1s and S 2p showed the bond of thallium with sulfur in TlS and Tl2S , but Tl2SO4, Tl(OH)3, S8 and Tl2O3 have been also found in the surface of the layers. The investigation of the film growth mechanism on different substrates by Scanning Atomic Force Microscopy revealed that the sorbtion-diffusion on the surface dominate the formation of the TlxSy crystallites formation. The photoconductivity of the films was investigated by the structure of planar electrodes and radiation by UV light.

14:30 oral

IN SITU ETCHING TREATMENTS OF EPITAXIAL LAYER BY USING DIFFERENT GAS COMPOSITION FOR MORPHOLOGICAL QUALITY IMPROVEMENT OF THE SURFACES.

Sonia De Angelis[1,2,3], LUCIANO SCALTRITO[1,3], SERGIO FERRERO[1,3], DENNIS PERRONE[1,3], FABRIZIO PIRRI[1,3], MARCO MAUCERI[2], STEFANO LEONE[2], GIUSEPPE PISTONE[2], GIUSEPPE ABBONDANZA[2], DANILO CRIPPA[4]

1. *Dipartimento di Fisica, Politecnico di Torino, corso Duca degli Abruzzi, 24, Torino 10129, Italy* **2.** *ETC srl, Z.I. Pantano D'Arci, Catania 95030, Italy* **3.** *Material and Microsystems Laboratori-χ Lab, Palazzo "L.EINAUDI", Lungo Piazza d'Armi, Chivasso, Italy* **4.** *LPE SPA, VIA FALZAREGO,8, MILANO 20021, Italy*

Contact: s.deangelis@etc-epi.com

Different homo epitaxial 4H-SiC commercial wafers were investigated after in-situ etching process in an Hot Wall Chemical Vapor Deposition (HWCVD) reactor. We have studied the effects of physical desorption due to different type and mixture of gases at the same process conditions in order to point out the morphology and the structural variations of epitaxial surfaces. As a result to select proper etching treatments.

An optical microscopy inspection was made to trace out a map of defect areas before and after etching treatments. We have analyzed the morphological evolution of the surface in every etching process step by means of marked area on the defect map. We also achived some other important information, concerning structural and morphological changing, by performing Atomic Force Microscopy and Micro Raman spectroscopy analysis on the same defect marked area.

The etched epi-layers showed a significant reduction of defects density and a good surface morphology, various gas treatments induced different variations in the morphological composition of the surface, above all in defect structure. We have tried to correlate morphological and structural changing as well as we used different gases type and mixture composition to perform etching treatments, therefore interesting results were found.

14:45 oral

Patterning of Surfaces by Polysilicon for Inducing Hydroxyapatite Growth by Laser-Liquid-Solid Interaction

Liliana Pramatarova[1], Emilia V. Pecheva[1], Radina Presker[2], Attila L. Tóth[3], Erike Horvath[3]

1. *Institute of Solid State Physics, Bulgarian Academy of Sciences (ISSP-BAS), 72, Tzarigradsko Chaussee blvd., Sofia 1784, Bulgaria* **2.** *Technical University, Walter Schottky Institute, Am Coulombwall 3, München D-85748, Germany* **3.** *Hungarian Academy of Sciences, Research Institute for Technical Physics and Materials Science, P.O.Box 49, Budapest H-1525, Hungary*

Contact: emily@issp.bas.bg

After oxygen, silicon (Si) is the second most abundant element in the environment and is present as impurity in most of the materials. The widespread occurrence of siliceous biominerals as structural elements in lower plants and animals suggests that Si plays a role in the production and maintenance of connective tissue in higher organisms [1]. It has been shown that Si is necessary in the bones, cartilage and in the formation of the connective tissue, as well as in some important metabolic processes [1]. Additionally, well known is that Si participates as SiO2 in the precipitation of apatite layers on bio-active glasses.

Polysilicon layers are deposited on glass substrates by subsequent or simultaneous RF magnetron sputtering of Al and a-Si in vacuum and as a result Si nanocrystallites are formed. The as-patterned surfaces are tested in terms of their reactivity towards inducing hydroxyapatite (HA) formation from simulated body fluid (SBF) by applying a method of laser-liquid-solid interaction (LLSI). Subsequently the samples are left in the same SBF in which they are irradiated with the laser for 4 hours. The grown structures are analyzed by SEM, LM, EDX, XRD, FTIR and Raman Spectroscopy.

Keywords

Hydroxyapatite, patterned surfaces, polysilicon, laser-liquid-solid interaction, simulated body fluid

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Wednesday Poster Session

Wednesday afternoon, 7 September, 15:50

Symposium G

Adhesion in building bonds - macro-, micro- and nanoscale

Welcome

Adhesive joints of the materials are of importance in many technical applications.
A number of adhesion type joints can be distinguished in constructions:

- bonding of a new concrete with an old concrete, bonding of pre-cast elements, bonding of steal with concrete,
- sealed joints e.g. dilatation (putties),
- joints which are formed during protection of the concrete surface by coatings,
- repairs and strengthening of constructions for example: filling of losses, cracks injections, strengthening with fiber reinforced polymers.

Adhesion is also important at the micro and nanoscale in the case of modification of existing and new developed composite materials (eg. textile concretes). Adhesion is defined as adherence of two materials being in contact. It is a result of intermolecular attraction forces appearing at the interface of the adhering materials. Their nature is often second order chemical bonds. Moreover, the adhesion strength can result from other accompanying phenomena like: primary bonds formation (chemisorptions), mechanical interlocking on the rough surface of substrate.

Recently, the construction repairing increased dramatically due to the fact that structures constructed during first decades after the Second World War reached a critical age of around 50 years. An adhesion between overlays and concrete substrate is one of the most important factors affect the reliability and durability of the system. Adhesion in building bonds has been incompletely described by existing adhesion theories. This is resulted from complexity of the adhesion phenomena in building bonds, particularly in repair systems. This was a leading idea to organize Symposium in the framework E-MRS Fall Meeting 2005 entitled "Adhesion in building bonds: macro-, micro- and nanoscale". The aim of the Symposium is exchange of multidisciplinary knowledge on the creation and characterization of adhesion in building bonds. The Symposium covers following main topics:

- factors influencing adhesion in building bonds,
- methods of characterization of relationship between adhesion and microstructure at macro-, micro-, and nanoscale,
- compatibility in building bonds: models and verification methods,
- durability of building joints created during repair, anti-corrosion protection and strengthening,
- destructive and nondestructive methods of adhesion evaluation for lab and in-situ application,
- application of computational science approach for modeling of adhesion phenomena.

It is visible from the Conference program that the 2005 E-MRS Fall Meeting is interdisciplinary. There is no doubt it create opportunity for very fruitful discussion among scientists from various fields of materials science and engineering. Many of technological solutions, seem to be to sophisticated for building industry at present, will be commonly used in the future. Fiber reinforced polymers and last nanotechnology applications are good examples of this type of successful technology transfer.

I hope that our Symposium and entire Conference will bring to all participants opportunity for valuable discussion and creation of new ideas, not only in the filed of adhesion joints. To all outstanding Members of Scientific Committee I would like to pass the words of respect and worm appreciation.

Lech Czarnecki

Invited speakers:

- **Prof. H.Rainer Sasse**, Bau Ingeneur Sozietät Aachen, Germany
- **Prof. Harald Schorn**, University of Dresden, Germany
- **Prof. Franco Sandrolini**, University of Bologna, Italy
- **Prof. Dionys Van Gemert**, Katholieke Universiteit Leuven, Belgium
- **Dr Wenzhong Zhu**,University of Paisley, Scotland
- **Prof. Wojciech Radomski**, Warsaw University of Technology, Poland

Scientific Committee:

- **Prof. Lech Czarnecki**, Warsaw University of Technology, Poland - chairperson
- **Prof. Jose Luis Barroso de Aguiar**, University of Minho, Portugal
- **Prof. Arnon Bentur**, Technion, Israel
- **Dr. Luc Courard**, University de Liege, Belgium
- **Prof. Dionys Van Gemert**, Katholieke Universiteit Leuven, Belgium
- **Prof. Peter C.Hewlett**, BBA British Board of Agrement, UK
- **Prof. Deon Kruger**, Rand Afrikaans University, South Africa
- **Prof. Andrzej S.Nowak**, University of Nebraska, USA
- **Prof.Yoshihiko Ohama**, Nihon University, Japan
- **Dr Antonio Porro**, NANOC, LABEIN, Spain
- **Prof. Wojciech Radomski**, Warsaw University of Technology, Poland
- **Prof. Michael Raupach**, RWTH Aachen, Germany

- **Prof. Franco Sandrolini**, University of Bologna, Italy
- **Prof. H.Rainer Sasse**, Bau Ingeneur Sozietät Aachen, Germany
- **Prof. Herald Schorn**, University of Technology Dresden, Germany
- **Prof. Johan Silfwerbrand**, Swedish Cement and Concrete Research Institute, Sweden
- **Dr Jakob Sustersic**, Institute for Research in Materials and Applications, Slovenia
- **Prof. Jacek Śliwiński**, Cracow University of Technology, Poland
- **Prof. Kyu-Seok Yeon**, Kangwon National University, Korea
- **Prof. Folker H.Wittmann**, Aedificat Institute, Germany
- **Dr Wenzhong Zhu, ACMC**, University of Paisley, Scotland

Organisers

- **Dr Andrzej Garbacz**, Warsaw University of Technology, Poland
- **Dr Bogumiła Chmielewska**, Warsaw University of Technology, Poland
- **Dr Luc Courard**, University of Liege, Belgium

Programme

Monday, 5 September

Session 1: Theories and engineering aspects of adhesion
Monday afternoon, 5 September, 14:00
Main Building, room 226
Lech Czarnecki, Makoto Kawakami presides

14:15 invited oral

Polymer Adhesion to Concrete - Theories and Engineering Aspects -

Rainer H. Sasse

Aachen University of Technology - Institute for Building Research (IBAC), Schinkelstr. 3, Aachen D 52056, Germany

Contact: die.sasses@t-online.de

The paper gives a general historical view over theoretical valuations for adhesion between concretes and polymers and relates these with results of laboratory tests.

The basic theories are presented: mechanical and specific adhesion (including molecular-physical and thermodynamical interpretation). Harmonies and differences between theories and experiments (laboratory one-parameter-studies and multi-parameter-tests with commercial concretes and polymers) are presented.

Considerations are made concerning boundary zones in the concrete substrate and in the polymer, which have other properties than the bulk materials. Such zones are not included in the classical theories.

14:45 invited oral

Experiments from the macro to the nanoscale

Harald Schorn

University of Dresden, Germany

Contact: Prof.Schorn@web.de

In civil engineering usually tests on a macroscale, on bodies of sizes between meters and centimeters are carried out to discover “material laws” used for structural design. To understand the mechanisms which take place in the material structure and which affect and determine all material properties, additionally tests on smaller scales are normal, e.g. measuring porosimetry of a cementitious material on the microscale. Optical microscopes are not useful. Avoiding sample preparation which would affect the phenomena being investigated, the surface of the sample is relatively rough and the depth of focus actually is insufficient.

New experimental technologies in microscopy were the base for further investigation on the macro- and on the nanoscale. Using an Environmental Scanning Electron Microscope (ESEM) even wet samples without necessity of sample preparation can be observed. This technology allows to develop observation methods for the process of hardening of cement paste. Especially a cement paste containing a polymer dispersion with particle diameters of less than 300 nm (PCC) was a subject of interest in research work.

Having observed the consolidation of PCC-materials it seems likely to try to investigate what happens in a stress state of this material. A miniature testing apparatus was produced which could be put into and used inside the ESEM. Microcrack propagation due to increasing tensile load and crack bridging behaviour of polymer-phase in PCC can be shown. The knowledge about the combined binder effect of both cement and polymer in PCC was highly improved. The contribution contains many impressive pictures taken from the observed samples.

15:15 oral

On the methods of bond strength measurements in concrete repair systems

Bogumila Chmielewska

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: B.Chmielewska@il.pw.edu.pl

Adhesion is a complex phenomenon which play significant role in modern building composite structures as well as in the case of repair of existing structures. In the paper adhesion joints created as a result of concrete repair or concrete protection are characterized. Methods used to measure a bond strength, which differ in the way of loading are shortly presented. The experiences with wedge splitting test applied for such measurements are especially described. The work presents comparative analysis of the bond strength measurements carried out using the following methods: wedge splitting test, tensile splitting test, direct tension and bending tests, pull-off test, slant test. The investigation was done for model repair systems consist of ordinary cement concrete substrate and mineral repair materials;two commercial repair materials were used. Additionally, the effect of concrete substrate quality and influence of primer was tested. Effect of various measurement methods on the obtained results of bond strength is discussed.

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster G-1

Surface free energy evaluation of building materials

Courard Luc, Frédéric Michel

University of Liege (ULg), B5, Liege B4000, Belgium

Contact: Luc.Courard@ulg.ac.be

The surface free energies and their components between two interacting surfaces are extremely important since not only do they dictate the strength of interaction, but also control processes like the stability of aqueous colloidal suspensions, wetting, spreading and adhesion. The interactions solid-liquid are mainly controlled by the interfacial surface tensions between the two phases. The characterization of the surface properties and especially the surface free energy components of the solid are, therefore, recognized as the key to understanding the mechanism of surface-based phenomena.

Concrete is a very complex material, not only what about concerns composition but also its evolution with time. As concrete can be considered as a colloidal suspension when in fresh state, surface interactions will firstly act on the dispersion of particles inside the concrete mix and promote the fluidity of the fresh material. The superplasticizers and Modifying Viscosity Agent are fundamental components, particularly for the development of Self Consolidating Concrete.

Another area of surface tensions action is the interface between formwork and cement paste. As the superficial layer of concrete is a critical zone with higher W/C ratio and defaults like air bubbles, it is also important to understand the basic phenomenons that will act on the quality of the surface and to select the most appropriate materials to avoid these defaults. Air is always present in fresh concrete and the aim of vibration techniques or textiles applied on the formwork is to prevent the formation or the stabilisation of the air bubbles in the surface layer of concrete. Data are needed to evaluate correctly the surface free energies of liquids and solids present in this three phases mix: air, cement paste, formwork.

Finally, concrete repair quality is based on interactions between old concrete and new material, at the levels of the creation and the stability of the interface.

15:50 poster G-2

Study the wettability of III-N thin films with different bond ionicity and surface layers polarity

L. Ostrovskaya[1], A. Vasin[2], O. Voznyy[3], Vitalij Deibuk[4], M. Sletov[4]

1. *V.Bakul Institute for Superhard Materials, NAS, 2 Avtozavodskaya, Kyiv 254074, Ukraine* **2.** *V.E.Lashkarev Institute of Semiconductor Physics of NAS of Ukraine, 45 Nauky Prospekt, Kyiv 03028, Ukraine* **3.** *Université de Sherbrooke, Sherbrooke J1K 2R1, Canada* **4.** *Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine*

Contact: vdei@chnu.cv.ua

The subject of this work was to study the effect of chemical bonding rebuilding on the wettability of III-nitrides (from BN to InN). Structural (bond ionicity) and surface properties, associated with their wettability and surface free energy, for 0.5- to 2-μm thick GaN, AlN, InN films deposited on different substrates (Si, α-Al_2O_3) by a pyrolytic deposition from complex ammonium compounds of Al, Ga and In halides have been studied. BN films with the different fraction of cBN (from 10 to 70% cBN) have been grown by the ion beam assisted deposition technique on Si (111) p-type substrate at the temperature 400 ^{o}C in argon and nitrogen medium.

The contact angles for the liquids of different polarities (water, μ = 1,84 D and glycerol, μ = 0,28 D) on these films were measured by a sessile drop technique (drop diameter 0,5 – 1 mm) in air at room temperature 20 ^{o}C. The surface energy was calculated by Fowke's equation using wetting angle measurement and was compared to the bond ionicity and the

films surface layers polarity.

The analysis of chemical bonding is made in different models (Phillips's ionicity, Vogl's polarity and Garcia-Cohen's charge asymmetry coefficient). The pseudopotential calculations of electronic charge distributions along the bonds and the charge asymmetry coefficients (BN - 0.35; GaN - 0.52; AlN - 0.61; InN - 0.64) allow us explain the wettability features of III-nitrides.

A correlation was noticed between the wettability and bond ionicity: the higher the bond ionicity of the film, the lower wetting angle values both for water and glycerol. Surface layers polarity also affects the wettability behavior: N-face of the film wetted better than Me-face. All the above results have been discussed along with the films surface energy parameter (its polar and dispersive components) as the surface-determined factors.

15:50 poster G-3

Improvement of wetting and bonding of SiO_2 ceramics and urea-urethane elastomer

Anna Boczkowska, Katarzyna Konopka, Kamil Babski, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: kako@inmat.pw.edu.pl

Ceramic-elastomer composites obtained via infiltration of porous ceramics by urea-urethane elastomers are a new class of materials with significant application opportunities. They combine the ceramic hardness and stiffness with the rubbery entropy-elasticity of elastomers.

The porous SiO_2 ceramic was infiltrated by urea-urethane elastomer synthesized by one-shot method from ethylene oligoadipate, 4,4'-diphenylmethane diisocyanate and dicyandiamide. The obtained elastomer contains polar, urea groups and highly polar nitrilimide side-groups in the hard segments. The existence of strong polar groups in every short hard segment influences the ceramic – elastomer adhesion by hydrogen bonds with hydroxyl groups available on the SiO_2 surface. The adhesion between ceramic and elastomer could be also improved by addition of coupling agents. Two kinds of coupling agents were used to improve the wetting and adhesion between ceramics and elastomer. The degree of wetting was estimated by the value of contact angle as a function of kind, amount and the way of introducing of coupling agent.

SEM observations of ceramic-elastomer composites showed that the elastomer infiltrated into the pores of ceramic matrix leading to the microstructure with percolation of the elastomer phase. Good infiltration is confirmed by low porosity of the samples. The compression tests of composites revealed that due to good adhesion between ceramics and elastomer the compression strength increases.

15:50 poster G-4

SURFACE MODIFICATION OF POLYCARBONATE BY PLASMA TREATMENT FOR OPTICAL APPLICATIONS

Argirios Laskarakis, Christoforos Gravalidis, Spiridon Kassavetis, Stergios Logothetidis

Polycarbonates (PC) are characterized as very attractive polymeric materials for an extensive use in optical technology applications. The use of plasma treatments has been successfully employed for the improvement of their surface properties and the increase of the adhesion of subsequent Anti-Scratch (AS) coatings deposited in order to advance the PC lens mechanical performance. In this work, we present a detailed investigation of the effect of Pulsed DC N_2 plasma treatment on the optical properties and the nanotopography of PC optical lens. The investigation of the PC surface modification during plasma treatment has been studied by in situ & real time Fourier Transform IR Spectroscopic Ellipsometry (FTIRSE) and Atomic Force Microscopy. These techniques revealed important information about the dominant mechanisms that take place during the plasma-surface interaction. The analysis of FTIRSE spectra showed the formation of a surface overlayer with thickness up to 500 nm adjusted by the plasma treatment, that is characterized by a polymer macromolecular chain scission in combination to chain crosslinking. Moreover, the study of PC surface nanotopography showed an initial increase of the roughness, followed by a reduction at higher values of the applied voltage and an elimination of the surface inhomogeneities (trenches) of the untreated PC lens, affecting the formation of the protective AS coating on the surface.

15:50 poster G-5

Influence of changing polymer modified bitumen microstructure on adhesion after ageing processes

Jan Król

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: j.krol@il.pw.edu.pl

Recently, polymers are useful materials for modification of bitumen to improve their technical properties, especially rheological ones. The literature data have been shown that, in same cases, increase of adhesion to aggregate has been additionally observed. However some authors have been indicated that an the effect of polymer modification on adhesion has not been significant.

In this paper the influence of short and long term ageing processes on polymer dispersion in polymer modified bitumen

was analyzed. The bitumen was modified by various amount of SBS elastomers. Rheological and technical properties were investigated. Microstructural changes of polymer dispersion were tested using a fluorescent microscope.

The influence of two types of ageing on changes of polymer dispersion distribution was characterized. Potential effect of polymer dispersion on adhesion was also discussed.

15:50 poster G-6

Adhesion of Fiber-Reinforced Concrete on Hardened Surfaces of High Strength Ductile Cementitious Composites

Makoto Kawakami[1], Ammar Hassan[1], Satoru Matsuoka[2]

1. *Akita University, Faculty of Engineering and Resource Science, Dept. of Civil and Environmental Engineering (Akita Uni.), 1-1 Tegata Gakuen-cho, Akita 010-8502, Japan* **2.** *Landes Co. Ltd., Project Development Department (Landes Co.), 630-1Otchiai Maniwa, Okayama 719-3192, Japan*

Contact: kawakami@ipc.akita-u.ac.jp

High strength ductile cementitious composite materials are gradually establishing a foothold in the application-field of construction engineering. Although these materials have improved properties, their cost is still comparatively high and they usually necessitate special manufacturing and curing procedures, which limits their use to precast elements. Consequently, one of the more economically feasible applications of these materials is using them in conjunction with ordinary concrete.

The application of the ductile cementitious composites together with ordinary concrete requires studying the bond characteristics between those two different materials. This paper reports the results of a research program of slant shear tests on cylinders made of the two materials.

The study focused on the adhesion of fresh concrete on hardened surfaces of high strength ductile cementitious composites. Slant shear tests were carried out on composite cylinders with a substrate of fiber-added ductile cementitious composites and an overlay of ordinary concretes with different fiber contents. The studied parameters of the tests were mainly the fiber content of the overlay concrete, the surface treatment and roughness of the cementitious material substrate, and the type of the cementitious material substrate.

The paper describes the tests and details the results. Failure stresses, bond strengths, and failure patterns were thoroughly discussed. The effects of each of the test parameters on the adhesion were also studied. A numerical analysis of the slant shear method was done and friction coefficients of the surfaces were calculated. The Mohr-Coulomb failure envelops under certain conditions were also presented and discussed. The paper outlines recommendations concerning surface treatment and the choice of materials for the different targeted applications.

15:50 poster G-7

Polyurethane coating for protecting concrete floors using parquetry with fillers originating from recycling

Joanna Ryszkowska, Janusz A. Rebis, Maciej Langner

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: januszr@inmat.pw.edu.pl

Very often the top layer of systems covering a concrete surface is made from polyurethanes. The polyurethane layer should have high tensile strength and abrasive wear resistance, because these materials are used in workshops, corridors, and warehouses. Polyurethanes are one of the most expensive commonly used polymers. That is why the Department of Materials Science and Engineering proposed using granulated polyurethane waste of different hardness as a filler for concrete coating.

The aim of this paper is to characterize the composites, which were synthesized with identical parameters as the system producer recommendations. Polyurethane layer was obtained from: two components polyurethane coatings and polyurethane recyclate, hardness 80^{o}ShA and density (1,2464g/cm^3) and 50^{o}ShD (1,2725g/cm^3). Recyclate, grain size 0,4-1,5 mm was obtained from polyurethane with high abrasive wear resistance. Poliols components with 12% to 30% wt. of polyurethane recyclate in room temperature were mixed. Into this mixture isocyanate component was added. The samples were conditioned for two weeks at standart conditions.

The mechanical properties of composites as tensile strength, ultimate elongation, hardness, and abrasive wear were investigated. Endurance properties were formulated during bending, tensile strength and ultimate elongation. SEM was used for structure investigation.

As a result of the research materials of higher resistance to abrasive wear and Young modulus with unchanged resistance to tensile strength, ultimate elongation and bending were obtained. Furthermore the permanent deformation of researched materials were decreased. Addition of recyclate, hardness 50^{o}ShD causes an increase in abrasive wear resistance to 150%, whilst the cost of the material is lowered.

15:50 poster G-8

Application of computer image analysis for evaluation of adhesion between bitumen and aggregate

Michał Sarnowski

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: M.Sarnowski@il.pw.edu.pl

The paper presents results of testing the adhesion between asphalt binder and aggregate.

Methods of testing adhesion between bitumen and aggregate were described.

In this work the influence of the aging of modified asphalt binders on adhesion was tested. Several types of polymer-modified asphalt and aggregate were investigated.

Two methods of aging: the Thin Film Oven Test (TFOT) and the Pressure Aging Vessel (PAV) were used.

Adhesion was determined by the modified boiling test.

Computer image analysis was used for qualitative and quantitative evaluation of adhesion between bitumen and aggregate.

Effect of the polymer modification on adhesion was discussed.

15:50 poster G-9

A new method for concrete surface roughness characterization by opto-morphology technique

Damien Schwall, Luc Courard

University of Liege (ULg), B5, Liege B4000, Belgium

Contact: Damien.Schwall@student.ulg.ac.be

The study of adhesion of repair materials on concrete structures implies a good knowledge of the influence of concrete surface treatment. The effects of surface preparation technique is rarely clearly described and parameterised: it is consequently difficult to point out the real influence of roughness on adhesion results. A large research project has been realized with regards to the influence of concrete substrate strenght and preparation technique efficiency. A new technique has been used in order to quantify the surface roughness of concrete: the projection “moiré” technique is an interfer-ometrical measurement method. The “moiré» phenomenon appears when two networks of light rays, made of equidistant lines - alternatively opaque and transparent -, are superimposed.

The technique of identification of relief is based on the deformation’s measurement of a parallel fringes pattern projected on a surface. A software analysis system RugoDS has been written and geometrical and statistical parameters have been calculated. Abbott's curves give interesting answers to differentiate surface preparation effects. Comparison between polished, scarified, sandblasted and hydro-jetted surfaces evaluation is presented.

15:50 poster G-10

Characterization of concrete surface geometry by laser profilometry

Katarzyna Kostana, Andrzej Garbacz

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: k.kostana@il.pw.edu.pl

Concrete surface needs to be properly prepared prior to repair of concrete structure. The main objective of concrete surface treatments is to remove deteriorated concrete or any type of layer that can cause a decrease in adhesion, as well as to enlarge the area of the contact zone to achieve a mechanical interlock with repair material. Various treatments are used for concrete surface preparation. As a result, different roughness levels are obtained. The concrete surface geometry can be characterized with various methods at different level of observation.

The aim of this work is the analysis of an effect of surface treatment on concrete surface geometry using laser profilometry. Several types of concrete preparation techniques were selected to obtain different levels of the surface roughness of concrete substrate. For each type of sample the following parameters of surface geometry were determined: waviness and roughness amplitude parameters, Abbott’s curve parameters and fractal dimension. Usability of the determined parameters for characterization of surface geometry was analyzed. The obtained results were compared with parameters of surface geometry previously obtained for the same samples using microscopic method and “sand” (macroscopic) method.

This contribution has been prepared in the framework of the research project granted by Dean of Civil Engineering Faculty, Warsaw University of Technology - grant number 503 G 1084 3705.

15:50 poster G-11

On the effect of concrete substrate roughness on stress wave propagation in repair systems

Tomasz Piotrowski[1], Andrzej Garbacz[1], Damien Schwall[2], Luc Courard[2]

1. *Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland* **2.** *University of Liege (ULg), B5, Liege B4000, Belgium*

Contact: tpiotrowski@student.ulg.ac.be

The necessity of repair efficiency evaluation is stressed in many standards and guidelines related to repair of concrete

structures. For this purpose, besides of pull-off test, non-destructive methods based on propagation of stress waves are usually recommended, e.g. ultrasonic methods and impact echo method.

In this work an effect of concrete substrate surface quality on stress wave propagation in repair system was investigated. Three substrate types, made from concrete of strength classes: C30/37, C40/50 and C50/60 were tested by impact-echo method. Four surface treatments (polishing, sandblasting, scabbling and hydro-jetting) were performed to obtain different quality of concrete substrate. The quality of the preparation was characterized on the basis of three main groups of parameters: surface geometry parameters, superficial concrete microcracking and adhesion strength. The surface geometry was characterized using patch test and opto-morphology technique. The quality of the superficial zone of concrete and cracks were systematically observed, in relation to the given surface treatment type. Commercial repair mortar was applied on concrete substrates after surface treatments. The impact-echo measurements and bond strength tests were carried out after 28 days of repair material hardening. The effects of concrete substrate strength and surface geometry characteristic on frequency spectrum were discussed. The potential relationships between parameters of stress wave propagation in repair system and the bond strength as well as the quality of bond interface were analyzed.

The research project was financially supported by the Government of Poland (KBN) and the Regional Government of Wallonia (DRI), Belgium. Part of experiments has been done in the framework of project granted by Polish Scientific Research Committee - grant number KBN 4 T07E 027 27.

15:50 poster G-12

Computer simulation of stress wave propagation in repair systems

Leslaw J. Kwasniewski[2], Andrzej Garbacz[1]

1. *Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland* **2.** *Warsaw University of Technology, Faculty of Civil Engineering, al. Armii Ludowej 16, Warszawa 00-637, Poland*

Contact: l.kwasniewski@il.pw.edu.pl

Recently, a growing interest in application of non-destructive techniques (NDT) for evaluation of repair efficiency is noted. For this purpose, stress wave based methods, e.g. impact echo, ultrasonic methods are commonly recommended. As a result of repair, a system containing two or more layers is created. Propagation of stress waves in this kind of system is complex phenomenon and depends on acoustic properties of repair material and its thickness as well as a quality of interface.

In this work finite element (FE) method was used to simulate stress wave propagation for various models of repair system. Ls-Dyna, an explicit finite element program dedicated for transient dynamics was applied. Several cases of impact-echo test with different parameters of layer system were analysed. Among considered model parameters were acoustic properties, boundary conditions, layer thickness of repair material and presence of delamination at the interface. Computer simulations of the stress wave propagation showed good correlation with the impact-echo principle. The effect of repair system parameters on wave propagation was discussed.

The research project was granted by Polish Scientific Research Committee - grant number KBN 4 T07E 027 27 (2004-2006).

15:50 poster G-13

Approach to strengthening design of RC structures with CFRP composites

Wojciech Karwowski

Warsaw University of Technology, Faculty of Civil Engineering, al. Armii Ludowej 16, Warszawa 00-637, Poland

Contact: w.karwowski@il.pw.edu.pl

Using CFRP materials lately became a very popular method of strengthening structures. Those materials are very often used when it is necessary to strengthen reinforced concrete. There are two types of CFRP materials used for that kind of strengthening – stripes and mats, and three types of cases – bending, shear (torsion), and confinement. The paper presents some problems, which may appear in anchorage zone of CFRP strips, which are used for strengthening bending elements. There are three components involved in the process – CFRP material, surface of strengthened structure and adhesive. CFRP material is the strongest part in this connection, while surface of strengthened structure is the weakest one. Adhesive, which is set between them, should firmly bond these two materials. All those factors should be taken into consideration while making calculations. Different methods are presented in the paper.

15:50 poster G-14

Weatherability of coating and grouting materials for repair and protection of concrete

Wiesława Głodkowska, Mariusz Staszewski

Technical University of Koszalin, Raclawicka 15-17, Koszalin, Poland

Contact: glod@wbiis.tu.koszalin.pl

The paper presents the results of tests of natural and accelerated weathering for chosen polymer and cement-polymer materials used in repair and surface protection of reinforced concrete structures. The tests comprised: a) crack resistance of

protective coatings, b) estimation of quality of compatibility of grouting materials with the concrete, c) tensile strength, tensile strain, Young's tensile modulus of elasticity of coating and grouting materials and their adhesion in tension to the concrete. On the basis of the tests carried out, functional relations have been observed between weathering of materials in natural conditions and weathering in accelerated conditions. Experimental relationships were fixed enabling the forecast of durability of materials included in the tests, and also for other similar materials to those tested, for repair and protection of concrete in regard to binder type. The usability of grouting and coating materials was determined by defining to what extent and for how long it might be acceptable to allow the worsening of material properties used in repair and for the protection of concrete against corrosion.

Tuesday, 6 September

Session 2: Environmental factors affecting adhesion

Tuesday morning, 6 September, 9:00
Main Building, room 226
Deon Kruger, Paweł Łukowski presides

9:00 invited oral

Dampness rise and repair systems in the restoration of ancient building materials

Franco Sandrolini

Università di Bologna, Dip. Chimica applicata e Scienza dei materiali - Engineering Faculty (DICASM), V.le Risorgimento 2, Bologna 40136, Italy

Contact: franco.sandrolini@mail.ing.unibo.it

Rising dampness in ancient walls is one of the most frequent problem in ancient buildings, which affects both building and restoration materials, thus leading to strong structural and surface damages. After outline of some peculiar, investigated cases (St. Marco Church in Venice, Pio Palace in Carpi, and other ones), the mechanisms and decay effects of rising water in the masonry are discussed from both laboratory and on site points of view. Some experimental results are finally discussed and evaluated with reference to the adhesion of the main materials systems frequently used in Italy for restoration of ancient materials of the architectural heritage.

9:30 oral

Technical methods of creation of adhesion level in repair systems

Lech Czarnecki

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: L.Czarnecki@il.pw.edu.pl

Repair of concrete structure results in formation of at least two-component system, i.e. concrete overlay/concrete substrate or repair material/repaired concrete. The adhesion between overlays and concrete substrate is one of the most important factors that affects the reliability and durability of repair. Recently, achievement of maximum bond strength is basic recommendation for various types of repair joints. The requirement for high adhesion arises from the higher tolerance on non-compatibility of properties of the both materials; in such a case, internal stresses involved with setting and shrinkage of the given repair material are usually higher as well.

In this paper, necessity of above recommendation has been discussed with regards to: repair material type, strength of concrete substrate and environmental conditions of bond service. The criteria for the creation of desired bond strength in relation to tensile strength of concrete substrate has been formulated. The possibilities of creation to attempt higher level of adhesion as well as economical and technical limitations of the adhesion improvement have been discussed.

The presentation has been prepared in the framework of Warsaw University of Technology grant - grant No. 504 G 1084 2815

9:45 oral

Humidity factors affecting adhesion of coatings on concrete

Klaus Littmann

Institut für Berufswissenschaften im Bauwesen, Universität Hannover, Herrenhäuser Strasse 8, Hannover 30419, Germany

Contact: klaus@littmann.de

The question of the durability of adhesion on moist and wet concrete surfaces addresses various matters to be dealt with in terms of engineering, building chemistry and building physics /1/. This paper looks at the topic from three different aspects.

Firstly there is the question as to how far current practice of measuring concrete moisture before the application of coatings is suitable for ascertaining the actual "coating compatibility" of the concrete. Different common methods are compared. An alternative procedure for measuring concrete mois-

ture is presented which is capable of making precise statements on surface moisture.

The paper continues with a short summarised evaluation of several research projects. The study was based on four different big research projects with a large number of sample bodies which were all treated with 3 different epoxy resin formulations. The evaluation gives hints on humidity parameters before, during and after application.

Finally, the paper looks at the damage mechanisms which can limit the durability of adhesion in the presence of water. These include:

· Physical processes:
- Mechanical influences, gas pressure, osmotic pressure
· Interface reactions
- Bottom coating layer
- Saponification or hydrolysis in the coating material
- Top surface layer
- Decomposition of the bonding agent
· Ageing processes

10:00 oral

Measure of repair system compatibility

Maciej Runkiewicz, Lech Czarnecki

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: maciej.runkiewicz@kajimaeurope.com

Bonded overlays constitute a common and versatile method for repair. Repair of concrete structure results in formation of a two-component system, i.e. concrete overlay/concrete substrate or repair material/repaired concrete Such system must be compatible to cooperate properly. In 1992 during the International Colloquium on Materials Science and Restoration held in Technical Academy of Esslingen the principles for repair material selection based on their compatibility were formulated. The research in this filed has been continued in Warsaw University of Technology and suitable compatibility models described by number of inequalities have been developed. These inequalities have been incorporated into the CCS software (Computer Compatibility System) developed on Warsaw University of Technology in cooperation with National Institute of Standards and Technology in 1995. The CCS presents graphically compatibility space for the given repair system. The compatibility software (CCS) allows to classify the situation in dichotomic way: compatible or incompatible. However, there is the need for compatibility (continues) measure. In this paper potential concepts of compatibility measure have been presented.

10:15 oral

ADHESION OF MINERAL REPAIR MATERIALS BASED ON EXPANSIVE BINDERS

Anna Halicka

Technical University of Lublin, Nadbystrzycka 36, Lublin 20-618, Poland

Contact: a.halicka@pollub.pl

Building repair materials should comply with specific requirements: compatibility with repaired material, high adhesion and durability. Additionally, in structural repair, it should actively contribute to bearing capacity.

In concrete structures, first requirement is met by materials based on mineral binders. When choosing the material satisfying to the conditions of adhesion, one can consider expansive binders. Their increase in volume is a result of the growth of ettringite crystals. They are built also in the joint, fill asperities of 'old' concrete surface and penetrate open pores. It results in increase of adhesion and eliminates consequences of shrinkage. Moreover, confinement of the expansion by adjacent parts of structure causes the pressure exerted to joint, increasing the bond strength.

In order to quantitatively evaluate the adhesion of expansive materials to 'old' concrete, two groups of tests were carried out. First, tensile strength of cylindrical concrete specimens made of two samples connected by means of cement grout was evaluated. Cement grouts were made with different binders (Portland cement with and without resin additives and expansive cement).

Furthermore, researches of bearing capacity of joint between ordinary and expansive concrete under combined stresses (shear and tension) were carried out. Two types of samples were used: sample with slant shear interface and sample with interface shaped as the surface of a truncated cone. These samples enabled to point out the influence of the volume changes of the concrete (shrinkage and expansion).

In the paper, the results of these tests are presented. The following conclusions are drawn:

- grout based on expansive binder used as a layer between two concrete samples ensure higher adhesion level than other tested materials,
- bond strength between ordinary and expansive concretes tested in combined stress state is higher than in the case of two ordinary concretes.

Session 3: Material factors affecting adhesion

Tuesday afternoon, 6 September, 14:00
Main Building, room 226
Andrzej Garbacz, José B. Aguiar presides

14:00 invited oral

Microstructure building in mortars modified with water-soluble polymers

Dionys Van Gemert, Elke Knapen

K.U.Leuven Civil Engineering Department (K.U.Leuven), Kasteelpark Arenberg 40, Leuven 3001, Belgium

Contact: Dionys.Vangemert@bwk.kuleuven.be

Usually, polymer-modified cement concrete or mortar is prepared by mixing polymer dispersions or redispersible polymer powders with the fresh mixture. The surface active agents, added to allow emulsification and stabilization of the dispersion during storage, hinder the cement hydration and the polymer film formation. Therefore, the addition of polymers in aqueous solution is aimed for. Water-soluble polymer molecules are supplied on a molecular scale, improving the approach of the relatively large cement grains by the polymers. This will allow the building up of composite polymer-cement microstructures on a nano-scale. In the absence of surface active agents, water-soluble polymers tend to require a lower proportion in order to be comparably effective as polymer dispersions. The addition of very small amounts of water-soluble polymers results in an improvement of the durability and the adhesion strength of the cementitious materials, which makes them appropriate as repair materials.

The effect of the addition of water-soluble polymers is twofold: the cement hydration reactions may be influenced and polymer film formation may take place. The microstructure building and the influence on the cement hydration reactions is studied by SEM investigation. An abundant crystal growth is noticed at the air void surfaces of the modified mortars where the presence of water-soluble polymers is expected because of their strong affinity to the gas-water phase.

Possible film formation is studied by examining the influence of the curing conditions on the flexural strength of mortar beams. Cement hydration requires a wet or moist curing, while a dry curing promotes polymer film formation. If film formation takes place, the contribution of the polymer film to the mechanical properties and the durability of the polymer film should be studied with respect to practical applications, in particular their resistance to re-dissolution in humid environment.

14:30 oral

Enhancing the adhesion of fresh to cured concrete by polymeric systems

Moshe Puterman

National Building Research Institute, Technion Israel Institute of Technology (NBRI), Technion City, Hajfa 32000, Israel

Contact: ptadar@techunix.technion.ac.il

The problem of the adhesion, or better to say: lack of adhesion, of fresh concrete to hardened concrete is well known. The discontinuity line formed between the two cast elements is a source of many deficiencies: it is a week zone mechanically and a potential "leak" zone as far as the waterproofing of the structure is concerned.

This work investigates the possible use of polymers as binders and adhesion promoters when applied on the line of the casing interruption.

The polymers used include:

- aqueous emulsion of polyvinyl-acetate, polyacryl-esters and styrene-butadiene-rubber
- epoxy resins and water based epoxy resins

The various materials were used as fresh (non-cured) or hardened (cured) systems on the first stage hardened concrete during the casting of the second stage fresh concrete.

The effect of the various polymer systems on the adhesion of the two concrete elements and their contribution to the contact between them was tested by their shear strength, flexure strength and pull-off strength of adhesion.

The presentation will report the results of the adhesion properties of the various treatments and applications using the polymers.

14:45 oral

Polymer-cement composites with improved adhesion to the concrete substrate

Paweł Łukowski

Warsaw University of Technology, Faculty of Civil Engineering, al. Armii Ludowej 16, Warszawa 00-637, Poland

Contact: P.Lukowski@il.pw.edu.pl

The paper deals with polymer-cement concretes (PCC) of improved adhesion to the concrete substrate. The classification and main uses of PCC materials in repairing and anti-corrosion protection of the concrete structures as well as in industrial floors, overlays and pre-cast elements have been briefly described. The types of polymer modifiers have been categorised. The influence of polymers on the material microstructure and mechanisms of possible improving of the material properties – particularly bond strength, tensile and flexural strength and tightness – have been discussed. The results of investigations, performed under the framework of the project granted by Polish State Committee for Scientific Research (No. 4 T07E 007 26), have been presented. At the initial stage of research several types of polymer modifiers have been tested; the polyacrylate dispersion (PAE) has been chosen for the further investigation as the most promising one. During the next stage the experiment has been carried out using statistical design of experiment. The material models (a composition – properties relationship formulated in the quantitative way) of polymer-cement mortar have been developed regard-

ing to adhesion to the concrete substrate, tensile strength and water absorbability, with polymer and cement contents as the material (input) variables. High values of determination coefficient R^2 (more than 0.75) have proved the good quality of the models. The statistical analysis of the models made possible to evaluate the synergic effects and the significance of the particular components of polymer-cement binder for creation of the bond strength and other technical properties of the material. This led to the finding that the polymer modifier and its co-operation with inorganic binder is more important than the Portland cement itself for some of those features. General conclusions about future needs in this field of research have also been formulated.

15:00 oral

Improvement of Textile Reinforced Concrete by Use of Polymers

Markus Schleser, Ulrich Dilthey

Welding and Joining Insititute RWTH-Aachen University (ISF), Pontstr. 49, Aachen 52062, Germany

Contact: sr@isf.rwth-aachen.de

For the improvement of the load bearing behaviour and the useful properties of concrete structures which are subject to bending and tensile stresses a steel reinforcement is, in general, stipulated. For reasons of corrosion protection, the steel reinforcement requires a concrete cover with a thickness of several centimetres. The demand for corrosion-resistant thin-walled parts is a motivation for the application of alternative reinforcement materials. Within the scope of the Collaborative Research Centre "SFB 532" (financed by the German Research Foundation DFG) the application of textiles made of polymer, carbon and alkali-resistant (AR) glass filaments in a fine concrete matrix are examined. In the long run it is the aim to reach the filament strength of the immersed textiles as thoroughly as possible. By reason of insufficient bond properties and damages which are a consequence of textile manufacturing processes, the utilisation of AR glass textiles in reinforced concrete components is approximately 30% under tensile load. Therefore the possibility of improving the activation of the filaments and thus the load bearing behaviour of textile reinforced concrete structures through the saturation of textiles and the modification of concrete with liquid polymers is discussed. Based on the accomplished tests, the utilisation of the textiles and the connected increase of the internal bond between the filaments duplicated the maximum component load-carrying capacity.

Session 4: Micro- and nanostructural evaluation of adhesion

Tuesday afternoon, 6 September, 15:50
Main Building, room 226
Courard Luc, Dionys Van Gemert presides

15:50 invited oral

Application of nanotechnology in construction materials

Wenzhong Zhu

University of Paisley, Advanced Concrete and Masonary Centre (ACMC), Paisley PA1 2BE, United Kingdom

Contact: wenzhong.zhu@paisley.ac.uk

Fundamental understanding at the micro/nano-scale of the behaviour of construction materials and their interaction with the environment are required to better explain/control properties and degradation processes. Such information is also vital in the engineering and design of novel construction materials with specific performance characteristics in mind. Advances in the field of nanotechnology have increasingly offered tools to study and control the fundamental building blocks (i.e. atoms, molecules and nanostructures) of many materials (e.g. carbon nanotubes, nanocomposites, etc.), and thus could provide a systematic and cost effective means to develop superior construction materials.

In the first part of this paper, a general review of recent developments and activities of nanotechnology application relevant to construction materials is given. The likely impact and the future trend/potential of nanotechnology application in construction materials are discussed. The second part of the paper reports a current study on nano/micro-scale mapping of mechanical properties of construction materials, particularly cement and concrete. Depth-sensing micro/nano-indentation technique is used to investigate the mechanical properties of various phases (or micro/nano-scale features) and the interfacial bond between different materials.

16:20 oral

Evaluation of fibre-cement interfacial properties by SEM-based push-out tests

Michał A. Glinicki[1], Jolanta Janczak-Rusch[2]

1. *Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT), Swietokrzyska, Warszawa 00-049, Poland* **2.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*

Contact: mglinic@ippt.gov.pl

SEM-based push-out tests were performed for interfacial characterization of fibre reinforced cement composites. The range of experiments covered strands of zirconia glass filaments of diameters ranging from 13 to 15 μm and cementitious matrices modified with various mineral additives.

The push-out tests were performed on thin slices, about 450-600 μm, cut out of the specimens perpendicularly to the direction of glass filaments. During testing a continuous re-

cording of load and displacement of the fibre was performed at a constant push velocity of about 0.2 µm/s. In order to evaluate a significance of wear of the interface a push-back testing was also performed. Simultaneous observation of push-out or push-back events was possible at SEM resolution rates. Test results revealed significant differences due to introduced matrix modifications as well as to aging of specimens. Both stress-based approach and energy-based approach was applied for the analysis of interfacial properties. Obtained results could be well correlated to the phenomena observed in flexural toughness testing and SEM/EDX and XRD data obtained in an accompanied research program.

16:35 oral

The effect of polymers on the properties of textile-reinforced composites;Is it an adhesion or friction effect ?

Moshe Puterman[1], Harald Schorn[2], Michael Raupach[3]

1. *National Building Research Institute, Technion Israel Institute of Technology (NBRI), Technion City, Hajfa 32000, Israel* **2.** *University of Dresden, Germany* **3.** *Aachen University of Technology - Institute for Building Research (IBAC), Schinkelstr. 3, Aachen D 52056, Germany*

Contact: ptadar@techunix.technion.ac.il

Glass fiber textiles, as well as textiles of other types, can be used as reinforcement for concrete elements. The reinforcing mash consist of woven rovings, each containing large amounts of fine fibers. However, the coarse cementitious binder does not penetrate very efficiently the inner parts of the roving and only the external filaments of each roving are in good close contact with the cementitious matrix.

The result is that only a small portion of the embedded filaments are the load bearing phase, whereas many (if not most) of the filaments are not contributing to the development of the expected mechanical properties.

It was the aim of this work to "activate" the inner parts of the roving by impregnation of polymers, in the form of aqueous nano-emulsions, into the textile. The main aim of te polymer addition was to improve the contact between the roving and the matrix, as well as bonding between the filaments themselves. Two types of polymers were used for this purpose:

- Film-forming, rubbery polymers
- non-film-forming, glassy polymers

both were tested in the wet and cured state.

The results, as presented by the flexure behaviour of the composite and pull-out properties of a single roving, suggest that the impregnated polymers have a substantial effect on the mechanical properties of the composite, with the non-film-former having a better contribution on the load bearing capacity of the system.

The results raise the interesting question: what is the main effect of the added polymer phase among the filaments: is it mainly an adhesion effect or a friction effect ?

16:50 oral

Digital analysis of microscopic images for crack system evaluation in concrete elements

Michał A. Glinicki, Agnieszka Litorowicz

Polish Academy of Sciences, Institute of Fundamental Technological Research (IPPT), Swietokrzyska, Warszawa 00-049, Poland

Contact: akwiat@ippt.gov.pl

Cracks in concrete arise at all stages of life of concrete structures, and can be induced by loads or volumetric changes due to plastic settlement, high curing temperature, various types of shrinkage and other deteriorating mechanisms such as frost, alkali aggregate reaction, etc. Inadequate curing leading to cracking or extensive microcracking could considerably decrease durability and service life of structures. A tool for crack identification and quantitative description of crack system in concrete was developed in order to aid a service life assessment of concrete elements in structures. The paper presents a new testing method and its significance for concrete permeability assessment. Samples for crack analysis were impregnated with epoxy resin containing fluorescent dye. The examination of crack system was performed in ultraviolet light using stereomicroscope and Image Pro Plus digital image analysis system on specimens cored out of several concrete structures. The process of digital image analysis consisted of extracting the various defect from the observed image, filtering operations and sorting the extracted defects on basis of shape analysis. Crack pattern analysis was performed on specimens cored out of several reinforced concrete floors, columns, slabs on ground, suspended parking slabs. The analysis of cracks in concrete cores resulted in interesting conclusions based on the crack widths distribution and crack localization in respect to steel reinforcement. The method was found very effective to support standard concrete diagnostics methods.

Wednesday, 7 September

Session 5: Effect of substrate quality

Wednesday morning, 7 September, 9:00
Main Building, room 226
Klaus Littmann, Moshe Puterman presides

9:00 oral

Freezing of Simple Fluids within carbon and silica nanotubes: a combined experimental and simulation approach

Sliwinska-Bartkowiak Malgorzata[1], Monika Jazdzewska[1], Benoit Coasne[2], Francisco R. Hung[3], Keith E. Gubbins[4]

1. *Adam Mickiewicz University, Department of Physics, Umultowska 85, Poznań 61-614, Poland* **2.** *CNRS - Universite Montpellier 2 (GES), Place Eugene Bataillon, Montpellier 34095, France* **3.** *Department of Chemical and Biomolecular Engineering, North Carolina State University, Raleigh NC 27695-7905, United States* **4.** *Department of Chemical and Biomolecular Engineering, North Carolina State University, United States*

Contact: msb@amu.edu.pl

Recent studies have shown a rich phase behavior associated with freezing and melting of host phases in porous materials. Depending on the adsorbate and the confining solid matrix, the transition temperature may be lowered or raised relative to the bulk value, and new surface-driven phases may intervene between the liquid and solid phases in the pore. We report experimental measurements and molecular simulation results of the freezing and melting behavior of carbon tetrachloride and krypton confined within pores of cylindrical geometry, specifically carbon nanotubes and silica MCM-41 of different pore sizes. Dielectric relaxation spectroscopy was used to determine the experimental melting points of confined carbon tetrachloride, and molecular simulations were performed using grand canonical and parallel tempering Monte Carlo techniques.

The transition temperatures and the structure of the confined phases are determined for pore sizes up to 5 nm. Our results for the largest carbon nanotube show that the adsorbate layers near the pore walls freeze at temperatures higher than the bulk freezing point, whereas the adsorbate in the inner regions of the pore experience a depression in the freezing temperature when compared to the bulk value. In contrast, only one transition temperature well above the bulk freezing point was obtained for carbon tetrachloride within a carbon nanotube with a diameter of 2.9 nm. The simulation results are in good agreement with the experimental measurements, and both suggest the presence of several inhomogeneous confined phases which are found to be stable over extended temperature ranges.

9:30 oral

An overview of the significance of crack widths on structural adhesion and degradation of cracked concrete

Andre C. Jooste[2], Deon Kruger[1]

1. *University of Johannesburg, Kingsway, Johannesburg 2006, South Africa* **2.** *Stewart Scott International, Main, Pretoria 0001, South Africa*

Contact: dk@ing.rau.ac.za

One of life's fundamental truths is that all concrete cracks with a resultant reduction in macro- and micro adhesion properties. Plastic, elastic, creep, shrinkage and thermal strains all contribute to the development of cracks in concrete members. Although these cracks may affect the adhesion integrity and life expectancy of a concrete structure, cracks are not the main cause of structural failure and degradation but play an important role in the propagation of other degradation processes. As will be shown in this paper there are certain myths about cracking that needs to be addressed. A classification of different non-structural cracks is given and the different causes of cracking and their influence on concrete durability are discussed. Attention is given to micro cracking, the repair of cracked sections and the so-called self-healing process of cracks smaller than 1 mm in width. The effect of crack widths on different durability properties is investigated by performing an intensive literature survey. The authors conclude that by means of appropriate design and detailing techniques, cracks can be limited to acceptable levels in terms of structural integrity and aesthetics.

9:45 oral

Influence of kind of mechanical concrete surface preparation on the adhesion of epoxy coatings

Guenther Roessler, Michael Raupach

Aachen University of Technology - Institute for Building Research (IBAC), Schinkelstr. 3, Aachen D 52056, Germany

Contact: roessler@ibac.rwth-aachen.de

If concrete surfaces should be coated they have to be prepared in such a way that the adhesion of the coating on the concrete will be durably sufficient.

Results of an investigation will be presented concerning adhesion of epoxy resin on concrete in dependence from concrete surface preparing method, roughness, concrete quality, viscosity of resin and temperature.

The results can be summarized as follwing: Concrete surfaces should be prepared mechanically in any way. Blasting with steel balls leads to the highest adhesion strength. If milling cutters must be used, for example if concrete must be re-

moved, the surface should be finished up with steel ball blasting. The temperature while welding an asphalt sheet or application of asphalt mastic does not moderate the adhesion strength.

10:00 oral

The influence of cement mortar and aggregates on adhesion forces of the protective coating to the concrete base

Monika Siewczynska, Jozef Jasiczak, Pawel Kolodziej, Adam Nietopiel

Poznań Technical University, pl. Marii Skłodowskiej-Curie 5, Poznań 60-965, Poland

Contact: monika.siewczynska@ikb.poznan.pl

The protective coatings (cement filler and polymer resin) are used in renovation of the concrete surface in order to fill the holes, to reinforce the underlaying surface or to protect the concrete structure from the environmental ageing. Those coatings should have a good adherence to the concrete base.

Pull-off tests were made to investigate adhesion of those coatings to the concrete. Then the analysis of the concrete cracking was performed, by tearing off the roller. The influence of cement mortar and aggregates during pulling-off the roller was pointed out. The tests were carried out on concrete cubes in which natural aggregates were replaced by granulated foamed polystyrene. The results were compared with ordinary concrete.

This approach was developed in order to fix the proportional contribution of the cement mortar and the aggregates to the adhesion through the pull-off tests.

10:15 oral

Study of bond between concrete layers with different rheological characteristic

Jakob Šušteršič[1], Igor Planinc[2], Andrej Kryžanowski[2], Andrej Zajc[1]

1. *irmalj1 (IRMA), Slovenčeva 95, Ljubljana 1000, Slovenia*
2. *University of Ljubljana, Faculty of Civil Engineering Ljubljana, Slovenia (FGG), Jamova 2, Ljublana 1000, Slovenia*

Contact: jakob.sustersic@guest.arnes.si

Some fundamental principles for studying the bond between concrete layers with regards to different rheological characteristics are discussed. This study is included into the Preliminary research project developed to find optimal technological- technical solutions for hydro-power plants construction in Blanca and Krško on the Sava river, Slovenia. Therefore, our sphere of interest is the study of bond between mass concrete and abrasion resistant concrete layer. These two concretes usually differ with regard to their mix-proportions, properties and rheological behaviour, particularly. These differences should lead to loss of bond between concrete layers and, consequently, the potential collapsing of the structure upper layer.

Session 6: Strengthening of concrete structures

Wednesday afternoon, 7 September, 14:00
Main Building, room 226
Bogumila Chmielewska, Jakob Šušteršič presides

14:00 invited oral

Methods applied for structural strengthening of concrete bridges

Wojciech Radomski

Warsaw University of Technology, Faculty of Civil Engineering, Institute of Roads and Bridges (IRB), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: W.Radomski@il.pw.edu.pl

A great part of bridge population in many countries, including Poland, requires to be structurally strengthened. During the last two decades, bridge strengthening has become one of the most technical and economical problems in bridge engineering.

Three fundamental cases leading to the necessity of the bridge strengthening are presented and discussed, namely:

• bridges showing a bad technical condition – restoration of their load-carrying capacity predicted in the original design;

• bridges showing a generally good technical condition – upgrading of their load-carrying capacity in accordance to the new requirements concerning utilization of the structures (e.g. growth of the live load level);

• bridges showing a generally non-satisfying or even bad technical condition – their further utilization requires the strengthening corresponding to the higher level of live loads than the designed level.

The paper is mainly devoted to the strengthening of concrete bridges.

The methods applied for their strengthening are classified according to the original author's criteria. The are classified into two fundamental groups, namely:

• the methods based on the redistribution of the internal forces in the structure (i.e. active methods);

• the methods not based on the redistribution of the internal forces in the structure (i.e. passive methods).

The methods applied are briefly characterized taking into account their cost and time of the required operations. Material solutions of the methods are presented in particular.

The newest methods, such as external prestressing and applications of the carbon fibre reinforced polymer (CFRP) products (i.e. strips and fabrics) are described in detail and exemplified by the case studies taken from the Polish bridge

experience. The use of the above methods are well developed in Poland.

Some research and practical conclusions are formulated at the end of the paper.

14:30 oral

Effect of temperature on systems for the reinforcement of concrete

José B. Aguiar, Aires F. Camões, Teresa J. Pimenta, Nelson F. Vaz, Marcelo M. Araújo

University of Minho (UM), Campus of Azurém, Guimarães 4800-058, Portugal

Contact: aguiar@civil.uminho.pt

This paper presents an experimental work using prismatic specimens of hydraulic mortar reinforced on the exterior with steel or CFRP plates. The adhesive used was an epoxy. Two series of tests were made. In the first one, the specimens were exposed seven days to temperatures between 20 ºC and 250 ºC, after which they were subjected to flexure tests at 20 ºC. In the second series, the specimens were exposed 24 hours to temperatures between 20 ºC and 60 ºC. The exposure to different temperatures was maintained during the flexure tests. The results of the tests show a decrease of adhesion strength with temperature. In the first series, the efficiency of the reinforcements decreases significantly above 50 ºC. In the second series, the significant decrease of the flexure resistance occurred above 40 ºC. The simultaneous action of temperature and load leads to a lower thermal resistance than the separate action.

14:45 oral

On the usability of wavelet analysis for nondestructive assessment of repair efficiency by impact-echo method

Andrzej Garbacz

Warsaw University of Technology, Faculty of Civil Engineering, Building Materials Eng.Division (BME WUT), Armii Ludowej 16, Warszawa 00-637, Poland

Contact: A.GARBACZ@IL.PW.EDU.PL

Impact-Echo method (I-E) is treated as the one of the most promising nondestructive techniques for assessment of concrete structures. This method can be also applied for evaluation of multi-layer systems created during repair of concrete. The stress wave generated in the I-E method has lower sensitivity to natural heterogeneity of concrete structure because of its lower frequency. Additional feature of I-E method is an application of frequency spectrum analysis obtained by fast Fourier transform of waveform. Recently, a new tool for signal analysis – wavelet analysis - is being implemented in NDT assessment, also in the case of concrete structures. It is a multiresolution time-scale methods which enables to extract information like instantaneous frequency, tiny echoes embedded in complex signal or to suppress noise.

In this work the effect of quality of interface: polymer-cement coating – concrete substrate on propagation of stress wave in I-E method was analyzed. The several repair systems were tested. They were differed in concrete surface quality (as a result of applying surface treatments) and presence of bond coat. For each repair system the wavelet analysis of impact-echo signals was performed. The effects of concrete surface quality and presence of bond coat on the results of the signal wavelet analysis were discussed.

The research project was granted by Polish Scientific Research Committee - grant number KBN 4 T07E 027 27.

Wednesday Poster Session

Wednesday afternoon, 7 September, 15:50

Symposium H

Filler metals, solders and joints of the next generation

Welcome

Today's complex requirements for structural components often cannot be entirely fulfilled by a single material. Hence, different materials (metal and ceramics, advanced composites with conventional materials) have to be combined to form joints. When joining dissimilar materials, such as metals and ceramics, problems resulting from the differences in physical and thermal properties of the material groups have to be overcome.

Joining materials is also a most important process when producing electronic components. However, even in the case when the materials are mostly the same, or at least similar physical properties, the miniaturization trend and the environmental issues (lead prohibition in electronics) are confronting the scientific and engineering communities with new challenges.

Novel filler metals and new solders have to be developed and reliable joints have to be produced to meet the specific requirements. Therefore, the correlations between the joint design, the microstructure and the properties have to be understood. Residual stresses play a crucial role, especially in case of joints of dissimilar materials. The internal and external interfaces in the combined materials strongly affect the joint reliability and need to be characterized and controlled. Furthermore there are still several open questions concerning joint design, processes and characterization. The symposium should bring together the scientific community from different fields (e.g. physics, chemistry, material science and engineering) as well as industry to review and discuss the relevant issues which are prerequisites for stimulate progress in this area.

The topics are:

- Filler metal and solder development
- Lead-free solder joints
- Joint of dissimilar materials
- Metal-ceramic joints Composite joints, Laminates
- Processing
- Thermomechanical behaviour and residual stresses
- Novel experimental techniques
- Theoretical modelling and numerical calculations
- Joint durability
- Interfaces and size effects

The session is supported by the COST531 action "Lead-free solders".

Organisers

- **Jolanta Janczak-Rusch**, EMPA, Duebendorf, Switzerland
- **John Botsis**, Laboratory of Applied Mechanics and Reliability Analysis, Swiss Federal Institute of Technology, Lausanne, Switzerland
- **Herbert Ipser**, Institut f. Anorganische Chemie-Materialchemie, Universitaet Wien, Wien, Austria

Programme

Monday, 5 September

Monday Poster Session
Monday afternoon, 5 September, 15:50

15:50	poster	H-1

ELFNET: A European Network on Lead Free soldering

Herbert Ipser[1], Clemens Schmetterer[1], Adolf Mikula[1], Jeremy Pearce[2]

1. *University of Vienna, Department of Inorganic Chemistry - Materials Chemistry, Waehringerstr. 42, Wien A-1090, Austria* **2.** *ELFNET, Unit 3, Curo Park, Frogmore, Hertfordshire AL2 2DD, United Kingdom*

Contact: herbert.ipser@univie.ac.at

The EU RoHS Directive requiring removal of lead hazard from electronics production processes in Europe by 2006 is now in force and is in line with similar initiatives in Japan, in the US, and in China, seeking greater sustainability. The necessary lead-free soldering technology exists and has been in development for a number of years but now requires urgent research and industry coordination on a pan-European scale in order to support the transformation of European industry and improve its competitiveness.

Several major EU, Regional and National projects are under way or proposed and industrial implementation has already begun. However, the technology is seriously lacking integration and synergy, particularly with regard to candidate countries, and there is no coherent knowledge base.

'ELFNET – European Lead-Free Soldering Network', is a Coordination Action that addresses these problems by using a new organizational model to create a network of research institutions and industry bodies from across Europe. It is a 3-dimensional structure with National Networks in 19 countries, recognized Technical Experts from each field and Industry Networks from each sector. It aims to mobilize and optimize the necessary critical mass of research and development in an industry-led initiative and to provide technical in-

formation to each Member State.

ELFNET has recently moved into its second year. It now holds a database of 100 lead-free research projects and 350 lead-free experts across Europe. In addition, information on state-of-the-art and research gaps has been compiled in various Reports, accessed on the website by members. With these reports and the first European Implementation Status Report now complete, ELFNET has identified a set of key issues where further effort is urgently needed. This will be dealt with by the new 'Issues & Solutions' initiative.

15:50 poster H-2

Interaction of Ag-In-Sn Solders with Palladium Substrates: A Phase Diagram Approach

Adela Zemanova[1], Ales Kroupa[2], Jan Vrestal[1], Herbert Ipser[3], Christoph Luef[3], Hans Flandorfer[3]

1. *Masaryk University, Faculty of Sciences, Kotlarska 2, Brno 61137, Czech Republic* **2.** *Academy of Sciences of the Czech Republic, Institute of Physics of Materials (IPM), Zizkova 22, Brno 61662, Czech Republic* **3.** *University of Vienna, Department of Inorganic Chemistry - Materials Chemistry, Waehringerstr. 42, Wien A-1090, Austria*

Contact: adel_zeman@yahoo.co.uk

In order to understand the interaction of lead-free Ag-In-Sn solders with palladium substrates one has to know the quaternary Ag-In-Pd-Sn phase diagram, and for that all the constituent binaries and ternaries. For this purpose, enthalpies of mixing were determined calorimetrically for liquid ternary Ag-Pd-Sn and In-Pd-Sn and quaternary Ag-In-Pd-Sn alloys. These experimental results were taken into account as an input for the optimization in CALPHAD - type phase diagram calculations of the above-mentioned two ternary phase diagrams. These were then combined with an earlier optimization of the Ag-In-Pd system as well as with literature data on the Ag-In-Sn system to obtain a first idea of the phase diagram of the quaternary Ag-In-Pd-Sn alloy system. The calculations were supported by a number of specifically devised experiments.

15:50 poster H-3

Comparison of wetting and mechanical properties of Cu/Sn-based solder couples produced under vacuum and under flux

Artur Kudyba[1], Natalia SOBCZAK[1], Rafal Nowak[1], Waldemar Radziwill[1], Herbert Ipser[2], Borys Mikulowski[3], Adolf Mikula[2], Hans Flandorfer[2]

1. *Foundry Research Institute (FRI), 73 Zakopianska Street, Kraków 30-418, Poland* **2.** *University of Vienna, Department of Inorganic Chemistry - Materials Chemistry, Waehringerstr. 42, Wien A-1090, Austria* **3.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: akudyba@iod.krakow.pl

Despite the fact of a large number of works on wetting properties of Pb-containing standard solders and candidate lead-free solder alloys, there is a great uncertainty about the role of testing atmosphere and procedure on wetting behavior of these alloys and how these factors affect the correlation between solderability and mechanical properties of solder joints.

In this study, the wetting of Cu substrates by several Sn-based lead-free solder candidates has been studied by a sessile drop method in air using standard flux or in vacuum applying different testing procedures. The structure and chemistry of solidified and cross-sectioned solder/Cu couples were examined by optical and scanning electron microscopy. Mechanical properties of the same couples were determined by push off shear tests.

The analysis shows that there is no important difference in properties of selected solder/Cu couples obtained in vacuum and in air with flux. For situations when the same combination of solder/Cu couple under identical testing conditions (time and temperature) showed dissimilar wetting and mechanical behaviors, it was noted that either the solder or the Cu substrate had oxidized regions, responsible for a local dewetting and for the structural defects, all contributing to pure wetting and weakening of the couples.

This work was done under the group project GP7 entitled "Prediction of New Generation Solders" and realized in the framework of European Concerted Action on "Lead-free solder materials" (COST 531). The authors are grateful to the Ministry of Science and Information Technology of Poland and the Foundry Research Institute for financial support.

15:50 poster H-4

Description of the mechanical performance of Cu-SnAgCu-Cu joints

Aissa Mellal[1], Thomas Rütti[2], John Botsis[1], Jolanta Janczak-Rusch[2]

1. *Ecole Polytechnique Federale de Lausanne (EPFL), Ecublens, Lausanne 1015, Switzerland* **2.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*

Contact: aissa.mellal@epfl.ch

Sn-4.0Ag-0.5Cu lead-free solder was used for the joining Cu plates of 1 mm in thickness. A combined theory-experiment approach was applied to describe the mechanical performance of the solder joints. Experimental data for solder joints with different gap widths (0.1-1mm) were obtained in tensile tests. The resulting stress-strain curves showed a decrease in tensile strength and an increase in yield strain with increasing gap widths. The joints with a gap thickness of 1mm were considered as reference joints for finite element simulations in which an elasto-visco-plastic behaviour of the solder was assumed. By comparing theoretical and experimental results (force-displacement characteristics) to the reference joints the constitutive behaviour of the solder was identified. The resulting model parameters were then used to predict the solder joint behaviour for defined geometrical constraints. Considering the inherent differences between specimens, due to processing and local defects, a good agreement between the theoretical and experimental data was found for the effect of the gap thickness.

15:50 poster H-5

Optimization of particle reinforced lead-free solders

Stefan F. Awietjan[1], Vinzenz Bissig[2], Krzysztof J. Kurzydlowski[1], Jolanta Janczak-Rusch[2]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Empa Materials Science and Technology (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*

Contact: stefan_a@wp.pl

The goal of this research was to optimize the structure and manufacturing process of a particle reinforced lead-free solder. The aim of the particles incorporation was to improve mechanical properties of the matrix solder, particularly the creep resistance. The main problem was a high porosity of the obtained composite solders samples and not homogenous particle distribution.

As a matrix solder Sn-4,0Ag-0,5Cu alloy in form of paste and powder was used. Three different particles types were used as a reinforcement; two types of nickel and copper particles. The particles were introduced by mechanical mixing, using a ball mixer under different mixing conditions. The amount of particles used was 5% wt.

The influence of particles type and mixing conditions upon the solders porosity was investigated. Two types of fluxes were added to the paste during mixing with particles and their influence examined. The Archimedes method was used for density measurements. Obtained composite solders microstructures were investigated with light and SEM microscopes.

The Lead-free composite solder joints and solder joints with different gaps were also prepared and tested. The optimal mixing and soldering parameters for composite solders were found. Obtained microstructure and particles arrangement is uniform. Composite samples with very low amount of pores were obtained.

15:50 poster H-6

Development of nanoparticle reinforced brazing filler metals and solders: the aspects of downscaling from micro to nanoscale

Vinzenz Bissig, Jolanta Janczak-Rusch

Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland

Contact: vinzenz.bissig@empa.ch

The composite approach is used to develop particle reinforced brazing filler metals and solders with tailored properties. For some applications the dimension of the particles and/or the brazing gap thickness has to be downscaled. Especially in case of microjoints, nanoparticles offer a possibility to modify the filler properties within a small volume. The experience gained by the development of composite fillers with microsized particles help by designing the nanocomposite fillers. However, a number of aspects have to be considered when downscaling from micro- to nanoparticle composite solders:

- the processing methods resulting in an uniform distribution of microparticles in a filler metal are do not ensure a homogenous distribution of nanoparticles in this filler (are not directly transferable)
- high specific surface area of nanoparticles increases the particle reactivity and may change the chemical composition of the base filler metal,
- even fine reaction layers (e.g. sub microns region) are in case of nanoparticles sufficient to converse them completely into reaction products,
- with the smaller size of particles added the viscosity of the liquid filer metal may increased by agglomerates of the reinforcement particles
- the “reinforcement” mechanisms differ for the micro- and nanosized particles.

Examples from own research are shown to illustrate these aspects.

15:50 poster H-7

Ti layer ion assisted deposition in metal ceramic bonding

Viorel T. Braic, Adrian E. Kiss, Mihai Balaceanu, Alina Vladescu, Mariana T. Braic

National Institute for Optoelectronics (INOE), 1 Atomistilor Str., Bucharest 77125, Romania

Contact: vbraic@inoe.inoe.ro

In active metal – ceramic bonding, reactions taking place between Ti and Al_2O_3 are of particular interest. The reliability of the joint depends on the metal-oxide reactivity and, consequently, on the interfacial microstructure.

The paper is presenting the results obtained in the vacuum brazing of alumina and copper parts using Ti as an interlayer between alumina and AgCu brazing alloys. Ti layer was deposited by vacuum cathodic arc method using ion bombardment with Ti ions originating from the highly ionized arc plasma, at a substrate temperature in the range of 250^0 C to 350^0 C. In order to control the shape of the deposited area, metallic Ti masks were used. As it is well known, the energy and intensity of the ion flux impinging on the substrate have a significant influence on the deposited film and interface structure. The higher the energy and density of the ions, the deeper the intermixing zone at the interface. The effects of the alumina substrate bombardment with medium energy Ti ions (0.5 ÷ 2.5 keV) – resulting from dc and pulsed biasing of the substrate during deposition – on some characteristics of the Al_2O_3/Ti interface were analyzed. XRD was used for phase identification at the interface. Elemental composition (AES), chemical nature and oxidation state (XPS) of the elements comprising the interface were also determined.

Different experimental conditions may affect the diffusion path in the Ti-Al-O system. In our case, the long term diffusion bonding is not applying, but it was observed that at low temperature deposition, Ti reacts with Al_2O_3 forming titanium oxides. The purpose of the present work is to investigate the interfacial reaction products in Ti/Al_2O_3 joints obtained in the context of diffusion taking place during Ti vacuum arc ion assisted deposition and during the brazing process (TiAgCu alloy formation). The characteristics of the interface were corroborated with the alumina/copper joint strength.

15:50 poster H-8

Model for intermetallic phase and intermetallic compound solidification during diffusion soldering

Waldemar Wołczyński[1], Jacenty Kloch[2], Krzysztof J. Kurzydlowski[3], Jolanta Janczak-Rusch[4], Edward Guzik[5]

1. *Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland* **2.** *Polish Academy of Sciences, Institute of Mathematics (IM PAN), Św.Tomasza 30, Kraków 31 027, Poland* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **4.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland* **5.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: nmwolczy@imim-pan.krakow.pl

Two modes of calculation are presented for both intermetallic phase and intermetallic compound formation during solidification. The presented equations consider a solute microsegregation resulting from partitioning, primary solute redistribution resulting from back-diffusion, secondary solute redistribution required by peritectic reaction and formation of intermetallic phase/compound resulting from peritectic reactions. The formulated set of equations is referred to the concept of undercooled liquid of the selected initial solute concentration formed due to dissolution of the liquid filler metal into the substrate. The solidification path is precisely defined in a given equilibrium phase diagram. The solidification path is accomplished within the infinitesimally small amount of the undercooled liquid, , according to the concept. The solidification occurs at the front of each growing sub-layer so that the undercooled liquid is forced to move from the substrate towards the axis of symmetry of the forming joint. The formulated equations realize the idea of moving undercooled liquid and are able to reproduce both amount of growing sub-layers as well as solute redistribution across the forming joint for which solidification was arrested. The confrontation of both theoretically predicted and measured profile of solute redistribution is shown for two solders Ni/Al/Ni and Fe/Zn/Fe. The current model is universal one and can also be applied to metastable phase diagram in order to emphasize that diffusion soldering occurs under metastable conditions.

15:50 poster H-9

Relationship between condition of deposition and properties of W-Ti-N thin films prepared by reactive magnetron sputtering

Andrian V. Kuchuk[1], Vasyl P. Kladko[1], Oksana S. Lytvyn[1], Anna Piotrowska[2], Roman Minikayev[3], R Ratajczak[4]

1. *V.Lashkaryov Institute of Semiconductor Physics NAS Ukraine (ISP), Nauky prosp., Kyiv 03028, Ukraine* **2.** *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland* **3.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland* **4.** *The Andrzej Sołtan Institute for Nuclear Studies (IPJ), Poland*

Contact: kuchuk@isp.kiev.ua

In this work we studied the relationship between condition of deposition (especially, nitrogen partial pressure) and material properties (chemical and phase compositions, microstructure, mechanical stress, surface morphology, resistivity, diffusion barrier performance), of W-Ti-N thin films.

Thin binary W-Ti and ternary W-Ti-N films were deposited by reactive magnetron sputtering from a W-Ti (30 at.%) target in Ar or Ar/N_2 discharges. A d.c. power density of 1.7 W/cm^2 was applied to the target and the partial pressure of N_2 was varied from 0 to 0.35 Pa at a total gas pressure of 0.5 Pa.

The investigation of W-Ti-N films by XRD, RBS, AFM, four-point probe and stress measurements showed that the structure, composition, resistivity, stress and barrier properties are strongly dependent on the nitrogen concentration in these films.

We found that nitrogen-free film was composed of a β-W matrix seeded with fine α-Ti precipitates. For $W_{65}Ti_{17}N_{18}$ films, the structure is a dense mixture of ultrafine crystallites of tungsten, TiN, W_2N and/or Ti_2N and amorphous phase. At high nitrogen concentrations $N > 30$ at.%, the structure is a mixed phase of W_2N and TiN. The resistivity of pure $W_{88}Ti_{12}$ film was 100 μΩcm and rose slowly up to 365 μΩcm for the ternary $W_{45}Ti_{10}N_{45}$ film. When the N_2 partial pressure reaches 0.35 Pa, the film resistivity is 850 μΩcm, which may be attributed to the reduction in crystalline size. The 100 nm thick amorphous $W_{65}Ti_{17}N_{18}$ film shows excellent barrier property preventing the interaction between Au and GaAs under annealing at 750° C.

Research was partially supported by grant from NATO (ref. NUKR.RIG.981275), and one author (A.V. Kuchuk) was supported by stipendium of NASU for young scientists.

15:50 poster H-10

Tensile deformation behaviour of the titanium alloy with hard elastic coating

Waldemar Ziaja, Jan Sieniawski

Rzeszow University of Technology, Rzeszów, Poland

Contact: wziaja@prz.edu.pl

The increasing demands for higher performance and more severe application conditions have been the main driver for wider use of titanium alloys for engineering components. The main reason for that is favourable combination of properties like high specific strength, good corrosion resistance and biocompatibility. On the other hand poor tribological properties restricted use of titanium alloys to non-tribological applications.

This drawback can be eliminated by modifying the surface layer of the alloy. Various surface engineering techniques including PVD and plasma nitriding can be used to produce metallic nitride coatings and more recently DLC coatings were successfully applied. However plastic deformation of the substrate leads to premature failure of the usually elastic coating when the high stresses are encountered. That is because coating and substrate materials posses significantly different strength properties i.e. Young's modulus. Additionally the difference in thermal expansion coefficient leads to development of high residual stresses during the process of coating deposition. Cracking of the hard coating leads to stress concentration and localized plastic deformation of the substrate that can modify macroscopic deformation behaviour of the system.

The life of the coating can be increased by introducing intermediate hardened surface layer reducing plastic deformation of substrate material. Main factors controlling deformation behaviour of the material with hard coating are: (i) the difference in Young's moduli between coating and substrate material, (ii) yield strength of substrate material, (iii) coating thickness, (iv) magnitude of residual stresses in the coating, (v) depth of the crack in the coating. In the work influence of these factors on tensile behaviour of titanium alloy with hard elastic coating was investigated by means of FEM method to enable tailoring the resistance to plastic deformation and increase load bearing capacity of this system.

15:50 poster H-11

Characteristics of welded joints and cracks occurred during pad welding of superalloy Inconel 713C

Maciej Lachowicz, Włodzimierz Dudziński

WROCŁAW, Wroclaw University of Technology, Institute of Materials Science and Applied Mechanics (PWr - IMMT), Smoluchowskiego 25, Wrocław 50 - 370, Poland

Contact: maciej.lachowicz@pwr.wroc.pl

The paper presents results of a metallographic examination of padding welds made by TIG method in almost unweldable casting superalloy Inconel 713C. During the research, microstructural changes were observed and identified in padding welds, in heat-affected zone and in cracked areas. The created phases were identified and their morphology characterised in the fused zone and in the cracked areas. In the research, the methods of light, scanning and transmission microscopy as well as X-ray structural analysis were applied. It was found that structure of the padding weld showed nanostructural features and the present cracks were caused by stresses resulting from low thermal conductivity of the alloy, high crystallisation velocity and interdendritic microporosity. The cracks observed in the heat-affected zone were caused, beside high thermal stresses, also by precipitates of secondary M6C type carbides with lower melting temperature and by creation of low-melting eutectics (γ\-γ\') with precipitates of secondary carbides MC and M_6C.

15:50 poster H-12

Structure and properties of joints of 14-5 PH steel and PM Fe-TiC composite using filler metal BNi2/MBF 20

Jerzy Nowacki

Technical University of Szczecin, Institute of Materials Science and Engineering (TUS), Piastów 19, Szczecin 71-310, Poland

Contact: jnowacki@ps.pl

Soldering tests of steel 14-5 PH and cermets - PM Fe-TiC composite plates using filler metal BNi2/MBF 20 on a nickel matrix were carried out. Steel 14–5 PH has high mechanical and corrosion resistance. Parameters of heat treatment of steel 14–5 PH are similar to parameters of soldering and heat treatment to the considered composites. This cermets belong to a group of sintered composites with a metallic matrix reinforced by particles of properties halfway between tool steel and sintered carbides. PM Fe-TiC composite are composites of a stainless steel matrix reinforced by titanium carbide particles.

Brazing process was done in vacuum according a special thermal cycle programme. On produced connections following was done: metallographic tests on an optical microscope and scanning microscope in transverse metallographic, quality and gravimetrical analysis, measurements of hardness and tensile strength test of a connection.

Solder shown good adhesion to a surface, no oxides and discontinuities both from a steel side and plates from PM Fe-TiC composite, and no cracks in plates being a result of brazing. The connection has a typical for binding metal BNi2 eutectic structure together with a zone of intermetallic phases in a separation plane steel – binding material and binding material – plates from PM Fe-TiC composites of a bonding matrix. Zones of intermetallic compounds differ clearly with a chemical composition and hardness from a bonding matrix. Obtained bindings have multiplayer structure.

In a separation plane PM Fe-TiC composite – binding material there are reaction zones having a chemical composition different from a binding material matrix. The reaction zone from a PM Fe-TiC composites side is rich in titanium, molybdenum and iron, in the bond

15:50 poster H-13

The Wetting Behaviour and Interface Structure of Unreinforced and Particle Reinforced Lead-Free Solders

Rafal Nowak[1], Artur Kudyba[1], Natalia SOBCZAK[1], Waldemar Radziwill[1], Thomas Rütti[2], Jolanta Janczak-Rusch[2], Borys Mikulowski[3]

1. *Foundry Research Institute (FRI), 73 Zakopianska Street, Kraków 30-418, Poland* **2.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland* **3.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: rnowak@iod.krakow.pl

The wetting behaviour of the SnCuAg solder on Cu and Ni substrates has been studied by a sessile drop method using the solder in the form of bulk material, solder paste or composite solder paste reinforced with 10% metallic particles (either Cu or Ni). Structural characterization of the interfaces and evaluation of their mechanical properties have been done on solidified sessile drop couples. The effects of particulate reinforcement, procedure used for the preparation of composite paste solder and testing conditions on the structure of the solder before and after wettability test as well as on interface structure of the solder joints were analized in order to explan different wetting behaviours and mechanical properties of corresponding joints.

This work was done under the group project GP7 entitled "Prediction of New Generation Solders" and realized in the framework of European Concerted Action on "Lead-free solder materials" (COST 531). The authors are grateful to the Ministry

of Science and Information Technology of Poland and the Foundry Research Institute for financial support.

Tuesday, 6 September

Parallel Session
Tuesday morning, 6 September, 9:00
Main Building, room 144

9:00 oral

Developing New Lead-free Solder Materials: COST 531, a Concerted European Action

Herbert Ipser

University of Vienna, Department of Inorganic Chemistry - Materials Chemistry, Waehringerstr. 42, Wien A-1090, Austria

Contact: herbert.ipser@univie.ac.at

A European Action on lead-free soldering has been initiated in 2002 under the COST Program of the EU. Its goal is the establishment of a database containing the relevant knowledge on possible lead-free solder materials and to provide the expertise for selecting particular materials for specific soldering purposes. By May 2005, 22 European countries have signed the corresponding Memorandum of Understanding, in addition, three non-European research organizations have been accepted as partners, with one application (Ukraine) pending.

The Action is structured into four Working Groups: WG 1 is responsible for experimental data on phase equilibria and thermochemical properties, WG 2 carries out the theoretical modeling of the corresponding phase diagrams, WG 3/4 investigates physical and chemical properties, and WG 5/6 is mainly responsible for reliability issues. Currently scientists from about 45 European research institutions are involved. WG 1 and 2 (together with SGTE) have identified nine quaternary systems as the immediate goal for a thermodynamic database; they comprise five possible ternary solder alloys together with relevant substrate materials. WG 3/4 and WG 5/6 cooperate in setting up a database on physical and mechanical properties of possible lead-free solders.

In our laboratory in Vienna we are currently working on a number of ternary systems to understand the interaction of Sn-Ag-Cu and Sn-Ag-In solders with Ni or Pd substrates. This research comprises experimental phase diagram investigations, thermodynamic measurements, and CALPHAD type optimizations. In addition, we are investigating various physical and mechanical properties of solder joints formed between Sn-Ag and Sn-Ag-In alloys and Cu contacts, in particular the so-called size effect, i.e. the influence of the size of the joint on the mechanical properties.

Research in Austria is supported by the Austrian Science Foundation under Projects No. P15620, P16495, and P17346.

9:30 oral

The Experimental and Theoretical Study of the Pd-Sn-Zn System

Jiri Vizdal[1,2], Ales Kroupa[1]

1. *Academy of Sciences of the Czech Republic, Institute of Physics of Materials (IPM), Zizkova 22, Brno 61662, Czech Republic* **2.** *Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Technicka 2896/2, Brno 61669, Czech Republic*

Contact: vizdal@ipm.cz

The experimental and theoretical study of the Pd-Sn-Zn system was carried out in the scope of this contribution. The knowledge of the phase diagram of this system is important from the point of view of the lead-free soldering as the Sn-Zn system is one of the candidate materials and Pd can be found in a substrate of the soldered joint. The binary systems Pd-Sn and Sn-Zn are currently assessed [1,2] whereas a few information about Pd-Zn exists but the first assessment of the Pd-Zn system was carried out recently [3].

Binary systems information was utilized for a calculation of the above mentioned ternary system prediction and the experimental alloys in significant regions were prepared. Authors have chosen Sn-Zn rich part of the phase diagram where calculations indicate the possibility of the miscibility gap presence in liquid phase. The samples were long term annealed at selected temperatures to obtain states close to the thermodynamic equilibrium and afterwards studied by SEM with EDX and WDX analysis. Experimental results and first Pd-Sn-Zn system modelling will be presented in this contribution.

This work is supported by the Ministry of Education of the Czech Republic under the project COST 531.002 and Grant Agency of the Academy of Sciences of the Czech Republic No. S2041105.

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Presented at international conference TOFA 2004, Book of Abstracts, Vienna, Austria, September 2004.

9:50 oral

Size and constraining effects in lead-free solder joints

Joël Cugnoni[1], Aissa Mellal[1], Jolanta Janczak-Rusch[2], John Botsis[1]

1. *Ecole Polytechnique Federale de Lausanne (EPFL), Ecublens, Lausanne 1015, Switzerland* **2.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*

Contact: joel.cugnoni@epfl.ch

Because of the tremendous developments in advanced processing technologies, the dimensions of contemporary electronic devices and interconnections have become smaller and smaller. Moreover, in recent electronic designs the solder joints may not only be electrical interconnections but may also play a significant role in the mechanical stability of the joint.

Due to the strong influence of geometrical constraints and processing parameters, the macroscopic stress-strain constitutive law of lead-free solder materials must be determined in the most geometrically and physically realistic conditions. In order to identify the elasto-visco-plastic constitutive law of Sn-Ag-Cu solders, a special optical strain measurement technique based on Digital Image Correlation has been developed to study the strain field in an idealized joint during a tensile test. Experimental results of the stress-strain relationship at room temperature of geometrically constrained Sn-Ag-Cu joints have been evaluated.

The measured strain field (Digital Image Correlation) and load data have then been used in an inverse numerical identification procedure to determine the true elasto-plastic constitutive law of the solder. The effects of geometrical constraints in a real solder joint with heterogeneous stress and strain fields are then studied by comparing the apparent (constrained) and true (non-constrained) stress-strain relationships. Experimental stress-strain relations for Sn-Ag-Cu joints of 0.25 mm , 0.5 mm and 1.0 mm thickness are presented and the constraining effects are discussed.

10:10 oral

Local fatigue in lead-free SnAg3.8Cu0.7 solder

Pascal P. Jud[1], Guenter Grossmann[1], Urs Sennhauser[1], Peter J. Uggowitzer[2]

1. *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland* **2.** *Laboratory of Metal Physics and Technology, ETH (LMPT, ETH), Wolfgan-Pauli-Strasse 10, Zurich 8093, Switzerland*

Contact: pascal.jud@empa.ch

In this study unified shear samples with a thin (100 µm) solder gap and an area of 10x10 mm^2 were investigated. The test setup was especially designed for pure shear stress. To understand the deformation behaviour the specimens were marked with various surface patterns. The patterns were etched and Pt deposited by Focused Ion Beam (FIB). It was found that the deformation of the solder is inhomogeneous in unidirectional shear tests and as well in cyclic testing. The deformation is concentrated in zones mostly parallel to the shear direction. The width of these areas found was between 5 to 20 µm by a length of 20 to several hundreds microns. The FIB etched patterns in the submicron range allowed investigating the deformation in detail with dislocation climbing, grain boundary sliding and recrystallization. With cross-sections done by FIB in the before mentioned zones it could be shown that there were no cracks generated during the primary and secondary creep.

Unidirectional shear creep tests showed an intense microstructure dependence of the shear creep rate. Two different microstructures were investigated. Water quenched samples, which showed a distinct finer microstructure with β-Sn phases of about one fourth of the ones of the slowly solidified samples. The water quenched samples exhibited a larger temperature sensitivity of the stationary creep rate and showed higher strain rates, especially at elevated temperature, compared to the slowly solidified samples.

In isothermal cyclic testing it could be shown by the FIB etched markers that there were as well zones with concentrated deformations established. There are two different mechanisms at work in the degradation of the sample. The first was found to form deformation zones and the second was the fatigue to rupture in these zones. The microstructure sensitivity is observed as well in the cycling tests. It was found that the quenched samples withstand less cyclic deformation.

Parallel Session

Tuesday afternoon, 6 September, 14:00
Main Building, room 144

14:00 oral

Evaluation of lead free soldering development by means of a holistic consideration

Matthias Tuerpe

Behr GmbH & Co. KG, Mauserstr. 3, Stuttgart 70469, Germany

Contact: matthias.tuerpe@behrgroup.com

In the engineering world, the way of thinking still is orientated towards step-by-step-processes. As one example, it is well known that the main costs are determined during and by the design. In the end, every division does her job without looking at the neighbouring demands. This includes university research where results are presented adding that now it

is up to the industry to work with it, the university job are fundamentals only.

But a succesful development needs the cooperation and knowledge of all different participants. The lonely researcher in his laboratory is history today. Connexions have to be understood and used as an important part of work.

With a general view on actual demands as well as the critcal weakness of today's procedures, one comes to the conclusion that a holistic approach only will lead to a change. For the development of such a model, brazing and soldering is one of the fields in research and industry well suited due to the fact that different people with different knowledge must cooperate all times in order to achieve success.

In the end, the existence of a holistic parameter as an intersection of interacting basic items can be derived. Based on a material approach, the material intelligence can be defined consisting material properties, manufacturing, service conditiones and organisational requirements.

For a confirmation of the model as well as the applicability in practice, the lead free soldering enforced by EU-regulations is taken. It is not sufficient to develop alloys for filler materials only. At the same time and very early, aspects of workability, reliability and commercial questions as the availability of alloying elements respectively must be taken into account.

For research and industry, the correlation of knowledge, information and communication is of increasing importance. Not the single result, but the classification inside a system is to be demanded.

14:30 oral

Microstructural evaluation during particles addition to lead-free solders

Paulina Unifantowicz[1], Jolanta Janczak-Rusch[2], Thomas Ruetti[2], Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Empa Materials Science and Technology (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*

Contact: paulau@interia.pl

The objective of the current research was the examination of microstructural features in lead-free solders and solder joints with incorporated particles. To improve the mechanical properties of solder joints the solders microstructures were modified by the addition of particles. Fine, evenly distributed particles can restrict grain growth, dislocation motion and crack propagation so as to strengthen the solder against creep and fatigue. The composite solders were produced by mechanical mixing and in-situ methods. Sn-3.5Ag and Sn-4.0Ag-0.5Cu solders were matrix alloys and Cu, Ni particles as well as Cu-coated glass spheres for reinforcement. The amounts of reinforcements were in the range of 1-15 vol. %. A comparison is made between the composites microstructures when various types, sizes and volume fractions of particles and different matrix alloys were used. The assessment criteria were the chemical reactions between the particles and the matrix, tendency for agglomeration and uniformity of the particles distribution. Simulation of the aging process was carried out to investigate the changes in the interfacial intermetallic layers with aging time.

The microstructures were investigated using SEM with an integrated EDX system and EPMA. Topographic images of the areas at the solder - substrate interface were derived from the AFM examinations. Tensile tests were conducted both for the bulk non-composite and the composite solder joints. The fractography of the tensile tested specimens is presented.

14:50 oral

Thermal oxidation study on lead-free solders of Sn-Ag-Cu and Sn-Ag-Cu-Ge

Sang Wan Cho, Yeonjin Yi, SeongJun Kang, Kwangho Jeong, C. N. Whang

Yonsei University, Institute of Physics and Applied Physics (IPAP), Seoul 120-749, Korea, South

Contact: dio8027@phya.yonsei.ac.kr

Recently, preventing environmental pollutions, lead-free(Pb-free) solders are about to replace tin-lead(Sn-Pb) eutectic solders. Sn-Ag-Cu alloys are leading candidates for lead free solders. However Sn-Ag-Cu alloys have some problems for manufacture. Among those, most critical problem is that Sn-Ag-Cu alloys are oxidized easily after aging at high temperature(150□). To prevent oxidation problem of Sn-Ag-Cu alloys, Sn-Ag-Cu-Ge alloys were developed, but the mechanism of thermal oxidation has not studied yet. So we report the surface oxidation mechanism of lead-free solders in this paper. This mechanism has been investigated after high temperature storage using X-ray photoelectron spectroscopy. It was founded that, in Sn-Ag-Cu-Ge alloy system, the solder surface is surrounded by GeO_2, and then the surface of metallic solder is protected by this thin GeO_2. Based on this result, we have performed the package level reliability test using solder ball of Sn-Ag-Cu-Ge alloy, and compared with normal Sn-Ag-Cu alloy.

15:10 oral

Microelectrochemical corrosion studies on lead-free Sn-Ag-Cu solder alloys

Oliver von Trzebiatowski, Jolanta Janczak, Thomas Suter

Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland

Contact: oliver.trzebiatowski@empa.ch

New solder alloys are being developed for electronic devices in order to replace traditional solder materials that contain harmful lead. A research group of the Empa is involved in developing new Sn-Ag-Cu based solder alloys. Particles are specially added in order to enhance the joint mechanical performance. The preliminary results show that there is a great potential for improvement of the mechanical properties of Sn-based solders especially of the creep resistance by the particle reinforcement. Whereas intensive studies focused on the physical, electrical and mechanical behaviour, no corrosion data are available at moment. Thus, the goal of this work is to characterize the corrosion behaviour of lead-free Sn-3.8Ag-0.8Cu alloys with and without different reinforcing particles. Electrochemical corrosion measurements in 1 M NaCl solution showed similar pitting potentials for all alloys, at around -430 mV (SCE). However, a quite strong variation of the corrosion potential and the passive current was observed. Although the new solder alloys show heterogeneous microstructures with reinforcing particles, intermetallic inclusions and pores, the corrosion behaviour is improved compared to the Pb-Sn standard solders. In order to further enhance the corrosion stability, microelectrochemical measurements are being performed to localize pitting initiation sites. It is assumed, that compositional and structural heterogeneities, like pores or particles trigger the onset of localize corrosion. An electrochemical microcell using microcapillaries filled with electrolyte is used to perform electrochemical tests on structures down to 0.5 μm. Preliminary microelectrochemical experiments on pore and particle free zones revealed a significant better corrosion behavior. The pitting potentials were shifted several 100 mV to higher values. At the moment we are trying to clarify the detrimental influence of pores and particles on the corrosion behaviour of Sn-Ag-Cu solder alloys.

Parallel Session

Tuesday afternoon, 6 September, 15:50
Main Building, room 144

15:50 oral

The effect of intermetallic compound on shear strength of diffusion soldered interconnections

Joanna Wojewoda, Paweł Zięba

Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland

Contact: nmwojewo@imim-pan.krakow.pl

The study presents capabilities of diffusion – soldering technology as an simple way to achieve mechanically stable interconnections which can be useful especially in electronic equipment. The joint is formed due to isothermal solidification and subsequent diffusion in solid state and it consisits of one or more intermetallic phases with melting temperatures much higher than the temperature used for the fabrication of the joint. The diffusion soldering is also a potential candidate for replacement of harmful Pb-Sn solders. In the present study the process was applied to join the copper substrates by the thin foil of low melting materials – either In-Bi (72 C) or In-Sn (119 C) eutectic alloys.

INSTRON tensile testing machine was used for shear strength tests of the joints fabricated in the broad range of the temperatures (100 – 300 C). Scanning Electron Microscopy investigation was used to identify the intermetallics present in the interconnection area and also to describe the fracture surface after the mechanical test.

16:10 oral

Transition from solidification to the first solid/solid transformation during diffusion soldering

Waldemar Wołczyński[1], Edward Guzik[2], Jolanta Janczak-Rusch[3], Dariusz Kopyciński[2], Jacenty Kloch[4]

1. *Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland* **2.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland* **3.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland* **4.** *Polish Academy of Sciences, Institute of Mathematics (IM PAN), Św.Tomasza 30, Kraków 31 027, Poland*

Contact: nmwolczy@imim-pan.krakow.pl

Formation of the Ni-Al-Ni and Fe-Zn-Fe joints in relation to the diffusion soldering is considered. The sequence of appearing intermetallic phases during diffusion soldering is observed by arresting solidification or first solid/solid transformation. The solute segregation measurements are performed to estimate the average solute concentration during solidification and first solid/solid transformation. A competition between metastable phase and stable phase growth during first period of solidification is considered. The period of time for a transformation of the liquid filler metal into technologically justified liquid solute solution is identified and analysed. The birth of the phase second in sequence (for the Ni/Al/Ni joint) or birth of the phase third in sequence (for the Fe/Zn/Fe joint) is revealed and described. The zero number degrees of freedom for both systems Ni/Al/Ni and Fe/Zn/Fe is considered from the view point of the Gibbs phase rule. Some suggestions for a solidification model together with solidification path are proposed. The first solid/solid transformation is observed and related to the previous period of solidification. The maintenance of the average solute content from solidification during the first solid/solid transformation is supposed and analysed. The first solid/solid transformation is described as a kind of chemical reaction and shown experimentally as consumption of the second phase in sequence (for the Ni/

Al/Ni joint) or third phase in sequence (for the Fe/Zn/Fe joint) by a dominant phase. The revealed consumption named as a “mantis” phenomenon is an independent behaviour of the system and occurs in sequence after solidification.

16:30 oral

Influence of alloying elements on diffusion welding of light weight hybrid joints

Jean Pierre Bergmann, Johannes Wilden

Technische Universität Ilmenau, Fachgebiet Fertigungstechnik, Neuhaus 1, Ilmenau 98693, Germany

The set of joints between dissimilar materials gained to a very important goal for joining specialists in the last decades. In automotive the increased interest in light weight concepts leads to set in a component contemporarily different light weight metals. Furthermore in other sectors of industrial application joining of dissimilar materials is a focus point. Diffusion welding represents a very attractive joining technology for advanced materials, especially when conventional fusion welding processes degrade the properties of the materials in the heat affected zone and in the melt pool, as it is performed at temperatures between 0,5 and 0,8 of the melting temperature.

Diffusion welding offers the unique possibility to achieve a metallurgical joining in the solid state without low melting filler, thus avoiding large brittle intermetallic phases, which form in the melt pool while cooling. In this paper the joining of aluminum to titanium will be presented. As aluminium can be dissolved till about 10 at.-% in the □-Ti crystal for temperatures below 550C, the formation of brittle intermetallic phase can be avoided. First investigations were performed on Al99,5 and cp-Ti, in order to understand the diffusion process for technical pure materials. In further investigations AA5xxx and AA6xxx are joined with cp-titanium (T<550C), in order to evidence the effect of alloying elements. The joints are evaluated by optical as well as SE-microscopy. EDX-analysis allows the definition of the diffusion zone as well as the composition. Comparable investigations when joining aluminium to magnesium are reported as well. Selected results upon mechanical behaviour of joints will be reported too, and confirm the suitability of the process for high strength applications.

16:50 oral

Practical progresses for novel fabrication of structural oxide/oxide ceramic matrix composites

Heinrich Kern[1], E. Stoll[1], P. Mahr[1], H-G. Krüger[1], A. R. Boccaccini[2]

1. *Institute of Materials Technology, Technical University of Ilmenau, Gebäude "Werkstoffe 1", Ilmenau 98693, Germany* **2.** *Imperial College London, Department of Materials, London, United Kingdom*

Contact: Kern@tu-ilmenau.de

Oxide/oxide-ceramic matrix composites (CMCs) are a new trend of materials that offer the advantages of ceramics like resistance to heat, erosion and corrosion, while adding toughness and thermal shock resistance. These materials are used where designers seek less downtime, reduced maintenance, lower operating cost, increased operating temperature, increased efficiency, lower emissions and reduced life-cycle costs. In the present work a method based on electrophoretic deposition techniques has been developed to fabricate continuous oxide fibre reinforced ceramic matrix composite. A new EPD cell was designed and built to warrant the improved infiltration of the oxide ceramic particles in several fibre mats used as reinforcement. For electrophoretic deposition of the ceramic component, a non-aqueous suspension consisting of alumina nanoparticles (□-Al2O3), ethanol and addition of 4-hydroxybenzoic acid was optimised by electro kinetic sonic amplitude (ESA) measurement. This kind of suspension is produced as a model to describe and characterize the mechanism of the electrical deposition to infiltrate oxide fibre mats (NextelTM 720). The composites exhibit a homogeneous matrix microstructure, characterised by a very high particle packing density, a convenient fibre content and relatively low porosity after sintering at 1300 C without holding time. The developed EPD cell allows for production of relatively large bodies with a diameter of 110 mm and “pseudo-ductile” fracture behaviour of the composite during the action of a reasonable strength value has been achieved. The electrophoretic deposition (EPD) method is presented as convenient alternatively technique to fabricate oxide/oxide CMC’s with improved high matrix density, high fibre content and suitable mechanical properties

Wednesday, 7 September

Parallel Session
Wednesday morning, 7 September, 9:00
Main Building, room 144

9:00 invited oral

Potential Vacuum Brazing – State of the art and developments from the perspective of a service contractor

Manfred Boretius

Listemann AG (Listemann), Gewerbeweg 18, Mauren 9493, Liechtenstein

Contact: m.boretius@listemann.com

Vacuum brazing is widely used to join sophisticated components for a broad range of industrial applications. Combining different materials by brazing offers new opportunities to design engineers to improve existing or to create new functions. Obviously a service contractor is engaged in all industrial application areas and has to handle all engineering materials. Beside full scale production he has to offer the ability to manufacture prototypes for evaluation as well as engineering resources to shorten customers time-to-market.

This paper will show the potential of vacuum brazing by case studies from mechanical engineering and tool industry as well as by development results in manufacturing high-performance abrasive tools and heat exchanger for gas turbine application.

9:30 oral

Comparison of three different active filler metals used for brazing ceramic-to-ceramic and ceramic-to-metal joints

Vinzenz Bissig, Jolanta Janczak-Rusch

Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland

Contact: vinzenz.bissig@empa.ch

Active brazing is a method for producing reliable and cost-effective ceramic-to-ceramic and ceramic-to–metal joints. The suitability of three different active filler metals Ag98.6In1Ti0.6, Cu74.5Sn14.0Ti10.0Zr1.5 and Ag59.0Cu27.25In12.5Ti1.25 for joining Si_3N_4/TiN composite ceramics for application at elevated temperatures (up to a maximum of 350C) was studied. Ceramic-ceramic and steel-ceramic joints were brazed and tested in bending as well as in tension up to 600C. Fractographic investigations were performed to investigate the joint failure mechanism. A Scanning Electron Microscope (SEM) with an integrated Energy Dispersive X-Ray Analysis (EDX) was used for microstructural analysis of the brazed zone. In addition, the micro-hardness was measured across the brazed zone (from one joining partner to other). The micro-hardness of the various phases of the brazing filler metal in the as-melted (in crucible) and as-brazed (in a joint) was compared.

The amount of the active element titanium has to be very carefully optimised under consideration of the properties of the joining partners. Increasing amounts of Ti up to a critical value improves the wetting behaviour of the filler metal on the ceramic. However, the variation of Ti content also changes the spreading behaviour of the brazing filler and strongly affects its reactivity. As a result, undesirable brittle reaction products may form at the interface between the metallic joining partner and the filler metal, thereby altering the properties of both of the metallic joining partner and filler metal. In particular, the brazing filler may lose its ductility which is essential for reducing residual stresses - a critical aspect in metal-ceramic joints.

9:50 oral

Fractography as a tool to optimize joint design and the brazing process

Jakob J. Kuebler, Vinzenz Bissig, Gurdial Blugan, Jolanta Janczak-Rusch

Empa Materials Science and Technology (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland

Contact: jakob.kuebler@empa.ch

A number of factors such as the brazing filler composition, brazing process parameters, gap thickness, properties of the joining partners, joint geometry, etc., can influence brazed joint performance. The complex relationships between these parameters make joint optimization a difficult task. The effects of some of these factors can be predicted, others have to be determined after processing and joint characterization. The fractographic analysis of joints provide important information on the failure origin and hence about the “weakest link” in the joint. The comparison of the crack path with the simulated stress distribution helps to distinguish between the processing oriented and design based failures.

Ceramic-ceramic and ceramic-metal joints were brazed using different Cu- and Ag-based active brazing filler metals and characterized by four point bending tests. Si3N4/TiN was used as the ceramic partner and 14 NiCr 14 steel as the metal joining partner. AgCuInTi, AgInTi and CuSnTiZr brazing filler metals with differing amounts of the active element titanium varying between 0.6 and 10.2 wt.% and different working temperatures and ductilities. To minimize the residual stresses the AgCuInTi filler was modified with SiC particles, the brazing gap thickness was controlled in the range between 0.1 and 1 mm.

From each brazing batch the specimens with the highest and lowest values of bending strength were fractographically analyzed. Different joint failure behavior patterns were observed depending on the filler metal used. High residual stresses and weakening of the ceramic partner near the braze-ceramic interface were found to be the main causes for the failure of ceramic-metal joints brazed with CuSnTiZr. The stresses were reduced and the strength optimized by applying a Cu-

interlayer. In the joints with AgCuInTi the influence of residual stresses on the mechanical strength could also be observed. Under these conditions the joints were successfully optimized.

10:10 oral

Attachment of carbon nanotubes to AFM tips: Strength of carbonaceous and metal containing joints

Ivo Utke, Stefan Fahlbusch, Vinzenz Friedli, Johann Michler

EMPA Materials Research and Technology (EMPA), Feuerwerkerstr. 39, Thun 3602, Switzerland

Contact: ivo.utke@empa.ch

Submicron- and nano-scale joining becomes increasingly important for assembly of individual nanowires into devices or experimental setups. For example, carbon nanotubes mechanically attached onto standard silicon cantilevers serve for high-resolution high-aspect atomic force microscopy (AFM). The joining is accomplished inside a scanning electron microscope (SEM). By means of x-y-z positioning tables the carbon nanotube is brought into physical contact with the AFM cantilever. The contact area is now scanned with the focused electron beam which leads to either the formation of a carbonaceous deposit due to the hydrocarbon contamination backpressure inside the microscope chamber or to a metallic deposit if metal-organic carrier gases are introduced into the SEM chamber. The mechanical stability of these deposits for joining purposes can be studied in the same setup by in-situ observation and simultaneous monitoring of the force-distance curve of the such modified cantilever.

Parallel Session

Wednesday afternoon, 7 September, 14:00
Main Building, room 144

14:00 invited oral

Composites with tailorable isotropic near-zero thermal expansion

Heinrich Kern, Christian Georgi

Institute of Materials Technology, Technical University of Ilmenau, Gebäude "Werkstoffe 1", Ilmenau 98693, Germany

Contact: Kern@tu-ilmenau.de

The satisfactory operation of various high technology systems is limited by the thermal expansion of individual components. Such components must be made of materials with zero thermal expansion or thermal expansion characteristics that match the attached components for optimum performance. Moreover, a variety of performance demands are additionally being placed on these systems. Com-posite materials are ideally suited to combine features of different materials, thus forming a material with desired properties. For example, commercial glass-ceramic materials with zero thermal expansion characteristics are composites of a crystalline phase with negative thermal expansion (NTE) and a glass phase with positive thermal expansion. However, glass-ceramic materials show some disadvan-tages due to low strength, low young's modulus, their brittleness and low thermal conductivity. More-over, glass ceramics are in situ composites, i.e. their compositions are limited to certain glass-forming systems.

Research on NTE materials has broadened considerably since the 1990s and was mainly intended to facilitate the development of new high-performance composite materials with tuneable thermal proper-ties. As for most applications isotropic thermal expansion behaviour is preferred, the main goal has been to find cubic materials that would exhibit isotropic NTE. Zirconium tungstate ZrW2O8 exhibits a cubic framework structure and NTE of large magnitude over its entire stability range. These unique properties have made it the most promising candidate material for new composites with controllable thermal expansion.

This paper is intended to summarize the results of research on the design, preparation and properties of composites with tailorable thermal expansion using ZrW2O8 and related framework oxides. It intro-duces some of the issues surrounding these NTE materials and their application in composites.

14:30 oral

On the Relief of the Residual Stresses in Ceramic-Metal Joints by a Layered Joint Structure

Matteo Galli[1,2], John Botsis[2], Jolanta Janczak-Rusch[1]

1. *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland*
2. *Ecole Polytechnique Federale de Lausanne (EPFL), Lausanne 1015, Switzerland*

Contact: matteo.galli@epfl.ch

The potential of active brazing filler metal modification by ceramic particle addition, with the aim of to reduce the cooling induced stresses and enhance the strength of ceramic metal joints, is studied. The addition of ceramic particles allows tailoring the mechanical properties of the filler metal and improving its mechanical behaviour at higher temperature, however simple reinforcing of the interlayer has a detrimental effect at lower temperature because, due to the filler strengthening, residual stress relief via plasticity is limited. Thus a layered joint structure is proposed: reinforced and not reinforced layers of different thicknesses and compositions are combined in an optimal manner to minimize residual stress.

Reinforced active filler alloys are produced by adding SiC particles to Ag-Cu-In-Ti alloy Incusil-ABA up to 30% vol.

and used in combination with the pure filler alloy for joining Si3N4-TiN ceramics to 14NiCr14 steel. The joints produced are tested in 4-point bending. Bending strengths of joints with different layouts (thickness and composition of layers) are compared with experimental data obtained for the same material systems brazed with single unreinforced filler metals.

The evaluation of residual stresses and the optimization of the joint layout are carried out by FE modelling; temperature dependent mechanical properties are assumed for all the constituents, the elasto-plastic properties of the reinforced filler metals are obtained by homogenization.

Both experimental and numerical results indicate as an optimal solution a “sandwich” layout with a central reinforced layer between two unreinforced layers in contact with the base materials; experimental data show that the average bending strength of such joints is 20% higher than that of joints brazed with a single layer of unreinforced filler metal.

14:50 oral

Residual Stresses, Thermomechanical Behavior and Interfaces in the Weld Joint of Ni-based Superalloys

Oleg Barabash, Rozaliya I. Barabash, S. A. David, G E. Ice

Oak Ridge National Laboratory (ORNL), One bethel Valley Road, Oak Ridge 37932, United States

Contact: barabashom@ornl.gov

The quasi single crystalline structure formation in the Ni-based superalloy welds was characterized by means of X-ray polychromatic microdiffraction together with orientation imaging microscopy, scanning electron microscopy, and optical microscopy. Two Ni-based superalloys with different crystallographic orientations, Rene N5 and CMSX-4, were studied. Periodic dislocation structure is formed during continuous movement of the melt zone in a thin Ni-based superalloy sheet. We observed oscillations in the dislocation structure formed under such conditions at both macro and micro scales. Depending on the temperature, the formation of dislocations is accompanied by the partial or complete dissolution of □' particles in the matrix. The distribution of the dislocation density at the macroscale is due to a symmetric temperature gradient perpendicular to the direction of melt zone movement. Maximal dislocation density correlates with the interface between the heat affected and fusion zones. Within the same macro regions, oscillations of dislocation density due to grouping at the micro scale were also observed. The typical micro scale length of dislocation density oscillations is related to the dendrite size and the conditions of local melting and solidification. It is possible to decrease the probability of stray grain formation in the fusion zone and to improve the quality of the weld by applying a special thermal treatment to the base material.

Wednesday Poster Session

Wednesday afternoon, 7 September, 15:50

Symposium I

High Pressure School '6 : High Pressure Techology of Nanomaterials.

Welcome

Scope of the High Pressure School:

- Application of high pressure for consolidation and production of nano-crystalline materials
- Modelling the structure of nanomaterials and mechanisms of their deformation
- Nanocrystalisation
- Characterisation of nanomaterials

Special topics:

- Nanostructured metals obtained by heavy plastic deformation
- Sintering nanocrystaline ceramics and composites
- Synthesis of nanopowders

Special Session:

- Working out the roadmap for the technology of nanostructured metalls and alloys with perspective 2015

Organisers

- **Julia Ivanisenko**, Institute of Nanotechnology, Research Center Karlsruhe, Germany
- **Michael Zehetbauer**, High Performance Materials Group - Nanocrystalline Materials, Materials Physics Institute, University Center Boltzmanngasse,Vienna University, Austria
- **Nikolay Krasilnikov,** Institute of Physics of Advanced Materials, Ufa, Russia and Ulyanovsk State University, Ulyanovsk, Russia
- **Witold Lojkowski**, Institute of High Pressure Physics, Polish Academy of Sciences, Warsaw, Poland
- **Bernard Kear**, Department of Ceramic and Materials Engineering, Rutgers University, Piscataway
- **Krzysztof Kurzydlowski**, Faculty of Materials Science, Warsaw University of Technology, Warsaw, Poland
- **Paul F. McMillan**, Christopher Ingold Laboratories, Department of Chemistry, University College London, United Kingdom

Proceedings

Proceedings of this Symposium will be published in "Solid State Phenomena", Trans Tech Publications.

Acknowledgements

This symposium is supported by:

- International Scientific Network: "Nano and Micro Technology", acronym: NaMic, Polish Ministry of Science and Information Society Technologies
- Center of Excellence PRENABIO at the Institute of High Pressure Physics, European Comission Contract
- Nanoroad SME Project, European Comission Contract
- Faculty of Material Science and Engineering, Warsaw University of Technology

Programme

Monday, 5 September

Monday Poster Session
Monday afternoon, 5 September, 15:50

Wednesday, 7 September

Synthesis of nanostructured metals
Wednesday morning, 7 September, 9:00
Main Building, Small Hall (Mała aula)
Yulia Ivanisenko presides

9:00 invited oral

The New SPD Processing Routes to Fabricate Bulk Nanostructured Materials

Ruslan Z. Valiev

Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation

Contact: RZValiev@mail.rb.ru

Since the mid-1990's the fabrication of bulk nanostructured metals and alloys using severe plastic deformation (SPD) has been evolving as a rapidly advancing direction of modern materials science that is aimed at developing materials with new mechanical and functional properties for advanced applications. The principle of these developments is based on grain refinement down to the nanoscale level by various SPD techniques. However, within recent years SPD techniques have been applied for producing bulk nanomaterials using some other principles, namely, SPD-consolidation of powders, including nanostructured ones, as well as SPD-induced nanocrystallization of initially amorphous alloys. This paper is focused on investigations and development of these new SPD processing routes enabling fabrication of fully dense nano-

crystalline metals and alloys with a mean grain size of 20-30 nm and homogenous microstructures. We consider physical principles of these routes and present results on the microstructural characterization of several nanocrystalline materials produced as well as on studies of their unique properties.

9:30 invited oral

Deformation-induced nanocrystallization in Al-rich metallic glasses

Nancy Boucharat[1], Rainer Hebert[1], Harald Roesner[1], Ruslan Z. Valiev[2], Gerhard Wilde[1]

1. *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany* **2.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation*

Contact: nancy.boucharat@int.fzk.de

Al-rich metallic glasses have attracted extensive attention due to occurrence of a primary crystallization reaction that yields microstructures composed of nanocrystal dispersions at extremely high number densities (up to 10^{23} m^{-3}) within an amorphous matrix. This specific microstructure with the resulting solute redistribution results in a remarkably high tensile strength. While thermal treatment is often applied for examining crystallization reactions, nanocrystal formation has recently been observed in metallic glasses during different deformation processes. We report here structural analyses of $Al_{88}Y_7Fe_5$ glass that has been subjected to high-pressure torsion straining (HPT) at room temperature. HPT straining is used as consolidation process that allows producing bulk materials from amorphous ribbons obtained by rapid quenching. It is shown that an extremely high number density of small Al nanocrystals develops already at room temperature when the sample is subjected to a large shear strain. Moreover, nanocrystals appear to be distributed homogeneously throughout the sample without any evidence of strong coarsening. Although the exact mechanism of deformation-induced nanocrystallization generates still controversial discussions, these results demonstrate that applying very large shear strains to marginally glass-forming vitrification products is a promising method for synthesizing homogeneous nanostructures of adjustable grain size. To analyze the mechanisms of deformation-induced crystallization in metallic glasses in detail, plastic deformations at lower strain such as cold-rolling or in-situ straining in the TEM have been performed at room temperature. Based upon these results, likely mechanisms that account for the formation of homogeneous ultrafine nanostructures using severe plastic deformation are discussed.

10:00 invited oral

On the Mechanisms Governing the Special Properties of SPD Nanometals

Michael Zehetbauer, Erhard Schafler

University of Vienna, Institute of Materials Physics, Vienna, Austria

Contact: zehet@ap.univie.ac.at

Besides the advantage that they can be produced in bulk shape, SPD nanometals show a number of particular properties which have not been achieved so far by nanometals produced by other methods. In spite of the advanced strength and fatigue life time they show a considerable ductility and fracture toughness, an increased rate of hydrogen ad- and desorption, and changes in phase boundaries (e.g. increases in solubility of alloying atoms a.s.o.). In principle, all these effects can be related to the existence, interactions and rearrangements of deformation induced lattice defects. While the enhancements of ductility and fracture toughness are caused by distortion-minimizing rearrangements of dislocations, the acceleration in hydrogen diffusion, and especially the changed parameters for the existence of certain phases, seem to be consequences of the presence of deformation induced vacancies. According to recent experiments conducted in SPD nanometals those reach concentrations which are even higher than those known from conventional plastic deformation. These results clearly favour the importance of the extended hydrostatic pressure in SPD deformation, rather than ideas that strong strain gradients being typical of SPD may be responsible for the increased storage of lattice defects in SPD nanometals.

Synthesis of nanostructured metals

Wednesday afternoon, 7 September, 14:00
Main Building, Small Hall (Mała aula)
Michael Zehetbauer presides

14:00 invited oral

Features of Microstructural and Mechanical Behavior in ECA Pressed Aluminium Alloys under High Strain Rate Superplastic Flow

Mikhail M. Myshlyaev

Russian Academy of Sciences, Baikov Institute of Metallurgy and Material Science (IMET RAS), 49 Leninsky prospect, Moscow 119991, Russian Federation

Contact: myshlyae@issp.ac.ru

The structure of rods subjected to the equal-channel angular (ECA) pressing have been studied by X-ray diffraction analysis, transmission electron microscopy, scanning electron microscopy, including back electron scattering diffraction and orientation image microscopy, technique. A fine-grained

structure has been shown to form in the process of pressing, finer grains forming at lower pressing temperatures. A largest number of grains demonstrate the formation of a dislocation substructure involving subgrains. A most developed substructure forms under pressing at elevated temperature.

A mechanical behavior under tension has been studied for ECA pressed samples having different structural states. Temperature and strain rate conditions to attain ultimate strains to failure have been defined for samples of each structural state. It has been shown that samples with a developed substructure are subject to a superplastic (SP) straining. Contrary to the expectations the ductility of finest-grained samples turned out low.

Mechanical behaviour of the alloys under constant tensile elongation rate and constant stress tensile creep has been studied in SP conditions. Multistage of SP flow has been shown. Dependencies of the true deformation rate on temperature, the true stress and true deformation for the hardening stage and the softening stage have been established. The activation energies and the coefficients of deformation rate sensitivity of stress, which characterize these stages, have been determined. Structural behavior has been studied.

It has been established that dynamical recrystallization on sub-grain level corresponds to hardening stage and dynamic recrystallization with participation of grain boundary sliding and migration corresponds to softening stage.

14:30 oral

The Effect of Dispersoids and Processing Route on Grain Refinement During Deformation of Aluminium Alloys deformed by ECAP

Marco Berta, Philip B. Prangnell

University of Manchester - School of Materials Science - Materials Science Centre, grosvenor street, Manchester M1 7HS, United Kingdom

Contact: m.berta@postgrad.umist.ac.uk

The effect of fine second-phase non-shearable dispersoids on the grain refinement of aluminium severely deformed by ECAE through routes A and Bc (+90degrees rotation) has been studied using a model Al-0.2%Sc alloy. The microstructure development with strain in the dispersoid containing alloy was compared to a single phase alloy, using high resolution EBSD analysis. The effect of the processing route was investigated for both materials using ECAE die angles of 90 degrees and 120 degrees.

The presence of fine dispersoids was found to inhibit the development of new high-angle grain boundaries and the formation of a fine grain structure with route A. This effect was enhanced by rotation of the sample using route Bc. The mechanisms of responsible for the different rates of grain refinement and their interaction with the processing variables are discussed.

14:45 invited oral

High Pressure Torsion of dual phase materials

Reinhard Pippan, Florian Wetscher, Andreas Vorhauer, Ilshat Sabirov

Erich Schmid Institute of Materials Science, OEAW (ESI), Jahnstrasse 12, Leoben A-8700, Austria

Contact: pippan@unileoben.ac.at

High Pressure Torsion (HPT) is a well known procedure to obtain nano- and sub-micrometer grained microstructures in bulk materials. The paper is separated into two parts.

The first one is devoted to the HPT technique itself. A careful analysis of the homogeneity of the deformation during HPT is performed in order to understand the peculiar behavior described in the literature. Even at large numbers of revolutions a nearly idealized torsion deformation is observed. Reasons for the reported deviation between observations and ideal torsion deformation are discussed.

The second part is devoted to HPT-deformation of dual phase materials. The behavior in this type of materials is more complex than in single phase materials. It varies from simple homogenization, fragmentation of one phase, to disintegration and supersaturation of the phases. Examples for the different mechanisms are presented.

15:15 oral

High strength and ductility of nanostructured Al-based alloy, subjected by high pressure technique

Dmitry A. Vorona[1], A Sharafutdinov[2], Nikolay A. Krasilnikov[1]

1. *Ulyanovsk State University (UlGU), L.Tolstoy, 42, Ulyanovsk 432700, Russian Federation* **2.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation*

Contact: davorona@mail.ru

The structure and mechanical properties of Al-based alloy 2024 after high-pressure torsion (HPT) was investigated. Alloy 2024 with homogeneous structure and grain size about 70 nm was obtained using HPT at 6 GPa pressure and 5 turns of anvils at room temperature. The nanostructured alloy at room temperature demonstrated very high UTS above 1100 MPa, and superplastic behavior at temperature higher then 300C. The microhardness of nanostructured alloy after superplastic deformation (1.5 GPa) was more than after standard treatment of coarse-grained alloy (1.2 GPa). The influence parameters of HPT and heat treatment on structure and deformation behavior of alloy were studied.

Opportunity of achievement in metals and alloys of combination high strength and good ductility opens perspectives of its

application in industry, particularly, for mirco-systems and for high-strength details with complex geometry obtained due to superplastic forming.

Wednesday Poster Session

Wednesday afternoon, 7 September, 15:50

15:50 poster I-1

The influence of hydrostatic extrusion on mechanical properties and tribological characteristics of an austenitic stainless steel

Julia A. Budniak[1], Malgorzata Lewandowska[1], Waclaw Pachla[2], Mariusz Kulczyk[2], Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: jbudniak@materials.pl

Austenitic stainless steels are widely used in various fields of engineering and biomedical applications. From this point of view, the improvement of their mechanical properties is of great importance. This can be achieved by grain refinement using one of the severe plastic deformation (SPD) methods. It has been recently shown that hydrostatic extrusion (HE) is one of efficient SPD methods. The aim of this work was to study mechanical properties and tribological characteristics of the 316 LVM austenitic stainless steel subjected to HE process.

The mechanical properties were characterized by microhardness measurements and compression tests. Wear properties were investigated using pin-on-disc tribometer under dry and lubricated conditions. The relation between friction coefficient and time was determined.

The results indicate that hydrostatic extrusion process significantly improves mechanical properties as well as wear resistant of the 316 LVM stainless steel. The results were discussed in terms of microstructural evolutions occurring during processing by hydrostatic extrusion.

15:50 poster I-2

Hydrostatic extrusion of intermetallic Ti-Al layers

Halina Garbacz[1], Waclaw Pachla[2], Tadeusz Wierzchoń, Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: haga@inmat.pw.edu.pl

Hydrostatic Extrusion (HE) as one of the methods of grain refinement of metallic materials down to the nanometric scale [1-4]. The main features of this method are large strain and very high strain rates (exceeding than 104 s-1). HE also offers the opportunity to obtain bulk materials in a variety of forms (rods and wires of complex cross-sections, small tubes). HE processing proved to be an efficient way to reduce the grain size in Ti. This grain refinement resulted in significantly improved mechanical properties.

The material examined in the study was commercially pure titanium with intermetallic Ti-Al layers. This material was subjected to HE at room temperature. The intermetallic layers were produced by magnetron sputtering followed by glow discharge assisted treatment. The HE of coated specimens resulted in substantial grain size refinement in titanium. As a consequence of such microstructure evolution the mechanical properties of this material are significantly improved. The intermetallic Ti-Al layers effected reduction of the pressure during extrusion of pure titanium and additionally increased microhardness and frictional wear of the material.

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4. M.Kulczyk, W.Pachla, H.Garbacz, M.Lewandowska, A.Mazur, K.J.Kurzydłowski: Material Science-Poland (2005) accepted

15:50 poster I-3

Microstructure refinement under high plastic strain rates in hydrostatic extrusion process

Krzysztof J. Kurzydlowski[1], Maria Richert[2], Beata Leszczyńska[2], Halina Garbacz[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: bleszcz@agh.edu.pl

Recent publications show that high strain rates influenced on the microstructure refinement similarly to large deformations. The strong tendency to microbands formation is observed in both cases. It was found that the width of microbands is very sensitive to the changes of deformation parameters. Especially, it has been observed that in the severely deformed materials the width of microbands is reduced to nanometric dimensions leading to the formation of nanometric microstructures.

In the process of hydrostatic extrusions, which has been used

in this work for deformation of cooper, large plastic strain is substituted by high strain rates exceeding 3.84*102. In all investigated samples the numerous microbands were found in the microstructure. The intensive intersections of microbands were observed. The width of microbands was noticed from 20 nm to about 400 nm. It means that locally, in the areas of microbands, the width of microbands achieved dimensions typical of the nanometric materials. A special feature was also the appearance of large areas of subgrains with the average dimension of about 200 nm. These areas were identified as the dynamically or post- dynamically recrystalized.

The large misorientations were found between the microbands and the surrounding materials. The appearance of large misorientations between the microbands and the surrounding material results in formation of high misorientation boundaries, which contribute to the microstructural and mechanical changes. The mechanism of creation of high misorientation in the microbands areas is probably different to the one operating during the process of dynamic recrystalization.

The results indicate the possibility of obtaining nanometric structure at lower deformations but higher strain rates.

15:50 poster I-4

The Influence of hydrostatic extrusion on microstructure of 6082 aluminum alloy

Pawel Widlicki[1], Halina Garbacz[1], Malgorzata Lewandowska[1], Waclaw Pachla[2], Mariusz Kulczyk[2], Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: pwidlicki@inmat.pw.edu.pl

Hydrostatic extrusion is one of the SPD methods for fabrication of ultra-fine grained alloys showing a significant increase in mechanical properties such as tensile strength and hardness.

Microstructure and properties of aluminum 6082 alloy after hydrostatic extrusion were investigated. Hydroextrusion was performed in three steps with cumulated true strains 1.34, 2.73 and 3.74 respectively. Microstructure observations were carried out using electron (SEM, TEM) and light microscopy. Within the framework of experiment grain and inclusions size, shape and distribution were investigated. The influence of cumulated true strain value on the microstructure homogeneity was also analyzed. The Hall-Petch relationship was determined for tensile strength and microhardness.

15:50 poster I-5

Influence of severe plastic deformation on PLC effect in Al 5XXX alloy

Joanna Zdunek, Pawel Widlicki, Halina Garbacz, Jarosław Mizera, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: jzdunek@inmat.pw.edu.pl

In the present work, Al-Mg-Mn-Si alloy (5483) in as received state and after severe plastic deformation process was used. The plastic deformation was obtained by the hydroextrusion, so three different true strain values had been received correspondingly 1.39, 2.77 and 3.79. All specimens were subjected to the tensile tests with strain ratio 8,3*10-4 s-1. The investigated material revealed an instability of plastic deformation in the form of serration on stress-strain curves (so called Portevin- Le Chatelier effect). It is noticeable that the grain size reduction effects on the character of the instability.

15:50 poster I-6

Combination of ECAP and Hydrostatic Extrusion for ultra-fine grain (UFG) microstructure generation in 99.98% nickel.

Mariusz Kulczyk[1,2], Waclaw Pachla[1], Anna Swiderska - Sroda[1], Nikolay Krasilnikov[3,4], Andrzej Mazur[1], Witold Lojkowski[1], Krzysztof J. Kurzydlowski[2]

1. *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **3.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation* **4.** *Ulyanovsk State University (UlGU), L.Tolstoy, 42, Ulyanovsk 432700, Russian Federation*

Contact: mariusz@unipress.waw.pl

Ultra-fine grained materials attract considerable attention, due to their high strength. They are usually obtained by severe plastic deformation processes. Extensive research is done to produce bulk nanostructured metals. The most effective method from the point of grain refinement is high pressure torsion, however it involves very small material volume. The goal of the present work is to demonstrate, that bulk, ultra-fine grained microstructure can be obtained in high purity nickel by combination of ECAP and hydrostatic extrusion (HE) processes. Deformation has involved eight times ECAP followed by eight times HE to 3mm wire with total true strain of ~12.5. The resulting microstructure is characterized by light and

TEM microscopy and mechanical properties. Addition of HE to ECAP-ed sample has led to significant grain size refinement, from 330nm to 160nm, and to apparent increase in the microstructure homogeneity. Microstructure transformation has led to appropriate improvement in strength properties, increase of yield stress by 78% in comparison to pure ECAP. Combination of ECAP and HE has enabled to achieve much better properties than application of each individual one and show to be promising in manufacturing of bulk UFG nickel.

15:50 poster I-7

STRENGTH OF COMMERCIAL ALUMINUM ALLOYS AFTER EQUAL CHANNEL ANGULAR PRESSING (ECAP) AND POST-ECAP PROCESSING

Maxim Y. Murashkin[1], Mikhail V. Markushev[3], Yulia Ivanisenko[2], Ruslan Z. Valiev[4]

1. *Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation* **2.** *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany* **3.** *Institute for Metals Superplasticity Problems, Russian Academy of Science, Ufa, Russian Federation* **4.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation*

Contact: maxmur@mail.rb.ru

Effect of thermomechanical treatment (TMT) that includes equal channel angular pressing (ECAP), further annealing, aging and rolling on ambient temperature tensile strength of heat hardenable and non-heat hardenable 6061 (Al0.9Mg0.7Si) and 1560 (Al6.5Mg0.6Mn) alloys has been investigated.

It has been stated that even after ECAP the alloys strength exceeds values observed after conventional strengthening processing.

Post-ECAP annealing of the 1560 alloy at temperatures above 2000C led to strength decrease and ductility enhancement. The strength of 6061 alloy can be additionally increased by TMT involving solution treatment before ECAP and post-deformation aging. Basing on the data of differential scanning calorimetry and dependences of hardness on temperature and time of annealing, optimal regime of the alloy post-ECAP aging has been established. As a result an increase of yield and ultimate tensile strength (YS and UTS) from 386 and 434 MPa to 434 and 470 MPa was obtained, respectively. Despite the increased strength the alloy elongation to failure remains high enough (~9%).

A possibility of the alloys additional processing by rolling has been investigated. After such a complex TMT it has been found out that the 1560 alloy strength in 2 mm processed sheets reached a record level (YS= 540 MPa and UTS=635 MPa) that was not observed in non-heat hardenable alloys earlier. For the 6061 alloy it is demonstrated that alloy YS and UTS make up to 475 and 500 MPa, whereas the elongation amounts to 7%.

Peculiarities of grain structure and texture evolutions as phase transformations due to TMT routes have been considered. The nature of the effects of ECAP and post-ECAP processing on the alloys mechanical behavior is discussed.

15:50 poster I-8

Mechanical properties of aluminium processed by equal channel angular pressing

Rafal Molak, Zbigniew Pakiela, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: rmolak@inmat.pw.edu.pl

The aim of this study was to investigate influence of large plastic strain on microstructure and mechanical properties of aluminium processed by severe plastic deformation (SPD) via equal channel angular pressing (ECAP) method. Polycrystalline high purity aluminium (99,99%) has been deformed at the room temperature by the ECAP in the range 1-12. Pressing velocity was 5 [mm/min]. The microstructure of aluminium was examined using light polarized microscope. To characterize mechanical properties we performed microhardness measurement and tensile tests. Obtained results showed strong interrelation between microstructure and mechanical properties.

15:50 poster I-9

Influence of the history of materials from the 2xxx series of aluminium alloys on structure and mechanical properties after deformation by ECAE method and annealing

Marzena Lech-Grega, Juliusz Senderski, Sonia Boczkal, Bartłomiej Płonka

The Institute of Non-Ferrous Metals Light Metals Division in Skawina (OML), Piłsudskiego 19, Skawina 32-050, Poland

Contact: zwlech@cyf-kr.edu.pl

Equal channel angular extrusion method has been used to deform AlCu(Si,Mg) alloys in the range of true strain $\varepsilon = 1.15 – 2.3$. The starting material for the investigation were samples obtained by extrusion from ingots and powder consolidation. The influence of the true strain on structure and properties was studied by optical and transmission electron microscopes and hardness measurement. Increasing of the strains led to refinement of structure and raised the properties. In structure,

the mutually crossing microbands were observed against the background with high density of dislocations. After additional annealing, the microstructure was composed of almost equiaxed grains. The hardness measurements show higher properties in material prepared by powder consolidation. The obtained results prove that there are differences in structure and properties of the same alloy processed by ECAE deformation method two different initial materials.

15:50 poster I-10

Equal Channel Angular Pressing (ECAP), its Effect on Structure and Properties of the Constructional Steel St3.

Maria Z. Borisova, Sofia P. Yakovleva, Afanasiy M. Ivanov

Russian Academy of Sciences, Institute of Physical-Technical Problems of the North, Yakut Branch, Siberian Department (IPTPN), Oktiabrskaya Str., 1, Yakutsk 677980, Russian Federation

Contact: bormaria@yandex.ru

The objective of this study was to describe two different ways for performing equal channel angular pressing of the constructional steel St3 and the relation between these two ways and steel's structure and properties. Recent research has focused on the importance of the investigation of how the ECA pressing influences the properties of the plastic metals, such as copper, aluminum and their alloys. However, further investigation of the effect of ECAP on steels is still needed. The investigation was performed with the constructional steel St3 which is widely used under northern conditions, being also the most available material. The billets were pressed by two different ways: Bc (after each pass a billet rotated 90 degrees around its longitudinal axis) and C (after each pass a billet rotated 180 degrees around its longitudinal axis). After two ECAP passes it was found out that grain size decreases from 16-18 μm to 4-6 μm. Difference in pearlite microstructure is displayed after Bc and C paths. At Bc path elongated pearlite's inclusions were obtained, while at C path involute pearlite's inclusions were observed. This difference is caused by different shear directions during second ECAP pass. At -40˚C, imitating the northern conditions, results for paths Bc and C were: σ_T=695 MPa, σ_B=705 MPa and σ_T=695 MPa, σ_B=720 MPa, respectively. No significant differences were observed between properties received by paths Bc and C. Therefore, both ways are suitable for practical use. ECAP is a good method of steel processing for it's use under northern conditions.

15:50 poster I-11

Combined SPD techniques for fabrication of nanostructured Ti rods for medical applications

Gulnaz H. Salimgareeva[1], Vladimir V. Latysh[2], Irina S. Semenova[2], Ruslan Z. Valiev[2]

1. *Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation* **2.** *Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation*

Contact: Sadikova_gh@list.ru

In this paper we present the results of investigation aimed at the development of technological processing methods of nanostructured materials for structural application. It is suggested to use a few types of deformation, including severe plastic deformation (SPD) and thermomechanical treatment, to process high physical mechanical properties formation in bulk billets.

The paper studies the effect of combined treatment regime on the microstructure and mechanical properties of semiproducts out of CP Ti. The preliminary deformation of CP Ti Grade 2 resulted in alpha-phase grain decrease from 50 mkm to 1 mkm, which contributed to the alloy deformability enhancement during ECAP. At the same time, the possibility appeared to decrease the number of ECAP passes leading to processing of homogeneous ultrafine-grained (UFG) state and, finally, to decreasing of structure anisotropy and the alloy properties enhancement.

The investigation considered the features of UFG structure formation in billets, evolution of mechanical properties at various stages of technological process. It was established that the formation of homogeneous UFG structure in semiproduct out of CP Ti enabled to enhance strength properties by 2-2.5 times and preserve sufficient ductility values.

15:50 poster I-12

Mechanical properties and deformation behaviour of ultra-fine grained nickel.

Nikolay A. Krasilnikov[2], Witold Lojkowski[1], Zbigniew Pakiela[3], Krzysztof J. Kurzydlowski[3]

1. *Polish Academy of Sciences, High Pressure Research Center (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Ulyanovsk State University (UlGU), L.Tolstoy, 42, Ulyanovsk 432700, Russian Federation* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: nikras@mail.rb.ru

Mechanical properties and deformation behaviour of ultra-

fine grained (UFG) Ni subjected to severe plastic deformation (SPD) are investigated. UFG Ni was characterized the homogeneous structure with the grain size 120 nm and high angle nonequilibrium grain boundaries. This state of nickel possesses significant ultimate tensile stress of 1270 MPa.

Investigation by HRSEM of deformation relief on the polished surface of the UFG Ni samples reveals the grain boundaries (GBs) steps already at small degrees of tension, that testifies to engaging of grain boundaries to deformation process at room temperature. After large degrees of the tensile tests the deformation relief revealed a network of crossed shear bands oriented at an angle in the range 35- 45 to the axis of tensile deformation. Shear bands propagated along GBs that are parallel to the plane of maximal shear stress. Formation of shear bands occurs due to strong shift and rotation of grain groups. This leads to a deformation mechanism involving collective relative displacement of grain groups, with extensive grain boundary sliding at room temperature. On the length scale of a few micrometers the material can be regarded as uniform and therefore the local strain distribution becomes more uniform than in coarse-grained materials. It is plausible that this mode of deformation may contribute to the enhanced ductility.

The features of deformation behaviour of Ni with the different grain size and various states of grain boundaries are also considered. Opportunity of achievement in metals and alloys of combination high strength and good ductility due to control of microstructure of materials opens perspectives of its application in industry, particularly, for mirco-systems and for details with complex geometry, obtained due to superplastic forming.

15:50 poster I-13

Microstructures and mechanical property of Ni processed by high-pressure torsion and their evolution upon annealing

Zhiqing Yang[1], Udo Welzel[2]

1. *Department of Physics, University of Antwerp, Antwerp, Belgium* **2.** *Max-Planck-Istitute for Metals Research (MPI), Stuttgart 70569, Germany*

Contact: zhiqing.yang@ua.ac.be

XRD, TEM, microhardness and thermal analysis were carried out on a series of Ni samples produced by high-pressure torsion (HPT) technique under a pressure of 7 GPa with rotation numbers of 0.5, 1, 3, 5, and 7. Evolution of microstructures and their inhomogeneity were investigated in detail. The local microstrain level showed dynamical oscillations as a function of the numbers of rotation of HPT, demonstrating dynamical evolution of lattice defects during the HPT procedure. Both XRD and TEM showed that there is sitll small diference in grain sizes even after 5 rotations of HPT, showing smaller grain sizes at the periphery region. The higher microhardness at the periphery region is the result of smaller grain sizes and higher density of lattice defects, comparing with the central region. Thermal treatment at a heating rate of 20K/min from room temperature to 473K did not result in decrease in microhardness, but increase by about 10 per cent for samples treated not more than 3 rotations of HPT. The increase in microhardness was contributed to further refinement of grain sizes, formation of more high-angle grain boundaries and more equilibrium state of grain boundaries during recovery. Recrystallization occurred by heating to higher temperatures, which resulted in decrease of microhardness. The nonequilibrium and high-energy grain boundaries were the main source of the stored deformation energy.

15:50 poster I-14

FEATURES OF BCC-METALS (V,Nb,V-Zr-C) STRUCTURE FORMATION AT COLD AND LOW TEMPERATURE DEFORMATIONS UNDER PRESSURE

Tatiana M. Teterina, V. P. Pilyugin

Russian Academy of Sciences, Ural Division, Institute of Metal Physics, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation

Contact: TatianaT@imp.uran.ru

The submicro- and nanocrystal condition of metals causes a great interest because of its special physico-mechanical properties. We investigated microstructure of deformed samples BCC of metals and alloys (monocrystalline Nb - 99.98 per cent, V - 99.92 per cent, macrocrystalline alloy V-Zr-C). Influence of deformation on hardening of V at 80K and 300K and Nb at 300K was studied.

Samples were deformed shear under pressure upon Bridgman anvils. Pressure at deformation was changed from 6 up to 12 GPa. Working temperatures were 300K and 80K. Structures of the deformed samples were studied with the help of the electronic microscopy method.

The sequence of structure changes from initial through increase of dislocation density to cellular-dislocation structure and transformations of last to nanocrystalline grain (NC) structure was revealed. NC structure of V and Nb is formed at achievement of true deformation e=7 and more with the most probable nanocrystalline grain of 20 nm sizes. Deformation up to e=10 and more does not cause changes of NC structure.

Deformation of metals at 80K also results in formation of NC structure, but with the smaller grain sizes and higher level of hardening. The maximum level of vanadium hardness after cold deformation (300K) makes 2.7 GPa from initial condition - 0.75 GPa, the hardness grows by 4.5 times after low temperature deformation (80K). The general level of niobium hardness grows by 1.7 times from initial after cold deformation. Measurements of niobium hardness have shown deform-

ation leads to steady growth of hardness up to e=9 at room temperature and the saturation stage of Nb hardness is not achieved unlike V in the same interval of deformations.

Structural changes of the V-Zr-C-alloy, as a whole, are similar to the changes, occurring in the V and Nb. However specific feature of the alloy is the behavior of carbides at deformation, so there are carbides broken on fragments and unchanged carbides after deformation.

15:50 poster I-15

Development of SPD technology for producing nanostructural materials

Mykola M. Bilousov

National Academy of Sciences of Ukraine, Donetsk Technical-Physical Institute, R. Luxemburg 72, Donetsk 83114, Ukraine

Contact: bil@hpress.dipt.donetsk.ua

The production of nanostructural materials is an important part of modern nanotechnology. The application of SPD technology is a necessary condition for the production of nanomaterials with specific properties. The idea of in-situ SPD technology consists in the continuous registration of external deformation parameters and the establishment of its relation with the internal nanostructural parameters.

The in-situ SPD technology of producing nanostructured materials is realized by plastic torsional deformation and pure shear deformation under high pressure. This technology provides in-situ method of deformation and formation nanocrystalline structures. Simultaneously, structure - sensitive parameters (such as, electricalresistance, acoustic emission) were investigated during the severe plastic deformation. In addition the direct X-ray (XRD and SAXS methods) study of the structural evolution was performed continuously during both compressive and shear deformation.

It is shown, that the plastic deformation of austenite of high-nitrogen steels (with CN=0.06ë0.57%N) in the fragmentation stage is accompanied by structural changes. This promotes the formation a nanocrystalline structure.

The in-situ SPD technology of producing the materials with submicro- and nanocrystalline structure by shear under high pressure is developed. The fragmentation and phase (g«a) transformation under high pressure and shear deformation is accompanied by structural modifications in the austenite of high-nitrogen steels. This promotes the shaping of complex parts with a nanocrystalline structure.

The application of in-situ SPD technology has made it possible to identify three stages of formation of NC structures by finding functional correlation between the critical shear stress and the start of plastic flow.

The interrelation was established between the external parameters (critical shear stress; stress at the initiation of plastic flow) and microstructural parameters (grain size, q

[ABSTRACT TRUNCATED TO 2000 LETTERS]

15:50 poster I-16

Formation of ultrafine gradient structure in hard magnetic alloy Fe-30%Cr-8%Co

Alexander Korznikov[1], Zbigniew Pakiela[2], Galia Korznikova, Krzysztof J. Kurzydlowski[2]

1. *Institute for Metals Superplsticity Problems, Russian Academy of Sciences, Ufa, Russian Federation* **2.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: korznikova@anrb.ru

The alloys of Fe-Cr-Co system belong to the group of hard magnetic materials of dispersion-hardening class. Low cobalt alloys alongside with economy of expensive cobalt offer a number of advantages. The most important of those is an open single α phase area. This feature allows the method of strain ageing to be applied for obtaining high coercivity state. It permits anisotropic permanent magnets to be produced by means of conventional metallurgical techniques including: quenching for fixing metastable α solid solution, preliminary ageing for decomposition of the solid solution into spherical α1 precipitations and α2 matrix, intermediate uniaxial deformation for elongation and orientation of α1 precipitations, giving rise to magnetic anisotropy, final thermal treatment.

The need for uniaxial cold deformation by 60-70% on the intermediate stage of formation of high coercivity state imposes serious limitations on this method due to the low deformation ability of the alloy in the preliminarily aged state. However it is known that grain size refinement enhances plasticity of brittle alloys. One way to refine the structure in bulk cylindrical billets without change in shape is hot torsion. Deformation by hot torsion allows the attainment of large plastic straining without failure. Due to inhomogeneity of deformation distribution along the radius, more refined structure is formed in the periphery parts of the billet causing plasticization of outer layers.

Using Fe-30%Cr-8%Co alloy it is shown that in a preliminarily aged state the room temperature plasticity of the material enhances due to the formation of gradient submicrocrystalline structure in a cylindrical billet by means of rotation in the temperature interval corresponding to dynamic recrystallisation. Owing to gradient structure the uniaxial deformation at room temperature was carried out without failure of the billet by means of grooved rolling to achieve magnetic anisotropy.

15:50 poster I-17

Influence of high presure hot compaction on microstructure of Al-Si-Ni-Mm alloys

Grzegorz Cieślak, Jerzy Latuch, Tadeusz Kulik

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: gcieslak@inmat.pw.edu.pl

The nanocrystalline Al-Si-Ni-Mm alloys have been intensively investigated in the recent years. Al – based alloys containing silicon (Si), rare earth metal (RE) and late transition metal (Ni), have high tensile strength and good wear resistance. Bulk nanostructured alloys were produced by ball milling of nanocrystalline ribbons followed by high pressure hot compaction.

Rapidly solidified ribbons were composed of a-Al crystals embeded in amorphous matrix.

The ribbons were produced by melt-spinning method in argon atmosphere.

Different temperatures in the range from room temperature to 350ºC and pressures of compaction up to 7.7 GPa were applied to improve the quality of compaction investigated alloys. X-ray diffraction, differential scanning calorimetry, transmission and scanning electron microscopy were used for characterisation of microstructure Al-Si-Ni-Mm alloys. The Vickers hardness was measured using a load of 1 kG.

15:50 poster I-18

Analysis of Deformation Behavior of Cu Processed by High Pressure Torsion

Vil D. Sitdikov, Rosa G. Chembarisova, Igor V. Alexandrov

Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation

Contact: svil@mail.rb.ru

Mechanisms of severe plastic deformation (SPD), realized with the help of high pressure torsion (HPT), have still remained a point for discussion. In the current investigation the 3D version of Estrin-Tóth dislocation model [1] has been applied for analysis of deformation behavior of HPT Cu. Typical experimental curves for modeling have been taken from [2].

It has been shown that the growth of the source activity and dislocation sinks in grain-cell walls is a possible explanation of peculiarities of Cu deformation behavior during SPD with the accumulated resolved shear strain up to 14. This growth leads to an increase in dislocation density and to a possibility of annihilation processes as well as change of the interaction character between dislocations. In particular, a fraction of active dislocation sources is increases by 4.3 times when increasing the imposed pressure from 0.8 GPa to 8 GPa. This is explained by constrains applied to work of dislocation Frank-Reed sources by a low cell size. The fraction of dislocations, which has left the cell body, increases by 2.3 times. This is connected with the limited length of free dislocation path. A growth of probability of annihilation processes by 1.6 times testifies to extremely non-equilibrium state of the material as a result of SPD. At the same time, the coefficient, which characterizes the interaction of dislocations alongside with growing of the imposed pressure, is increased by 2 times.

1. L.S. Tóth, A. Molinari, Estrin Y., J. Eng. Mater. Technol. 2002, 124:71.

2. M. Zehetbauer, H.P. Stüwe, A. Vorhauer, E. Schafler and J. Kohout. Advanced Engeneering Materials, 2003, 5:5.

15:50 poster I-19

A change in dislocation density during equal-channel angular pressing of Cu: experiment and simulation.

Nariman A. Enikeev, Igor V. Alexandrov

Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation

Contact: carabus@mail.rb.ru

Samples of Cu processed by equal-channel angular pressing (ECAP) up to 12 passes were investigated by x-ray analysis. Dependencies of the x-ray dislocation density on the imposed strain were obtained. The dependencies were analysed in terms of dislocation model of deformation behaviour of f.c.c. metals at large strains. This approach was developed on the base of Estrin-Toth model [1], into which certain modification were introduced. The results of calculations are in reasonable agreement with experimental data. The suggested approach may assist in prediction of microstructure parameters evolution during severe plastic deformation.

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15:50 poster I-20

MD simulation of atomic structure of nanocrystals

Marcin Wojdyr, Bogdan F. Palosz

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: wojdyr@if.pw.edu.pl

Molecular dynamics method with empirical interatomic potentials is used to simulate the structure of ZnS nanoparticles.

The difference in crystal lattice structure, strains and internal pressure in the grain interior and in the surface shell is analyzed as a function of the grain size up to 2r=5 nm. MD predictions of x-ray diffraction are compared with experimental results.

15:50 poster I-21

Analysis of homogeneity of texture formation processes under high pressure torsion

Marina V. Zhilina[1], Igor V. Alexandrov[1], Jan T. Bonarski[2], Andrey V. Scherbakov[1]

1. *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation* **2.** *Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland*

Contact: zh_mv@mail.ru

As it is known, high pressure torsion (HPT) leads to formation of ultrafine-grained states with a unique complex of physical and mechanical properties [1]. However, the problem of homogeneity of structure and texture, being formed at different distances from the torsion axes and corresponding to the areas with a different accumulated strain values, have still remained unsolved.

The current work presents the results of the conducted experimental X-ray structural studies of the preferred orientations homogeneity in copper billets, subjected to HPT. HPT was realized by deforming of disk-shaped billets 10 mm in diameter and 0.7 mm thick under the imposed pressure of 5 GPa with a number of rotations, equal to 1, 5 and 10, at room temperature. Four selected areas, located at equal distances from each other in one of the billet's radii, were investigated. The size of areas consisted 0.6 mm in diameter. The results are presented in a form of pole figures (PF) (111), (110) as well as volume fractions of the main texture components LaboTex texture software was used for calculations and graphic presentation of the obtained results [2].

It was established that the crystallographic texture in the initial state is rather chaotic, but it is homogeneous along the billet's radius. The preferred orientation, which can be described as a pure shear texture is formed during the process of HPT after a different number of rotations. At the same time, one may observe increasing the texture intensity of the main texture maxima with raising the number of rotations and smearing the texture alongside with retiring from the billet's center.

1. Valiev R.Z., Islamgaliev R.K., Alexandrov I.V., Bulk nanostructured materials from severe plastic deformation, Progr. Mater. Sci. (2000) 45, pp. 103-189.

2. Pawlik K., Ozga P.: LaboTex: The Texture Analysis Software, 'Göttinger Arbeiten zur Geologie und Paläontologie', SB4, 1999.

15:50 poster I-22

Texture Development in Model Al-Li Alloy Subjected to Severe Plastic Deformation

Bogusława Adamczyk-Cieślak, Jarosław Mizera, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: badamczyk@inmat.pw.edu.pl

The texture of Al – 0.7 wt. % Li alloy processed by two different methods of severe plastic deformation (SPD) has been experimentally investigated by X-ray diffraction technique, and analyzed in terms of the orientation distribution function (ODF). It was found that severe plastic deformation through both equal channel angular extrusion (ECAE) and hydrostatic extrusion (HE) results in ultrafine grained structure in Al – 0.7 wt. % Li alloy. Microstructure (grain shape and size) of the materials produced via SPD methods strongly depends on the applied technological parameters and method. The texture of investigated alloy was different in both cases due to change of deformation mode.

15:50 poster I-23

X-RAY ANALYSIS OF HIGH PRESSURE TORSION INDUCED NANOSTRUCTURES IN TI AND NI

Ascar R. Kilmametov[2], Ruslan Z. Valiev[1,3], Igor V. Alexandrov[1]

1. *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation* **2.** *Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation* **3.** *Institute of Physics of Advaced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation*

Contact: ascar@mail.rb.ru

High pressure torsion (HPT) is a widely used severe plastic deformation technique to form ultrafine-grain (nanostructure) states in metals and alloys. The most promising physical and mechanical properties one can expect performing new metastable nanostructures by HPT as well as combining HPT with other methods to process nanocrystalline materials. In the present work X-ray investigations including texture analysis of commercially pure Ti and Ni subjected to HPT have been presented.

It was revealed that the applied pressure growth during severe plastic deformation of commercially pure Ti leads not only to substructure refinement with increase of dislocation density

and microstrain level but also to phase alpha-omega transition at room temperature. The coexistence both of alpha and omega phases (the latter known as high pressure one) in the ratio approximately of 1:3 has been obtained after high pressure removing.

Two different initial states of Ni, namely, coarse-grained and electrodeposited nanocrystalline were used for comparative studies of microstructure characteristics after HPT. Texture analysis discovered a new type of crystallite orientation distribution in nanocrystalline state. Unlike to "traditional" case of sharp axial texture forming in cubic symmetry metals prevailed pole density close to a unit has been observed. Received crystallographic texture data has been concluded to be experimental evidence of the changed plastic deformation mechanisms in nanocrystalline Ni subjected to HPT.

15:50 poster I-24

Application of diamond anvil cell techniques for studying of plastic deformations in Ti - alloys at pressures up 50 GPa

Olga V. Savina, Alexey N. Babushkin, Artemiy A. Popov, Ivan V. Sukhanov

Ural State University, Department of Physics, Lenin Av., 51, Ekaterinburg 620083, Russian Federation

Contact: Savina_olga@rambler.ru

Progress in understanding of properties of materials at high pressure in many respects is connected to diamond anvils technique. We developed and investigated a technique of megabar range pressure valuation, applicable in the opaque high pressure diamond anvil cell (DAC) with anvils of the rounded cone-plane type made of synthetic carbonado-type diamond, consisting of dielectric grains of synthetic diamonds in layers of conducting materials. These anvils are good conductors, permitting measurement of the electrical properties of samples placed between the anvils in DAC by using the anvils as the electrical contacts to the sample.

The purpose of our researches is to reveal the features of phase transformations in Ti - alloy under high pressure.

We report the results of an investigation of the TEMF of Ti - alloys at pressures 15 -50 GPa and room temperature in DAC. It was shown that reversible changes of TEMF take place at pressure increasing and decreasing.

15:50 poster I-25

Using solid state infiltration methods for severe plastic deformation

Aikaterini Zi[1], Yuri Estrin[1], Ralph J. Hellmig[1], Michael Kazakevich[2], Eugene Rabkin[2]

1. *Clausthal University of Technology, Department of Materials Science and Technology, Clausthal-Zellerfeld, Germany* **2.** *Technion - Israel Institute of Technology, Dept. of Materials Engineering, Haifa 32000, Israel*

Contact: aikaterini.zi@tu-clausthal.de

ECAP (equal channel angular pressing) is a well-known method of severe plastic deformation used to produce ultrafine grained materials. The dimensions of ECAP specimens are usually in the centimeter range. For producing high strength wires or fibres having a diameter in the micrometer/ millimeter range, downscaling of the ECAP process may be a viable option. To achieve this, several kinds of experiments have been carried out.

For downscaling to micrometer range, porous steel discs can be used as processing tool. In this case, a solid state infiltration method as a variant of the known forcefill process can be used. Severe plastic deformation is introduced due to material flow through the tortuous channels inside the porous pre-form leading to grain refinement depending on processing conditions.

To obtain specimens with a typical dimension in the millimeter range, the forcefill approach taken above was altered using die channels produced by conventional or ultrasonic drilling. The tool geometry used is equivalent to conventional ECAP using a multi-channel die.

Results on aluminium for several channel dimensions and processing conditions will be presented and discussed.

15:50 poster I-26

SiC-Zn nanocomposites obtained using high-pressure infiltration technique

Anna Swiderska - Sroda[1], Grzegorz Kalisz[1,2], Ewa Grzanka[3,4], Stanisław Gierlotka[3], Svetlana Stelmakh[3], Nathalie C. Herlin-Boime[5], Bogdan F. Palosz[3]

1. *Polish Academy of Sciences, High Pressure Research Center (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **4.** *Warsaw University, Faculty of Physics, Hoza 69 Str., Warszawa 00-681, Poland* **5.** *CEA-Saclay, Bat 522, Gif-sur-Yvette 91191, France*

Contact: annas@unipress.waw.pl

Nanocomposites with a primary nanocrystalline SiC matrix and a secondary nanocrystalline Zn phase were synthesized in toroid-type cell, under pressure up to 8 GPa at temperature up to 1650^{o}C using high-pressure infiltration technique. The advantage of our technique is that in a single, continuos process (i) the powder is compressed to form the matrix with nanopores (ii) the nanopores are filled with a second phase (iii) the second phase, here Zn, crystallizes in the nano-scale. The key limitation of this technique is that the pores in the matrix need to stay open during the entire process of infiltration. We used SiC nanopowders with different granularity (particle size from several to hundreds of nanometers) and mean crystal size in the range 10 - 60 nm. The porosity studies of the green bodies prepared from the investigated powders (2 and 8 GPa, room temperature) showed that: (i) in all samples open porosity was maintained, (ii) in the powder with particle size in the range of several nanometers fractal structure of the powder promoted the infiltration process. The nanocomposites, which we have obtained showed microstructure with two phases distributed (mixed) homogeneously on the nano-scale. Process conditions and powder granularity influenced the crystal size of the second phase. The mean grain size of Zn varied from 20 to 100 nm and was smaller in the composites obtained with finer matrix, under higher pressure at lower temperature. The volume fraction of Zn reached 20 % independently of the powder morphology and p – T conditions. The strength and specific surface of the interfacial boundary influence the mechanical properties of the composites. The microhardness HV_{02} of our nanocomposites varied from 5 to 16 GPa and rose up with an increase of pressure and temperature of the infiltration process and with the powder refinement.

15:50 poster I-27

CBN-TiN nanocomposites; structure and mechanical investigations

Wioletta Gorczyńska-Zawiślan[1], Ewa Benko[1,2], Piotr Klimczyk[1]

1. *Institute of Metal Cutting, Wrocławska 37A, Kraków 30-011, Poland* **2.** *University of Bielsko-Biala, Willowa 2, Bielsko-Biała 43-309, Poland*

Contact: wioletta.gorczynska@ios.krakow.pl

The nanomaterials gives opportunity to obtain bulk nanocomposites with better mechanical properties than conventional materials. The aim of this work was to obtain the superhard materials – cBN-TiN nanocomposites and to study them basic mechanical properties. This nanocomposites were sintering under high pressure (7-8 GPa) and high temperature (1400-1800 ^{0}C) conditions. The microstructure observations were performed by using scanning electron microscopy (SEM) and also density, chemical phase compositions (XRD) were investigated. Mechanical properties as microhardness and Young`s modulus were determined. The preliminary fabrication processes of these nanocomposites and its results are presented.

15:50 poster I-28

NANOCRYSTALLINE CEMENTED CARBIDES SINTERD BY THE PULSE PLASMA METHOD

Andrzej Michalski, Dariusz Siemiaszko

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

WC-Co carbides are usually produced by sintering with the participation of a liquid cobalt phase. However, the sintering of nanocrystalline WC-Co powders in the presence of a liquid cobalt phase results in the WC grains growing out. For example, if a powder with an initial grain size of about 10nm is subjected to conventional sintering, the WC grain size usually increases to about 500nm, and even with the use of growth inhibitors – to about 300nm.

The study presents the results of examinations of the structure and properties of nanocrystalline cemented carbides consolidated by the pulse plasma sintering method (PPS). The sintered nanocrystalline carbides were produced of a mixture of WC5wt.% Co powders with an average WC grain size of about 100nm. The sintering process was carried out at a temperature of 1200^{o}C under a load of 50MPa for 5min. The thus sintered carbides had an average crystallite size of about 120nm, a density of 15.2g/cm^{3}, hardness of 2000HV30 and the stress intensity factor K_{IC} = 12.6MPa*m$^{1/2}$. The hardness and resistance to brittle fracture of the WC5wt.%Co carbides sintered by PPS are higher than those of carbides sintered by the conventional methods with the participation of a liquid cobalt phase. For example, a conventionally sintered carbide (WC+6wt.%Co) with an average WC grain size of 700nm has a hardness of 1860HV30 and a stress intensity factor K_{IC} of 8.6MPa*m$^{1/2}$.

15:50 poster I-29

PULSE PLASMA SINTERING OF A NANOCRYSTALLINE Cu POWDER

Marcin Rosinski, Andrzej Michalski, Dariusz Siemiaszko, Jakub Jaroszewicz, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: ninmar@inmat.pw.edu.pl

The nanocrystalline copper powders produced by the reaction of the CuO reduction with hydrogen, were consolidated using the pulse plasma sintering (PPS) method. The sintering process was carried out at temperatures between 450 and 600^{o}C under a load of 60MPa for 5min. The average crystallite size of the sinters thus obtained was about 80nm. The sinters pro-

duced at 450°C had a relative density of 87%, and those sintered at 600°C – 92%; their hardness was 215HV0.1 and 195HV0.1, respectively, which means that it is threefold as high as that of solid copper with a nanocrystalline grain size of about 50μm. The resistivity of the sinters was about 0.103μΩm i.e. about tenfold greater than that of coarse-grained solid copper.

15:50 poster I-30

NANOCRYSTALLINE NiAl-TiC COMPOSITES SINTERED BY THE PULSE PLASMA METHOD

Marcin Rosinski, Andrzej Michalski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: ninmar@inmat.pw.edu.pl

The paper presents the results of examinations of nanocrsytalline NiAl-TiC composites produced from nanocrystalline powders. The powders were prepared by mechanical milling and subjected to pulse plasma sintering (PPS). The effects of the sintering process parameters, such as the heating rate, and of the homogeneity of the powder were taken into account. The analysis of the consolidation results chiefly included the characteristic features of the composites, such as the density, hardness, resistance to brittle fracture and grain size. The grain size was measured by X-ray diffraction (XRD) using the Hall-Williamson method and transmission electron microscopy (TEM).

Our experiments show that by using pulse plasma sintering, it is possible to produce sinters with a density very close to theoretical, and with their nanocrystalline structure being preserved. The NiAl-TiC (40wt.%) sinters had a hardness of 1070HV1 and 1000HV30, and the resistance to brittle fracture $K_{IC} = 7.5MPa*m^{1/2}$.

15:50 poster I-31

NANOCRYSTALLINE Cu-Al_2O_3 COMPOSITES SINTERED BY THE PULSE PLASMA TECHNIQUE

Marcin Rosinski, Andrzej Michalski, Jakub Jaroszewicz, Dariusz Siemiaszko, Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

The paper presents the results of examinations of the structure and properties of the nanocrystalline Cu-Al_2O_3 composites with the two different Al_2O_3 contents: 10vol.% and 20vol.%. The composites were produced using a mixture of copper and Al_2O_3 powders with an average crystallites size of about 60nm in Cu and about 40nm in Al_2O_3. The powders were consolidated by pulse plasma sintering (PPS) at a temperature of 650°C under a load 60 MPa for 5min. Irrespective of the volumetric content of Al_2O_3, the relative density of the composites is about 92%, and the average Cu crystallite size is about 80nm. The hardness of the composites varies with the volumetric content of Al_2O_3, and is equal to 250HV0.1 with 20% of Al_2O_3 and to 220HV0.1 with 10% of Al_2O_3. The Cu-20%Al_2O_3 composite has a resistivity of 0.386μΩm and the composite with 10% of Al_2O_3 - a resistivity of 0.149μΩm.

15:50 poster I-32

High-pressure Induced Structural Decomposition of RE-doped YAG Nanoceramics

Dariusz Hreniak[1], Stanisław Gierlotka[2], Witold Lojkowski[2], Wiesław Stręk[1], Piotr Mazur[1], Robert Fedyk[2], Robert Pazika, Robert Pązik, Agnieszka Opalińska

1. *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: hreniak@int.pan.wroc.pl

Abstract. The preparation of transparent nanoceramics from nanocrystalline $Y_3Al_5O_{12}$ (YAG) powders doped with rare-earth ions has been described and the results of investigation of the structure and morphology have been presented. Decomposition of YAG nanocrystals into $YAlO_3$ (YAP) was observed. The temperature and pressure for the decomposition was much lower than that reported for larger crystals. The transformation was connected with grain coarsening. The influence of the method of preparation of the YAG nanopowders on the final transparency of the nanoceramic produced was determined. Preliminary results of the dependence of luminescence properties on the structural transformation of the nanograins are presented.

15:50 poster I-33

Nanocrystalline SiC compacts obtained by sintering of laser synthesized nanopowders under extreme pressures

Nathalie C. Herlin-Boime[1], Yann Leconte[2], Stanisław Gierlotka[3], Grzegorz Kalisz[3,4], Anna Swiderska - Sroda[3], Ewa Grzanka[3,5], Svetlana Stelmakh[3], Bogdan F. Palosz[3]

1. *CEA-Saclay, Bat 522, Gif-sur-Yvette 91191, France* **2.** *CEA-Saclay, Saclay 91191, France* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **4.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **5.** *Warsaw University, Faculty of Physics, Hoza 69 Str., Warszawa 00-681, Poland*

Contact: herlin@drecam.cea.fr

The laser pyrolysis technique was used to synthesize SiC nanopowders from a mixture of SiH_4 and C_2H_2. The experimental parameters (gas flow rates, laser power) were tuned in order to obtain powders with different characteristics (grain and crystallite sizes, structure). Batches of SiC nanopowders with narrow size distribution and average grain size controlled in the range 15-90 nm were obtained. Detailed structural analyses have been performed on the initial powders. Sintering experiments were performed under extreme high pressure in a broad range of pressure and temperature conditions (up to 8 GPa and 1900C). Dense sintered SiC with average grain size between 20 and 200nm were obtained. Studies of microstructure, showed a significant decrease of porosity with an increase of the sintering pressure. Diffraction studies of the sintered material did not show any significant grain growth below 1800oC. However, they also revealed a high level microstrains in the compacts. Both higher temperature and pressure lead to an increase of the hardness of the sintered materials. Larger grains, either because of the starting material or because of the recrystallization during sintering, generally lead to the softer compacts. In the sintered materials the volume fraction of the atoms located at grain boundaries can be up to 20-30% of total sample volume, assuming that thickness of boundaries is about 0.5-1 nm. Therefore, the effect of grain boundaries on the volume properties of the sintered materials composed of really small crystallites can be very strong.

15:50 poster I-34

Production and sintering of diamond particles with nanometric nickel coating

Evgueni Ekimov[1], A.F. Pal[2], A.N. Ryabinkin[2], A.O. Serov[2], V.E. Fortov[3], A.S. Ivanov[4], A.N. Starostin[4], R.A. Sadykov[1], N.N. Mel'nik[5], Adam Presz[6]

1. *Institute for High Pressure Physics (IHPP), IHPP, Troitsk 142190, Russian Federation* **2.** *M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation* **3.** *Institute for High Energy Densities RAS (IHED RAS), Izhorskaya 13/19, Moscow 125412, Russian Federation* **4.** *Russian Research Center "Kurchatov Institute", Moscow 123182, Russian Federation* **5.** *P.N. Lebedev Physical Institute RAS, Moscow 119991, Russian Federation* **6.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: ekimov@ns.hppi.troitsk.ru

The physical and chemical processes occurring at high pressures (8-9 GPa) and temperatures (up to 2400^0 C) in the system of diamond particles of 3 – 5 μm in size with addition of nickel in amount of 1 – 2 % vol were investigated. Nickel was deposited on the surfaces of diamond particles as the layers near of 10 nm thickness without sp^2-carbon formation in the final product. The deposition was performed by means of the dusty plasma technique in the RF plasma. The coatings with two different types of surface morphology – flat dense or dendritic were obtained depending on the plasma parameters. In the course of the diamond composite powder sintering the processes of the nickel recrystallization and the graphitization of diamond were observed at the temperatures more than 800^0 C. At the sintering temperatures more than 1400^0 C the graphite-like carbon is present in the diamond compacts as two phases with different interplanar spaces d_{002}. The absolute decrease of non-diamond carbon content in the specimens and relative increase of the graphite-like carbon with "low" interplanar distance as the sintering temperature increase were established.

15:50 poster I-35

Synthesis and properties of GaAs nano-composites

Grzegorz Kalisz[1,3], Ewa Grzanka[1,2], Anna Swiderska - Sroda[1], Stanisław Gierlotka[1], Dariusz Wasik[4], Maria Kamińska[4], Bogdan F. Palosz[1]

1. *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Warsaw University, Faculty of Physics, Hoza 69 Str., Warszawa 00-681, Poland* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **4.** *Warsaw University, Institute of Experimental Physics (IEP UW), Hoza 69, Warszawa 00-681, Poland*

Contact: kalisz@unipress.waw.pl

Semiconductor nanocomposites consisting of primary phase in the form of hard nanocrystalline SiC matrix and the secondary nanocrystalline semiconductor phase were obtained by high-pressure zone infiltration. The technique was similar to that recently used by us for synthesis of superhard and metal-ceramic nanocomposites based on nanocrystalline diamond and SiC powder. The synthesis process occurs in three stages: (i) the nano-powder of SiC is compacted, (ii) the temperature is increased above the melting point of GaAs and the pores are being filled with liquid, (iii) upon cooling GaAs nanocrystallites grow in the pores. The process is performed under pressures up to 8GPa and temperatures up to 1600K. Under extreme pressures the liquid phase penetrates the pores of sizes smaller than 10 nm. Synthesis of nanocomposites was performed using toroid-type high-pressure apparatus in Warsaw (sample size φ 4x5 mm). Commercially available nanocrystalline SiC and powders synthesised at CEA, Saclay, from gaseous mixtures of silane with hydrocarbons were used as SiC matrices.

X-ray diffraction studies were performed using laboratory diffraction equipment. Phase analysis, grain size distribution function and also macro- and micro-strains present in the synthesised materials were examined. Microstructure of the syn-

thesised materials was characterised using Scanning Electron Microscopy (SEM) equipped with microprobe. Far-infrared reflectivity and Raman spectroscopy techniques showed characteristic LO and TO phonons of both GaAs and SiC materials and allowed to trace built-in strains.

15:50 poster I-36

Electrical properties of nanocrystalline ZrO2 at high-pressure

Anna N. Trefilova[1], Alexey N. Babushkin[1], Witold Lojkowski[2], Agnieszka Opalińska

1. *Ural State University, Department of Physics, Lenin Av., 51, Ekaterinburg 620083, Russian Federation* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: Trefilova@mail.ru

We studied correlation between the sizes of crystallite and resistance ZrO_2 at the pressures 22 - 50 GPa and temperatures 77 - 400 K. Nanocrystalline praseodymium doped zirconia powders were produced using a microwave driven hydrothermal process under pressures up to 8 GPa. Nanopowders of zirconia with Pr in solid solutions having Pr contents of 0.5 mol %. The bulk material sample of zirconia having Y_2O_3 contents of 5 mol %. Size of crystallites changed from 10 to 500 nm.

The dc resistance measurements were carried out in a diamond anvil cell rounded cone-plane type.

At a pressures of about 30-37 GPa the ZrO2 resistance decreases by 3-4 orders of magnitude. It's found that the transition pressure of ZrO_2 depends on crystallite size.The smaller the crystals the smaller the transition pressure. The reduction of transition pressure was observed to 10 nm. However at 10 nm the transition pressure rises steeply.

It is possible to suspect, that the surface effects essentially change ZrO_2 conductivity mechanism at high pressures.

At the pressure of about 40-48 GPa anomalies in the pressure dependence of the resistance and of the parameters which depend on the concentration, mobility, and activation energy of the charge carriers are found. This permit to suppose the high-pressure induced phase transformation at 40-48 GPa.

Furthermore we obtain that the activation energy depends on the crystallite size. The smaller the crystals the higher activation energy.

We studied relaxation processes in ZrO_2 under the high pressures and the room temperature.The analysis of experimental data has shown that the time function of electric resistance most precisely described by exponential function. It can be seen, that relaxation times depend on pressure and crystallite size.

15:50 poster I-37

Pressure- assistance lateral nanostructuring of the epitaxial silicon layers with SeGe quantum wells

Irina V. Antonova[1], Mitrofan B. Gulyaev[1], Vladimir A. Skuratov[2], Andrzej Misiuk[3], Peter Zaumseill[4]

1. *Institute of Semiconductor Physics SB RAS, pr. Lavrentieva 13, Novosibirsk 630090, Russian Federation* **2.** *Joint Institute of Nuclear Research, Dubna, Russian Federation* **3.** *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland* **4.** *Institute for Semiconductor Physics, Frankfurt(Oder), Germany*

Contact: antonova@isp.nsc.ru

Nowadays epitaxial silicon layers with SiGe quantum wells are increasingly important due to possible new application in nano- and microelectronics. In such structures SiGe layers are under strong compressive stress and Si layers are under tensile stress depending on the Ge content. It is well known, that point defects and stress are two main factors effect on Ge and doping impurity redistribution in heterostructure. Diffusion coefficient of Ge into strained Si layer is increased in 2-3 orders of magnitude in comparison with that in fully relaxed Si [1]. Low fluence high energy ion implantation (HEII) allows one to add a periodical lateral gradient of stress and defects in epitaxial layer. Moreover, HEII can cause formation of vertically ordering nanodots or nanoclustors [2] and can be used for development of methods of local diffusion of Ge or doping impurities in SiGe/Si heterostructures. In the present study the comparison of Ge redistribution in epitaxial SiGe/Si superlattice under annealing at different hydrostatic pressure and in the case of low dose HEII in superlattice with subsequent annealing at atmospheric pressure were made. Structures were characterized by capacitance-voltage measurements, secondary ions mass spectroscopy and atomic force microscopy. It was found that in the case of HEII the redistribution of impurities is drastically increased. Whereas uniform high pressure cause less pronounced effect. Details and mechanism of HEII and hydrostatic high pressure impact on impurity redistribution in epitaxial SiGe/Si superlattice are discussed in the report.

The work was supported in a part by Russian Foundation for Basic Research, grant 05-02-16479.

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15:50 poster I-38

Sintering of icosahedral quasicrystalline Al-Cu-Fe powders using high quasihydrostatic pressure

Mykola O. Yefimov, Yuly V. Milman, Dina V. Lotsko, Sergey V. Ulshin, Izabella I. Timofeeva, Miroslav V. Karpets, Olexander I. Bykov

Institute for Problems of Materials Science, 3, Krzhizhanovsky, Kyiv 03142, Ukraine

Contact: yefimov@ipms.kiev.ua

The processes accompanying the sintering of quasicrystalline Al-Cu-Fe powders were investigated. Pure Al-Cu-Fe powders and powders alloyed by 0.3 wt.% Sc and 11 wt.% Cr were chosen for studies. Quasicrystalline powders were produced by water atomization technique. The technology of sintering with high quasihydrostatic pressure was used. High-temperature sintering under high quasihydrostatic pressure was realized in the high-pressure cell of "lentil" type. Sintering was carried out at 700 C during 2 min using the pressure range from 2.8 up to 7 GPa. The optimum pressure of preliminary cold pressing (0.6 GPa) and high temperature pressing (3-4 GPa) were determined. The compacts with porosity < 2% were obtained. The phase compositions of compacts were studied by X-ray diffractometry and SEM techniques. Microhardness was measured by Vickers indenter at a load of 2 N. Using the original technique developed by authors the plasticity characteristic δ_H of the sintered quasicrystalline compacts was determined from the microhardness values as well.

The annealed Al-Cu-Fe and Al-Cu-Fe-Sc compacts were obtained in the single-phase quasicrystalline state whereas the Al-Cu-Fe-Cr compacts - in the single-phase approximant state.

It is revealed that microhardness of sintered compacts noticeably lowers with the growth of the sintering pressure, whereas line broadening in X-ray patterns of compacts increases. The analysis of line broadening in X-ray patterns has shown a progressive growth of the density of phason defects in quasicrystalline lattice with increasing the sintering pressure.

The stress-strain curves were obtained for all sintered compacts. The stages of strengthening and softening are observed for all deformation curves. However, the softening stage on deformation curve of Al-Cu-Fe-Cr compacts (approximant phase) is shorter than for the samples of the quasicrystalline phase. In all sintered compacts the δ_H=0.67-0.70.

15:50 poster I-39

The amorphous phase decomposition in Fe-based alloys after severe plastic deformation

Danila V. Matveev[1], G. E. Abrosimova[1], A. S. Aronin[1], S. V. Dobatkin[2], O. G. Rybchenko[1], E. V. Tatiyanin[2,3], I. I. Zverkova[1]

1. *Russian Academy of Sciences, Institute of Solid State Physics, Chernogolovka 142432, Russian Federation* **2.** *Russian Academy of Sciences, Baikov Institute of Metallurgy and Material Science (IMET RAS), 49 Leninsky prospect, Moscow 119991, Russian Federation* **3.** *Institute for High Pressure Physics, Russian Academy of Sciences, Troitsk 142092, Russian Federation*

Contact: matveev@issp.ac.ru

Fe-based amorphous alloys show high magnetic properties. Last years metallic glasses have attracted the interest as precursors for production of nanocrystalline state. Nanocrystalline structure created by primary crystallization of melt-spun amorphous Fe-based ribbons exhibits excellent soft magnetic properties. Small addition of Cu and Nb deteriorate potential properties of nanocrystalline Fe-Si-B alloy. However nanocrystalline structure does not form at the crystallization of Fe-Si-B and Fe-B alloys at heating. Nowadays, formation of nanocrystalline structure was found to occur under severe plastic deformation [1]. Recently we have formed nanocrystalline structure in amorphous Fe81B13Si6 alloy by high pressure torsion at different temperatures. The nanocrystalline structure was found to form at achievement of definite strain. The phase composition, crystal size, morphology and distribution of crystals in the sample depend on a strain and treatment temperature. The minimal size of the obtained crystals is 5-10 nm. In this work we try to form the nanocrystalline structure by high pressure treatment of amorphous Fe80B20 and Fe84B16 alloys.

Amorphous Fe80B20 and Fe84B16 alloys were prepared by melt quenching. The samples were exposed to pressure of 4 and 6 GPa at room temperature in a toroid-type chamber using hexagonal BN as pressure transmitting medium. Structure of the samples was studied by X-ray diffraction and transmission electron microscopy. X-ray diffraction patterns of as-prepared samples were typical for metallic glasses. A position of first diffuse halo corresponds to 2□ = 57.09 for Fe84B16 and 2□ = 57.28 for Fe80B20 (FeK□ radiation). The shift of the position of first diffuse maximum points to a change of a radius of first coordination sphere with boron concentration. After high pressure treatment the shape of diffuse peak slightly changes and it could be described only as a sum of two Gauss functions with the first of them corresponding to amorph

[ABSTRACT TRUNCATED TO 2000 LETTERS]

15:50 poster I-40

Influence of interface region and surrounding media of quantum dots on exciton binding energy.

Anton A. Grigoriev[1], Vladimir G. Litovchenko[2,3]

1. *V.Lashkaryov Institute of Semiconductor Physics NAS Ukraine (ISP), Nauky prosp., Kyiv 03028, Ukraine* **2.** *Institute of Semiconductor Physics (ISP), Nauki pr., Kyiv 03028, Ukraine* **3.** *Institute of Semiconductor Physics NAS Ukraine, Kyiv, Ukraine*

Contact: grigorev@isp.kiev.ua

We present theoretically investigation of excitonic effects on the energetic structure of silicon quantum dots in silicon oxide matrix. Calculations were performed taking into account the electron-hole Coulomb interactions, expanded interface area, leakage of electronic density from quantum dot and experimental values of barrier high.

The interactions of electrons and holes are strongly enhanced in ultrasmall "quasiopen" quantum dots because of decreasing effective permittivity caused by influence of dots surrounding media.

Dependences of exciton binding energy, work function (electron affinity) and energy of optical transfers from quantum dot diameter have been obtained. The achieved results demonstrate notably difference to the parameters achieved from well-established idealized case (sharp and infinit barriers) to the ultrasmall (~1.5 nm) dots.

Usage of renewed by us effective media approximation allows us to predict some principal new physical effects, such as negative electron affinity (electrons localization outside the dot), which can be useful for electron field emission applications.

Comparison with recent experimental on Si-nanostructures embeded into SiOx insulator and created in porous surface layer show good quantitative agrrement.

Such structures consider as perspective in consumer optoelectronics and as field emmiter for construction of flat panel displays.

Thursday, 8 September

Characterisation and nanostructure evolution

Thursday morning, 8 September, 9:00
Main Building, Small Hall (Mała aula)
Joerg Weissmueller presides

9:00 invited oral

Transmission Electron Microscopy Investigations of Phase Transitions in SPD Materials

H. Peter Karnthaler, Christian Rentenberger, Thomas Waitz

University of Vienna, Institute of Materials Physics, Vienna, Austria

Contact: hans-peter.karnthaler@univie.ac.at

When methods of transmission electron microscopy (TEM) are applied to nanostructured alloys processed by severe plastic deformation (SPD) like high pressure torsion (HPT) special methodological points have to be considered. In HPT materials the density of grain boundaries is so high that in the TEM images frequently overlapping grains are causing moiré patterns that must be interpreted correctly. In the following, two examples are given of studying phase transitions in HPT materials. In polycrystalline L12 ordered Ni3Al a two phase structure is formed by HPT deformation at about 8000 %. The structure consists of veins of disordered nanograins that are embedded in the ordered coarse-grained structure. The observations show that inhomogeneous deformation leads to localized disorder and a high density of dislocations. Dynamic recovery causes a heterogeneous formation of the nanocrystalline structure. The nanocrystalline veins showing only a weak texture grow by the formation of nanograins generated at their interface. At strains above 50.000% the whole volume transforms to the nanocrystalline structure [CR & HPK, Acta Mater. 53 (2005) 3031]. In NiTi, a shape memory alloy, the phase transformations caused by the HPT deformation were investigated. Prior to HPT the specimen was in the cubic B2 austenite phase whereas after a HPT deformation of about 1000%, strain induced B19' martensite containing shear bands of an amorphous phase were observed. The latter fill the whole volume at larger strains. Since the amorphous phase formed by HPT contains several nanocrystalline debris that can act as nucleation sites, it can be successfully used to make a bulk nanocrystalline structure by devitrification. The nanocrystalline structure of NiTi processed this way shows a new size-dependent martensitic transformation path based on atomic scale twinning that is not observed in the large-grained material [TW & HPK, Acta Mater. 52 (2004) 5461].

9:30 invited oral

Structure of nano-crystals: a key to understanding the unique properties of nano-materials

Bogdan F. Palosz

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: palosz@unipress.waw.pl

A key problem of "nanoscience" is understanding what is the origin of the difference in the properties between nano- and single crystals. In contrast to single- for nano-crystals one cannot disregard the fact that there is a large number of atoms located at the surface and, consequently, one has to accept that the crystallographic structure of a very small size particle may deviate from that in the bulk crystals. In a tentative model, a nanocrystal might be presented as a two-phase system formed by the grain core and the surface shell, both having characteristic dimensions.

There are no standard procedures established for performance of structural analysis of a nanocrystal. The problem is that its complex structure cannot be well represented by a unit cell. Only for this reason the methods which are used for elaboration of powder diffraction data based on Bragg equation should not be applied for examination of nanocrystalline materials. The values of lattice parameters calculated from individual Bragg reflection differ between each other, and because they vary with the diffraction vector Q, they are rather "apparent lattice parameters", alp's. Although Bragg equation is not fulfilled for very small crystals, it might be practical to base analysis of real structure of nanocrystals referring to Bragg type scattering and concentrate on interpretation of the deviations of diffraction images of nanocrystals from those corresponding to a perfect crystal lattice.

Examples of examination of distribution of strains among nano-grain core and surface shell has been examined at ambient conditions, but also under high pressures and high temperatures are given based on X-ray and neutron scattering experimental data. Advantages of the analysis referring to reciprocal and real spaces of nano-structures are discussed.

10:00 invited oral

Application of XRD diffraction methods for determination of grain size distribution in nanoparticles

Roman Pielaszek, Witold Lojkowski

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: roman@pielaszek.pl

During XRD measurement, X-ray detectors (counters) register scattered photons with a finite efficiency. This results in experimental noise proportional to the square root of the intensity, according to well known properties of Bernoulli distribution.

In standard cases signal/noise ratio suffices for X-Ray phase analysis (determination of crystalline phases) and basic grain size approximation by measurement of peak width. However, further investigations of a fine structure of the peak profile may be affected by high noise amplitude.

In the present paper we discuss determination of the size distribution of nanocrystals by analysis of the fine structure of the peak profile. Influence of the experimental noise and resulting accuracy of the method will be investigated. We will estimate photon flux (X-ray tube/synchrotron) needed for successful evaluation and give a practical directions on optimal experimental setup. Dispersion of the size distribution parameters (i.e. dispesion of average grain size and dispersion of dispersion of the grain sizes) will be given as a function of experimental noise level.

11:00 oral

Simultaneous measurement of nanoprobe indentation pressure and photoluminescence of quantum dots

Kazunari Ozasa[1,2], Mizuo MAEDA[1], Masahiko HARA[1], Masane OHASHI[3], Yuan-Hua LIANG[3], Yoshio ARAI[3]

1. *Institute of Physical and Chemical Research (RIKEN), 2-1 Hirosawa, Wako-Shi, Saitama 351-0198, Japan*
2. *CREST-JST, 3-1-6-5 Shibuya, Tokyo 150-0002, Japan*
3. *Saitama University, 255 Shimo-ookubo, Sakuraku, Saitama 338-8570, Japan*

Contact: ozasa@riken.jp

Nanoprobe indentation onto the surfaces of solid materials can produce a very high pressure (strain) more than 20-80 GPa for the nm-scale areas. When the nanoprobe is made from an optical fiber, the optical properties such as photoluminescence (PL) or reflectance of the samples under a high pressure can be measured with nm-scale resolutions. However, simultaneous measurements of high pressure and optical properties are generally difficult for nanoprobe indentations, since the indentation force is too small for direct measurements.

We have succeeded to measure simultaneously the nanoprobe indentation-force (pressure) and the PL of InGaAs/GaAs quantum dots (QDs) at low temperatures (down to 10K), by employing a small resistance-bridge load-cell. Indentation force as low as 200 uN can be measured, which corresponds to approximately 62 GPa at the sample surface. The PL of InGaAs/GaAs QDs was measured through the 1μm aperture at the apex of the optical-fiber-nanoprobe, indented onto the surface within the elastic limits. The strain-enhanced fine PL peaks originating from single QDs were observed reproducibly, only when a certain range of GPa pressures were produced by the nanoprobe indentation. Blue shifts of fine peaks were observed with the PL enhancement, suggesting that the strain effects are responsible for the PL enhancement.

By the model calculation of nanoprobe indentation with elastic deformation theory and strain Hamiltonian, we figured out the strain distribution and energy-level shifts, and found that the potential minimum for light holes is formed in GaAs be-

neath the nanprobe, which causes the PL enhancement through hole accumulation into the QDs. Since the simultaneous measurement of indentation force and PL peaks enable us to analyze the nanoprobe-induced strain (pressure) distribution, it contributes much to the application of nanprobe-modulation spectroscopy, where we can measure the nm-scale optical properties of the samples.

11:15 oral

Microstructural Evolution in Mechanical Alloying and Hot Pressing of Aluminium and 316 Stainless Steel Powder Blend

Indranil Manna[1], Asis Samanta[2], Partha P. Chattopadhyay[2], Witold Lojkowski[3], Hans-Jörg Fecht[4,5]

1. *Indian Institute Of Technology,Kharagpur (IIT), Kharagpur, India* **2.** *B E College, Howrah 711103, India* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **4.** *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany* **5.** *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany*

Contact: imanna@metal.iitkgp.ernet.in

Mechanical alloying is a solid-state non-equilibrium mechano-chemical synthesis technique for producing nanocrystalline or amorphous metallic alloys, intermetallic phases, compounds and composites. The process involves a complex sequence of deformation, fragmentation, cold welding, dynamic-polygonization and grain refinement. Thus, milling in a planetary ball mill could simulate the microstructural changes anticipated at the surface of bulk components exposed to severe wear, erosion and mechanical deformation. Indeed, the microstructural changes associated with railroad steel during high-speed train movements could successfully be simulated by subjecting the same material to mechanical attrition (or an accelerated process of wear) in high-energy mechanical milling under predetermined conditions.

AISI 316 stainless steel and similar ferrous materials are often coated on Al based light alloys to improve wear resistance. Therefore, ball milling of pre-alloyed AISI 316 stainless steel and elemental Al powder blend in different proportion may simulate the microstructural evolution at different locations of the alloyed zones extending from the coated surface (pure stainless steel) to the underlying bulk (Al-alloy).

The present study aims to examine the phase evolution in blends comprising different proportions of stainless steel and Al (0, 25, 65 and 85 wt. %) powders during high-energy ball milling though x-ray diffraction analysis, scanning electron microcopy and high-resolution transmission electron microscopy. An attempt has also been made to study the mechanical property of the bulk samples obtained by hot pressing the ball milled powder blend at suitable temperature and pressure. In the present paper, we will present our results on the microstructural changes and mechanical property and discuss the utility of consolidation of amorphous/nanocrystalline powders by high-pressure technique to develop engineering components.

11:30 oral

Evolution of precipitate coarsening reaction in a nanostructured Fe-Ni-Mn maraging alloy

Syamak Hossein Nedjad[1], Mahmoud Nili Ahmadabadi[2], Tadashi Furuhara[3], Tadashi Maki[3]

1. *Sahand University of technology (SUT), Sahand New Town, Tabriz, Iran, Tabriz 51335-1996, Iran* **2.** *University of Tehran (UT), North-Kargar, Tehran 14395-731, Iran* **3.** *Kyoto University, Kyoto 606-8501, Japan*

Contact: nili@ut.ac.ir

Fe-Ni-Mn maraging alloys show discontinuous coarsening of f.c.t. θ-NiMn precipitates along prior austenite grain boundaries (PAGBs) during isothermal aging. Heavy cold rolling of a solution annealed Fe-10Ni-7Mn (wt. %) maraging alloy and subsequent aging treatment at 773 K were found to result in the formation of a nanostructured material. Hardness measurement and transmission electron microscopy were used to study the aging behavior and microstructural evolution of the nanostructured alloy. At early stages of aging, an ultrafine grained structure was observed showing fine matrix precipitates. However, at later stages of aging, severe coarsening of precipitates was found. Consequently, a lamellar duplex microstructure composed of conventional precipitation hardened laths and ultrafine grained ferrite was identified in an intermediate aging time. Coarse f.c.t. θ-NiMn precipitates were identified among ultrafine grained ferrite. At longer stages of aging, evolution of the coarsening reaction were found to result in the development of the ultrafine grained ferrite regions.

Keywords: Nanostructure: Cold Rolling: Maraging: Precipitation: Coarsening

11:45 oral

SPD processed alloys as efficient vacancy-hydrogen systems

Arkadiusz K. Wieczorek, Maciej Krystian, Michael Zehetbauer

University of Vienna, Institute of Materials Physics, Vienna, Austria

Contact: arkadiusz.wieczorek@univie.ac.at

Severe Plastic Deformation (SPD) is a very effective method to produce bulk nanostructured metals [1], under conditions of enhanced hydrostatic pressure and/or of a relatively low deformation temperatures. Such a heavy plastic deformation produces several crystal lattice defects like dislocations, grain

boundaries, and also vacancies. In comparison with conventional cold work techniques, SPD methods achieve a much higher concentration of vacancies [2].

The presence of vacancies (i) significantly accelerates atomic diffusion [3], (ii) enables to trap up to six hydrogen atoms per vacancy, and (iii) can even create ordered vacancy sublattice [4,5]. These features can be important for a hydrogen storage technique in solid state getting more effective and safe than hydrogen storage as a gas or a liquid. One of the most attractive systems is Mg – MgH_2 because of its ability to absorb hydrogen up to 7.6 wt.%, its low cost and abundance. However, sluggish desorption kinetics due to rather low hydrogen diffusion in magnesium hydride has prevented its practical use until now. The creation of additional lattice defects, particularly vacancies in the Mg structure by means of SPD methods may yield marked improvements of diffusion as well as of H_2 storage capacity, although much work is still needed to fully understand processes taking place in metal-vacancy-hydrogen systems.

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12:00 oral

Buried nano-structured layers in high temperature-pressure treated Cz-Si:He

Andrzej Misiuk[1], Barbara Surma[1,2], Jadwiga Bak-Misiuk[3]

1. *Institute of Electron Technology (ITE), Al. Lotników 32/46, Warszawa 02-668, Poland* **2.** *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **3.** *Polish Academy of Sciences, Institute of Physics (IFPAN), al. Lotnikow 32/46, Warszawa 02-668, Poland*

Contact: misiuk@ite.waw.pl

The effects of treatment of helium implanted Czochralski grown silicon (Cz-Si:He, He ion dose up to 10(17)cm(-2),at energy up to 300 keV) at HT up to 1400 K under hydrostatic pressure, HP, up to 1.2 GPa are investigated by transmission electron microscopy, photoluminescence and X-Ray methods.

The treatment at up to 920 K under HP results in a creation of buried nano-structured layers containing thin-walled He-filled cavities and bubbles.

The Cz-Si:He samples treated at even higher HT indicate the presence of dislocated (defected) buried layers; their structure depends strongly on HP applied.

HP affects diffusivity of implanted helium and of implantation - induced point defects and thus promotes a creation of smaller but more numerous cavities / bubbles as well as of other defects near the range of implanted helium ions.

Synthesis of nanostructured metals

Thursday afternoon, 8 September, 14:00
Main Building, Small Hall (Mała aula)
Ruslan Z. Valiev presides

14:00 invited oral

FORMATION OF SUBMICROCRYSTALLINE STRUCTURE IN TITANIUM ALUMINIDES AND THEIR MECHANICAL PROPERTIES

Gennady A. Salishchev

Institute for Metals Superplsticity Problems, Russian Academy of Sciences, Ufa, Russian Federation

Contact: gensal@imsp.da.ru

Intermetallic materials based on titanium aluminides have attracted wide-spread attention for potential use in high temperature applications because of their outstanding properties including high specific strength, good creep and corrosion resistance. However their applications have been limited by low ductility, so that generally hot working can only be accomplished at temperatures above 1000-1200^{o}C. A reduction in the temperature range in which the alloys are superplastic would be commercially attractive. This can be achieved through microstructure refinement to a submicron grain size.

In the present work formation of submicrocrystalline (SMC) structure in different TiAl ($L1_0$ superlattice) and Ti_3Al (DO_{19}) based alloys during hot working and their mechanical properties were studied. A method of production of a SMC structure (d<1 μm) in massive work-pieces based on initiation of dynamic recrystallization during hot working has been developed. The method involves continuous grain refinement due to dynamic recrystallization at a decreasing deformation temperature. A microstructure with a grain size of 0.1 to 0.4 μm and no porosity was produced in different TiAl and Ti_3Al based alloys. Partial disordering was detected in a Ti_3Al alloy with the SMC structure. The grain refinement hardened the intermetallic alloys at room temperature (RT). In a fully ordered Ti_3Al alloy RT ductility increased when the grain size decreased, while the ductility of a partially disordered SMC Ti_3Al and TiAl alloys was close to zero. Low temperature superplasticity (SP) of intermetallic alloys was studied. The effect of grain size, chemistry, phase composition, temperature and strain rate on elongation, flow stress and strain rate sensitivity were investigated. Low-temperature SP behavior was compared with high-temperature SP. As an example of practical application it will be shown the pack rolling for production of sheets with SMC structure from $TiAl_2Nb$ based alloy.

14:30 oral

FORMATION OF SUBMICROCRYSTALLINE STRUCTURE IN MOLYBDENUM BY MULTIPLE ALL-ROUND PRESSING

Konstantin V. Ivanov, Ivan P. Mishin, Yury R. Kolobov

Institute of Stregth Physics and Materials Science of SB RAS, Tomsk, Russian Federation

Contact: ivanov-ru@yandex.ru

It is known that one of the methods for ductilization of refractory metals, e.g. W and Mo, and for decreasing the temperature of ductile-to-brittle transition is grain-size refinement. During the recent decade, methods for grain size refinement employing severe plastic deformation were being developed intensively. It has been shown that grain size reduction of Mo can be effectively achieved by high pressure torsion (HPT). HPT-treatment results in the formation of submicrocrystalline structure (average grain size ~0.2 μm) in molybdenum and in residual porosity removal. However, HPT-samples have very limited size, which renders impossible their practical application. In view of the above, we studied the feasibility to form submicrocrystalline structure in molybdenum using severe plastic deformation by multiple all-round pressing (so-called abc-pressing) which offers a number of advantages over the other methods of severe plastic deformation. For example, abc-pressing allows one to obtain submicrocrystalline structure in bulk samples, it is less labour-intensive and is superior to equal channel angular pressing in that it requires no specific equipment and setup, which is a very attractive feature in view of possible commercial applications.

It is established that molybdenum in the as-received state has the columnar structure with cavities between the columns. Grain size in the cross-section is about 5 μm. After abc-pressing at elevated temperature the cavities on the metallographic section are not observed. The electron microscopic investigations reveal submicrocrystalline structure (d~0.5 μm). As a result of the abc-pressing the microhardness value rises by 1.5 times relative to the as-received state. It is found that thermal stability of the submicrocrystalline structure produced by abc-pressing is significantly lower relative to that produced by high-pressure torsion. The physical reasons for the above unusual difference are discussed.

14:45 invited oral

Hydrostatic Extrusion as a method to produce UFG and NC metals

Waclaw Pachla

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

Contact: wacek@unipress.waw.pl

Formation of ultra-fine grained (UFG) or nanocrystalline (NC) structures requires a very large deformations performed at relatively low temperatures (to prevent dynamic recrystallization). To maintain the material integrity during the severe plastic deformation (SPD) a high pressure must be imposed on the workpiece. These conditions require the special methods of mechanical deformation to be applied. Some of them, commonly used, are: high pressure torsion (HPT), equal channel angular pressing (ECAP), cyclic extrusion compression (CEC), cumulative rolling (CR) and multiple forging (MF). They all impose some limitations on researchers, as small volume or limited length, low homogeneity of strain or susceptibility to cracking. Recently, the hydrostatic extrusion (HE) has shown to be an effective SPD method (see EMRS'2005 Conference contributions). It will be demonstrate, that HE satisfies the bulk nanostructures generation criteria, i.e.: high plastic homogenous strain at low deformation temperatures under high imposed (hydrostatic) pressure. Examples of the effective grain refinement in several treated metals and alloys, as aluminium and aluminium alloys, copper, iron, nickel, titanium, stainless steel, and others will be presented. Simultaneous improvement of mechanical properties, such as hardness, tensile strength and yield stress due to refinement and transformation of microstructure during HE will be discussed. Capability of HE process in further increase of strain during deformation improving the properties of metals and alloys will be presented.

Acknowledgment: This communication reports the results of the project supported by the Ministry of Science and Informatics 4 T08A 045 25, 4 T08A 027 24 and PBZ-KBN-096/T08/2003. The author wishes to acknowledge contribution made to this communication by Prof. K. J. Kurzydlowski, Dr. M. Lewandowska, Dr. H. Garbacz, M.Sc. A. Mazur and M.Sc. M. Kulczyk.

15:15 oral

MECHANISM OF GRAIN REFINEMENT IN ALUMINIUM ALLOYS IN THE PROCESS OF HYDROSTATIC EXTRUSION

Malgorzata Lewandowska

Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland

Contact: malew@inmat.pw.edu.pl

Hydrostatic extrusion (HE) is one of the methods that enable relatively high strains. Its unique features are three-axial compressive stresses within the billet and high strain rates. This results in a rapid processing under high pressure and may provide a new opportunity for suppressing recovery and possible grain growth in heavily deformed materials. This work describes the microstructure evolutions and mechanisms of grain refinement in aluminium alloys processed by this deformation method.

The materials used in this study represent different series of aluminium alloys and include heat-treatable (2XXX and 7XXX series) and non heat-treatable (1XXX series) alloys. The microstructure observations were performed by using TEM and SEM techniques. Next, the revealed microstructures were described quantitatively in terms of the grain size and shape. The distributions of misorientations between neighbouring grains were also calculated on the basis of crystallographic orientations of the grains measured from Kikuchi lines obtained in TEM.

It has been found that the processing by HE results in a significant grain refinement and the formation of large fraction of deformation induced high angle grain boundaries (HAGBs). From the quantitative analysis of the material microstructure and grain boundary characteristics, the contribution of different mechanisms of grain refinement (e.g. continuous dynamic recrystallization, grain rotation) was analysed. These results were compared to aluminium alloys processed by other severe plastic deformation methods. Moreover, one of the aspects of microstructural analysis was the evolution of size, shape and distribution of second phase particles (precipitates and intermetallics). Their influence on the process of grain refinement and HAGB distribution was discussed.

Synthesis of nanostructured metals

Thursday afternoon, 8 September, 15:50
Main Building, Small Hall (Mała aula)
Nikolay A. Krasilnikov presides

15:50 invited oral

Features of CEC: Method, Structure & Materials Properties

Maria Richert

AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland

Contact: mrichert@uci.agh.edu.pl

The Cyclic Extrusion Compression (CEC) is one of the methods of severe plastic deformation (SPD), which are used for the nanomaterials production. The CEC method allows deforming materials to the arbitrarily large deformations without the change of the initial shape of sample. The large hydrostatic compressive stresses are exerted during the deformation, preserving the sample cracking. Using the CEC method the nanomaterials from Cu and aluminum alloys were produced. It was found that, after exerting of true deformation of about j=14, only some part of sample changed into the nanomaterial, while the remainder volume still showing the ultrafine microstructure. The nanometric microstructure creates generally inside the areas of intersecting microbands. Large misorientation was found between the microbands and the surrounding materials, which facilitated the formation of boundaries of nanograins. The hardness of samples increased with the increase of deformation, however only to the some level of about 100 MPa, which appeared as the some boundary hardening of metallic nanomaterials. The slow increase of hardening, above of about the true deformation j=4, suggested the activation of the softening processes. Probably due these processes the arbitrarily increase of hardening is impossible. During the range of small hardening changes still the refinement of nanograins was observed, indicating that grain refinement exactly not fit with the changes of properties.

16:20 invited oral

Features of Twist Extrusion: Method, Structures & Material Properties

Yan Y. Beygelzimer, Dmitry Orlov, Sergey Synkov, Victor M. Varyukhin, Alexey Reshetov, Alexandr Synkov

Donetsk Physics & Technology Institute of the National Academy of Sciences of Ukraine (DonPhTI NASU), 72 R. Luxembourg St., Donetsk 83114, Ukraine

Contact: orlov@donec.net

As it was shown during last decade, severe plastic deformation (SPD) is very effective method of grain refinement aimed to ultra-fine grained and nanostructured materials obtaining.The basic SPD techniques are High Pressure Torsion (HPT) and Equal Channel Angular Extrusion (ECAE).Recently they were developed several new techniques: 3D forging, Accumulative Roll Bonding, Twist Extru-

sion (TE), Constrained Groove Pressing, Repetitive Corrugation and Straightening, etc. Twist extrusion was proposed by Prof.Beygelzimer in 1999 and is being developed by all the authors during last 5 years.Under TE each billet's cross-section is deformed similarly to thin disk under HPT.But unlike it, the deformation is cyclic: at first billet becomes twisted at some angle in one direction, and then - re-twisted at the same angle in opposite direction.In comparison with other SPD techniques, TE has few essential features.Plane of simple shear is perpendicular to a billet axis (in contrast to 45-60 degrees in ECAE).Intense flows of material being deformed within cross-section of a billet.During the processing billet's surface expands by ~80% in twisted part and shrinks to original size upon exiting it.Size of distorted areas of a billet is smaller than under ECAE.Possibility to handle profile billets including those with axial channel.Relative simplicity of TE installation into standard tool due to constant extrusion axis.Our experiments on primary and recycled Al alloys, Cu, Ti, and Ni-base superalloys showed effectiveness of TE in both grain refinement and mechanical properties improving.So, we obtained in the above metals ultra-fine grained structure quite homogeneously distributed in a billet cross-section and length.The mechanical properties are also uniform.This is confirmed by Microhardness and tension tests data.For Ti alloys we observed anisotropic (in longitudinal and transversal directions) mechanical properties.But this anisotropy was eliminated by following rolling.

16:50 oral

The role of the weak points in the corrosion resistance of hydrostatically extruded steel porducts

Dominika Klassek[1], Thomas Suter[2], Waclaw Pachla[3], Krzysztof J. Kurzydlowski[1], Oliver von Trzebiatowski[2], Mariusz Kulczyk[1,3]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Empa, Materials Technology and Research (Empa), Überlandstrasse 129, Dübendorf 8600, Switzerland* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: Dominika.Klassek@materials.pl

Hydrostatic extrusion (HE) can be used to produce nanostructured materials by applying high hydrostatic pressure, to obtain a homogenous microstructure within entire volume of the processed material. Hydrostatically extruded materials exhibit significant increase in the mechanical properties such as hardness and yield stress. The goal of this project is to achieve also an improvement in corrosion resistance of the 316LVM stainless steel by decreasing the grain size down to the nanometer scale by applying HE.

The corrosion behaviour of 316LVM steel has been examined in NaCl solutions with different chloride concentrations. Large scale polarization and etching tests showed that the corrosion resistance of hydrostatically extruded 316LVM steel decreased in comparison to as received specimens. Etching test in VA2 solution indicated that additional weak points, other than MnS inclusions, could be present in the microstructure. They might be related with stresses introduced during HE. In order to relax this stresses, hydrostatically extruded steel has been annealed. Small area measurements in different zones on the rod specimens were carried out in order to find out which weak points are responsible for the corrosion resistance of 316LVM steel. No significant change between centre and edge of the rods was observed. However, preliminary tests performed on cross sections parallel to the HE direction, showed improved corrosion behaviour compared to the measurements performed perpendicular to the HE direction. These results indicate, that MnS inclusions which were deformed during HE, might trigger the onset of corrosion on 316LVM steel.

Friday, 9 September

Nanopowders and nanocomposites

Friday morning, 9 September, 9:00
Main Building, Small Hall (Mała aula)
Bogdan F. Palosz presides

9:00 invited oral

Role of plastic deformation in sintering powders

Yuly V. Milman[1], Alexander Slipenyuk[2]

1. *Institute for Problems of Materials Science, 3, Krzhizhanovsky, Kyiv 03142, Ukraine* **2.** *Institute for Problems of Materials Science, National Academy of Sciences of Ukraine, Krzizanovsky, Kyiv 38(044), Ukraine*

Contact: milman@ipms.kiev.ua

The development of physical concepts and analysis of experimental results have shown that intensive densification of powders in sintering is determined not only by diffusion processes, but by shape changing due to plastic deformation as well. In the absence of external stresses, plastic deformation is caused by the forces of capillary interaction, which are essential for the assembly of sintering particles. The elaborated classification of temperature ranges of plastic deformation includes three types: hot deformation (at temperature $T>T_r$, where T_r is the recrystallization temperature), warm deformation (at $T^*<T<T_r$, where T* is the characteristic deformation temperature) and cold deformation at $T<T^*$. The sintering without loading is possible in the temperature ranges of hot and warm deformation only, i.e., at $T>T^*$. For metals as a rule $T^*<T_D$, where T_D is the temperature at which the velocity of diffusion is sufficient for the process of sintering. There-

fore the minimum sintering temperature for metals is determined by the value of T_D . But in covalent crystals and ceramics $T^*>T_D$ usually. Therefore the temperature of intensive densification for these materials is determined by the value of T* even for nano-size powders. Severe plastic deformation intensively accelerates the sintering process in the range of warm deformation. Increasing dislocation density during severe plastic deformation leads to the accelerated mass transfer due to growth of the vacancies density and due to the accelerated diffusion along the dislocations lines. In some cases the severe plastic deformation makes possible the consolidation of metastable powders: nanostructured, amorphous and quasicrystalline in which compacting must be carried out at relatively low temperatures. The examples of successful application of a severe plastic deformation for sintering of rapidly solidificated powders are given.

9:30 oral

Sintering of nanopowders under high pressure

Stanisław Gierlotka[1], Robert Fedyk[1], Grzegorz Kalisz[1,3], Ewa Grzanka[1,2], Anna Swiderska - Sroda[1]

1. *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Warsaw University, Faculty of Physics, Hoza 69 Str., Warszawa 00-681, Poland* **3.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: xray@unipress.waw.pl

Ceramic materials with nanometer crystallite size are expected to play an important role in the technologies of the future. While search for an industrial-scale method of sintering of nanometric powders is still underway, laboratory-scale samples of nanoceramics can be effectively prepared by sintering under pressure. Using torroid-type high pressure equipment we sintered a range of nanometric powders of various chemical nature, such as YAG (yttrium aluminum garnet), GaN, SiC and others. Sintering pressures were in the range 2-8GPa, and sintering temperatures were selected according to the thermodynamic properties of the materials being sintered. I most cases compacts with almost 100% of the theoretical density could be obtained with the crystallite size in the sintered body nearly equal to that of the initial powder. Controlled grain growth could also be achieved. Hardness of the samples was equal, or sometimes even higher than the reference values. Parameters of the sintering process: pressure, temperate and time had a clear effect on the microstructure and physical properties of the compacts with temperature being the most critical factor. The way the starting powders were synthesized and the way the materials were treated before sintering also greatly influenced microstructure and thus the properties of the compacts. All samples sintered under exhibited considerable residual microstrain. Apart from general considerations a number of material-specific factors e.g. phase transitions were found to affect the sintering process. The compacts sintered were used to demonstrate specific properties of bulk nanomaterials not found in conventional ceramics.

9:45 oral

Influence of Surface Oxidation on Photoluminescence Property from Nanocrystalline Silicon Particles in Pure Water

Keisuke Sato[1,2,3], Koichi Shinoda[3], Satoshi Yanagisawa[3], Kenji Hirakuri[3]

1. *Tokai University, Kitakaname, Hiratsuka, Kanagawa 259-1292, Japan* **2.** *Dept. of Applied Science, Tokai University, Kitakaname, Hiratsuka, Kanagawa 259-1292, Japan* **3.** *Dept. of Electronic and Computer Engineering, Tokyo Denki University, Ishizaka, Hatoyama, Hikigun, Saitama 350-0394, Japan*

Contact: sato@f.dendai.ac.jp

Nanocrystalline silicon (nc-Si) particles have attractive characteristics for a luminescence phenomenon. The luminescence color of nc-Si particles is dependent strongly on the particle size because the reduction of particle size lead to the increase of luminescence energy. The particle size can be freely adjusted by the oxidation onto the surfaces of nc-Si particles. Here, we report the photoluminescence (PL) property from the surface oxidized nc-Si particles in pure water.

The nc-Si particles were formed in a silicon dioxide (SiO_2) layer by cosputtering of Si chips/SiO_2 targets and subsequently annealing at 970 ^oC. The SiO_2 layer surrounding the nc-Si particles, then, was etched by the hydrofluoric steam treatment. After etching, the nc-Si particles were uniformly dispersed in pure water by the ultrasonic wave treatment. The samples were evaluated by using a high resolution transmission electron microscope, fourier transform infrared and PL measurements.

The nc-Si particles with an average size of approximately 2.5 nm were existed in pure water. The surfaces of nc-Si particles were adsorbed with hydrogen and oxygen atoms. The nc-Si particles emitted a red light with a peak at 720 nm. When the nc-Si particles were immersed in pure water during 250 hours, the average size of nc-Si particles reduced from 2.5 nm to 2.3 nm by the increase of oxidation quantity on the surface. This was caused by the replacement of Si atoms existing mostly outside region of nc-Si particles and oxygen atoms which dissolve in pure water. The reduction of particle size resulted in the variation of luminescence color from red light (720 nm) to orange light (650 nm).

10:00 oral

EXAFS and SAXS analysis in forming of metal nanoparticles in water-in-scCO$_2$ microemulsions

Masafumi Harada

Nara Women's University, Kita-Uoya Nishimachi, Nara 630-8506, Japan

Contact: harada@cc.nara-wu.ac.jp

The environmentally benign, nontoxic, and nonflammable supercritical fluids such as water and carbon dioxide (sc-CO_2) are the two most abundant and inexpensive solvents. Water-in-scCO_2 dispersions in the form of microemulsions offer new possibilities in waste minimization for the replacement of organic solvents in separations, reactions, and materials formation processes. In this study, we have tried to synthesize silver (Ag) nanoparticles by the photo-reduction of $AgClO_4$ and cupper (Cu) nanoparticles by the chemical reduction of $Cu(ClO_4)_2$ using reducing reagent such as $NaBH_4$ in the presence of fluorinated surfactants in water-in-scCO_2 microemulsions. Furthermore, we have investigated the reduction process of metal ions and the average particle size of the particles by means of in-situ UV-Vis and EXAFS as well as their aggregates in the microemulsions by means of in-situ SAXS measurements.

Water-in-scCO_2 microemulsions containing $AgClO_4$ and $Cu(ClO_4)_2$ were synthesized in a high-pressure SUS 316 cell which contained fluorinated surfactant FAR-2 or PFNA aqueous solution, as well as $AgClO_4$ (4.3×10^{-4} mol), $Cu(ClO_4)_2 \cdot 6H_2O$ (1.58×10^{-3} mol) and $NaBH_4$ aqueous solution, respectively. The water-to-surfactant molar ratios (w) were ranging from w=5 to w=1000. The cell was then kept at 35 C and 25MPa for 120 min with continuous stirring to form a single-phase microemulsions. In the case of Ag, after stirring, the microemulsions were irradiated with UV light to perform the reduction of Ag ions to form Ag particles. On the other hand, in the case of Cu, during the stirring, the reduction of Cu ions to form Cu particles by $NaBH_4$ occurred. The EXAFS and SAXS measurements were performed using BL-9A and BL-15A, respectively, at the Photon Factory, High Energy Accelerator Research Organization (KEK-PF), Japan. EXAFS spectra of Ag-K and Cu-K edge were collected to estimate the particle size of Ag and Cu during the reduction process, respectively.

10:15 oral

Fabrication and micro-structure characterization of Al2O3/Ni-P composites with interpenetrating phases

Jakub Michalski[1], Tomasz Wejrzanowski[1], Stanisław Gierlotka[2], Jerzy Bieliński[3], Katarzyna Konopka, Tomaz Kosmac[4], Krzysztof J. Kurzydlowski[1]

1. *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **3.** *Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland* **4.** *Josef Stefan Institute, Ljubljana, Slovenia, Slovenia*

Contact: jmichalski@inmat.pw.edu.pl

Fabrication of 3D interpenetrating ceramic-metal composites through the use of high pressure sintering of ceramic powder coated with Ni-P nanoparticles using electroless chemical plating is be reported. Electroless nickel plating method was used to produce nanometric layer of Ni-P spherical nanoparticles (size range approximately 20-50 nm) on the entire grain surface of the ceramic powder. The coated powders were consolidated via hot pressing (HP) process and pressureless sintering after cold isostatic pressing (CIP) process. SEM, TEM and XRD techniques were used to investigate the influence of the consolidation conditions (temperature and pressure) on the microstructure of composites and in particular the morphology of metallic Ni-P phase. The homogeneity of the interpenetrating network structure was measured via computer image analysis and compared with the results of resistance measurements. The results indicate that the use of electroless nickel plating and high pressure during the consolidation process allows for fabrication of uniform 3D interpenetrating continuous metal ceramic composites. The metal phase fully fills the space between alumina grains (open and closed pores). Depending on the consolidation conditions it is possible to obtain dense or porous materials where the metal phase could have nano or micro structure.

11:00 invited oral

Synthesis of diamond based superhard nanocomposites

T. Waldek Zerda

Texas Christian University (TCU), 3000 W. Bowie, Fort Worth TX 76133, United States

Contact: t.zerda@tcu.edu

Reactive sintering of diamond-SiC nanocomposites is possible by thorough mixing of diamond and silicon nanosize

powders or by ball milling mixtures of diamond and silicon powders. Properties of nanocomposites were characterized by scanning electron microscopy, XPS, Raman scattering and x-ray diffraction. Diamond-SiC nanocomposites had remarkably high fracture toughness and were significantly harder than the sintered pure nanocrystalline diamond compacts. Broadening of x-ray lines was explained in terms of plastic deformations and average sizes of diamond and silicon carbide crystals. Results were compared with those obtained for pure diamond and silicon carbide nanocrystals after high pressure, high temperature treatment. Graphitization of diamond was also discussed.

11:45 oral

Synthesis of diamond particle under Alkaline hydrothermal conditions

Nakamichi Yamasaki, Kazunori Yokosawa, Sergiy Korablov, Kazuyuki Tohji

Tohoku University, Sendai, Japan

Contact: yamasaki@mail.kankyo.tohoku.ac.jp

Small particles were produced from trichloroethane and hydrogenated diamond seeds mixture under alkaline hydrothermal conditions. These products were very hard, transparent and unsoluble in the acid mixture used for purification of commercial diamond. Only sp^3-bond (1332 cm^{-1}) was observed in Raman spectrum. It means that the product is a diamond. Despite the XRD pattern also signified pure diamond, it is difficult to divide products from seeds diamond. Therefore, cubic boron nitride was used as seeds. Similar hydrothermal products were observed by SEM photographs, and Raman spectrum of this sample signified that the residue composed of cubic boron nitride and diamond. Thus, it is without controversy proven fact that diamond was synthesized under high pressure alkaline hydrothermal conditions.

Chlorinated organic compound (trichloroethane) is dechlorinated in alkaline hydrothermal solution, and intermediate active carbon species are polymerized on the surface of seeds as diamond. Without diamond/cBN seeds the produced carbon was amorphous. Therefore, the structure of seeds surface supports the formation of diamond phase in area that is far from thermodynamical stability of diamond.

These experimental conditions were considerably lower (pressure up to 1 GPa and temperature 573 K) than the parameters of commercial synthesis of diamond. Commercial diamond is synthesized at over 6 GPa and over 1900 K, therefore, this diamond is very expensive. But, our experimental conditions are relatively inexpensive, and are easy to produce the diamond in large scale as well as large polycrystalline diamond.

12:00 oral

"Synthesis of nanopowders by electric discharge assisted mechanical milling".

Andrzej Calka, D Wexler

University of Wollongong, Wollongong NSW, Australia Faculty of Enginneering, University of Wollongong, Wollongong NSW 2522, Australia

Contact: andrzej_calka@uow.edu.au

Electric discharge assisted mechanical milling can be applied to the synthesis of a range of fine powder products, including nanocrystalline powder particles and individual nano-particles and nano-fragments. Variables include the starting powder sample size; electric arc parameters, such as arc length and arc voltage/current; mechanical milling parameters and gas atmosphere. Recently, the new processing variable of electric discharge frequency (Hz and kHz range) has been added to the technique. In this presentation we describe the results of the current experimental program underway, to investigate phase transformations and/or particle fragmentation during discharge milling, and to optimise processing parameters required for the synthesis of particular high surface area particles, nanostructural powders and nano-particles. We describe microstructural, morphological, and phase changes induced during the often competing processes of fragmentation into nano-particles, agglomeration of powder particles, and particle melting and/or sintering. Specific investigations include; (i) the modification of carbon base materials, where discharge milling under controlled conditions has resulted in the transformation of graphite into amorphous carbon, and the formation of graphite nanostructures (nanotubes and exotic nanofragments), (ii) the transformation of silicon and boron into nanofragments by discharge milling and (iii) phase transformations and nanoparticle formation induced in inorganic oxides, including the transformation of hematite into nanostructural magnetite, and finally into nanoparticles of iron.

Phase transformations

Friday afternoon, 9 September, 14:00
Main Building, Small Hall (Mała aula)
Gennady A. Salishchev presides

14:00 invited oral

Surface stress, the lattice parameter of small crystallites and its response to pressure

Joerg Weissmueller, Jürgen Markmann

Forschungszentrum Karlsruhe, Institut für Nanotechnologie, Herrmann-von-Helmholtz-Platz 1, Karlsruhe 76344, Germany Universität des Saarlandes, Technische Physik, Saarbrücken, Germany

Contact: Joerg.Weissmueller@int.fzk.de

Nanometer-sized particles or polycrystalline solids with a nanometer grain size will usually exhibit a lattice parameter which differs from that of the same substance in coarse-grained form. This observation testifies to the fact that considerable forces, originating from the surfaces or grain boundaries, act on the crystal lattice. In fact, pressures of several GPa can be induced in this way, and experiments on nano-scale solids show millimeter-scale movements as a response to changes in the surface-induced pressure. Intriguingly, it is found that the pressure can be tensile as well as compressive, depending on the material and the state of its surface. One can derive rigorous and general results relating the surface-induced pressure to the surface forces. An important - and sometimes neglected - consequence of these considerations is that the cases of fluids and solids require separate treatment: in fuids, the Young-Laplace equation relates the jump in the pressure across a surface to the mean curvature and the surface tension, whereas in solids there are two equations, both involving the surface stress tensor: one for the local jump in the tangential component of the stress, and a second for the average of the full stress tensor over the microstructure. The second result also provides a relation between the pressure and a scalar surface stress, which is readily applied to the analysis of lattice parameter data. While surfaces are often considered as two-dimensional objects, recent experiments suggest - counterintuitively - that it is relevant to allow for a surface to undergo deformation in the third direction, that of its normal. This leads to a pressure-dependence of the surface stress, and to an apparent size-dependence of the compressibility of nanoparticles when subjected to an external pressure, which does not imply that the lattice becomes softer or more compliant at small size.

14:30 invited oral

Phase transformations in pearlitic steels induced by severe plastic deformation.

Yulia Ivanisenko[1], Ian MacLaren[2], Xavier Sauvage[4], Ruslan Z. Valiev[3], Hans-Jörg Fecht[5]

1. *Forschungszentrum Karlsruhe, Institut für Nanotechnologie, Herrmann-von-Helmholtz-Platz 1, Karlsruhe 76344, Germany* **2.** *University of Glasgow, Department of Physics and Astronomy, Glasgow, United Kingdom* **3.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, Ufa 450000, Russian Federation* **4.** *University of Rouen, Institute of Material Research, Saint-Etienne du Rouvray, France* **5.** *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany*

Contact: ivanissenko@int.fzk.de

An overview of the number of unusual phase transformations taking place in nanocrystalline pearlitic steels in conditions of the severe deformation, i.e. combination of high pressure and strong shear strains will be given.

Strain induced cementite dissolution is a well-documented phenomenon taking place at cold plastic deformation of pearlitic steels. Recently new results which can shed an additional light on mechanisms of this process were obtained thanks to 3D AP and HRTEM investigations of pearlitic steel following the high pressure torsion (HPT). It was shown that the process of cementite decomposition starts with depleting of carbides with carbon, which indicates that deviation of chemical composition of cementite from stoichiometric one is the main reason for thermodynamic destabilisation of cementite at plastic deformation. Important results were obtained concerning the distribution of released carbon atoms in ferrite. It was experimentally confirmed that carbon segregates to dislocations and grain boundaries of nanocrystalline ferrite.

Another unusual phase transformation taking place in nanocrystalline peartitic steel during room temperature HPT is a stress induced \alfa\-->γ\

transformation, something which never occurs at conventional deformation of coarse grained iron and carbon steels. This was concluded to have occurred due to a reverse martensitic transformation. Atomistic mechanism and thermodynamic of the transformation, as well as issues related with stability of reverted austenite will be discussed.

15:00 oral

Kinetic and thermodynamic factors leading to dissolution of cementite in pearlitic steel subjected to severe plastic deformation under pressure

Witold Lojkowski[2], Yulia Ivanisenko[1], Hans-Jörg Fecht[1,3]

1. *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **3.** *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany*

Contact: wl@unipress.waw.pl

Mechanical alloying encompasses many different processes: high pressure torsion, mechanical milling, ECAP (Equi-Channel Angular Processing), shot penning, hydrostatic extrusion, and also friction processes taking for example place on the surface of railway rails. Besides structure refinement to the nano-scale these processes in many cases lead to phase transformations. For instance in pearlitic steels, they lead to dissolution of the cementite phase. In this paper we review shortly the thermodynamic and kinetic factors governing this process in particular and the formation of new phases during mechanical alloying and in general. It is shown how phase equilibrium is locally changed by high strains resulting from elastic incompatibility and nonequilibrium structures of the interfaces between precipitates and matrix. In addition, it is shown that mechanically induced flow of material can lead

to accelerated dissolution kinetics. This process may be seen as wear at the interface between the precipitate and the surrounding it matrix.

15:15 oral

Application of diamond anvil cell techniques for studying nanostructure formation in bulk materials directly during severe plastic deformation

Alexey N. Babushkin[1], Artemiy A. Popov[2], Ivan V. Sukhanov[1]

1. *Ural State University, Department of Physics, Lenin Av., 51, Ekaterinburg 620083, Russian Federation* **2.** *Ural State Technical University (UPI), Ekaterinburg, Russian Federation*

Contact: alexey.babushkin@usu.ru

We have applied diamond anvil cell technique to study the formation of new states in metals, alloys and nanostructured materials directly during severe plastic deformations (pressure range 15 - 50 GPa, room temperature).

Well-known, that at compression of a material in diamond anvils, the sample get by not less than three steps of deformation. At small stress deformation has elastic character. Growth of pressure conducts to plastic deformation of a sample and change of its geometrical sizes. At exceeding by pressure of some limit owing to the actions of forces of friction locking a material in the field of compression, the sample appears in new area of elastic deformations which top limit is limited to durability of a material of anvils. That is at compression of a sample in diamond anvils it is exposed severe plastic deformations.

One of problems of researches at high plastic deformations is revealing the transformations proceeding in a sample, is direct during deformation. Use of techniques of diamond anvils allows fixing transformations into a material directly during deformation. As sensitive features we use thermoelectromotive force and arbitrary heat transfer.

We report results of the investigation of the beta titanium alloy Ti-15-3. For the first time it is revealed that in the beta titanium alloy Ti-15-3 there are probable irreversible structural transformations at ~ 30 and ~ 42 GPa. For the first time the possibility of studying time dynamics of structural transformations in titanium alloys at room temperatures is shown. One of the probable reasons of irreversible changes can be formation in an alloy a nanoscale structure.

This work is supported in part by CRDF BRHE under grant EK-005-X1

Roadmapping workshop “Nanometals for SMEs with eh time perspective 2015”

Friday afternoon, 9 September, 15:30
Main Building, Small Hall (Mała aula)
Witold Lojkowski presides

15:50 oral

Current trends in the development of metallic nanomaterials: results of a study carried out within the EC project Nanoroad SME

Witold Lojkowski[1], Małgorzata Chmielecka[1], Agnieszka Daniszewska, Mariusz Kulczyk[1,2], Dmytro Kuzmenko[1]

1. *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **2.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland*

Contact: wl@unipress.waw.pl

The purpose of this project is to identify metallic nanomaterials that could be of great importance for Small and medium Enterprises in the near future. To reach that goal, we performed a patent and literature research to identify the nanostructured metals that are the subject of most extensive research in the recent years. We also interviewed experts in that field. In this talk we will present the results of this study, indicating the advantages of the new nano-materials and the main barriers for their applications. We will also show problems with categorization of such materials.

The hottest research topic in that respect is synthesis and application of metallic nanoparticles, first of all silver nanoparticles. However there is a great potential also in the application of nanostructured metals produced by heavy plastic deformation. The presentation will be followed by a "Roadmapping workshop" where we will try order the nanostructured metals and their technologies according to the expected commercialization chances.

The project is carried out with support of the EC project "Nanoroad SME"

16:20 oral

Workshop on Nanomaterials displayed in Technology Roadmaps

Oliver Schwandner

Fraunhofer Institute for Manufacturing Engineering and Automation (FhG IPA), Nobelstrasse 12, Stuttgart 70569, Germany

Contact: oms@ipa.fhg.de

The European funded project Nanoroad SME (www.nanoroad.net) aims to develop Technology Roadmaps in the domain of nanomaterials. With the help of these Roadmaps SME from different industrial sectors such as Automotive and Aerospace, Health/Medical and Energy will be able to adjust their capacities and concentrate on future Nano-trends. Knowledge from more than ten leading institutes and other facilities across Europe will be bundled in a

database. The information will be filtered and displayed according to the special needs of companies.

Nanometals is one selected material field within the project. Together with you as experts in this field coming from a research or an industry background a workshop will be held within the framework of the E-MRS Fall Meeting in Warszaw.

Prof. Witold Lojkowski of the Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS) will be chairing this workshop and will present the results of our survey on these nanomaterials based on the last five years EU research, patents search and a study of scientific literature. With your help and expertise we would like to complete the future view on possible applications and developments in this material field.

Saturday, 10 September

Modelling of structure and deformation behaviour of nanocrystalline materials

Saturday morning, 10 September, 10:00
Faculty of Material Science, Aula (room 212)
Maria Richert presides

10:30 invited oral

Modeling of Mechanical Properties of Nanocrystalline Ceramics

Izabela A. Szlufarska

University of Wisconsin at Madison, Department of Materials Science and Engineering, 1509 University Avenue, Madison, WI 53706, United States

Contact: izabela@engr.wisc.edu

Multimillion-atom molecular dynamics simulations have been performed on parallel computers to study atomistic mechanisms of nanoindentation of nanocrystalline ceramic thin films, understanding of which is critical for the design and fabrication of nanocrystalline materials with enhanced mechanical properties. We have implemented a sintering scheme involving high pressure and high temperature to create a realistic two-phase microstructure with crystalline grains and a disordered intergranular phase. We have found that the increased volume fraction of highly disordered intergranular films manifests itself in novel deformation mechanisms as compared to nanometals. Simulation results on nanocrystalline silicon carbide show that the interplay between grain rotation, cooperative grain motion, sliding at grain boundaries and intergranular deformation combine to produce a unique load-displacement response. We predict a crossover from continuous cooperative grain response to discrete intra-grain plasticity at a critical depth that is a fraction of the grain size.

11:30 invited oral

Deformation of Nanocrystaline Materials: Do we need computer simulations to understand it?

Herbert D. Gleiter

Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany

Contact: herbertgleiter@hotmail.com

Today, computer simulations are a frequently used method to model e.g. atomic processes in materials. The aim of this talk will be to point out the potential as well as the limitations of this approach by considering computer simulations of the deformation processes in nanocrystalline metals. The results of simulations of deformation processes will be critically compared with recent experimental results, pointing out under which circumstances computer simulations are likely to yield new insight.

12:10 invited oral

Synergies between simulations and experiments in nanocrystalline metals

Helena Van Swygenhoven

ASQ division- Materials and Reasearch, Paul Scherrer Institute (ASQ-PSI), PSI-Villigen, Villigen 5232, Switzerland

Contact: helena.vs@psi.ch

Atomistic simulations have provided unprecedented insight into the structural and mechanical properties of nanocrystalline materials, highlighting the role of the non-equilibrium grain boundary structure in both inter- and intra-grain deformation processes. One of the most important results is the capability of the nanosized grain boundary to act as source and sink for dislocations, in other words a deformation mechanism that does not leave behind dislocation debris. The dislocation activity suggested by molecular dynamics for four different nc-fcc metals, Al, Cu, Ni and Au are discussed in terms of the inherent restrictions and caveats of the simulation technique and in terms of material properties such as the generalized stacking fault energy curves. Details about the nucleation and propagation mechanism and their relation to grain boundary structures and grain boundary misorientations will be presented.

Simulations should however be taken as a guidance for experiments and therefore have to be validated. In the second part of this talk, several experimental investigations will be presented that are designed to validate the results of the simulations, such as stress relaxation testing at different temperatures and a new type of in-situ X-ray diffraction experiment recently developed at the Swiss Light Source. This in-situ experiment allows following in a time-resolved way the peak shift and the peak broadening during deformation. Measure-

ments show that for electrodeposited Ni with a mean grain size of 30nm, peak broadening is reversible upon unloading, demonstrating the absence of remaining dislocation debris. However in ultra-fine grained Ni synthesized by High Pressure Torsion the peak broadening is irreversible, similar to what is observed for coarse grained metals and is to be expected when a dislocation network is built up during deformation.

12:50 invited oral

Science to Business: Nanotronics

Andreas Gutsch

Degussa AG, Creavis Technologies & Innovation, Paul-Baumann-Straße 1, Marl 45772, Germany

Contact: andreas.gutsch@degussa.com

Innovative solution systems for electronics applications in collaboration with universities and industrial cooperation partners could be developed using a new business concept, called ‘Science to Business’.

It is based on integrating a close network between the fundamental research at the universities and the industrial application and marketing expertise. As it can significantly reduce the time from invention to market-ready product, this approach has clear competitive advantages. For this reason, Degussa AG, Düsseldorf, the world's leader in specialty chemicals, opened its new Science-to-Business Center Nanotronics at the Marl Chemical Park in April 2005.

Scientists from universities and research institutes, from small- and medium-sized enterprises, and from Degussa will all work in close cooperation in the Science-to-Business Center. The Center will also temporarily integrate the employees of universities and research institutes, thereby combining excellent scientific expertise with industrial project management and the infrastructure of an advanced chemical site. Furthermore, the Science-to-Business Center will strengthen practical scientific education, since the acquired know-how will flow directly back to the participating universities and research institutes.

Degussa is focusing on "Nanotronics" for its first Science-to-Business Center, the close interaction between nanotechnology and electronics. The Science-to-Business Center will primarily develop electronic systems and elements whose core components consist of new types of electrically functional nanomaterials and nano-coatings. Some of the fields of application include printable electronics, low cost displays, flexible solar cells and mobile energy systems.

Part II. Satellite events

Laser Ceramic Symposium

Welcome

The scope of the First Laser Ceramic Symposium includes methods of the fabrication and properties of ceramics prepared from nanopowders. Currently, the ceramics fabricated from nanopowders are extensively investigated and proposed to be used in the field of optoelectronics and modern quantum electronics as novel lasing and scintillating materials. Particularly, ceramics prepared from rare-earth and transition ions doped oxides are the most promising materials for high power laser devices. The aim of this Symposium is to present and discuss the recent advances in development and research on nanocrystalline ceramics characterized by high optical quality. The topics will include both fabrication of sinterable nanopowders and ceramics, and their laser, optical and other physical properties. Also other emerging applications of such ceramics will be covered by the Symposium, like scintillators, LEDs etc.

Organisers

Alexander A. Kaminskii (co-chair), Institute of Crystallography, RAS, Moscow

Wiesław Stręk (co-chair), Institute of Low Temperature and Structure Research, PAS, Wrocław

Ken-ichi Ueda (co-chair), Institute for Laser Science, University of Electro-Communications, Japan

Witold Łojkowski, Institute of High Pressure Physics, PAS, Warsaw

Dariusz Hreniak, Institute of Low Temperature and Structure Research, PAS, Wrocław

Proceedings

After peer-review procedure and the final acceptance submitted papers will be published in a special issue of Optical Materials.

Acknowledgements

The Symposium will be supported by:

- International Scientific Network: "Nano and Micro Technology", acronym: NaMic, Polish Ministry of Science and Information Society Technologies

- Center of Excellence PRENABIO at the Institute of High Pressure Physics, European Comission Contract

Programme

Monday, 5 September

Highly transparent laser ceramics
Monday afternoon, 5 September, 14:00
Main Building, room 206
Wiesław Stręk presides

14:00 invited oral

Current status of laser ceramic research: technology, characterization and apllication

Ken-ichi Ueda

Institute for Laser Science, University of Electro-Communications, 1-5-1 chofugaoka Chofu, Tokyo 182-8585, Japan

Contact: ueda@ils.uec.ac.jp

14:45 invited oral

Nonlinear properties of crystalline laser ceramics based on cubic oxides

Alexander A. Kaminskii

Institute of Crystallography, Russian Academy of Sciences, Moscow, Russian Federation

Contact: kaminalex@mail.ru

15:15 oral

Recent progress in the synthesis of transparent laser ceramics

Yoël Rabinovitch[1,2], Daniel Tétard[1], Jean-François Baumard[1], Jean-Claude De Miscault[2]

1. *Laboratoire Sciences des procédés céramiques et des traitements de surface, 123, avenue albert thomas, Limoges 87000, France* **2.** *Compagnie industrielle des Lasers, Orléans, France*

Recent work has successfully demonstrated the potential application of highly transparent ceramics as amplifying medium in laser cavity. Major results have been obtained by Japanese teams such as high output laser power, various doping agents (Nd, Yb), etc.

Nevertheless, considering the strategic weight of this technology, it is believed that a local industrialization of laser ceramics should be done in Europe. To realize this purpose, we have developed a new process to synthesize highly transparent Nd :YAG ceramics in large sizes using high purity available raw materials. The last results obtained open the way to promising laser ceramics under various shapes.

Monday Poster Session

Monday afternoon, 5 September, 15:50

15:50 poster LCS-1

Index of Refraction for Nano-Ceramic Materials

Artur Bednarkiewicz, Piotr Mazur, Dariusz Hreniak, Wiesław Stręk

Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland

Contact: A.Bednarkiewicz@int.pan.wroc.pl

The index of refraction for Nd^{3+} and Eu^{3+} doped $Y_3Al_5O_{12}$ ceramics was measured and compared with that of bulk material. The Sellmeier equation $n^2(\lambda[\mu m])=A+B\lambda^2/(\lambda^2-C)$ was found to have A, B and C equal to 1, 1.44156, 0.06716 and 1, 1.36429, 0.05849 for Nd:YAG and Eu:YAG respectively, in comparison to single Nd:YAG crystal 1, 2.2779 and 0.01142. At 614nm where Eu^{3+}: $^5D_0$ --> $^7F_2$ appears the index of refraction for ceramics is n=1.617 whereas for YAG single crystal it is n=1.830. At 909nm where Nd^{3+}: $^4F_{3/2}$ --> $^4I_{9/2}$ appears the index of refraction for ceramics is n=1.604 whereas for single YAG crystal it is n=1.819.

The Einstein's coefficient A_i for electric-dipole transitions depends on index of refraction according to $\chi^{ABS}_{ED} = (n^2+2)2 / 9n$ and according to $\chi^{EMI}_{ED} = n(n^2+2)2 / 9$ for absorption and emission respectively. Thus one may expect the absorption coefficient to be reduced in nanocrystals based materials in comparison to bulk one. The lower index of refraction measured for ceramics, should reduce the absorption coefficient down to ~84% of the value for both Nd^{3+} and Eu^{3+} doped YAG single crystal. One may also expect the lifetimes to be longer in nanocrystals based materials in comparison to bulk one. The measured indexes of refraction for Eu^{3+} or Nd^{3+} doped ceramics reduces the χ_{ED} for luminescence down to ~66% of the value for YAG single crystal and may lead to 1.5 times increased decay constants. These effects could be one of explanations of the long lifetimes observed by us in Nd^{3+} and Eu^{3+} doped nanopowders. The measured lifetimes for 0.5 and 1%Nd:YAG nanopowders exceeded 400μs (485 and 420μs respectively), however for the ceramics the lifetimes decreased down to 230 and 210 μs respectively. We thus suppose the reabsorption of radiation could also play a role in highly scattering medium like powders.

15:50 poster LCS-2

Excited states dynamics in the ternary ytterbium halides

Joanna Cybinska[1], Janina Legendziewicz[1], Georges Boulon2, Gerd Meyer[3]

1. *Wroclaw University, Faculty of Chemistry, 14 F. Joliot-Curie, Wrocław 50-383, Poland* **2.** *Laboratoire de Physico-Chimie des Matériaux Luminescents, UMR 5620 du CNRS (UCBL), 10 rue A.M. Ampere, Villeurbanne 69622, France* **3.** *Institut für Anorganische Chemie, Universität zu Köln, Greinstrasse 6, Köln D-50939, Germany*

Contact: jcybinska@poczta.fm

Interest in Yb^{3+}-doped materials has been increasing with advances of high-performance in GaAs laser diode which are ideally suited for pumping the Yb^{3+} ion between 905 and 980 nm [1]. Yb^{3+} is indeed the most promising ion that can be used in a non-Nd^{3+} laser in the near-IR.

The purpose of this work is to present and discuss spectroscopic properties of Yb^{3+} in K_2LaX_5 : (X= Cl, Br, I) hosts grown by the Bridgman method according to the procedure described in [2, 3]. The structural characterization was made for the obtained crystals.

Spectroscopic measurements useful for laser applications, high resolution absorption, emission spectra as well as decay time at 293 K, 77 K and 4 K were investigated. The interpretation of Stark levels were done by using Raman and IR absorption spectra to select electronic and vibronic lines. Upconversion process by non-radiative energy transfer to RE unexpected impurity ions as well as Yb^{3+} pairing and clustering are expected in these hosts.

[1] T.Y. Fan, IEEE J. Quantum Electron 29 (1993) 1457.

[2] G. Meyer, Th. Staffel, Z. anorg. Allg.Chem. 532 (1986) 31

[3] J. Legendziewicz, J. Cybinska, G. Meyer, Optical Materials, 24 (2003) 197

15:50 poster LCS-3

Optical study of PLZT films on Al_2O_3 substrate

Maria J. Gomes, Anatoli Khodorov

University of Minho (UM), Campus de Gualtar, Braga 4710057, Portugal

Contact: mjesus@fisica.uminho.pt

In the composition range near the morphotropic phase boundary La-modified lead zirconate titanate (PLZT) ferroelectrics show the enhanced ferroelectric, electro-mechanic and electro-optic properties that make the material to be very interest for application in electronic and electro-optic devices. In this work, we prepared PLZT films of composition 9/65/35 on Al_2O_3 substrate with sol-gel method and studied their optical

and structure properties. The dielectric function was calculated by fitting the transmittance and reflectance spectra of the films measured in the wave-length region 220-2600 nm. The optical study revealed appearing of anomalous behaviour of dielectric function below the absorption edge when the perovskite ferroelectric phase was achieved during crystallization. The shift of absorption edge as well as evolution of band tailing with film thickness were studied. The correlation between optical and structure properties of the films was carried out and discussed.

This work was supported by FCT (grant SFRH/BPD/11675/2002).

15:50 poster LCS-4

Analysis of the optical properties of Pr^{3+}-Yb^{3+} ions in codoped transparent oxyfluoride glass ceramics. Application for 1300 nm optical amplifier

felipe manuel rosa gonzalez, Inocencio R. Martín, Fernando Lahoz, José M. Cáceres, Víctor Lavín, Jose M. Calvilla-Quintero, Sara González-Pérez

Universidad La Laguna, La Laguna 38206, Spain

Contact: frosag@ull.es

A study of the optical properties of Pr^{3+}-Yb^{3+} ions in oxyfluoride glass and glass ceramics has been carried out. The transparent oxyfluoride glass ceramics were obtained just by thermal treatment of the glass ($30SiO_2+15Al_2O_3+29CdF_2+22PbF_2+(4-x-y)YF_3+xPrF_3+yYbF_3$) at 470º C for 36 h to precipitate nanocrystallites without loss of transparency. An important feature of these materials, relevant to devices such lasers and amplifiers, is that rare-earth dopants preferentially segregate into the fluoride nanocrystals phase when cerammed, and thus reside low phonon energy sites.

The cross section emissions at 1300 nm corresponding to the $1G_4$->$3H_5$ transition of Pr^{3+} ions have been obtained and compared for the glass and glass ceramics samples. Due to the ceramming process the linewidth of this transition is reduced about 12 % and the spontaneous emission probability is increased about 18 %. Consequently, an increase of the emission stimulated cross section by a factor 1.4 is obtained.

This interesting transition coming from the $^1G_4$ level of Pr^{3+} ions could be efficiently excited from energy transfer process from Yb^{3+} ions. So, the energy transfer processes among Yb^{3+} and Pr^{3+} ions under excitation at 980 nm have bee extensively studied. From the fits of the fluorescence decays of Yb^{3+} ions it could be concluded that the most of rare earth ions are placed in the nanocrystalline environment and participate in an efficient energy transfer process from Yb^{3+} to Pr^{3+} ions.

15:50 poster LCS-5

Synthesis, characterization and optical properties of lutetium double phosphates doped by ytterbium ions

Malgorzata Guzik[1], Janina Legendziewicz[1], Wladyslawa Szuszkiewicz[2], Jorma Hölsä[3]

1. *Wroclaw University, Faculty of Chemistry, 14 F. Joliot-Curie, Wrocław 50-383, Poland* **2.** *Institute of Chemistry, Economy Academia, 118/120 Komandorska, PL-53-345 Wrocław, Poland, Poland* **3.** *University of Turku, Department of Chemistry, FIN-20014 Turku, Finland, Poland*

Contact: goguzik@poczta.fm

Compounds containing Yb3+ ions have potential application as laser devices and they have some advantages over the Nd3+ ion as laser emitting center. There is no cross-relaxation process thus no concentration quenching. In addition, the intense and broad Yb3+ absorption bands are well suited for IR laser diode pumping.

This paper is devoted to the comparison of two different procedures to obtain the new ytterbium doped alkali metal lutetium double phosphates and their characterization. The mentioned compounds were synthesized by solid state reaction and M3PO4 (where M= Na, Rb) and lanthanide phosphates were obtained by two different methods. Thermogravimetry, FT-infrared and absorption spectroscopy were performed to characterize the samples. X-ray diffraction was used to identify different phases. Crystal structure of lutetium double phosphates rubidium salt is isostructural with hexagonal yttrium double phosphates, while lutetium double phosphate sodium salts crystallize in structure a little different that of orthorombic yttrium compound. The absorption, reflection, emission and excitation spectra were measured at room temperature, 77 and 4K in the IR and UV ranges and were compared with the results obtained for samples of the same type containing yttrium instead lutetium in the same matrices. The luminescent spectra of double phosphates were measured with different excitation line, also by using synchrotron radiation. The low temperature absorption spectra in the region of 2F7/2 -> 2F5/2 transition together with the IR spectra were used to assign the vibronic components. Energy levels diagram for excited states in both types of the crystals were proposed.

Moreover, the emission and excitation spectra using synchrotron radiation were investigated and the origin of the emission were described.

15:50 poster LCS-6

Yttrium oxide nanoparticles preparation

Tasoltan T. Basiev, Vasilii A. Konyushkin, Pavel P. Fedorov, Sergei V. Kouznetsov, Sergei V. Lavristchev, Vjatcheslav V. Osiko, Alexey M. Samartzev, Valerii V. Voronov

General Physics Institute, Vavilov Str. 38, Moscow 117942, Russian Federation

Yttrium oxide crystallized in cubic system and characterized by high thermoconductivity and transparence in the IR-range. Y_2O_3 nanoparticles are precursors for laser ceramic fabrication.

Nanoparticles of yttrium oxide formation were investigated using soft chemistry methods, i.e. yttrium oxide dissolution in acids (nitric, hydrochloric) followed by pH-regulation by the ammonium solution titration or by the urea hydrolysis under heating. The obtained gel-like precipitations of the precursors lost their volatile components during drying and heating. The follows chemical reactions took place in the nitrate processes:

$Y_2OH)_5NO_3 \times nH_2O$ --- $Y_2(OH)_5NO_3 + nH_2O$ (n=2-3),

$Y_2(OH)_5NO_3$ --- $Y_4O_5(NO_3)_2 + 5H_2O$,

$Y_4O_5(NO_3)_2$ --- $2Y_2O_3 + 2NO + 1.5O_2$,

at the first step finished at 150 ^oC, the second – at 340 ^oC and the third – at 500-550 ^oC.

The coherent dispersion scattering regions sizes of Y_2O_3 particles are 30-40, 56-72 and 90-130 nm after thermal treatment at 500, 750, 900 ^oC respectively and are the function of first deposition conditions (composition and solutions electronic microscopy data provides the strong multilevel agglomeration particles formation. Y_2O_3 particles form follows the precursors particles form. The addition of the surface active compounds to the initial solution allows changing the deposited particles form.

The ultra-sonic effect (frequency 22 kHz, power 100 W) during precursor precipitation in the cavitations zone leads to plates formation (size 1×1 μm and thickness approximately 20 nm). Without surface active compounds the plates grow into the agglomerates with the linear size approximately 1 μm. Thermal treatment of precursors allows fabrication quasi 2D particles of cubic yttrium oxide.

The conditions of globular 13-130 nm Y_2O_3 nanoparticles fabrication were determinated.

15:50 poster LCS-7

Preparation and sintering of nanometric oxides and YAG powders derived from nitrates by precipitation and co-precipitatin method

Zdzislaw M. Librant[1], Helena Węglarz[1], Tadeusz Łukasiewicz[1], Krzysztof Haberko[2]

1. *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warszawa 01-919, Poland* **2.** *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

Contact: Zdzislaw.Librant@itme.edu.pl

Ammonium hydrogen carbonate was applied as precipitant for production of yttrium and neodymium oxides and YAG nanopowders from solution of nitrates. Loose agglomerates of oxide powders derived from proper precursors have crystallite sizes well below 50 nm. Powders were calcined up to 1100^oC, milled, mixed in proper proportions for solid state reaction synthesis, and densified by isostatic pressing at the pressure of 200 MPa. For the solid state reaction synthesis of YAG and 1at.%Nd:YAG, commercial Taimei alumina was applied. X-ray diffraction analysis have shown at the low calcination temperatures, presence of YAP phase, which disapear at 1000^{o}C and only YAG phase remains. Powder compacts were sintered in vacuum (10^{-6}Tr) within the temperature range of 1700-1780^oC and holding time up to 60 hours. Several samples were prepared with 0,5% of tetraethyl orthosilicate (TEOS) as sintering aid. After grinding and polishing of sample surfaces good translucency and even transparency was found. As a quality indicator of microstructure of produced samples, optical transmission spectra in the infrared wavelength region was measured in relation to YAG and Nd:YAG single crystals. Only 70% of transmittance of YAG single crystal at the upper wave region was achieved. Positive effects of added sintering aids (TEOS) on final microstructure was not found.

15:50 poster LCS-8

Co-precipitation synthesis of Nd:YAG nanopowders: the effect of joint thermal and mechanical treatment

Maria Luisa Saladino[1], Eugenio Caponetti[1], Filomena Serra2, Stefano Enzo[2]

1. *Università di Palermo (UNIPA), Viale delle Scienze, Pallermo 90128, Italy* **2.** *Università di Sassari, Sassari, Italy*

Contact: saladinoluisa@libero.it

Nanopowders of Yttrium Aluminium Garnet (YAG) doped with neodymium ions were obtained by the co-precipitation method from the reaction of aluminium and yttrium nitrate

and neodymium oxide with ammonia giving samples of nominal stoichiometry $Nd_X:Y_{(3-X)}Al_5O_{12}$ (where X = 0, 0.02, 0.05, 0.10, 0.19, 0.38, 0.72). After washing and drying, the hydroxide precursors were heat treated for different times and temperature. Differential Thermal Analysis curves (DTA) from room temperature up to 1000 C show the presence of exothermal events accompanying phase transformation phenomena. X-ray diffraction (XRD) investigations conducted with a high-resolution powder diffractometer on the specimens calcined at high-temperature show that the samples are single phase garnet except for the highest concentration of Nd, where a minor hexagonal $YAlO_3$ phase may also be present. Precise determination of the cubic garnet lattice parameters as a function of the Neodymium content according to the Rietveld method shows a parabolic increase from the value of 12.022 (±2) Å for $X_{Nd} = 0$ up to 12.128 (±2) Å for $X_{Nd} = 0.72$. The line broadening analysis after correction for instrumental factors indicates that the average crystallite size is in the range 50-70 nm. Results will be presented and discussed concerning the role of a mechanical treatment on the amorphous powder in order to skip kinetically the formation of metastable phases and to favor precipitation of the garnet with consistent neodymium content.

15:50 poster LCS-9

New routes for the synthesis of Al-doped ZnO transparent nanomaterials

Madalina L. Popescu[1], Robert R. Piticescu[1], Roxana M. Piticescu[1], Claude J. Monty[2], Marioara Abrudeanu[3], Adrian M. Motoc[1]

1. *Institute for Non-ferrous and Rare Metals, 102 Biruintei Blvd., Pantelimon 73957, Romania* **2.** *Procédés,Matériaux et Energie Solaire (PROMES), Avenue Professeur Trombe, Font-Romeu 66120, France* **3.** *University of Pitesti, Pitesti, Romania*

Contact: mpopescu@imnr.ro

Nanocrystalline ceramic and composite materials present a set of highly improved and novel mechanical, electronic, optical, catalytic or bioactive properties over the traditional materials. The implementation and utilisation of these new materials is strongly dependent on the microstructure and surface nanochemistry characteristics investigation and modelling. New processes for the synthesis and sintering are required to be developed to control and optimize the chemical composition, component distribution, crystalline and grain sizes;

In our paper we present some results on the synthesis of Al – doped nanomaterials via two routes: hydrothermal synthesis and vaporisation – condensation in a solar furnace.

The influence of the synthesis parameters on the nanopowders characteristics is discussed and results are compared with thermodynamic predictive calculations.

The evolution of mean crystallite sizes of powders obtained in the two synthesis routes is modelled.

Pressed pellets made from the hydrothermal synthesized powders were used to deposit thin films by RF sputtering. Some characteristic properties of these films are finally presented.

The authors acknowledge the support received in the frame of an ECO-NET Project managed by EGIDE –France, FP6 Infrastructure Project SOLFACE and a NATO fellowship.

15:50 poster LCS-10

Synthesis and interaction of lanthanide oxide nanoparticles of with amorphous SiO_2.

Małgorzata Małecka, Leszek Kępiński

Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland

Contact: M.Malecka@int.pan.wroc.pl

Synthesis of inorganic material nanoparticles by water in oil (W/O) microemulsion method enables to produce small particles with narrow size distribution. Using this technique, we obtained well-defined 4-5 nm nanoparticles of precursors of lanthanide oxides (La_2O_3, CeO_2, Pr_6O_{11}, Tr_4O_7). Interactions of lanthanide oxide nanoparticles with SiO_2 were studied on example of Pr_6O_{11}-SiO_2 system by transmission electron microscopy (TEM) and X-ray diffraction (XRD). Two different preparation methods were used. In the first, precursor of praseodymium oxide was supported on amorphous SiO_2 by impregnation, followed by calcination in oxygen at 400^0C, until Pr_6O_{11}-SiO_2 system (10 % mol Pr_6O_{11} – 90 % mol SiO_2) was formed. As revealed by TEM the sample contained small Pr_6O_{11} nanocrystals (4-5 nm) partly aggregated into clusters (10-100 nm). In the second case nanoparticles of praseodymium oxide precursor were immobilized into silica during synthesis, which prevented their agglomeration. As a result of calcination in oxygen at 400^0C small amorphous particles of Pr^{+3} oxide (4-5 nm) uniformly embedded in SiO_2 were obtained. Solid state reactions and formation of Pr silicates in both systems were explored in oxidizing atmosphere at temperatures 800^0C, 1000^0C and 1100^0C.

15:50 poster LCS-11

Preparation and electrical properties of the BaTiO3 nanoceramics

Robert Pązik[1], Dariusz Hreniak[1], Wiesław Stręk[1], Grzegorz Paściak[2], Jacek Chmielowiec[2], Witold Lojkowski

1. *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland* **2.** *Electrotechnical Institute, Division of Electrotechnology and Materials Science (IEL), M. Skłodowskiej-Curie 55/61, Wrocław, Poland*

Contact: R.Pazik@int.pan.wroc.pl

The new method of fabrication of the $BaTiO_3$ nanoceramics obtained from the sol-gel derived nano-powders is reported. The energy band gap was estimated to be 1.26eV from the absorption reflectance spectra. Preliminary studies of the size effect on the electrical conductivity of the nanoceramics measured at different temperature are presented. Interesting phenomena was found, with increase of temperature at the range of 350-500^{o}C the conductivity increases with the simultaneous decreasing of the grain size of nanoceramic sample. Above 500^{o}C generally the grain size effect on the conductivity is negligible but samples with bigger grains have got slightly higher conductivity. All samples shown semiconducting properties.

15:50 poster LCS-12

Preparation and characterisation by XRD and TEM of ZnO based magnetic helionanos

Claude J. Monty[1], Jules KOUAM, Adrian M. Motoc[2], Francois SIBIEUDE, Benjamin MARTINEZ, Felipe SANDIUMENGE, Lluis BALCELLS, Jordi Arbiol[3,4]

1. *CNRS laboratoire Procedes, Materiaux et Energie solaire (PROMES), BP5 Odeillo, Font Romeu 66120, France* **2.** *Institute for Non-ferrous and Rare Metals, 102 Biruintei Blvd., Pantelimon 73957, Romania* **3.** *Electronics Department, University of Barcelona, C\ Martí i Franqu?s, Barcelona 08028, Spain* **4.** *Serveis Cientificotècnics, University of Barcelona (SCT-UB), Lluis Sole i Sabaris, 1-3, Barcelona 08028, Spain*

Contact: monty@promes.cnrs.fr

M (M=Co, Mn, Fe) doped ZnO nanophases (0D-NANOS and 1D-NANOS) have been prepared by vaporisation-condensation in a solar reactor (helionanos formed by solar PVD) from targets of known initial composition.

The heliophases and initial powders have been characterized by X-Ray Diffraction analysis (XRD) and Transmission Electron Microscopy (TEM).

A detailed analysis of the XRD spectrum has been performed to estimate :

1 - diffraction peaks position shifts and shape anomalies

2 - the average size of coherency domains (nanophases average grain size)

The first point led to discuss eventual variations of the lattice parameter as a function of composition, presence of stresses and distribution of grain sizes.

The second point led to discuss the influence of parameters such as the initial composition and the pressure inside the reactor during the solar PVD process.

Magnetic properties of 0D-NANOS-Zn1-xCoxO have been studied : these nanomaterials exhibit an original behaviour which will be discussed.

15:50 poster LCS-13

Electrical Conductivity of Cubic/Tetragonal Phase Stabilized Nanocrystalline La2O3 doped ZrO2

Paramasivam Thangadurai, Sinnanadar Ramasamy, Arumugam Chandra Bose

University of Madras, Department of Nuclear Physics, Guindy Campus, Chennai 600025, India

La_2O_3 doped nanocrystalline zirconia (ZrO_2) was prepared by chemical co-precipitation method for the 3, 5, 8, 10, 15, 20 and 30 mol% concentrations of La_2O_3 . X-ray diffraction (XRD) showed that the as-synthesized samples contain only monoclinic phase. Annealing at higher temperatures lead to tetragonal/cubic structural phase stabilization. Sintering the samples at temperature 1173 K stabilized the tetragonal and cubic phases. Grain sizes were found to lie between 10 and 13 nm. Electrical conductivity studies were performed on the cubic/tetragonal phase stabilized La_2O_3 -ZrO_2 by complex impedance spectroscopy. The conductivity increases up to the dopant concentration 10 mol% and then decreases with further increase in La2O3 concentration. Initial increase in conductivity is correlated to the stabilization of the cubic phase and the subsequent decrease in the conductivity with the dopant content is interpreted on the basis of the oxygen-ion movement model. Electrical conductivity has contributions from grain and grain boundary regions. But the grain boundary conductivity is slightly higher than the corresponding grain conductivity. Higher grain boundary conductivity shows higher diffusion coefficient for the atoms on the surface of the ZrO_2 grains. The possible mechanism of the oxygen ion conduction in the La_2O_3 stabilized zirconia (LSZ) is reported.

Tuesday, 6 September

Highly transparent laser ceramics (part 2)
Tuesday morning, 6 September, 9:00
Main Building, room 206
Alexander A. Kaminskii presides

9:00 invited oral

Ceramic Lasers of Normal and Composite Type

Akio Ikesue[1], Kunio Yoshida[2], Tomosumi Kamimura[2], Yan Lin Aung[1], Taira Takunori[3]

1. *Poly-Techno Co., Ltd., 2-4-1 Mutsuno Atsutaku, Nagoya 456-8587, Japan* **2.** *Osaka Institute of Technology, Osaka 535-8585, Japan* **3.** *Institute for Molecular Science (IMS), 38 Nishigonaka, Myodaiji, Okazaki 444-8585, Japan*

Contact: poly-ikesue@s5.dion.ne.jp

(Normal Type)

For the important four-level solid-state laser host, a YAG ($Y_3Al_5O_{12}$) single crystal, a small content of fluorescent Nd^{3+} ions are used as active species in the 1064 nm wavelength. In recent years, a Nd-doped YAG laser has been applied widely in various industrial capacities, including medical operation, metal processing, and other applications.

The present author demonstrated in 1994 that the effective laser oscillation was performed successfully for the first time in the world using polycrystalline Nd:YAG. The present work can be summarized as follows.

(1)High-quality Nd:YAG ceramics were fabricated successfully by simple solid-state reaction under vacuum sintering.

(2)Such properties of Nd:YAG ceramics as thermo-mechanical properties were nearly the same as those of Nd:YAG single crystal by Cz method.

(3)Optical loss of Nd:YAG ceramics was 0.9%cm^{-1}, as low as that of Nd:YAG single crystal.

(Composite Type)

We have developed successfully most advanced composite laser media which perfectly bonded both single crystal and polycrystalline ceramics. The bonding interfaces between single crystal and ceramics become almost "Stealth" or " Seam-less" conditions, because the interface has no interstice and migrates towards polycrystalline ceramics by sudden grain growth. The advanced composite was confirmed almost optically perfect bonding by interferometory.

To obtain higher laser function, the composite laser using single crystals was developed in 1998, however several problems existed in conventional composite because of unperfected bonding. The narrow interstices ($<\lambda$) partially exist in interface of conventional composite composed of single crystals, so it has some problems such as optical loss, thermal diffusion, lifetime etc. We challenge to fabricate new composite laser media having more improved bonding interface, which composed of polycrystalline ceramic and single crystal.

9:30 invited oral

Yb-doped laser ceramics and the laser oscillations

Kazunori Takaichi[1], Hideki Yagi[1,2], Masaki Tokurakawa[1], Akira Shirakawa[1], Ken-ichi Ueda[1], Shunsuke Hosokawa[2], Takagimi Yanagitani[2], Alexander A. Kaminskii[3]

1. *Institute for Laser Science, University of Electro-Communications, 1-5-1 chofugaoka Chofu, Tokyo 182-8585, Japan* **2.** *Takuma works Konoshima Chemical co. Ltd., 80 koda takuma-cho mitoyo-gun, Kagawa 769-1103, Japan* **3.** *Institute of Crystallography, Russian Academy of Sciences, Moscow, Russian Federation*

Contact: takaichi@ils.uec.ac.jp

Ytterbium-doped materials are very attractive for laser-diode-(LD-) pumped solid-state lasers. The Yb-doped materials have high quantum efficiency, no concentration quenching, and long lifetime. The broad absorption band in the near-IR region is suitable for pumping with a LD, and the broad emission band is able to generate tunable laser oscillations and ultrashort pulses. So far, many Yb-doped laser materials have been researched and are used for high power lasers and ultrafarst lasers.

Since 1998, it has been possible to fabricate highly transparent ceramics for solid-state lasers by a vacuum sintering technique and nanocrystalline technology. Our fabrication method is fundamentally different from any press methods, for example the cold isotropic press method and hot isotropic press methods, because no pressure is applied through the whole fabrication process. As a result, our ceramics have good homogeneity in terms of grain size and less internal strain. Additionally, this method is suitable for the fabrication of large sized products and production in large numbers.

We have been developing highly transparent Yb-doped $Y_3Al_5O_{12}$ and RE_2O_3 (RE = Y, Sc, and Lu) ceramics for solid-state lasers. From spectroscopic point of view, there is no difference between an Yb:$Y_3Al_5O_{12}$ single crystal and the ceramics. For a RE_2O_3, the ceramics also has same spectroscopic properties as the single crystals.

The LD-pumped Yb:$Y_3Al_5O_{12}$ and Yb:RE_2O_3 ceramic lasers have been demonstrated. For an Yb:$Y_3Al_5O_{12}$, a 940-nm LD was used for the excitation. In the cw Yb:RE_2O_3 lasers, they were pumped at the zero-phonon line (ZPL) of around 976-nm wavelength. ZPL pumping has a few advantages: larger absorption cross section, lower quantum defect. In a ZPL-pumped cw Yb:Y_2O_3 laser, the extraction efficiency of 72 % was obtained at 1078 nm. An LD-pumped cw mode-locked ceramic laser oscillation was carried out. The pulse duration of 430 fs was obtained.

10:00 oral

Highly transparent YAG ceramics

Hideki Yagi[1,2], Yanagitani Takagimi[1], Takaichi Kazunori[2], Ken-ichi Ueda[2]

1. *Takuma works Konoshima Chemical co. Ltd., 80 koda takuma-cho mitoyo-gun, Kagawa 769-1103, Japan* **2.** *Institute for Laser Science, University of Electro-Communications, 1-5-1 chofugaoka Chofu, Tokyo 182-8585, Japan*

Contact: yagi@konoshima.co.jp

The recent development of ceramic laser materials is probably the most important innovation in the field of laser material fabrication technology.

We developed highly transparent YAG ceramics by means of modern ceramic technique including the vacuum sintering and nanocrystalline technology. Scattering losses were greatly reduced in our laser ceramic, and the optical properties of the ceramics are even better than those of the single crystal YAG.

The fabrication method of ceramics involves the following. First, YAG oxide powder was fabricated by wet chemical reaction and calcinating. Next, the power was ball-milled with solvent, and milled slurry was put into a gypsum mold and dried to obtain a desired form. Finally, the materials were sintered in vacuum for several hours. After annealing, highly transparent YAG ceramics were obtained. As a result of this method, great progress has been made on the development of Nd:YAG ceramic lasers. The efficiency of the Nd:YAG ceramic laser has been shown to be comparable to or better than that of single crystals at 100 W level, and the CW laser output power reached up to 1.4 kW. The microstructure and optical properties of these ceramics will be discussed.

Moreover, we fabricated Cr4+:YAG, Er:YAG, Nd:Cr:YAG and Nd:YAG - YAG composite ceramics for saturable absorber and gain medium. The microstructure, optical properties and laser characteristic of these ceramics will be reported.

10:15 oral

Preparation and spectroscopic properties of LuAG:Tm and LuSAG: Tm

Yikun Liao, Danyu Jiang, Yaming Ji, Jianlin Shi

Shanghai Institute of Ceramics, Chinese Academy of Sciences, 1295 Dingxi Road, Shanghai 200050, China

Contact: liaoyikun@mail.sic.ac.cn

Tm-doped LuAG and LuSAG precursors were synthesized through a co-precipitation process, using ammonium hydrogen carbonate as precipitator. Single-phase cubic LuAG/Tm and LuSAG/Tm phosphors were obtained after calcinations at 1000°C and 1200°C, respectively. These powders could be easily sintered into corresponding transparent ceramic LuAG/Tm and LuSAG/Tm in H_2 atmosphere at 1850°C. The PL excitation and emission spectra were recorded for both phosphors and ceramics. All emission spectra (λ_e x = 355 nm) show blue emissions at 455 and 460 nm corresponding to the Tm^{3+} transitions $^1D_2 \rightarrow {}^3F_4$ and $^1G_4 \rightarrow {}^3H_6$.

Glass-ceramics lasers

Tuesday afternoon, 6 September, 14:00
Main Building, room 206
Ken-ichi Ueda presides

14:00 invited oral

Glass-ceramics and ceramics: new opportunities for laser materials

Michel Mortier[1], Amina Bensalah[1], Geraldine Dantelle[1], Gilles Patriarche[2], Daniel Vivien[1]

1. *Ecole Nationale Supérieure de chimie de Paris (ENSCP), 11 rue P. et M. Curie, Paris 75005, France* **2.** *Laboratoire de Photonique et de Nanostructures, CNRS Route de Nozay 91460 Marcoussis, France, France*

Contact: michel-mortier@enscp.fr

While rare-earth-doped crystals exhibit most of the optical and thermomechanical properties required to get high power laser beams, their maximum size is limited. Also, their maximum doping level is limited by the activator ions segregation undergone during the growth processes from the melt.

Glasses can be synthesised in various shape and without the same size limitation. However, their mechanical properties are not sufficient and their thermal conductivity does not allow enough heat evacuation during laser operation. Also, the maximum doping level is limited due to rare-earth (RE) clustering tendency. Moreover, the optical properties of the RE ions suffer from inhomogeneous broadening.

In this study, we have studied fluoride materials. With respect to the usual oxide type materials, a fluoride material can offer a low phonon environment favourable to enhance the radiative rate and quantum efficiency. The excited states lifetime are increased.

A first alternative route to single crystals has been investigated. This consists in using oxyfluoride glass ceramics in which RE ions benefit of a crystalline and fluoride environment. These materials are obtained using glass synthesis technique and thermally activated crystallisation of nanocrystallites thank to RE nucleating effect. With thermomechanical properties higher than glasses and crystal like optical properties, the glass ceramics are competitive materials with an easy shaping and fabrication in the air. Two approaches developed in the lab will be discussed, either PbF2 phase inside germanate glass and CaF2 phase inside silica glass.

Finally, transparent ceramics prepared from nanopowders are a logical outcome for high power laser materials. Many res-

ults have been recently obtained with YAG and RE sesquioxides activated with Nd and Yb. We will present here preliminary results in which a fluoride route is investigated using CaF2 as a model material for ceramming processes.

14:30 oral

Optical properties of rare earth doped oxyfluoride glass-ceramics

Fernando Lahoz, Inocencio R. Martín, Víctor Lavín, Ulises Rodríguez, José M. Cáceres, Fernando Rivera, Javier Plata, Sara González-Pérez

Universidad La Laguna, La Laguna 38206, Spain

Contact: flahoz@ull.es

Oxyfluoride glass-ceramics are obtained when a precise thermal treatment is applied to a precursor glass. This thermal treatment causes that fluoride nanocrystals precipitate in the vitreous matrix. X-ray diffraction measurements have been performed and reveal that two phases coexist in the glass-ceramic. One is vitreous and has an oxyfluoride composition and the second one is crystalline and provides a fluoride environment. In fact, the macroscopic properties of the glass-ceramic are characteristic of the aluminosilicate glassy phase except the optical properties of the rare-earth (RE) ions, which are characteristic of low-phonon energy fluoride crystals. The fluoride nanocrystals have low-phonon energy and, as a consequence, the probability of nonradiative relaxation processes is expected to be low, giving place to longer excited state lifetimes than in the oxyfluoride precursor glass. A very efficient IR to visible upconversion process is reported in Ho^{3+} doped glass-ceramics. Three strong emission bands in the primary color components, red, green, and blue give place to an intense white emission light, which is observed by the naked eye under IR excitation at 750nm. The excitation mechanism of this efficient upconversion emission is studied and it was found to be due to a photon avalanche process.

Finally, laser action in Nd^{3+} doped gass-ceramics has been studied. A relatively low pump threshold of about 18 mJ was detected. Losses due to UV and visible upconverted emission inside the laser cavity have been observed.

14:45 oral

Stimulated emission from cobalt-containing glass ceramics

Alexander M. Malyarevich[1], Yuri V. Volk[1], Konstantin V. Yumashev[1], Olga S. Dymshits[2], Alexander V. Shashkin[2], Alexander A. Zhilin[2]

1. *International Laser Center, Minsk, Belarus* **2.** *S.I. Vavilov State Optical Institute, St-Petersburg, Russian Federation*

Contact: malyar@ilc.by

Transparent glass ceramics have a wide traditional spectrum of applications. Possibility to dope these materials with rare-earth and transition-metal ions provides their perspectives as lasing media. Near-zero thermal expansion coefficient, high thermal stability and thermal conductivity together with flexibility of synthesis procedure make glass ceramics as a material of choice in comparison with crystalline media. Our paper presents stimulated emission properties of zinc- and magnesium- aluminosilicate glass ceramics doped with different concentrations of cobalt ions.

It is shown that the absorption and luminescence properties of these glass ceramics are defined mainly by tetrahedrally coordinated Co^{2+} ions located in $ZnAl_2O_4$ gahnite and $MgAl_2O_4$ spinel nanocrystals, respectively, formed in glass ceramic matrix during double-stage thermal treatment procedure.

The studied glass ceramics have broad and intensive luminescence band in visible due to $^4T_1(^4P) \rightarrow {}^4A_2$ transition of Co^{2+} ions. Luminescence decay time has considerable dependence on cobalt ions concentration and varies from 90 to 280 ns for 0.5 and 0.1 weight % of CoO in zinc-aluminosilicate glass ceramics, respectively. Magnesium-aluminosilicate glass ceramics shows less pronounced variation in luminescence decay time – from 25 to 40 ns for the same change of cobalt concentration.

Differential transmission spectra measurements after pump at 532 nm allow to register stimulated emission of cobalt ions in the range around 650-700 nm. Values of stimulated emission cross-sections in this spectral range were evaluated and for cobalt ions in zinc- aluminosilicate glass ceramics they are as high as 2-5 10^{-19} cm^2.

Perspectives of cobalt-doped glass ceramics as laser active media are analyzed taking into account various synthesis conditions of these materials.

15:00 oral

Concentration dependence of Ni^{2+} luminescence in MgO

Nina Mironova-Ulmane[1], Ilmo Sildos[3], Janis Grabis[2]

1. *Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia* **2.** *Riga Technical University, Miera 34, Riga, Latvia* **3.** *Institute of Physics, University of Tartu, Riia street 142, Tartu EE-2400, Latvia*

Contact: ulman@latnet.lv

It is known that nickel ions doped magnesium oxide MgO:Ni^{2+} is the excellent potential as tunable infrared laser in the 1300-1500 nm. However in order to optimize MgO:Ni for the best operation and evaluate its potential, it is necessary to have data on the luminescence as a function of temperature and concentration of nickel ions.

We present results of luminescence spectra of Ni^{2+} ions for single crystals MgO and ceramics preparation from nanosized

MgO:Ni. The single crystals MgO:Ni^{2+} in this investigation were grown by the chemical transport reaction methods and by melting. The ceramics MgO:Ni fabricated from nano-powders which received by evaporation of raw power in a radio-frequency plasma. The luminescence spectra with concentration of Ni c_{Ni} = 0,5%; 1% and 5 % single-crystals was studied in the range 460-1500 nm which includes all d-d electronic transition in ions Ni^{2+} at the temperatures from 10 to 300K. Luminescence spectra of MgO:Ni consist of three broad band. The band at 12500 cm^{-1} is related to the $^1E_2(D) \rightarrow {}^3A_{2g}(F)$ electrical dipole transition, the band at 18000 cm^{-1} is related to the $^1T_{2g}(D) \rightarrow {}^3A_{2g}(F)$ electrical dipole transition whereas the band at 8000 cm^{-1} is related to the $^3T_{2g}(D) \rightarrow {}^3A_{2g}(F)$ magnetically dipole transition. The intensity of luminescence of green and red band decreases when the temperature increases. Besides, the integral intensity of band in infrared luminescence depends strongly on the c_{Ni} and excitation wavelength. In the region of the concentration where we do not observe infrared luminescence of exchange-coupled of Ni^{2+} pairs, the infrared luminescence intensity does not depend on measurement temperature. At higher c_{Ni} can see luminescence of Ni^{2+} pairs, but where the number of the exchange-coupled pairs is relatively small, the infrared luminescence intensity of the zero-phone line for the single Ni ions remains constant below 70K and decrease exponentially above 100 K.

15:15 oral

Characterization of radiation defects in transparent oxides

Gianguido Baldinozzi, David Simeone, Michael Dollé, Dominique Gosset

Ecole Centrale Paris, SPMS, UMR CNRS-ECP (ECP), Grande voie des vignes, Chatenay-Malabry 92295, France CEA Saclay, DMN/SRMA (CEA), CE Saclay, Gif-sur-Yvette 91191, France

Contact: baldinozzi@spms.ecp.fr

It is now well established that irradiation can trigger displacive and order disorder phase transitions in metals and alloys. Recent works have clearly shown that these transitions can also exist in irradiated ceramics. The elementary mechanisms leading to these structural modifications will be discussed in two large gap materials: pure monoclinic zirconia and $ZnAl_2O_4$ spinel. The irradiation can strongly affect their structures and their microstructures, leading to a nanostructuration. This can present potential useful consequences for tailoring their physical properties.

Nanostructured ceramics and powders

Tuesday afternoon, 6 September, 15:50
Main Building, room 206
Witold Lojkowski presides

15:50 invited oral

Ceramic micro solid-state lasers

Takunori Taira

Institute for Molecular Science (IMS), 38 Nishigonaka, Myodaiji, Okazaki 444-8585, Japan

Contact: taira@ims.ac.jp

Transparent ceramic laser materials, which were fabricated by a solid-state reaction method, offer numerous advantages over melt growth methods, including faster production times, solid solution allowing the fabrication of multi-phase transition materials, highly homogeneous materials and the ability to engineer profiles and structures before sintering. Much progress has been made in improving the optical quality from ceramics, as well as exploring new materials [1-3]. Successfully developed concentrated Nd:YAG ceramics was opened the way for drastic heat reduction by directly upper laser level pumping [4, 5]. Recently, the advantages of Yb:YAG materials for high-power and highly efficiency lasers are well recognized [6-8]. In this research, we'd like to discuss about high power edge-pumped single crystal Yb:YAG/ceramic YAG composite microchip laser and ultrafast Yb:$Y_3ScAl_4O_{12}$ ceramic laser. The author would like to thank Dr. M. Tsunekane, Dr. J. Saikawa, Dr. T. Dasucalu, Prof. I. Shoji, Dr. N. Pavel, Dr. Y. Sato, Dr. V. Lupei, Dr. A. Ikesue, and Prof. K. Yoshida. This work was supported in part by the CREATE-JST Fukui Association for Industry and Technology contract and the Grant-in-Aid for Scientific Research A No.15206073 from the ministry of Education, Culture, Sports, Science, and Technology of Japan.

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16:20 invited oral

Optical investigations of RE-doped YAG nanoceramics

Wiesław Stręk[1], Dariusz Hreniak[1], Piotr Mazur[1], Artur Bednarkiewicz[1], Witold Lojkowski[2], Robert Fedyk[2]

1. *Polish Academy of Sciences, Institute of Low Temperature and Structure Research, Okolna 2, Wrocław 50-422, Poland* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: W.Strek@int.pan.wroc.pl

The optical properties of rare-earth (Eu^{3+}, Tb^{3+}, Nd^{3+}) doped $Y_3Al_5O_{12}$ nanoceramics characterized by different grain sizes in the range 25 – 100 nm and different concentration of rare-earth ions were investigated. The method of fabrication and structural investigations are presented. The influence of doping level on the luminescence spectra and structural properties has been observed. The emission spectra were measured as a function of excitation power. The mechanisms leading to the hot emission in Nd^{3+}:YAG nanoceramics are discussed.

16:40 oral

Yttria stabilized zirconia nanocrystals luminescence

Donats Millers[1], Larisa Grigorjeva[1], Krisjanis Smits[1], Witold Lojkowski[3], Agnieszka Opalińska[2]

1. *Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia* **2.** *Polish Academy of Sciences, High Pressure Research Center (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: dmillers@latnet.lv

The Yttria stabilized Zirconia nanocrystals were synthezed by microwave driven hidrothermal method. The luminescance spectra, decay kinetics and luminescence intensity dependence on temperature were measured for different size nanocrystals. Steady-state luminescence was excited by X-rays, time-resolved luminescence study was prepared under pulsed laser beam (4,66eV, 2ns) and under pulsed electron beam (270 keV, 10ns) excitation. It is shown the luminescence spectra are similar for all nanocrystal sizes, whereas the intensity of luminescence is nanocrystal size dependent. The origin of this dependence was interaction between electronic excitations and intrinsic defects. The luminescence decay kinetics (both, under laser and electron beam excitation) reveals the fast initial decay followed by slower one. The decay of luminescence did not obey a simple exponential or hyperbolic rule, indicating more complicated recombination process. The time–resolved spectra and decay kinetics analysis confirms more than one kind of recombination centers were involved. Possible models of luminiscence centres and recombination processes are discussed. The luminescence intensity and decay time dependence on temperature gaves an evidence that two processes – the luminescence center destruction and non – radiative electron transitions – were responsible for luminescence quenching observed. The relative contribution of these processes in luminescence quenching depends on nanocrystal size.

16:55 oral

Time-resolved luminescence of nanostructured ZnO

Larisa Grigorjeva[1], Donats Millers[1], Aleksej Kuzmin, Robert Kalendarev, Witold Lojkowski[2], Anita Tomaszewska-Grzęda

1. *Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia* **2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland*

Contact: lgrig@latnet.lv

ZnO has been attractive material for potential applications, for example, lasers and optoelectronic devices. The excitonic luminescence with decay times in ps range will be observed up to room temperature. It is expected high quantum yield for fast luminescence due to exciton quantum size effects on nanostructured ZnO. The ZnO nanocrystals and thin films were synthesized and characterized by FTIR, x-ray diffraction and SEM methods. The luminescence spectra and decay kinetics were studied under pulsed laser (266 nm, 2 ns) and pulse electron beam (10 ns 250 keV) excitation. Nanocrystalline powders with controlled grain size 10-40 nm was prepared by microwave driven hydrothermal process using different chemical reactions. Nanostructured ZnO thin films were prepared using i) dc magnetron sputtering technique in mixed Ar/O2 atmosphere from metallic zinc target on Si substrates. After deposition, the samples were heat treated at about 1000C for one hour. Thus obtained films had thickness of about 500 nm and were not preferentially oriented, according to XRD; ii) on Si substrates using atmospheric pressure chemical vapor deposition (APCVD) starting from a mixture of ZnO and graphite powders. Such films had larger thickness of about few microns and were oriented along c-axis in (002) direction.

The photoluminescence spectra of nanopowders and both types of films consist of broad defect band at 1.9-2.4 eV region and excitonic luminescence band at about 3.2 eV. The decay kinetics had complex structure with more than two exponent.

17:10 oral

Nd:Y_2O_3 nanopowders for laser ceramics

Yurii Kopylov, Valery Kravchenko, Anatolii Komarov, Zoya Lebadeva, Vladimir Shemet

Fryazino Institute of radioengineering and electronics Russian Academy of Sciences (FIRE RAS), Vvedenskogo sq., Fryazino 141190, Russian Federation

Contact: ylk215@ire216.msk.su

Y_2O_3 is a promising material for laser oxide ceramics. The Japan team obtained recently Y_2O_3:Nd and Y_2O_3:Yb laser ceramics using chlorides water solutions for preparation of starting oxide nanopowders. We used different salt solutions to obtain the powders, doped with Nd. Mixtures of Y and Nd nitrates, oxalates and carbonates were used. Two ways were applied to adjust the solution pH values to Y hydroxide deposition (around 8): 1) addition of urea to the salt solution with subsequent heating to 80-100^oC; 2) addition of ammonium hydroxide solution to the salts' solution. Grain agglomeration, their forms, sizes and surface area depend critically on the experimental conditions. Grain sizes in the range 10-200 nm and surface areas in the range 10-140 m^2/g were obtained. Y_2O_3 powder were obtained by heating hydroxide precursors in air at temperatures 900-1200^oC, and the oxide grain surface area was in the range 50-100m^2/g. Plates type grains were formed when carbonates' solutions were used, whereas nitrates and citrates gave more uniform three dimensional grains. Pellets were prepared from the nanopowders using both uniaxial pressure around 2000 kg/m^2 and pouring of water-nanopowder mixture into gypsum moulds. Vacuum sintering of the pellets at 1700-1800^oC gave transparent ceramic samples. Results of electron microscopy of the samples, Nd absorption and emission spectra are presented.

17:25 oral

The Luminescent Investigation of Laser Nanomaterials Nd: Y_2O_3 and Nd :YAG.

Vladimir I. Solomonov, Alfiya V. Rasuleva, Vladimir V. Osipov, Maxim G. Ivanov

Institute of Electrophysics of RAS (IEP), Ekaterinburg, Russian Federation

Contact: plasma@iep.uran.ru

The pulsed cathodoluminescence of the initial material and products of synthesis of the laser nanoceramic (Y_2O_3: Nd and YAG: Nd) was investigated. The technology of synthesis was as follows. The initial coarse powder of both oxide and garnet had the average particle size 100 mm and purity 99.99 %. It was pressed into the pellets-targets and annealed at 1200^oC. The nanopowder with the particle size about 20 nm was obtained by laser evaporation of the targets. The nanopowders were calcinated at 900^oC. The nanoceramics were received by the magnetic – pulsed compaction of nanopowders with the further sintering at temperature up to 1700^oC. The luminescence of the initial material and products was investigated in the visible spectral region (350-750 nm) with the help of the setup CLAVI [1]. The irradiation by the electron beam of the powders and pellets took place at the room temperature in air. The duration of the e-beam was 1.5 ns. The exposure dose for one pulse was about 1 kGy.

In the spectrum of the initial powder Y_2O_3 the distinct impurity bands of the uncontrolled rare-earth ions with the quantity less than 10-2 atomic percent were found. In the pellets prepared for the laser evaporation from the powder mixture of Y_2O_3 with Nd_2O_3 content 1% mol. and 3.6 % mol, the amplification of the impurity bands was observed. The band broadening of ions Nd^{3+} is dominated in the both nanopowders and nanoceramics.

The pulsed cathodoluminescence method was shown to be an effective one to control the change of the properties of the materials at all the stage of the synthesis.

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17:40 oral

Synthesis of Al-doped ZnO nanomaterials with controlled luminescence properties

Robert R. Piticescu[1], Roxana M. Piticescu[1], Claude J. Monty[2], Witold Lojkowski[3], Larisa Grigorjeva[4]

1. *Institute for Non-ferrous and Rare Metals, 102 Biruintei Blvd., Pantelimon 73957, Romania* **2.** *Procédés,Matériaux et Energie Solaire (PROMES), Avenue Professeur Trombe, Font-Romeu 66120, France* **3.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland* **4.** *Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia*

Contact: rpiticescu@imnr.ro

Nanocrystalline ceramic and composite materials present a set of highly improved and novel mechanical, electronic, optical, catalytic or bioactive properties over the traditional materials. The implementation and utilisation of these new materials is strongly dependant on the microstructure and surface nanochemistry characteristics investigation and modelling. New processes for the synthesis and sintering are required to be developed to control and optimize the chemical composition, component distribution, crystalline and grain sizes.

Due to the combination of interesting piezoelectric, electric, optical and thermal properties ZnO-doped nanomaterials are of high interest for multifunctional applications in gas sensors, ultrasonic oscillators or transparent electrodes in sol-

ar cells. In our paper we present the results on the synthesis of zinc oxide powders with different Al content by two different procedures: hydrothermal route and evaporation-condensation in a solar furnace.

The influence of the synthesis parameters on the chemical and microstructural characteristics of nanophases synthesized in the two methods has been systematically studied using chemical methods, XRD, BET, picnometric density, XPS, SEM and TEM. The first results on the obtaining of bulk ceramics by sintering and thin films by electrophoresis deposition are also presented.

The authors acknowledge the financial support in the frame of the ECO-NET project “Modelling and verification of electrochemical and electronic properties of multifunctional nanomaterials” financed by EGIDE-FRANCE.

Round Table Discussion

Welcome

The Round Table Discussion is a special Session within the framework of the EMRS 2005 Fall Meeting in which ideas for the future of European Universities will be discussed. There will be two invited talks given by Marcel van der Voorde, from Max-Planck Institute, Germany, and Paul W. Gilgen, from EMPA, Switzerland. The invited audience will consist of influential Conference participants, Rectors and Deans of Polish Universities and others with potential impact on the future development of Universities. The aims of the meeting are:

- to present new ideas for the European Universities,
- to obtain feedback from participants,
- to prepare a document for wide distribution,
- to start a movement to reform the Universities so that Europe becomes really competitive in the world.

The Round Table Session will be held on Thursday 8th September, 2005 from 9.00 to 11.30 in the Senate Room (Room Nr 124) in the Main Building of Warsaw University of Technology, Plac Politechniki, 1, Warsaw

Programme

9.00 - 9.10 Opening

9.10 - 9.30 Marcel van der Voorde: *Future Vision on Europe's Universities*

9.30 - 9.50 Paul W. Gilgen: *From science to market - a key process for the technology-based renewal of economies*

9.50 - 10.20 Coffee Break*(Room 123)*

10.20 - 11.30 Brain storming session

- The main points to be discussed will be displayed on the screen.
- There should by no more than 3 main points, 20 min for each.
- The audience will be asked to state opinions about the highlighted issues.
- Brainstorming techniques are recommended:
- Flipcharts will be used and aspects noted
- Amendments will be made on the flipcharts
- Closing remarks

Programme

Thursday, 8 September

Round Table Discussion
Thursday morning, 8 September, 9:00
Witold Lojkowski presides

9:00 invited oral

Future Visions on Europe's University - Based Research

Marcel Van de Voorde

University of Technology Delft, Rotterdamseweg 137, Delft 2628 AL, Netherlands

Contact: vandevoorde@mf.mpg.de

Universities need to be restructured: "the life in an ivory tower belongs to the past"! The University structures and operation from the last centuries do not comply anymore with the needs imposed by the consumer, technology, and society of the 21st century.

Visions for the University of the Future will be described and essential changes in structures and management are highlighted, on the basis of the following themes:

i) The educational system in Europe still relies on two paradigms: the classification of the different sciences according to Auguste Comte and the hierarchy of lectures/tutorials/practices in our pedagogic practices. The real need is for young students/scientists to know who to move forward when faced with a real-world problem (on a scientific- and/or technical topic), they have never met before, on a real-world-time-scale, and a real world budget. The rigid faculty structures need to be dismantled with the creation of small- and medium sized clusters for education and University Centres of Excellence for Research.

ii) The trans-disciplinarily principle will occupy an important role in future Universities, Research Centres and Industry. In future, post graduates have to operate in a trans-disciplinary environment, unknown in the past! The inter/ trans-disciplinarily of to day is the "specialty" of to morrow!

The University leaders should recognize that 1) teaching is largely for students who will not become future academics, and for careers that don't exist yet and 2) research and teaching must be linked so that students will be ready for the new ideas of knowledge that research will provide.

The new millennium will see us enter an era of novelties in medicine, transport, society ….. With new tools, new insights and understanding, and a developing convergence of the disciplines of physics, chemistry, materials science, etc we may dear to dream of novel and superior products and systems that were, until the 21ste century, the stuff of science fiction.

iii) The traditional models of Governance and Management of Universities are no longer appropriate; we need to consider new models.

The managing autonomy at institutional level and the leader-

ship for entrepreneurial and innovative Higher Education Institutions (Rector + Senate) must be linked to accountability and control of use of public funds tostakeholders (Executive Council).

In future, the actors should be taken at 1) EU-level, 2) National level and 3) Institutional level.

There is a pressing need for drafting "Principles of Governance and Management" that provide a European reference underlying the academic and social responsibilities of Universities.

iv) The University will become the "thinking tank" of the future European Industry; the academic research should be curiosity driven. To be excellent, competitive and wealth creative on an international scale requires:" strong science" – "strong industry" -and "well connected". This will result in frontier research, mobility of scientists and technologists and effective knowledge sharing but it requires the creation of new partnerships between Universities and Industry, to develop innovations and discoveries. Multiple models for collaboration and technology transfer will be presented.

v) The role of the European Union – 7th (2006 – 2013) Framework Programme + European Research Council, National- and Regional Governments in this important matter will also be mentioned.

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