
The European Materials Conference



E-MRS 2003
FALL MEETING

European Materials Research Society
Fall Meeting

Warsaw University of Technology
Warsaw (Poland)

15th – 19th September, 2003

Conference Organisers:
Polish Materials Society
High Pressure Research Center, Polish Academy of Sciences
Institute of Physics, Polish Academy of Sciences
European Materials Research Society, E-MRS

THE E-MRS FALL MEETING CONFERENCE CHAIRMEN:

K. Kurzydłowski,	Warsaw University of Technology
W. Łojkowski,	High Pressure Research Center, Polish Academy of Sciences
A. Mycielski,	Institute of Physics, Polish Academy of Sciences
P. Siffert,	European Materials Research Society, France

Welcome

We are pleased to welcome delegates to the 2003 Fall Meeting of the European Materials Research Society, E-MRS, being held at Warsaw University of Technology.

This event is only the second time E-MRS has held a Fall Meeting, in addition to the Annual Spring Meeting, held each June in Strasbourg, France. The 2003 Spring Meeting marked the twentieth anniversary of the Society which now holds the largest materials science conference in Europe. The 2003 Spring Meeting consisted of 16 parallel symposia and was attended by nearly 2000 participants.

The inaugural E-MRS Fall Meeting in 2002 was held in three locations: Moscow, on Software Development for Process and Materials Design; Warsaw, Interfacial Effects and Novel Properties of Nanomaterials; and Zakopane, on Solid Solutions of the II-VI Compounds, Growth, Characterization and Applications.

The great success of the 2002 Meetings, both in scientific quality and organization, led to the E-MRS deciding to hold this year's Fall Meeting in Warsaw. The organizers have followed the theme 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 2003 Fall Meeting will endeavour to continue these themes and further enhance the contacts between scientists from the European Union Candidate Countries and other Eastern European Countries with those from Western Europe, North America and Asia.






The 2003 Fall Meeting comprises seven parallel symposia and 3 Plenary Sessions which are being held in the morning on the first three days of the Conference 15th, 16th and 17th September. During the opening Plenary Session the Professor J. Czocharlski Gold Medal will be presented to Professor Dr. Walter Heywang.

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.

ACKNOWLEDGEMENTS

THE E-MRS FALL MEETING 2003

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	Polish Ministry of Science and Information Technology
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	<p>High Pressure Research Center "UNIPRESS" Polish Academy of Sciences, SPUB MSN</p>
	EUROPEAN MATERIALS RESEARCH SOCIETY
	Institute of Physics, Polish Academy of Sciences
	Faculty of Materials Science, WUT

Local Organizing Committee

W. Łojkowski (1)
M. Lewandowska (2)
R. Pielaszek (1)
L. Perchuć (1)
A. Opalińska (1)
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- (1) High Pressure Research Centre, Unipress, Polish Academy of Sciences,
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Conference Secretariat

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Conference Location

Warsaw University of Technology
Plac Politechniki 1
00-661 Warsaw
Poland

CONFERENCE PROGRAMME

SYMPOSIUM A, Room 219:

Novel Wide Bandgap Materials for Optoelectronic and Electronic Applications

SYMPOSIUM B, Room 134:

Development of Methods for Characterizing the Microstructure of Novel Materials

SYMPOSIUM C, Room 213:

5th International Workshop on Molecular Beam Epitaxy and Vapour Phase Epitaxy Growth Physics and Technology

SYMPOSIUM D, Room 144:

Colossal Magnetoresistance – New Materials and New Ideas

SYMPOSIUM E, Room 206:

Materials for Medical Applications

SYMPOSIUM F, Small Hall (Mała Aula):

Functional Nanostructured Materials for Optoelectronics and other Applications

SYMPOSIUM G, Room 208:

Bulk and Graded Nanometals

PRE-SCHOOLS:

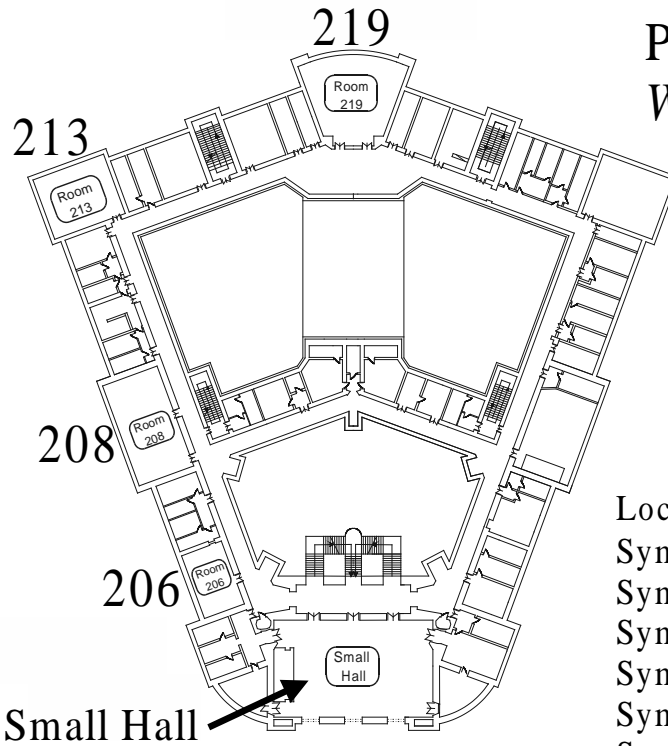
▷ **Syposium B Pre-School** Development of methods for Characterisation of the Microstructure of Novel Materials

▷ **Symposium F&G Pre-School** Nanocrystalline Materials: Fabrication Structure, Modelling and Applications

▷ **Vth High Pressure School on High Pressure Methods for Biotechnology**

Location of lecture rooms

Politechnika Warszawska
 Warsaw University of
 Technology



Second floor

First (ground) floor

Locations:

Symposium A: 2nd floor, Room 219

Symposium B: 1st floor, Room 134

Symposium C: 2nd floor, Room 213

Symposium D: 1st floor, Room 144

Symposium E: 2nd floor, Room 206

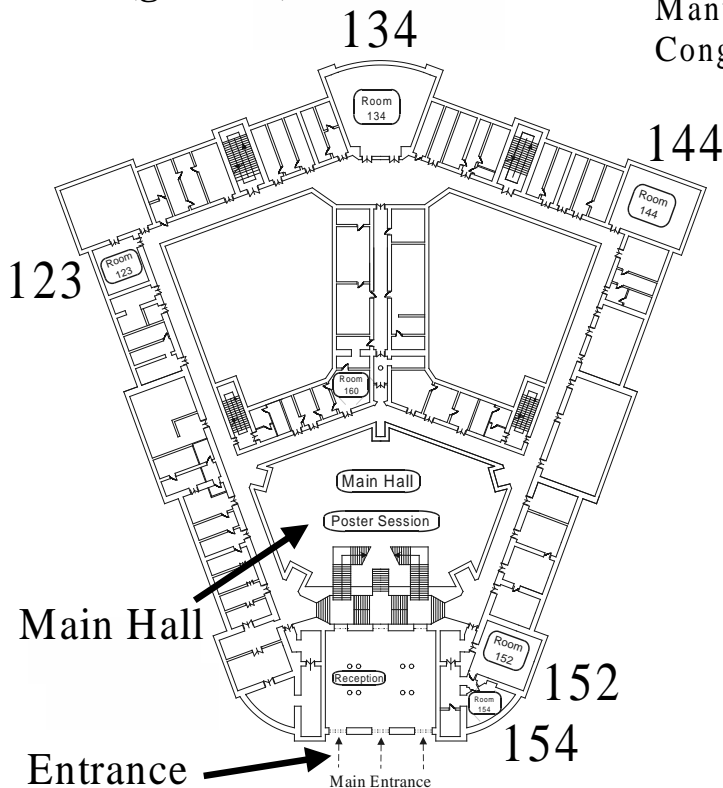
Symposium F: 2nd floor, Small Hall

Symposium G: 2nd floor, Room 208

Poster Session: Main Hall (Duza aula)

Manuscript Office: 1st floor, Room 152

Congressor Office: 1st floor, Room 154



Introduction

The E-MRS 2003 Fall Meeting will consist of the three Plenary Sessions, 1 Joint Session, on Wednesday morning 17th September, and 7 separate scientific symposia which will run concurrently.

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

The Opening Ceremony will take place on Monday 15th September at 8:30 am. During the first Plenary Session the ceremony to award the Professor Jan Czochralski Gold Medal to Professor Doctor Walter Heywang, of Siemens AG, will take place. Professor Heywang is a pioneer in the development and application of the Czochralski Method of Crystal Growth on which so much of the semiconductor industry is based.

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 doctor's 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 referee's reviews) in the Polish and English languages. The journal is devoted to materials problems, and is particularly concerned with metallic materials, various forms of plastics, ceramics and composites related to their application, and also promotes modern developments in surface engineering. During the last two years, the PMS was the co-organiser of the following conferences:

1. Symposium on "Intermetallic Alloys", Warsaw, 2000
2. The XVIth Physical Metallurgy and Materials Science Conference ADVANCED MATERIALS & TECHNOLOGIES, Jurata, Poland, 2001
3. The XIth International Conference on Electron Microscopy of Solids, Krynica, Poland, 2002
4. Z.S. Basiński Symposium on Advances in Materials Plasticity, Kraków, Poland, 2002
5. "Obróbka powierzchniowa" (Surface treatments), Kule-Częstochowa, 2002

6. The 2nd International Conference on Environmental Degradation of Engineering Materials, Bordeaux, France, 2003
7. Seminar on “Problems in designing research and teaching projects in the field of materials engineering”. This seminar is organised annually at various research centres in Poland with the aim of exchanging individual experiences, to coordinate results and establish mutual contacts between the institutions and research groups. These efforts seek to modernise and adapt their research and teaching activities to the modern requirements in the light of advances made in science and technology throughout the world and to benefit from the possibilities available in Poland.

The Evolution 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 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, justifying 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 the official approval for the establishment of a technical university provided that the funds needed to build and furnish it, an additional 2.5 million roubles, would be collected 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 the retreat, most of the movable property of the Polytechnical Institute (i.e., libraries, experimental and laboratory equipment, etc.) was taken to Russia.

In the interwar period, a few thousand engineers graduated from the Polytechnical Institute, 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.

Prof. Jan Czochralski (1885-1953) - Poland's most famous materials scientist.

Prof. Jan Czochralski was born on 23 October 1885 in Kcynia, a small town in Western Poland, at that time under Prussian administration. He attended the elementary and secondary schools in Kcynia.

At the beginning of the 20th century Jan Czochralski moved to Berlin where, from 1907 -1917, he worked as an engineer - in Allgemeine Elektrizitäts Gesellschaft (AEG): At the same time he received further chemical and metallurgical education (Charlottenburg Technical University) and education in fine arts (Berlin University). In 1917 Jan Czochralski organized the well-equipped research laboratory of Metallbank und Metallurgische Gesellschaft syndicate in Frankfurt am Main of which he was its director until 1928. He was one of the founders and the president of the German Scientific Society of Metal Sciences and was elected the honorable member of International Society of Material Sciences in London. He was a consultant of the great metallurgical firms (Boffors; Skoda, Creusot). In 1929 he moved to Warsaw, being invited by the Polish President, Prof. I. Mościcki to become ordinary professor of metallurgy and metal research in the Chemistry Department at the Technical University in Warsaw. He was honored with the title of the doctor honoris causa of Technical University of Warsaw. He organized the Institute of Metallurgy and Metal Sciences of the Warsaw Technical University and Metallurgical Department of the Chemical Research Institute. Both research laboratories have played a significant role in the progress of Polish industry and metal sciences. The Second World War stopped the research work of Prof. Czochralski. During this time he organized the wide support for his co-workers and for the people of Polish culture and the Polish underground army. After the war Prof. J. Czochralski returned to the native Kcynia to run the small drug firm BION. He died in Poznań hospital on 22nd April 1953 and was buried in the family tomb in Kcynia.

Professor Jan Czochralski carried out his scientific research, which concerned a wide range of subjects (light alloys, metal corrosion and inclusions, diagrams of recrystallization, anisotropy of mechanical properties of monocrystals, X-ray - methods in metal sciences). His studies on the velocity of metal crystallization and the method of monocrystal growth (especially in August 1916) made him famous and gave him a distinguished position in science. The Czochralski method of pulling crystals and of the melts still in used today as the simplest and commonly used crystal growth methods in the semiconductor industry.

Prof. J. Czochralski was also interested in literature, music and painting. All his activities were mainly the results of his self-education which prof. Czochralski, one of the most famous Polish scientists, regarded as the best way to success. The name of Jan Czochralski is one of the best known names encountered in the world's material science literature.

In 1999 a primary school in Kcynia was named after Prof. Jan Czochralski. In 2000 the Foundation for Materials Science Development, Poland established Prof. Jan Czochralski Gold Medal for achievements in material science and asked E-MRS for cooperation to promote J.Czochralski international achievements as well as the Medal.

Professor Jan Czochralski Gold Medal

The Foundation for Materials Science Development, based in Kraków, Poland, took the decision in 1998 to establish and fund the Professor Jan Czochralski Gold Medal. The purpose being to promote and recognize achievements in the field of materials science and to enhance the name and lifetime work of Czochralski, a distinguished scientist and inventor, and often regarded as the founder of the modern materials science.

Nominations are received by the Foundation from various interested bodies and the recipient is proposed by the Jury and confirmed by the Foundation's Executive Committee. The agreement between the Foundation and E-MRS includes holding the presentation ceremony the Czochralski Gold Medal during the Plenary Session of the E-MRS Meetings.

The first Professor Jan Czochralski Gold Medal was awarded to Dr. Paul Siffert during the E-MRS Symposium devoted to materials science activities in Central Europe held in Kraków in November 2000. Professor Kazimierz Flaga (Poland) was the recipient of the Czochralski Gold Medal presented during E-MRS Spring Meeting in Strasbourg in June 2001.

The European Materials Research Society and the Polish Foundation for Materials Science Development have jointly agreed to honour Professor Dr. Walter Heywang with the Czochralski Award and Gold Medal for both, his basic scientific research and development work in the field of electronic semiconductors, especially the innovative growth and characterisation of silicon.

With this Czochralski Award and Gold Medal should at the same time be recalled and recognized the great merits of Siemens AG for their epoch making industrial development of electronic grade silicon. Without this pioneering work nearly 50 years ago, there would be no microelectronic industry today. Professor Dr. Walter Heywang was leading the Siemens Central Research Laboratories during this crucial period.

Prof. Dr. Walter Heywang, who will be awarded the Professor Jan Czochralski Gold Medal

Walter Heywang was born in 1923 in Germany. He was awarded a Masters Degree in 1948 and graduated with a Ph.D. at the University of Würzburg with theses in the field of semiconductors and superconductors.

He joined Siemens in 1950, where he was involved in a variety of successful R&D projects:

- Ferroelectric ceramics, revealing and explaining the influence of grain size on dielectric properties. This has become the basis for a novel type of ceramic capacitors;
- Semiconductors grade silicon, developing new means for material characterization mainly concerning bulk and interface recombination respectively trapping;
- Grain boundary effects in different semiconductor materials (Si, SiC, ZnO, ferroelectrics);

- Explanation of the resistivity anomaly in doped BaTiO₃ ceramics and bringing these so-called ptc-resistors to production and to the market place;
- Special sensors, sensors systems and activators including the use of phase transitions;
- Bringing LEDs and surface acoustic wave devices to production including the necessary application oriented development;
- Contributions to the development of lasers, especially semiconductor lasers, with a Patent on Injection excitation in 1957

During his career at Siemens (1950-1988) he had to take increasing responsibility. In 1976 he became head of the Corporate R&D Laboratories with about 1000 coworkers. These laboratories have to cover the entire electrotechnics research in which Siemens AG is engaged.

In addition to these activities within Siemens he became an Associate Professor at the Technical University of Munich in 1969. He received an Honorary Doctor of Engineering degree from the University of Lund and the Wilhelm Exner Medal from the Austrian Engineering Association.

He is member of the German Physical Society, The German Society of Electrical Engineers (VDE/ITG) and fellow of IEEE. He was on the advisory board of different research institutions and companies and a member of the steering committee of the European Solid State Device and Circuits Conference, which he has developed from Germany.

He has edited the series HALBLEITERELEKTRONIK (SEMICONDUCTOR ELECTRONICS published by Springer) together with R. Müller and is author or co-author of 3 of its 21 volumes. He is author of about 90 scientific publications.

Timetable of Events

Sunday 14th September

19:00 – 21:00 Welcome reception, Physics Building

Monday 15th September

08:30 – 08:45 **OPENING CEREMONY** Small Hall (Mała Aula)
Chairman: Professor Paul Siffert, General Secretary of E-MRS

PLENARY SESSION Small Hall (Mała Aula)

Chairman: Professor Paul Siffert

08:45 – 09:30 **Krzysztof Kurzydłowski** – Warsaw University of Technology
Professor Czochralski – Distinguished Scientist and Inventor,
Founder of Modern Materials Science

09:30 – 09:45 **Ceremony to award the Professor Jan Czochralski Gold Medal to Professor Dr. Walter Heywang**

09:45 – 10:30 **Thomas A. Weber** – National Science Foundation, Arlington, U.S.A.
Funding of Materials Science Research at the
United States National Science Foundation

10:30 – 11:00 **Coffee Break** Main Hall (Duża Aula)

Chairman: Dr. Peter A. Glasow, E-MRS, IUMRS Imm. Past President

11:00 – 11:45 **Detlef Hommel** – University of Bremen, Germany
Short Wavelength Optoelectronic: Status and challenges

11:45 – 12:30 **Izabella Grzegory** – High Pressure Research Center, Warsaw, Poland
Gallium Nitride Single Crystals Grown Under
High Pressure for Optoelectronics Applications

12:30 – 14:00 **Luncheon**

14:00 **INDIVIDUAL SYMPOSIA**
See plan of WUT for the location of each symposium

19:00 – 21:00 **POSTER SESSION** Main Hall (Duża Aula)

Tuesday 16th September**PLENARY SESSION Small Hall (Mała Aula)****Chairman: Professor Marcel H. Van de Voorde****09:00 – 09:45 William G. Stirling** – European Synchrotron Radiation Facility (ESRF)
Grenoble, France

Materials Science at the European Synchrotron Radiation Facility

09:45 – 10:30 Albert Fert – Unité Mixte de Physique CNRS-Thales, Orsay, France
Recent Developments in Spin Electronics and Perspective**10:30 – 11:00 Coffee Break** Main Hall (Duża Aula)**11:00 INDIVIDUAL SYMPOSIA**

See plan of WUT for the location of each symposium

19:00 Dinner – Polish cuisine – Novotel Forum**Wednesday 17th September****PLENARY SESSION Small Hall (Mała Aula)****Chairman: Dr. Thomas A. Weber****09:00 – 09:45 Mathias Werner** – Deutsche Bank AG, Berlin 10117, Germany
Markets and Economical Aspects of Nano- and Microtechnology
Related Products**09:45 – 10:30 Peter Gluhe** – GFD Gesellschaft für Diamantprodukte GmbH
Ulm 89081, Germany

Diamond – an Emerging Material for Medical and Technical Applications

09:00 – 10:30 SYMPOSIUM C – see programme of Symposium C (Room 213)**10:30 – 11:00 Coffee Break** Main Hall (Duża Aula)**11:00 INDIVIDUAL SYMPOSIA A-E**

See plan of WUT for the location of each symposium

SYMPOSIA F & G SPECIAL JOINT SESSION

Small Hall (Mała Aula)

19:00 – 21:00 POSTER SESSION Main Hall (Duża Aula)

SPECIAL JOINT SESSION OF SYMPOSIA F & G

Small Hall (Mała Aula)

PERSPECTIVES OF NANOTECHNOLOGY**Chairman: Dr. Thomas A. Weber****11:00 – 11:45 Marcel H. Van de Voorde** – University of Technology
Delft, Netherlands**11:45 – 12:30 Leonard Fass** – GE Medical Systems, Slough, SL1 4ER, U.K.
Nanotechnology in Medicine**12:30 – 14:00 Luncheon****Chairman: Professor Witold Łojkowski****14:00 – 14:30 Hans-Jörg Fecht** – Ulm University, Ulm 89081, Germany
High Performance Nanoscale Materials and Bulk Metallic Glass**14:30 – 15:00 Antoaneta Folea** – European Commission, Brussels, Belgium
Participation of Accession Candidate Countries to the FP6 – Priority 3**15:00 – 15:30 Ottilia Saxi** – The Institute of Nanotechnology (IoN), Stirling, U.K.
Nanotechnology Start-ups in Europe**15:30 – 15:50 Coffee Break** Main Hall (Duża Aula)**Chairman: Dr. Mathias Werner****15:50 – 16:20 Giovanni Baldi** – Colorobbia Italia Spa
Laboratorio di Ricerca Avanzata, Italy
Synthesis of Inorganic Nanoparticles and their Application
on Ceramic Substrates**16:20 – 16:50 Martin Zachau** – OSRAM GmbH (OSRAM), München, Germany
Nanomaterials for Lighting**16:50 – 17:20 Andrzej Siemaszko** – Institute of Fundamental Technological Research
Warsaw, Poland
Structural Funds in Poland and Polish Research
Potential in Nanotechnology**19:00 – 21:00 POSTER SESSION** Main Hall (Duża Aula)

E-MRS Fall Meeting 2003
Book of Abstracts

Plenary session

Monday, September 15th

08:30 - 09:00

Opening Address and Czochralski Award

Main Building, Small Hall (Mała aula)

09:00 - 10:30

Plenary session - Hermann Grimmeiss

Main Building, Small Hall (Mała aula)

9:00 - 9:45

Professor CZOCHRALSKI - Distinguished Scientist and Inventor, Founder of the Modern Materials Science

invited
plenary

Krzysztof Kurzydowski²⁾, Stanislaw Mankowski^{1,3)}

1) Warsaw University of Technology, Koszykowa 75, Warsaw 00-662, Poland

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

3) Rector of Warsaw University of Technology, Poland

Prof. CZOCHRALSKI was born in Central Poland in 1835 and died 50 years ago in Poznan. He obtained engineering degree in Berlin and started to work as assistant to famous scholar Wichard von Moellendorf. Right from the beginning of his career he took interest in what can be now described as physical metallurgy and pioneered research in plastic deformation of single crystals. Also contributed to development of modern crystallography.

In 1929 received an honorary degree from Warsaw University of Technology and a year later accepted professorship position with the Faculty of Chemistry at this school. Organized Department and later Institute of Metallurgy and Metal Science. His major interest continued to be with processing of metals and in particular he researched crystallization. Research in this field, carried out in Germany, resulted in invention of the famous CZOCHRALSKI method for growing large mono-crystals, which paved the way to modern technologies of electronic materials. He also studied elastic, plastic properties of metals and their corrosion resistance. Professor CZOCHRALSKI was a pioneer in what is now described as technology transfer. His inventions match excellence of scientific achievements. Obtained a number of patents in Germany and Poland.

Professor has lived in turbulent times. Holding two citizenships, Polish and German, and acting successfully both within business and academia he made a great number of fans and enemies. The scientific foundations built at WUT by CZOCHRALSKI proved to be resistant to historical misfortunes. Faculty of Materials Science and Technology, the leading materials research institution in Poland is proudly continuing tradition of metallurgy and metal science at Warsaw University of Technology. This Faculty is also one of the leaders in technology transfer taking fully with the reach achievements of its famous founder.

9:45 - 10:30

Funding of Materials Science Research at the US National Science Foundation

invited
plenary

Thomas A. Weber¹⁾

1) National Science Foundation (NSF), The National Science Foundation, Arlington, Virginia 22230, United States

The United States National Science Foundation (NSF) is an independent agency of the United States government. NSF currently has a budget of over five billion dollars and is responsible for funding all areas of fundamental science and engineering except for science and space based astronomy. Within NSF, more than \$350M is spent on materials. In January of 2000, President Clinton announced the National Nanotechnology Initiative. Currently the US is spending \$850M on this initiative of which NSF accounts for \$220M. This talk will detail the areas of science and engineering funded by the Division of Materials Research (DMR) within both the core science areas and the initiative.

The Division of Materials Research has also recently launched an initiative to encourage collaboration between US and foreign scientists. Currently there are programs in place between NSF and 6 countries in the Americas, 17 European countries, the European Science Foundation and the European Union. Details about the first few competitions will be given and the necessary steps to be taken for European Science to avail themselves of such collaborations.

10:30 - 11:00

Coffee break

Main Building, Main Hall (Duża aula)

Plenary session - continued - Paul Siffert

11:00 - 12:30

Main Building, Small Hall (Mała aula)

Short wavelength optoelectronics: Status and challenges

Detlef Hommel¹⁾

11:00 - 11:45

1) University of Bremen, Otto-Hahn-Allee, Bremen 28359, Germany

invited
plenary

Optoelectronic in the blue-UV region based on AlGaIn/GaN/InGaIn structures is developing rapidly and first DVD players with a 410nm laser diode (LD) inside (Blue Ray Disc, 23 GB) made by Sony are now available in Japan. Nevertheless big problems remain to be solved for nitride laser diodes. One is to go to shorter wavelength than 360nm the other to cover wavelength longer than 480nm. Till now electrically pumped lasing of nitride structures was only possible within this spectral range.

Only ZnSe-based structures have shown lasing between 480 - 560nm. New approaches for stable green lasing will be discussed including CdSe/ZnSSe quantum dot laser and a first monolithic green surface emitting laser (VCSEL). The quantum dot laser shows under constant current degradation a significantly higher long term stability compared to a quantum well LD emitting at the same wavelength.

Another interesting topic is to realize single photon emitter operating at room temperature for quantum information technology and quantum cryptography. Here the II-VI system CdSe/ZnMgSSe has serious advantages compared to the classical III-V compounds like InAs/GaAs.

In the talk the state of art of these different kind of short wavelength light emitters will be presented and critically discussed from the scientific and application point of view. All kinds of laser diodes mentioned above have been realized in Bremen as well as single phonon emitters up to 200K.

Gallium nitride single crystals grown under high pressure for optoelectronics applications

11:45 - 12:30

Izabella Grzegory¹⁾

invited
plenary

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

The most important advantage of the growth of GaN from high temperature gallium solutions under high nitrogen pressure is that the crystals are almost free of dislocations (100 cm^{-2}). The low density of dislocations is of crucial importance for the high power optoelectronic devices based on GaN and their ternaries. Therefore, the application of the pressure grown crystals as substrates for epitaxial deposition of blue-violet InGaIn laser diode structure should give high quality devices. This will be shown by comparing of some physical properties of the quantum structures grown on both near defect free GaN and typical GaN/Al₂O₃ substrates containing high density of dislocations. Also the recent progress in the construction of high power blue and violet lasers on the pressure grown substrates achieved at the HPRC will be reviewed.

The most important disadvantage of the high pressure growth of GaN is that the method gives individual 100 mm thick platelets of 10-15 mm in their lateral size. The strong anisotropy of the crystallization process will be discussed in terms of two dimensional nucleation on particular crystallographic planes to show that this is rather an intrinsic property of the system. The relations between supersaturation and the nucleation rates explain both the anisotropy and difficulties with control of the growth in directions parallel to the c-axis of the crystal. The application of directional crystallization methods is a way to increase the rate of stable growth of near dislocation free GaN in c-direction. The results of both the high pressure solution growth and the low pressure HVPE (Hydride Vapor Phase Epitaxy) with the use of GaN platelets as substrates are very promising and will be also presented.

Lunch break

12:30 - 14:00

Tuesday, September 16th

Morning session - Marcel H. Van de Voorde

09:00 - 10:30

Main Building, Small Hall (Mała aula)

Materials Science at the European Synchrotron Radiation Facility

William G. STIRLING¹⁾

9:00 - 9:45

1) European Synchrotron Radiation Facility (ESRF), Grenoble BP220-38043, France

invited
plenary

X-rays have long been one of the major techniques used in the characterisation and investigation of materials. With the advent of dedicated synchrotron light sources a large, and increasing, number of new methods has become available for studies in materials science.

In this talk, the wide range of experimental techniques available at Europe's most advanced synchrotron light source, the European Synchrotron Radiation Facility (ESRF), will be summarised. The ESRF, which is funded by a total of 17 Member and Associate countries, operates a 6 GeV electron storage ring producing very intense x-ray beams with energies from below 1 keV to several hundred keV. Each year over 3000 scientific users carry out some 1200 experiments on the 40 beamlines at ESRF; a large number of these beamlines are involved in materials science research.

Illustrative examples from methods including diffraction, EXAFS, XMCD and imaging will be described, and future developments and trends discussed.

9:45 - 10:30

Recent Development in Spin Electronics and Perspective

Albert Fert¹⁾

invited
plenary

1) Unité Mixte de Physique CNRS-Thales, Domaine de Corbeville, Orsay 91404, France

Spin electronics (or spintronics), at the interface between magnetism and electronics, is a field of research in considerable expansion. The basic concept of spin electronics is the control of spin currents in nanostructures, and this concept has already been exploited for the giant magnetoresistance of the multilayers and in magnetic tunnel junctions. I will review the most recent developments, with a particular focus on the reversal of magnetic moments by spin transfer from a spin-polarized current and spintronics in semiconductor-based heterostructures. I will also discuss the potential of these developments for applications and the general perspective for the exploitation of the spin degree of freedom in electronic devices.

10:30 - 11:00

Coffee break

Main Building, Main Hall (Duża aula)

Wednesday, September 17th

09:00 - 10:30

Morning Session - Thomas A. Weber

Main Building, Small Hall (Mała aula)

9:00 - 9:45

Markets and Economical Aspects of Nano- and Microtechnology Related Products

invited
plenary

Mathias Werner²⁾, **Stephan Mietke**²⁾, **Jürgen Ilgner**²⁾, **Hans-Jörg Fecht**¹⁾

1) Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany

2) Deutsche Bank AG, Microtechnology Innovation Team, Unter den Linden 13/15, Berlin 10117, Germany

Nanotechnology is gaining more and more interest even in the financial community. From a commercial point of view nanomaterials and nanotools - that cover for example analytical instruments and to some extent, equipment for the semiconductor industry are already in the market or have the best opportunity to be commercialized soon.

At the beginning of the value chain are materials. A good example for a currently rapid development are carbon nanotubes. The success or failure of carbon nanotubes depends on their market applications. Carbon nanotubes could play a pivotal role in the upcoming nanotechnology age if their remarkable electrical and mechanical properties can be exploited. Nanotubes offer significant advantages over most existing materials such as carbon fibers including unimagined strength-to-weight ratio, attractive mechanical properties and very interesting field emission properties. Mass market applications include the reinforcement of polymer nanocomposites and field emission displays. Nanotube molecules might line up to form a light, strong wire or a composite that could be unbeatable as a material for making lightweight vehicles for space air and ground. These figures of merit may be realistic but in terms of cost are yet commercially unattractive. However, recent developments indicate a technology breakthrough which allows a mass production of carbon nanotubes. Several predictions indicate that the global production increases from the few kilogram range to more than 100 tons this year. Furthermore, a significant price reduction is expected.

In general, the term nanotechnology is badly defined and useful market figures - with a few exceptions - do not exist so far. An interesting part is the borderline between microsystems and nanotechnology. In certain microsystems

applications - e.g. for hard disk drive heads based on giant magnetoresistance read heads - nanotechnology plays already a significant role in a multi-billion dollar market place.

Diamond- an emerging material for medical and technical applications

Peter Gluche²⁾, André Flöter²⁾, Hans-Jörg Fecht¹⁾

1) Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany

2) GFD Gesellschaft für Diamantprodukte mbH, DaimlerChrysler research center, Wilhelm-Runge-Str. 11, Ulm 89081, Germany

9:45 - 10:30

invited
plenary

Diamond possesses outstanding material properties. Especially the high mechanical stability in combination with the high thermal conductivity and the low coefficient of friction makes diamond an attractive material for many applications. In the past 7 years, novel CVD-processes for diamond growth have been developed and optimized, resulting in the fabrication of polycrystalline and fine grained diamond films of high mechanical strength. Furthermore, the recent progress in plasma structuring of diamond films, allows to fabricate diamond parts of complex shape and micron resolution. As a first product, diamond scalpels for eye and microsurgery, were introduced into the market in 2001. Modern surgical methods require sharp micro cutting tools. In order to decrease the recovery times of patients, there's a trend towards ambulatory surgical techniques. However, these treatments require less traumatic wounds. Especially in the field of microsurgery, like eye- and neurosurgery, the demand of ultra sharp cutting tools is therefore continuously increasing. A first major step towards this trend was the introduction of diamond keratomes for eye surgery, made from natural gem stones approx. 35 years ago. Since diamond is the hardest of all known materials and has outstanding mechanical properties, it was possible to fabricate micro cutting tools having an outstanding and long lasting sharpness.

The transfer of the research results into a stable and reliable production will be described. An overview of the market for cutting tools for micro surgery will be given. The novel fabrication method, based on CVD diamond deposition and plasma structuring techniques, will be discussed and advantages of the new fabrication method in comparison to conventional fabrication methods, including market aspects, will be given. The next generation of diamond products, which are still under development, will be presented.

Coffee break

Main Building, Main Hall (Duza aula)

10:30 - 11:00

Symposium A

Novel Wide Bandgap Materials for Optoelectronic and Electronic Applications

Organisers

- **Marek Godlewski**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Stanisław Krukowski**, High Pressure Research Center, Polish Academy of Sciences, Warsaw, Poland
- **A. Matkovskii**, Lviv Polytechnic National University, Lviv, Ukraine
- **Andrzej Suchocki**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Adam Zakrzewski**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Proceedings

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(International Workshop on ADVANCED SPECTROSCOPY OF MODERN MATERIALS)

Monday, September 15th

13:50 - 14:00 **Opening address - Marek Godlewski**
Main Building, room 219

14:00 - 15:30 **Afternoon session - Marek Godlewski**
Main Building, room 219
Nitrides I

14:00 - 14:45 **AlGaN/GaN based optical and electrical sensors**
Oliver Ambacher¹⁾

invited oral *1) Center of Micro- and Nanotechnologies, Technical University Ilmenau (ZMN), Gustav-Kirchhoff-Str. 7, Ilmenau 98693, Germany*

Electronic transport in semiconductors that possess high internal spontaneous and piezoelectric polarization opens up a new field of pyroelectronics and pyrosensors. The pyroelectric character of group-III-nitrides with wurtzite crystal structure yields a novel degree of freedom in designing and tailoring devices for modern micro- and nanoelectronic applications. Furthermore spontaneous and piezoelectric polarization induced surface and interface charges can be used to develop very sensitive but robust sensors for the detection of ions, gases, polar liquids and UV-light. We present a review of both theoretical and experimental studies of spontaneous and piezoelectric polarization present in AlGaN/GaN heterostructures as well as the electronic transport properties of polarization induced two-dimensional electron gases which are formed at the AlGaN/GaN interface due to the difference in the total polarization of two adjacent III-nitride layers. We demonstrate that the two-dimensional electron gases (2DEGs) achieved without modulation doping are very suitable as channel of high electron mobility transistors optimally suited for high power and high frequency applications. In addition we review recent achievements concerning optical and chemical sensor applications of undoped AlGaN/GaN heterostructure field effect transistor devices. We summarize experimental results on the optical effects of AlGaN based heterostructures for highly selective detection of UV-light, the sensitivity of GaN Schottky diodes and gas sensitive field effect transistors for the detection of hydrogen and hydrogen containing gases as well as the performance of AlGaN/GaN based ion sensitive field effect transistors for highly sensitive pH measurements in electrolyte solutions.

14:45 - 15:30 **Thermal effects and current instabilities in group III-Nitride-based devices**
Alexander E. Belyaev¹⁾

invited oral *1) Institute of Semiconductor Physics NAS Ukraine, Kiev, Ukraine*

GaN and related materials have recently attracted a lot of interest for applications in high-power electronics capable of operation at elevated temperatures and radiation environment. Group III-Nitrides and their ternary and multi-ternary systems offer numerous advantages. These include wider bandgaps as well as excellent transport and thermal characteristics. The remarkable properties of Group III-Nitrides have led to rapid progress in the realization of a broad range of electronic devices. Despite the rapid progress in the upper frequency limits and RF power levels of three-terminal devices the representatives of two-terminal devices, in particular, resonant-tunnelling diodes (RTDs) still play an important role in many system applications at frequencies above about 50 GHz. If RTDs could be produced in the Group III-Nitride system, a number of novel possibilities exist for development and exploitation. If RTDs can be made in Group III-nitrides, a whole new high-power microwave technology could be realised. Thus, the goal of this communication is two-fold. First, we address experimental and theoretical study of a two-dimensional electron gas (2DEG) transport at low and moderate electric fields. In particular, we concentrate on hot-electron effects and self-heating phenomena. Separation between these effects is important to clarify the thermal budget of devices. The second point concerns with growth conditions influence on performance of nitride-based quantum tunnelling devices. Potential reasons for the instabilities in the I-V characteristics are discussed.

15:30 - 15:50 **Coffee break**

15:50 - 17:20 **Afternoon session - continued**
Main Building, room 219
Nitrides II D. Hommel

15:50 - 16:35 **Unusual properties of nitrogen containing III-V semiconductors; ab-initio calculations**

invited oral

Izabella Gorczyca¹⁾, Niels E. Christensen²⁾, Axel Svane²⁾

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

2) *Institute of Physics and Astronomy, University of Aarhus, Aarhus, Denmark*

The most interesting features of nitrides (GaN, AlN, InN, BN) and related alloys (InGaN and GaAsN) are presented considering their electronic, structural, optical and chemical properties. Ab-initio calculations of the band structure and total energy are performed by the linear muffin-tin orbital method (LMTO) method.

Studies of the energy band structures of nitrides, the pressure-induced phase transitions and lattice vibrations at high pressure indicate the role of specific features of the nitrogen atom and the role of the Ga-3d and In-4d semicore states.

The energy levels of native defects (vacancies and antisites) and impurities (C, Zn, Mg, O, Be), their formation energies and the effects of pressure on these properties are calculated. For the cation vacancies, which are the most stable native defects in n-type material, we compare their behavior in the nitrides and in GaAs, finding substantial differences in the pressure dependence of the defect level positions and formation energies. Comparing the substitutional impurities GaN:Mg and GaN:Be, we find differences in their high pressure behavior, in agreement with the high pressure measurements of the light emission performed on GaN:Be and GaN:Mg samples.

Observed peculiarities of InGaN and GaAsN band structures are explained in terms of ab-initio calculations:

1. unusually strong decrease of the InGaN bandgap and its pressure coefficient when indium is incorporated is related to the dramatic change of the valence band shape caused by strong hybridization of In(p,d) and Ga(p,d) wave functions.
2. The introduction of small amounts of nitrogen modifies strongly the conduction bands in GaAsN. The lowest conduction band exhibits strong non-parabolicity, and the fundamental gap, E_g , decreases with x. The Γ , L and X states mix and form new low-energy states. Pressure affects the electronic properties of GaAsN, and it influences the electron effective masses and their dependence on x.

Optical properties of low-dimensional systems based on group-III nitride ternary and quaternary alloys.

16:35 - 17:20

Pierre LEFEBVRE¹⁾

invited oral

1) *CNRS - Universite Montpellier 2 (GES), Place Eugene Bataillon, Montpellier 34095, France*

In low-dimensional systems based on hexagonal group-III nitrides, the optical recombination of electron-hole pairs is tributary to the complex interplay of internal electric fields and of the localisation of carriers, at the scale of a few nanometers. A microscopic model is proposed that accounts for these intricate influences and for the resulting remarkable scaling law that was observed by time-resolved photoluminescence (TRPL) in a variety of InGaN/GaN quantum well and quantum dot systems: once plotted in a reduced-time scale, the photoluminescence decay keeps a constant, nonexponential shape. The time-dependent screening of the internal field by high densities of electron-hole pairs is analysed. The paper also presents TRPL results proving that it is possible, by using quaternary alloys, to produce quantum structures for which the internal electric field is minimized.

Poster session

19:00 - 21:00

Theoretical study on electronic, positron and structural properties of Ga- **$_{1-x}\text{Al}_x\text{N}$**

19:00 -

Yarub Al-Douri

poster

Electronic and positron charge densities and band structures of $\text{Ga}_{0.5}\text{Al}_{0.5}\text{N}$ using the empirical pseudopotential method (EPM) are investigated. For the ternary alloy $\text{Ga}_{1-x}\text{Al}_x\text{N}$, the virtual crystal approximation (VCA) is coupled with the pseudopotential method. The energies along Γ , X and L of $\text{Ga}_{1-x}\text{Al}_x\text{N}$ alloy as a function of the mole fraction are calculated. Angular correlation of positron annihilation radiation (ACPAR) along different crystallographic directions in $\text{Ga}_{0.5}\text{Al}_{0.5}\text{N}$ is calculated. Other quantities such as ionicity character and bulk modulus by means of recent models [1, 2] with respect to the mole fraction are discussed.

A-1

[1] Y. Al-Douri, H. Abid, A. Zaoui and H. Aourag, *Physica B* 301, 295, 2001.

[2] Y. Al-Douri, H. Abid and H. Aourag, *Physica B* 305, 186, 2001.

Self-heating and microwave noise in AlGaIn/GaN**L. Ardaravicius¹⁾, J. Liberis¹⁾, A. Matulionis¹⁾, L.F. Eastman²⁾, J.R. Shealy²⁾, A. Vertiatchikh²⁾**

19:00 -

1) *Semiconductor Physics Institute, A. Gostauto 11, Vilnius LT-2600, Lithuania*

poster

2) *Cornell University, 425 Philips Hall, Ithaca NY 14853, United States*

The influence of self-heating effects to hot-electron noise is investigated at 373 K and 293 K in nominally undoped

A-2

AlGaIn/GaN two-dimensional electron gas (2DEG) channels subjected to a strong pulsed electric field applied parallel to the heterointerface. The 2DEG channel was induced by spontaneous polarization and piezoelectric fields.

Hot-electron noise measurements were performed in the frequency band near 10 GHz where contributions due to generation-recombination noise and $1/f$ fluctuations were negligible. The gated modulation-type radiometric setup was used for investigation of the current fluctuations in the bias direction. The bias pulses ranging from $4 \mu\text{s}$ to $0.5 \mu\text{s}$ were chosen. At a high bias, the thermal quenching of hot-electron effects is observed. As a result, the associated kinetic processes (hot-electron sharing, for example) are shifted towards higher electric fields. In particular, the raise in lattice temperature shifts the onset field for the noise due to occupancy fluctuation to appear.

In the delay-time dependent noise measurements, the decrease of the hot-electron noise temperature upon the delay time is observed when the bias pulse is on. To estimate the average lattice temperature, the microwave noise thermometry method [1,2] is applied. The lattice temperature was estimated through backward extrapolation of the delay-time dependent noise temperature $T_n(t)$ measured after the voltage switch-off. The method is discussed and compared with other techniques.

1. M. de Murcia, E. Richard, J. M. Perraudin, A. Boyer, A. Benvenuti, and J. Zimmermann, *Semicond. Sci. Technol.* 10, 515 (1995).

2. H. Hartnagel, R. Katilius, and A. Matulionis, *Microwave Noise in Semiconductor Devices*, Wiley, New York, 2001, p. 86-90.

19:00 - poster A-3 **Schottky Barrier and Aging Effect studies in Au(Cu)/p-CdTe Contacts**
Yevgen O. Bilevych¹⁾, Andriy V. Sukach¹⁾, Volodymyr V. Tetyorkin¹⁾, Volodymyr M. Popov²⁾
 1) *Institute of Semiconductor Physics NAS Ukraine, Kiev, Ukraine*
 2) *Physical and Technological Research and Certification Centre "Microanalytics" (PTRCCM), 3 Severo-Siretskaya, Kiev 04136, Ukraine*

CdTe is regarded as a promising material for X-ray detectors. Knowledge of the Schottky barrier formation in p-CdTe is insufficient. Partly it can be explained by such intrinsic physical properties of p-CdTe as low temperature conductivity, instable surface and poor mechanical characteristics.

In order to clarify possible mechanisms of the Schottky barrier formation the influence of the surface treatment on physical properties of Au(Cu)/p-CdTe contacts were investigated. The contacts to p-CdTe were prepared on (110) CdTe single crystals with hole concentration $p=(6-10) \cdot 10^{15} \text{ cm}^{-3}$ and Hall mobility $80-120 \text{ cm}^2/\text{Vs}$. The surfaces were prepared by wet chemical etching prior to metallization using etchants composed from three components. In sum, seven etchants were investigated. The barrier height was determined from the photoemission current measurements. SIMS and SEM measurements have been carried out to characterize compositional structure at the interface before and after aging.

In freshly prepared contacts SIMS measurements demonstrate pronounced deviation from the stoichiometry at the interfaces caused by wet chemical etching. The multi-level pinning of the Fermi level at the metal-CdTe interface was experimentally observed in these contacts. It has been found that the barrier height can be regulated in a wide range of energies for surfaces treated in different etchants (from approximately 0.2 eV up to 0.8 eV when measured from the valence band maximum).

Photoemission current and SIMS profile measurements were performed in freshly prepared, six-month and one-year old contacts in order to study aging effects. The barrier height has been found to change substantially during the storage of contacts under normal laboratory condition. In one-year old contacts only two barriers close to 0.6 eV and 0.8 eV were observed in investigated contacts. Experimental results are discussed in terms of formation of native defects at the interface caused by surface treatment.

19:00 - poster A-4 **Optical properties of CuInTe₂ single crystals by photoacoustic spectroscopy**
Abdeslam Bouloufa¹⁾, A. Messous¹⁾, A. Zegadi¹⁾, M.V. Yakushev²⁾, R.D. Tomlinson
 1) *Labo.CCNS, Dpt. Electronics, University of Setif (UNIV.), Route, setif 19000, Algeria*
 2) *Physics Department, Salford University, Salford M5 4WT, UK, United Kingdom*

This paper reports the optical properties of the photovoltaic material CuInTe₂ (CIT) as characterized by photoacoustic spectroscopy (PAS), who yields direct information on non-radiative absorption processes which are usually associated with the band structure and defect-related energy loss mechanisms.

Measured spectra of p-conducting CIT single crystals are given using a high resolution near infrared photoacoustic spectrometer of the gas-microphone type. The data is used to evaluate the absorption spectra, the band gap energy, and ionization energies of defects that have been observed in the tail of the spectra (1).

Ionisation energies of intrinsic defects are derived from the appropriately determined values of the gap energy. Different energy levels have been observed. The shallow level assigned to the antisite defect Cu_{In} and two levels were assigned to the vacancies of $\text{Cu}(\text{V}_{\text{Cu}})$ and $\text{Te}(\text{V}_{\text{Te}})$. The PAS technique reveal the presence of populations of

deep defects in our samples (3).

The obtained results confirm the efficiency of the technique in detecting the non-radiative defects known to dominate the properties of this compound. A discussion on the relation between the composition of the samples and the intrinsic defect chemistry in this compound is also given. Finally, we give a comparison between the results we have obtained and those published in the literature (3, 4).

References:

- 1) A. Zegadi, A. Boloufa, N. Mazouz, B. Barka, E. Ahmed, A. E. Hill and R. D. Tomlinson. PV in Europ. Photovolt. Sol. Energy Conf., Rome 11-17 Oct. Italy, 81 (2002)
- 2) A. Zegadi, M.A. Slifkin, M. Djamin, R. D. Tomlinson and H. Neumann, Sol. Stat. Commun. 83, 591 (1992)
- 3) M. V. Yakushev, H. Neumann and R.D. Tomlinson, Cryst. Res. Technol. 30, 121 (1995)
- 4) A. Zegadi, A. Bouloufa, M. Rouha, A. Messous M. V. Yakushev and R. D. Tomlinson. 17th Europ. Photovolt. Sol. Energy Conf., Munich 22-26 Oct. Germany, 1062 (2001)

Synthesis and luminescent investigation of KTiOPO_4 and $\text{NaTi}_2(\text{PO}_4)_3$ crystals doped with rare earth ions

Oksana V. Chukova¹, Roman Boyko², Pavlo Nagorny², Sergiy G. Nedilko¹

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- 2) Kyiv National Taras Shevchenko University, Department of Chemistry, Volodymirskaya Str. 64, Kyiv 01033, Ukraine

Modern device technology reveals increasing interest to materials with integrated functional possibilities. The potassium titanyl phosphate KTiOPO_4 (KTP), for example, is a perspective material for similar application. Thus, it is possible to use nonlinear optical properties of the doped KTP crystal for light generation and as nonlinear material at the same device.

This paper presents results of spectral-luminescent investigation of the phosphate crystals doped with the RE ion impurities. The crystals were synthesized from melt of mixtures of the corresponded oxides taken in optimal proportions. Choice of activated ions was caused not only by the fact that RE ions are the main "laser" activator impurities. It was also taken into account that RE impurities stabilize the crystals structure.

Investigation of the spectral-kinetic (absorption, luminescence) properties of pure (inactivated) and doped single crystals and polycrystalline samples of the potassium titanyl phosphate KTP:RE^{3+} (RE = La, Eu, Nd) and sodium titanium phosphate $\text{NaTi}_2(\text{PO}_4)_3$ (RE = Sm, Dy) was carried out in 4,2 - 300 K temperature region. Luminescence bands and decay kinetic of matrix emission and of inner emission of the RE ions were observed. It was shown on an example of the impurity europium ions (Eu^{3+}) that RE ions can arrange two different positions in the phosphate matrices. This is explained by unisocharged arrangement of the rare earth ions on the sites of titanium ions with 4+ charge.

19:00 -
poster
A-5

Deep UV Detection by CVD diamond position sensitive devices

Gennaro CONTE¹, Maria C. Rossi, Giovanni Mazzeo, Stefano Salvatori, Viktor Ralchenko²

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- 2) Moscow, General Physics Institute (RAS), 38 Vavilov, Moscow, Russian Federation

In the last few years the request for high performance ultraviolet (UV) detectors has shown a growing of interest. These devices have been realized with traditional semiconductors (i.e. silicon) principally for the low cost of the material, owing to the well developed technology, which compensates for the difficulties to adapt a material with a low energy bandgap for such kind of applications. On the other hand, there are fields where only new, high energy bandgap, materials may be used (i.e. UV sun study, which needs a device with high discrimination ratio between visible and UV radiation). Nowadays, the intrinsic limitations of this material can be overcome by CVD diamond. The dynamic performance of photoconductive devices based on this material has been a topic over which many efforts have been spent. Very recently it has been proposed the use of photoconductive CVD diamond devices for pulsed UV radiation in the field of ULSI photolithography. Indeed, the needs of decreasing the active dimension of the electronic devices has led to the use of excimer laser radiation for the photoresist exposition. For such kind of application, the principal request is for a good stability and very high MTBF, requirements not completely satisfied by silicon based devices. Diamond, with its threshold for radiation damage around 100 mJ/cm², meets the specifications. The dynamic performance, noise, gain and band-width of PSDs made by CVD diamond will be presented and correlated with the material quality.

19:00 -
poster
A-6

Thermodynamic stability of partially ordered $\text{Si}_{1-x}\text{C}_x$

Vitaliy G. Deibuk¹, Yuriy G. Korolyuk¹

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The practical importance for electronic industry of material systems which are compatible with standard Si processing technology the growth as well as the characterization of the $\text{Si}_{1-x}\text{C}_x/\text{Si}$ structures have been extensively

19:00 -
poster
A-7

pursued in the last years. More recently, the optical and electronic properties of novel $\text{Si}_{1-x}\text{C}_x$ and $\text{Si}_{1-x-y}\text{Ge}_x\text{C}_y$ alloy layers on Si substrates have attracted much interest. Such systems may provide high quality materials which extend the range of Si-based heterostructures. Nevertheless, the thermodynamic properties, stability and structure properties of $\text{Si}_{1-x}\text{C}_x$ alloys is still not will understood.

In this work we report the theoretical study of epitaxially strained $\text{Si}_{1-x}\text{C}_x/\text{Si}(001)$ alloys. Our investigations were based on NVT molecular dynamic method with three-particle Tersoff's potential of interactions with mirror-boundary conditions.

It allows us to obtain information on the influence of external biaxial stress on the ordering. We calculated and analyzed the enthalpy of mixing epitaxially strained ZB(zinc-blende)-ordered and RB(rhombohedral)-ordered $\text{Si}_{1-x}\text{C}_x$ alloys. The bond-length of C-C, Si-C and Si-Si bonds as a function of the compositions is build by using the radial-distribution functions. The diagram of stability of ordered $\text{Si}_{1-x}\text{C}_x/\text{Si}(001)$ alloys is studied. It's shown that ordered and biaxial strain increase the stability. In this way, we also gain further insight into the effect of biaxial strain and alloying on intrinsic properties of the silicon-carbon alloys materials. Our results are in a good agreement with recent experimental data.

19:00 - poster
A-8 **Optical characteristics and structural ordering of quasi - two - dimensional TlGaS_2 crystals doped by rare-earth ions.**

Gadjibala I. Abutalybov¹⁾, Sevda Z. Dzhafarova, Nailja A. Ragimova

1) Institute of Physics, Azerbaijan National Academy of Science, G.Javid, 33, Baku AZ143, Azerbaijan

The present paper shows the results of complex experimental studies of optical phenomena, electron-paramagnetic resonance, Raman scattering, and heat capacity in TlGaS_2 single crystals doped with various concentrations of rare-earth (RE) ions. The authors were the first to discover that optic parameters of TlGaS_2 single crystals can be improved by increasing the degree of structural ordering of the cation sublattice doping them by RE ions. The impurity ions with the ionic radii close to the radii of the main cations (Tl^{3+} , Ga^{3+}) and charges of main cations in the area of rather low concentrations were shown to exert an ordering effect on the cation sublattice of a congruent TlGaS_2 crystal.

In this case the crystal resistance to laser radiation is also observed to grow. The effect of diminishing photorefractive while the crystal is doped, apparently, due to increasing efficiency of the radiating recombination of photo-excited carriers without their being captured to deeper levels, and correlates well with the discovered ordering of the cation sublattice and both of them are observed for the same, relatively narrow, range of concentrations and the type of doping impurities. The efficiency of such a recombination determines the intensity of luminescence in doped crystals. And greater luminescence is attained by alloying them in the concentration range of RE, ions in which the alloyed single crystals possess the maximum degree of structural ordering and increased resistance to optic damage.

19:00 - poster
A-9 **UV-written channel waveguides in Er-doped Bi_2O_3 -based glass**

Angelique Favre¹⁾, Eric Lee, Vasilis Apostolopoulos, Corin B. Gawith, Chao-Yi Tai, Elizabeth Taylor, Yuki Kondo²⁾, Fumihito Koizumi³⁾

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2) Asahi Glass Co. Ltd, Kanagawa-ku, Yokohama 221-8755, Japan

3) Asahi Glass Co.Ltd, Yokohama, Japan

The increase of the information transmission capacity using wavelength division multiplexing (WDM) transmission systems requires low cost compact amplifiers for metro use. Er-doped planar waveguide amplifiers are thus envisaged.

We are currently investigating a novel erbium-doped oxide glass based on Bi_2O_3 , which exhibits broadband emission and negligible concentration quenching up to 13,000 ppm of Er concentration.

In this study, we report the fabrication of channel waveguides in a Bi_2O_3 -based glass. Erbium doped channel waveguides were fabricated using direct laser writing (with a UV beam at 244 nm) into a bulk Er-doped glass.

The fabrication technique and results will be presented. The mode profile, loss, change in refractive index will be reported.

19:00 - poster
A-10 **In-depth and in-plane profiling of InGaN-based laser diodes and heterostructures**

Marek Godlewski^{1,3)}, E. M. Goldys⁴⁾, Matthew R. Phillips²⁾

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2) University of Technology, Sydney (UTS), Broadway, Sydney 2007, Australia

3) College of Science, Cardinal S. Wyszyński University, Warsaw, Poland

4) Division of Information and Communication Sciences, Macquarie University, Sydney, Australia

We employ cathodoluminescence (CL) technique for evaluation of in-depth and in-plane instabilities of light emission in series of InGaN quantum well structures, including laser diode structures. Heteroepitaxial and homoepitaxial

InGaN/GaN MOVPE-grown structures are studied. Stimulated emission under electron beam pumping in a conventional CL set up is achieved. We study light emission properties from laser structures and their relation to microstructure details. Large in-plane fluctuations of light emission are also present for excitation densities larger than the threshold densities for the stimulated emission. The latter indicates that potential fluctuations are not fully screened in the active regions of laser structures even at large excitation densities.

This work was partly supported by the grant numbers 5 P03B 007 20, 5 P03B 123 21 of KBN and by the DENIS program of European Union (G5RD-CT-2001-00566).

Cathodoluminescence study of n-type doped GaN epilayers and GaN/InGaN quantum well structures

19:00 -

Marek Godlewski^{1,4}, **Elzbieta Lusakowska¹**, **E. M. Goldys⁵**, **Matthew R. Phillips³**, **T. Bottcher²**, **S. Figge²**, **Detlef Hommel²**

poster

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

A-11

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

3) *University of Technology, Sydney (UTS), Broadway, Sydney 2007, Australia*

4) *College of Science, Cardinal S. Wyszyński University, Warsaw, Poland*

5) *Division of Information and Communication Sciences, Macquarie University, Sydney, Australia*

In this communication we discuss light emission properties of n-type doped GaN epilayers and InGaN/GaN quantum well structures. We evaluate the influence of n-type doping of these layers and structures and of the doping level on structural quality of the samples studied, the observed light emission intensity and in-plane emission instabilities. We confirm reported previously strong enhancement of light emission from doped samples and discuss possible mechanisms of the enhancement. Large- and small-scale light emission inhomogeneities are demonstrated based on the results of cathodoluminescence, scanning electron microscopy and micro-photoluminescence experiments. The present study indicates possibility of saturation of dislocations at high doping level or at large excitation density.

This work was partly supported by the grant numbers 5 P03B 007 20, 5 P03B 123 21 of KBN and by the DENIS program of European Union (G5RD-CT-2001-00566).

Compensation mechanisms in magnesium doped GaN

19:00 -

Marek Godlewski^{1,3}, **H. Przybylinska¹**, **E. M. Goldys⁴**, **J. P. Bergman⁵**, **B. Monemar⁵**, **I. Gregory²**, **S. Porowski²**

poster

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

A-12

2) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

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4) *Division of Information and Communication Sciences, Macquarie University, Sydney, Australia*

5) *Dept. of Physics and Meas. Techn., Linköping University, Linköping, Sweden*

In this communication we discuss compensation processes in magnesium doped GaN epilayers and bulk samples. We show that doping with magnesium very efficiently compensates shallow donor centers present in GaN. The observed effect is by far more efficient than expected from the comparison of concentrations of donor and acceptor centers introduced to a sample. Present study demonstrates a significant enhancement of potential fluctuations in doped samples. We evaluate the influence of p-type doping on these potential fluctuations from Kelvin probe measurements. Large- and small-scale light emission fluctuations are demonstrated. Micro-photoluminescence (PL) study indicates an unusual anti-correlation between the intensities of excitonic and defect-related emission processes in p-type doped structures and also the presence of so-called hot PL. Hot PL was observed only in the case of strongly compensated p-type samples, which we relate to strong potential fluctuations in these samples. We discuss possible mechanisms of compensation processes based on the results of our recent optically detected magnetic resonance investigations.

This work was partly supported by the grant numbers 5 P03B 007 20, 5 P03B 123 21 of KBN and by the DENIS program of European Union (G5RD-CT-2001-00566).

Study of Long-Term Stability of Ohmic Contacts to GaN

19:00 -

Krystyna Golaszewska², **Eliana Kaminska²**, **Anna Piotrowska²**, **Renata Kruszka²**, **Andrian Kuchuk²**, **Ewa Papis²**, **Roman Szeloch¹**, **Pawel Janus¹**, **Teodor Gotszalk¹**, **Adam Barcz^{2,3}**, **Andrzej Wawro³**

poster

A-13

- 1) Wrocław University of Technology, Wybrzeże Wyspiańskiego, Wrocław 50-370, Poland
 2) Institute of Electron Technology (ITE), al. Lotników 32/46, Warsaw 02-668, Poland
 3) Institute of Physics PAS (IF PAN), Warsaw, Poland

In view of the success of GaN-based photonic and electronic devices we have performed a study of long-term stability of ohmic contacts to GaN. The specific focus of the work was on thermally stable metallizations using transition metal nitrides (ZrN, NbN, TiN). Because of their high electrical conductivity and thermal stability these compounds are materials of choice for diffusion barriers in metallization systems. Moreover, transition metals have been shown effective in providing low-resistivity contacts to n-type GaN (by depleting its superficial film for nitrogen and creating thus a highly doped subcontact region) and p-type GaN (by extracting hydrogen from the subcontact region). Our studies involved accelerated lifetime testing and thermal conductivity studies of metal/GaN ohmic contacts. The approach adopted for aging studies was based on the following premises. First, the degradation of an ohmic contact during the operation of the device is most likely caused by microstructural changes within the contact region. Therefore, the main issue in assessing the long-term stability was to identify the key interfacial transformation for good ohmic behaviour, as well as "the weakest point" of the contact microstructure. Aging was performed by means of annealing up to 150°C over a period of 300 hours. Thermal conductivity measurements were made using a home-built scanning thermal microscope, enabling to couple the topographic image with the thermal conductivity information.

A correlation between aging characteristics and thermal conductivity measurements has been established. The difference between GaN and metallization conductivity of 6 - 10 W/m/K for the most reliable ohmic contacts has been found. These contacts were characterised by featureless surface morphology after lifetime test.

Work partially supported by grants from European Commission G5RD-CT-2001-00566-DENIS and the State Committee for Scientific Research 7T11B 009 20.

19:00 - poster A-14 **The interaction of Pr₂O₃ with 4H-SiC(0001) surfaces**
Andriy Goryachko¹, Ioanna Paloumpa¹, Guido Beuckert¹, Karsten Henkel¹, Patrick Hoffmann¹, Dieter Schmeißer¹, Yevgen Burkov¹

1) Brandenburg Technical University, Department of Applied Physics/Sensorics (BTU), Erich Weinert Str. 1, Cottbus 03044, Germany

We prepare thin films (<3nm) of Pr₂O₃ films on 4H-SiC(0001) surfaces by a wet chemical process involving aqueous solutions of Pr(NO₃)₄. We use surfaces covered by a native oxide and such with the oxide being removed by HF dip. We determine the roughness of these films by AFM studies and their chemical composition by XPS measurements. Synchrotron radiation photoelectron spectroscopy (SR-PES) is used to study the core levels in more detail. We report on the thickness dependent variations in the O1s core level and determine the relative abundance of graphitic carbon in the interface regime from the C1s emission. The Si2p core level emission is analysed in detail and reveals that a silicate is formed at the interface and the thickness of that interfacial layer is determined. Our data are compared to corresponding studies of the Pr₂O₃ interaction with Si(100) surfaces /1,2/.

/1/ H.J.Osten, J.P.Liu, H.J.Muessig, Appl.Phys.Lett. 80 (2002) 297.

/2/ D. Schmeisser, Comp.Materials Science, in press.

19:00 - poster A-15 **Fabrication and characterization of SiC/Si heterojunction diode.**
Taupin Héloïse¹, Philippe MARIE¹, Laurent PICHON², Richard RIZK¹

1) Laboratoire d'Etudes et de Recherches sur les MATériaux (LERMAT), bd Maréchal Juin, Caen 14050, France, Métropolitain

2) Groupe de Recherche en Informatique, Image, Instrumentation de Caen (GREYC), bd Maréchal Juin, caen, France, Métropolitain

Thin silicon carbide films have been obtained by reactive magnetron sputtering at various temperatures under hydrogen rich plasma. Infrared absorption spectroscopy measurements reveal a crystalline fraction that increases from about 40% to 80% when the deposition temperature is increased from 300 to 600°C. X-ray diffraction measurements show evidence of SiC nanocrystals embedded in an amorphous matrix. Moreover, preliminary dark conductivity measurements show a transport property behavior that appears quite compatible with the evolution of the structural features. Finally, SiC/Si heterojunctions based on Sb-doped SiC layers were fabricated and characterized, showing a relatively high rectification behavior together with a low leakage current.

19:00 - poster A-16 **Self-Assembly Synthesis of Hexagonal Boron Nitride Buckling Nanowires at Room Temperature**
Ming-Chan Lu², Hsi-Lien Hsiao¹, Po-Wen Lin¹, An-Ban Yang¹, L-J Chen²

- 1) Department of Physics, Tunghai University (THU), P.O. Box 803, Tunghai University, Taichung 407, Taiwan, Province of China
- 2) Department of Materials and Science, National Tsing Hua University, Hsinchu, Taiwan, Province of China

Hexagonal phase boron nitride buckling nanowires were successfully synthesized even at room temperature by electron-cyclotron-resonance chemical vapor deposition technique through the upstream Ar plasma cracking of nitrogen, hydrogen and diborane mixed gases. The self-assembled nanowires exhibit peculiar sponge-like shape in the scanning electron microscope images. High resolution transmission electron microscope images demonstrate that the growth axis of nanowires is perpendicular to [0002] direction. It is believed that these hexagonal phase boron nitride nanowires are nucleated and self-assembled in the gas phase. The length of these buckling nanowires are observed to range from 100nm to 200nm. Cross-sectional transmission electron microscope images indicate that these nanowires tend to lay on the substrate. In the meantime, spherical amorphous boron nanoparticles with diameters ranging from 40nm to 150nm were also observed in the samples. Moreover, intense UV light emission peaked at 310nm (4eV) was observed while the samples illuminated by 266nm laser beam. Synchrotron radiation excitation luminescence measurements reveal sharp 201nm (6eV) absorption band dominate this strong light emission. This observation indicated that the 4eV UV luminescence seems come from the deep levels transitions and possibly corresponding to the high density dislocations exist in the buckling nanowires structures.

Optical and magnetic resonance investigations of ZnO crystals doped with transition metal ions

19:00 -

Vitalii Y. Ivanov¹⁾, Marek Godlewski^{1,3)}, S. Yatsunenکو¹⁾, A. Khachapuridze¹⁾, Z. Golacki¹⁾, Maciej Sawicki¹⁾, A. Omel'chuk²⁾, M. Bulany⁴⁾, A. Gorban'⁴⁾

poster

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

A-17

2) Institute of Semiconductor Physics (ISP), Nauki pr., Kiev 03028, Ukraine

3) College of Science, Cardinal S. Wyszyński University, Warsaw, Poland

4) Dnepropetrovsk National University, Dnepropetrovsk, Ukraine

In this communication we present recent results of photoluminescence (PL), photo-ESR and optically detected magnetic resonance (ODMR) studies, performed on series of crystals: ZnO, ZnO:Mn, ZnO:Co, ZnO:Fe, ZnO:Ti, ZnO:Cr and ZnO:Cu. All samples were grown by chemical vapor transport technique and were doped with transition metal impurities into the melt.

We focused on the comparison of optical and magnetic properties of various crystals with different composition and doping level. In particular we discuss influence of doping with transition metal impurities on PL and magnetic resonance spectra. We found that two of the TM ions studied (Cr and Co) we found that TM is present in two charge states: 2+ and 3+, respectively.. Both impurities can be recharged with light of appropriate energy and such recharging processes we monitored by observing their in ESR signals.

Moreover, we found that ZnO:Mn crystals with Mn fraction up to 5% exhibit a weak ferromagnetic behavior at low temperatures.

Optical and ODMR study of GaN-based HEMT structures

19:00 -

Vitalii Y. Ivanov¹⁾, Marek Godlewski^{1,2)}, A. Khachapuridze¹⁾, S. Yatsunenکو¹⁾, Yu. Sveshnikov³⁾, A. Arendarenko³⁾

poster

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2) College of Science, Cardinal S. Wyszyński University, Warsaw, Poland

A-18

3) ELMA-Malachit, Moscow, Russian Federation

Processes of carrier localization, their scattering and trapping at impurities, but also at localization states, are very important for understanding transport mechanisms in III-nitrides based device oriented structures. In this communication we discuss results of PL and ODMR studies of GaN/AlGaIn HEMT structures. We studied undoped and Si-doped GaN and AlGaIn layers grown by MOCVD in (0001) orientated sapphire substrates. We start with discussion of PL spectra and then correlate their properties with growth conditions. For example, a parasitic yellow emission is observed in the samples grown with 30 nm GaN nucleation layer, containing high dislocation density.

We also found two new magnetic resonance signals in the ODMR spectra with g factors equal about 1.7 and 0.9. Their possible origin is proposed.

Results of the ODMR and PL investigations are compared with electrical measurements.

Impact of post-growth thermal annealing on emission of InGaIn/GaN multiple quantum wells

19:00 -

Saulius Jursenas¹⁾, Yung-Chen Cheng²⁾, Shih-Wei Feng²⁾, C.C. Yang²⁾, Cheng-Ta Kuo³⁾, Jian-

poster

A-19

Shihn Tsang³⁾

1) Institute of Materials Science and Applied research (IMSAR), Sauletekio al. 9, Vilnius 2040, Lithuania

2) Graduate Institute of Electro-Optical Engineering and Department of Electrical Engineering, National Taiwan University 1, Roosevelt Road, Sec. 4, Taipei, Taiwan, Province of China

3) Advanced Epitaxy Technology Inc., Hsinchu Industrial Park, Hsinchu, Hsinchu, Taiwan, Province of China

InGaN-based multiple quantum wells (MQWs) are the key structures for high efficiency, long lifetime violet, blue, and green light emitting diodes and laser diodes. During the epitaxial growth of InGaN/GaN structures and during the fabrication of devices, active layers undergo several high-temperature treatments. The distribution of the indium composition and the strain in quantum wells may be changed due to spinodal decomposition and interdiffusion of indium and gallium across the interface of quantum well and barrier after these thermal treatments. This can lead to a change of emission properties of MQWs.

Here we present results on temperature-dependent site-selective photoluminescence, photoluminescence excitation and time-resolved luminescence study of three In_{0.15}Ga_{0.85}N/GaN MQWs with well-widths of 2, 3, 4-nm, upon thermal annealing at 800 °C for 30 min. Our results imply on nontrivial dependence of MQWs emission properties upon thermal annealing. Blueshift of luminescence and pronounced changes in the absorbance are attributed to domination of spinodal decomposition for the thin wells. While pronounced redshift in luminescence and increase in the excitation lifetime is attributed to prevalence of strain-induced field-effect for the thicker MQWs. These changes are related to the enhancement of indium segregation and rise in the built-in electric field effect of as-grown samples of various thicknesses.

19:00 -

poster

Nanotechnology methods for wide forbidden zone semiconductors (boron, carbon, their compounds) preparation.

P Kervalishvili

A-20

The one of the main goals of nanotechnology is to take the building blocks such as atoms, molecules or their clusters, understanding their properties and build new materials - films, layers and ingots, new tools and devices.

The high-temperature semiconductors with the wide forbidden zone are one of the most preferable materials for modern nano technology-based electronics. Among them the main role can play the very interesting and important materials based on boron and carbon as well as their compounds. These complicate substances with unique structural properties and difficult technology for their film's preparation are promising because of their electric and physical properties as well as mechanical hardness and chemical resistance. According to several research works carbon and boron crystals are building of clusters, the essential structural elements containing 4, 12, 60, and 84 atoms. These nanoelements due to necessary thermodynamic conditions are transforming to amorphous or crystalline films, layers and other deposits, which can have some advanced properties.

Following to classical ideas of particles formation and growth and in correspondence with the so-called atomistic process of conception, atoms being the germs of the solid phase unite in aggregates (molecules, clusters) with atoms quantity depending on atoms potentials. Using the established or last years-new approach to the mechanism of cluster's formation it is easy to show that during the formation of small particles the appearance of small particles analogous to so-called fractal clusters very often takes place. Such volume clusters consisting of separate clusters of lesser dimensions have much more low density than the matrix substance. Formation of semiconductor materials using the clusters based growing mechanisms actually is carried out by experimental methods, among which it should be noted: supersonic outflow of vapors into vacuum, thermo-, laser- and plas

19:00 -

poster

Investigation of thermo- and photoluminescence in heterostructures PbS/ZnTe

Halyna Khlyap¹⁾, V. Blashkiv¹⁾, L. Bochkariova²⁾

1) State Pedagogical University, 24 Franko str., Drohobych, 82100, Ukraine, Ukraine

2) Jaroslavl State University, 14 Soviet str., Jaroslavl, 150014, Russia, Russian Federation

A-21

The abstract reports first results of studying thermoluminescent (TL) properties of heterojunctions n-PbS/p-ZnTe assembled for designing cooled focal plane arrays and active elements operating in near-IR spectral range. Heterostructures were prepared by molecular-beam epitaxy of lead sulfide films on monocrystalline (110)-ZnTe wafers. Electrical measurements performed in wide temperature range (77 - 300 K) were demonstrated the sufficient effect of 2D space-charge region localized at the interface. TL was excited by samples enlightening from the side of lead sulfide film as well as from the substrate side by 550 nm and 700 nm light sources and N₂-laser. The data obtained show that the energy depth of radiation centers is 0.31 - 0.49 eV. Photoluminescence (PL) was excited by UV-light source from PbS film side. PL spectral distribution was presented by gaussian-like curve with maximum localized in 500 - 620 nm range.

19:00 -

poster

Advanced transparent beryllium ceramics for ionizing radiation dosimetry

Victor S. Kijko¹⁾, Sergei V. Gladkovsky¹⁾, Ivan A. Dmitriev¹⁾, Andrei A. Sofronov¹⁾, Yuri N. Makurin¹⁾, Alexander L. Ivanovsky²⁾

A-22

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2) *Institute of Solid State Chemistry of the Urals Branch of RAS (ISSC), Pervomaiyskay, 91, Ekaterinburg 620219, Russian Federation*

At present BeO ceramics due to the unique complex a physicist-chemical characteristics are broadly used in radio-electronics, gas lasers production, as well as in ionizing radiation dosimetry. In connection with that the development of production technology of fine-grained transparent BeO ceramics, possessing high quantum luminescence output and relatively small flashing period, acquires particular urgency.

Samples of transparent BeO-based ceramics were produced by high temperature vacuum pressing at $T=1520$ K and the value of pressure 30 MPa during 15-20 minutes up to slump completion. The density of samples formed 3,01-3,02 g/cm³ and was close to theoretical. Lithium carbonate (0,4-1,0 wt.), boron oxides (0,01-0,5 wt.%) and rare-earth elements (REE) were used as additives for ceramics characteristics improvement.

It is shown that boron oxides doping gives a positive influence on microstructure and optical features of transparent beryllium ceramics. REE admixtures promote a quantum luminescence efficiency raise because of additional account of oxygen vacancies formation.

It is stated that studied ceramics possess high sensitivity to ionizing radiation and maintains transparency in the broad spectral range $\lambda = 0,1 - 9,0 \mu\text{m}$). Thereby, conducted studies have shown high efficiency of transparent BeO ceramics for ionizing radiation dosimetry usage.

The role of admixture phase in raising of 4,9 eV luminescence intensity peak attributed to the formation of additional optically active centres (oxygen vacancies) on BeO microcrystals surface and bringing about the formation of F-centres is determined.

The presented investigation was carried out under the support of RFBR of Russian Academy of Science (project # 1-03-96430) and federal "Integration" program (project IO965).

Room temperature growth of c-axis oriented ZnO films on various substrates

Hyoun Woo Kim¹, Nam Ho Kim¹

1) *Inha University, School of Materials science and engineering, 253 Yong Hyun Dong, Nam Ku, Incheon 402-751, Korea, Republic of*

19:00 -

poster

We have demonstrated the growth of ZnO thin films with c-axis (002) orientation at room temperature on Si(100), SiO₂, and amorphous ZnO substrates by the radio frequency (RF) magnetron sputtering method. We investigated the structural properties of ZnO thin films deposited on various substrates with varying the RF power. X-ray diffraction analysis revealed that increasing the RF power helped to decrease the compression stress and to increase the grain size, regardless of the substrate material. Scanning electron microscopy indicated that the structural morphology of the ZnO films were not dependent on the substrate material.

A-23

Electrical properties the five-component chalcogenides of silver

Olga L. Kobeleva¹, Viktor B. Zlokazov¹, Nina V. Melnikova¹, Larisa L. Nugayeva¹, Leonid Y. Kobelev¹, Yakov L. Kobelev²

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2) *Institute of Metal Physics, Ural Division of Russian Academy of Sciences, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation*

19:00 -

poster

A-24

The development of modern crio-electronic requires the creation of new the semiconductors materials, including materials, which to combine the superionic properties at low temperatures with other properties (optical, magnetic et al). In USU the five-component chalcogenides AgGePbS_{3x}Se_{3(1-x)} (x=0.3-0.7) were synthesised and their electrical properties by a method impedance spectroscopy are investigated. The compounds with x=0.4-0.7 have grey colour and metal shine, compound with x=0.3 have grey dim colour.

Godographs of an impedance are characterised by presence of two precisely divided areas - low-frequency (electrode processes) and high-frequency (processes in volume of a sample). Researches were carried out on frequency 1592Hz, which, as follows from the impedance diagrams, belongs to HF, in the field of temperatures 78K-400K. Temperature de-pendencies of conductivity the synthesised compounds have a Arrhenius type, that is characteristic for ionic semiconductors.

For x=0.5 the conductivity at T=300K is equal 7.6 mS/m. The conductivity of compounds with x=0.4-0.7 on 1-2 order is less, than for compounds which containing As instead of Pb, is comparable to conductivity AgGeSbS₃ and on some orders less, than at AgGeSbSe₃. For x=0.3 the conductivity is comparable on size to conductivity AgGeSbSe₃. In compounds with x=0.7 at T~420K is observed piroelectric phase transition. All connections are the mixed ionic-electronic conductors with temperature of began the ionic of carry 220K-300K, the share ionic conductivity depends from x (50-99.5%).

It is possible, that other ratio of sulphur and selenium in compounds will result in the further growth of conductivity and downturn of temperatures of a beginning the ionic of carry. Also it will be interesting to investigate copper-containing chalcogenides.

The research was made possible in part by Award CRDF (No. REC-005).

Diffusion and diffusion induced defects in GaN

19:00 - poster
A-25 **Dmytro Kolesnikov¹⁾, Witold Lojkowski¹⁾, Christian Jäger³⁾, Wolfgang Jäger³⁾, M. Bockowski¹⁾, Rafal Jakiela²⁾**

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

2) *Institute of Physics Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw, Poland*

3) *Technische Fakultät Christian-Albrechts-Universität Kiel, Kaiserstr. 2, Kiel D-24143, Germany*

GaN and its solid solution with InN and AlN have become very popular for the last 10 years. It is connected with the possibility of using it in the optoelectronics. Diffusion processes in GaN play a very important role. Annealing at high temperatures is useful for many technological processes such as removing of defects after implantation, doping or electrical contact activation. The process of laser degradation is connected with diffusion of impurities. This process depends strongly on structural quality of the epitaxial layers.

The aim of this work was to obtain the first diffusion coefficients for different elements in GaN at the temperatures higher than decomposition temperature of GaN and to understand the mechanism of mass transport in this material.

It was found that the diffusion of impurities brings the V-shape defects in GaN. The mechanism of diffusion and defects formation in GaN was proposed. The diffusion coefficients and the activation energies for Mg, Zn, Ni and Au diffusion in GaN were calculated.

Optical characterization of GaN doping superlattices as grown, hydrogen implanted and annealed.

19:00 - poster
A-26 **Herbert Willi Kunert²⁾, D J. Brink²⁾, M. Hayes²⁾, J. Malherbe²⁾, L. Prinsloo³⁾, J. Barnas¹⁾**

1) *Institute of Physics, Adam Mickiewicz University, Umultowska 85, Poznań 61-614, Poland*

2) *Department of Physics, University of Pretoria., University str., PRETORIA 0084, South Africa*

3) *Department of Chemistry, University of Pretoria, Pretoria 0001, South Africa*

GaN doping superlattices (DSL's) were grown epitaxially (MBE) with eight periods of n-GaN/p-GaN (50nm/50nm) doped: $1 \times 10^{18} \text{cm}^{-3}$ (n-Si) and $1 \times 10^{18} \text{cm}^{-3}$ (p-Mg) with a thin AlN nucleation layer (20nm) on a 2-inch single-side-polished sapphire (0001) wafer. Low temperature photoluminescence and Raman spectroscopy (PL), (RS)-respectively were used to characterize the as grown and hydrogen implanted (dose = $10^{12} - 10^{17} \text{cm}^{-2}$, Energy 1MeV), samples. From the as grown sample the PL yields the well known DAP's band. In addition, in the region 2.1-2.8 eV (where normally the YL band is observed in bulk Mg-doped GaN) we monitor several mini, well-resolved separate bands these do not resemble the YL band. Temperature and laser power dependence do not affect the energy position of these peaks. Implantation decreases the intensities of most bands but does not induce new optically active transitions. Annealing at 900C reveals a broad solid unstructured YL band centred at 2.3 eV.

In bulk GaN there are six allowed Raman active modes. In the GaN DSL's reference sample we observe vibrational modes at 428cm^{-1} (sapphire substrate mode), 572cm^{-1} -E₂(high) - like which normally is centred at 572cm^{-1} , and a mode at 738cm^{-1} which originate from the A₁(LO)- 735cm^{-1} bulk GaN. Implantation at low doses shifts the modes slightly while higher doses influence mostly the second order vibrational modes. We observe two implantation enhanced modes at 1400 and 1440cm^{-1} . The half-width of the strongest mode at 572cm^{-1} increases with implantation dose indicating structural damage.

Applying the Ploog-Dohler approach to the GaN DSL's, the quantum mechanical calculations on the lifetime of carriers yields a large value (compared with GaAs DSL's). Mechanisms for the origin of the annealing-induced YL band and the observation of eight mini bands are discussed. Raman selection rules for the second order modes in the GaN DSL's are discussed.

Morphology and structural perfection of vapor-grown PbI₂ crystals

19:00 - poster
A-27 **I. V. Kurilo¹⁾, O. V. Rybak¹⁾**

1) *Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine*

PbI₂ single crystals have considerable potential for use in optical information recording systems, nonlinear optical devices, and x-ray and gamma detectors. The purpose of this work was to assess the effect of growth conditions on the habit of PbI₂ grown from the vapor phase in the presence of excess iodine. The growth conditions of PbI₂ crystals were optimized using thermodynamic analysis of the equilibrium vapor-phase composition in the Pb-I₂ system and theoretical and experimental studies of mass transport: source temperature, 750-800 K; deposition temperature, 630-660 K; iodine overpressure, 4-10 kPa; growth time 2-4 h. The structural perfection of the crystals was examined by optical and SEM techniques and x-ray diffraction. By varying growth conditions, platelike, ribbon, needle, twinned, and dendritic crystals were prepared, as well as combinations and intergrowths of these habits. In most cases, under optimal growth conditions, we obtained platelike or ribbon crystals. The growth habit was responsive to the thermal conditions, which determine the stoichiometry of the growing crystal. Typically, the platelets and ribbons had the same orientations. It is believed that, in vapor growth, platelike morphology is due to the lateral growth of needle crystals, acting as growth leaders. Platelike crystals may result not only from

the vapor-liquid-solid mechanism but also from layer-by-layer growth by the vapor-solid mechanism. The observed twinning may be due to the significant supersaturations during growth, thermal stress, and second-phase inclusions. The amount of twinning was found to rise with increasing Pbl2 vapor pressure.

Energy transfer to Er³⁺ ions in silicon-rich-silicon-oxide: efficiency limitations.

19:00 -

Dmitry Kuritsyn¹⁾, A Kozanecki¹⁾, H. Przybylinska¹⁾, W Jantsch²⁾

poster

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2) *Institut für Halbleiterphysik Universität Linz, Altenberger Str. 69, Linz A-4040, Austria*

A-28

A detailed investigation of the excitation mechanisms of the ⁴I_{13/2}-⁴I_{15/2} intra-4f-shell luminescence of Er³⁺ ions near 1.5 μm in silicon-rich silicon oxide (SRSO) is reported. SRSO:Er was produced by high dose implantation of Si⁺ ions into SiO₂ layers grown on silicon, followed by Er⁺ implantation at an energy of 800 keV. Samples were thermally annealed up to 1100°C to create different types of defects and Si-nanocrystals (nc-Si). Photoluminescence excitation (PLE) spectra were obtained using optical parametric oscillator (OPO) and Ti:Sapphire laser in the range 420-680 and 720-830 nm, respectively.

The results show that in SRSO:Er two mechanisms of excitation are in competition: a resonant one and via defects. The resonant channel of excitation of Er³⁺ 4f-shell which gives a maximum luminescence yield for Er doped silica without additional Si is suppressed by the silicon excess related defects; resonant peaks disappear with increasing of annealing temperature of SRSO. We suggest that the dominant factors, which limit the excitation efficiency of Er³⁺ in SRSO, are distance dependence of the transfer rate and little spectral overlap of the interacting states. Employing density of states function for nc-Si, Er³⁺ absorption function and spectral overlap integral, the quantitative estimation of overall efficiency of the system nc-Si:Er³⁺ for multipole interaction between localized in nc-Si (or nc-Si/SiO₂ interface) e-h pairs and 4f-states of Er³⁺ is presented. The dependence of the Er³⁺ emission on excitation duration were also studied. For one electron-hole pair being generated by excitation pulse we show that one nc-Si can excite only one or less of Er³⁺ ions in its vicinity.

Studies on sub-band gap absorption in AlGaIn photoconductors and solar-blind photodetectors

19:00 -

Vadim B. Lebedev¹⁾, Irina Cimalla¹⁾, Oliver Ambacher¹⁾

poster

1) *TU-Ilmenau, Institut für Festkörperelektronik, FG Nanotechnologie, Gustav-Kirchhoff-Str. 1, PF 100565, Ilmenau 98684, Germany*

A-29

A large variation in the direct band gap of wurtzite AlGaIn alloys is very suitable for light detecting devices which are sensitive in the 220 - 365 nm spectral range and have an ability to detect a signal in a narrow spectral band with photon energies close to a high background radiation.

In this work we present studies on AlGaIn photoconductors and high performance AlGaIn photodetectors based on a combination of three epitaxial AlGaIn layers with different alloy composition acting as optical filter, isolator, and detector layer, respectively. A peak responsivity for this device has been achieved at 280 nm with a range of sensitivity between 240 and 300 nm. An AlN layer has been used as an isolating barrier between the AlGaIn optical filter and the AlGaIn photodetector top layer.

Samples were grown by plasma assisted molecular beam epitaxy on c-plane sapphire substrates. For the accurate measurement of the sub-band gap absorption, spectral photocurrent measurement (SPC) and photothermal deflection spectroscopy (PDS) have been applied to characterise the defect structure of the epilayers. The advantage of CPM and PDS, in comparison to other optical techniques (particularly transmission) is their high sensitivity down to $\alpha \sim 10^{-7}$, where α is the absorption coefficient and d the thickness of the film. Process dependent variations in direct band-gap energies, Urbach tail widths, bulk and surface defect densities in the films are provided as a measure of composition and structural quality of epitaxial layers. A difference of more than one order of magnitude in the SPC above the band gap has been measured in back- and top-illuminated UV-photodetector heterostructures demonstrating the functionality of the integrated filter (cut-off energy ~ 4.85 eV). The UV/visible contrast of more than four orders of magnitude and in addition a difference in the PDS and SPS absorption spectra of more than one order of magnitude has been observed.

Growth and Characterisation of AgIn₅S₈ and CuIn₅S₈ Thin Films

19:00 -

Liudmila V. Makhova¹⁾, Igor Kononov¹⁾, Rüdiger Szargan¹⁾

poster

1) *Willhelm-Ostwald-Institut für Physikalische und Theoretische Chemie, Universität Leipzig. (WOI), Linnestr.2, Leipzig 04103, Germany*

Spinel CuIn₅S₈ and AgIn₅S₈ materials are perspective for application in optoelectronic semiconductor devices. For the first time, thin films of these compounds were grown and investigated. The films were deposited by sequential process on Mo-coated soda lime glass. This process consist of sulfur vapor sulfurization of a metallic alloy precursor. We found that good adhesion to the substrate (Mo-coated glass) could be achieved by introducing a Ti layer between the substrate and the spinel layer. We observed a good electrical conductivity through this contact. From analysis of the XPS data, the information about the interface spinel/Ti and composition of the

A-30

surface was gained. The influence of preparation conditions, namely of the phase composition of the precursor alloy, of the sulfurization temperature and time on the stoichiometry in the bulk and at the surface of the spinel layers have been investigated by XRD, XRFA and XPS methods. Moderate roughness of the films and a crystallite size of about 300-400 nm were observed in the AFM images.

19:00 - **Growth and Radiation Induced Color Centers in Oxide Laser Crystals**
poster **A. Matkovskii^{2,4)}, D. Sugak²⁾, P. Potera⁴⁾, A. Suchocki¹⁾, Ya. Zhydachevskii²⁾, A. Durygin¹⁾, A. Shakhov³⁾**

A-31

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4) *Institute of Physics, University of Rzeszow, Rzeszow, Poland*

The present work contains results on study of color centers in YAlO_3 (YAP) and $\text{Gd}_3\text{Ga}_5\text{O}_{12}$ (GGG) crystals both pure and activated with various dopants. By the origin the color centers in the crystals studied can be divided on growth CC (observed in as-grown crystals) and induced CC (created in crystals as a result of temperature treatments or irradiation).

The growth coloration of GGG crystals is caused by $[\text{V}_\text{O}\text{V}_{\text{Ga}}]^-$ complexes and Fe^{3+} impurity (absorption near 37000 cm^{-1}), complex centers $[\text{CaF}^+]$, $[\text{CaO}^-]$ and $[\text{Gd}_{\text{octa}}\text{O}^-]$ (absorption near 29000 cm^{-1}) as well as F-centers (23000 cm^{-1}).

The growth coloration of YAP crystals (wide absorption band in the $48000\text{--}20000\text{ cm}^{-1}$ range with several maxima) is caused by color centers formed as a result of deviation of crystal composition from the stoichiometric one ($R=\text{Y}_2\text{O}_3/\text{Al}_2\text{O}_3 > 1$) and presence of uncontrolled impurities such as Ca, Mg and Fe.

By the time stability at room temperature the induced CC can be divided on stable and transient CC. By the mechanism of formation they can be divided on ionization and radiation displacement ones. The stable coloration is caused by recharging of growth CC present in the crystals as well as formation of the radiation displacement defects by the impact mechanism.

The stable CC in the crystals studied can be O^- centers localized near defects in cation sublattice, F-centers and $[\text{V}_\text{O}\text{V}_\text{Ga}]^-$ complexes. The transient CC can be F^+ -centers and self-trapped O^- centers that absorb near 15000 cm^{-1} . Influence of activator ions on the processes of CC formation is analyzed.

GGG:Nd active media are more radiation resistant in comparison with YAP:Nd crystals, therefore they and can be used in solid-state lasers for space applications.

19:00 - **Stimulated emission in InGaN/GaN structures with different quantum well width**

poster **Saulius Miasojedovas¹⁾, Saulius Jursenas¹⁾, Genadij Kurilčik¹⁾, Arturas Zukauskas¹⁾, Shih-Wei Feng²⁾, Yung-Chen Cheng²⁾, C.C. Yang²⁾, Cheng-Ta Kuo³⁾, Jian-Shih Tsang³⁾**

A-32

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2) *Graduate Institute of Electro-Optical Engineering and Department of Electrical Engineering, National Taiwan University 1, Roosevelt Road, Sec. 4, Taipei, Taiwan, Province of China*

3) *Advanced Epitaxy Technology Inc., Hsinchu Industrial Park, Hsinchu, Hsinchu, Taiwan, Province of China*

Light-emitting and laser diodes for green to near-UV region are based on quantum structures with InGaN active region. However, characteristics of devices with multiple quantum wells (MQWs) are not clear so far. One of the most important characteristics of MQWs is the well width. With increasing the well width the emission from MQWs is affected not only by quantum confinement, but by intricate processes due to spinodal decomposition of In and built-in electrical field as well. To maximise spontaneous and stimulated emission, underlying physics should be cleared out and the growth conditions and structure of the MQWs are to be optimised.

Here we present results on high-excitation luminescence spectroscopy in $\text{In}_{0.15}\text{Ga}_{0.85}\text{N}/\text{GaN}$ MQWs with various well width. High excitation conditions results in screening of built-in electrical field by free carriers and allowed us to determine the influence of In segregation on optical properties. Optimal quantum well thickness for InGaN/GaN lasing structures is obtained for the well width $d = 3\text{ nm}$. With increasing the well thickness, indium-rich clusters increase in size that results in an occurrence of deeper localization states with longer depopulation time and conventional degradation of the material. Meanwhile in thin wells, increased role of the interfaces states results in enhanced nonradiative recombination.

19:00 - **1.54 μm Luminescence quenching in erbium-doped hydrogenated amorphous silicon deposited by D.C. Magnetron Sputtering**

poster **KECHOUANE Mohamed²⁾, MOUHEB Ouiza²⁾, BIGGEMAN Daniel¹⁾, TESSLER Leonardo¹⁾**

A-33

- 1) Instituto de Fisica "Gleb Wataghin", UNICAMP, C. P. 6165, 13083-970 Campinas, SP, Brazil, Campinas 6165, Brazil
- 2) Laboratoire "Couches Minces et Semiconducteurs" - U.S.T.H.B. - Faculté de Physique - BP 32 16111 El-Alia - Bab-Ezzouar - Alger, BP 32 El-Alia - Bab-Ezzouar, Alger 16111, Algeria

Erbium was doped hydrogenated amorphous silicon (a-Si:H) using D.C. magnetron sputtering from a silicon target partially covered by tiny metallic erbium chunks. This doping technique allows high doping levels of erbium concentration (1%) which is two orders of magnitude higher than in ion implanted crystalline silicon. After annealing under oxygen atmosphere at 350 °C, a-Si:H<Er> films with variable optical gap (1.14 to 2 eV) exhibits efficient room temperature at 1538 nm. The PL intensity was found to decrease with the decrease of the optical gap of the a-Si:H<Er> film.

The observed PL lines at 1549.6, 1538 and 1529.7 nm have quite similar temperature dependence, indicating that both are related to the same erbium site. The Intensity of these PL lines at room temperature was 6, 15 and 12%, respectively, of that at 15K. This result suggest that a-Si:H is a suitable host for efficient Er³⁺ emission at room temperature.

a-Si:H has an erbium solubility higher than 10²⁰ cm⁻³ and a greatly reduced thermal quenching of the erbium PL due to a less efficient excitation energy transfer. The erbium luminescence is encouraged by the existence of an insulating environment around the erbium, due to the presence of oxygen into a-Si:H films. Oxygen is known to enhance the PL by increasing the number of radiative Er³⁺ optical centers and to produce an increase in the band gap.

The study of the evolution of the PL intensity, according to the excitation power of the laser, permits to reveals the existence of two distinctly excitation mechanisms in erbium implanted and in erbium doped a-Si:H.

In order to characterize the change in a-Si:H structure, we are interested to the effects induced by the thermal annealing used in order to activate the Er³⁺ optical centers. We found that the increase in the optical gap and the electrical resistivity of the a-Si:H<Er> films produced at low temperature substrate is more important than that deposited at 240 °C.

[ABS

[ABSTRACT TRUNCATED TO 2000 LETTERS]

Low dimension porous silicon based heterostructures for possible optoelectronic and accessory applications

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Active films coatings on porous silicon (PS) surface with semiconductors and dielectrics such as oxide (Al₂O₃, SiO_x), chalcogenide materials (ZnS, CdTe, GaSe), polymer films were created. Possibility of optocouple creation for the wide spectral range (as light emitter and detector) using PS was considered. Photoluminescence, electroluminescence, photosensitivity of porous silicon were investigated. Also it was used such additional methods as photo-TSD, photo- and cathodoluminescence, photovoltaic effect, volt-current characteristics method.

Investigated structures on the PS base have been possessed in the noticed photovoltaic sensitivity in the range of 1-3 eV and have been sensitive to the IR- and X-rays at the TSDC regime in the range of 77- 450 K.

Current-voltage characteristic investigation of single and double heterostructures were showed its magnificent sensitivity to the mechanical strains. We have investigated dependencies of the relative current change which depend on the pressure unit from tension sets on the heterostructure.

So it was indicate that obtained PS-silicon heterostructures possess photovoltaic properties in the wide spectral range. All these structures were possessed light-emitting properties under laser beam exciting and electron beam. Accessory application porous silicon heterostructures may be connect with sensitivity to mechanical strains.

19:00 -

poster

A-34

Local complex emission centers in the wide band gap materials: theoretical predictions and experimental diagnostic possibilities

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The paper concerns mainly to the statement of the study of a role of complex defects to determine emission (luminescence) properties of the oxide wideband gap optical materials.

Luminescence centers (LC) of materials constructed by cation and anion sublattice with the last formed by molecular groups are considered as containing both the "core" of the center and the neighborhood elements of the crystal matrix. The "core" can be simple, one centered, or two centered. The first is either admixture atom, molecule (charged or not) or own defect of matrix. The second is complex center that combines the simple "core" that determines principal characteristics of electron-vibronic transitions of the LC and some either own defects of matrix or admixtures. The interaction between two components of complex center determines the spectral - kinetic characteristics of the complex centers on the whole.

The electronic system of the complex LC is united, that especially concerns of the excited electronic states, besides it should to be considered as non-adiabatic system which is described in the frames of Jahn Teller effect (conjugated electron-vibronic states). It is particularly important when the "core" of LC is a molecule. These centers were formerly called by us as "ion-polaron" pairs.

19:00 -

poster

A-35

These properties result in a sensitivity of the LC to changing of structure, composition, presence of impurities and defects, temperature and other external factors.

The paper draws attention to experimental observation of mentioned properties for some complex oxide materials (chromates, phosphates, tungstates,) via:

- Zero-phonon lines (ZPL) behaviors;
- Temperature dependencies of luminescence kinetics;
- Local phase transitions;
- Formation of microcrystalline inclusions on the basis of "own" chemical components

19:00 -

GaN related LED and LD on Si substrates

Suzuka Nishimura¹⁾, Satoru Matsumoto¹⁾, Kazutaka Terashima²⁾

poster

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2) *Materials Science and Technology, Shonan Institute of Technology, Fujisawa Kanagawa, Japan*

A-36

Blue LED and LD have attracted much attention of many device and material researchers. The GaN crystals are usually grown on Al₂O₃ crystals, so the device structure is complicated and cleavage face is not apparent. We have studied the growth of GaN on Si substrates by using BP as a buffer crystal. The objectives are

- 1, to make low cost devices,
- 2, to grow cubic type GaN on Si to fabricate insensitive devices for stress during laser action,
- 3, to fabricate OEIC devices in the near future.

We have grown GaN/BP on 2inches in diameter Si (100) substrates by using MOVPE machine. The BP crystal and GaN crystals are epitaxially grown on Si. The substrates were markedly flat by

the role of buffer crystal. If the buffer was not sufficient, the stress during crystal growth was too high to curve the substrates in the presence of many slips. The photoluminescence signal demonstrated the grown GaN crystals was mainly cubic type. The crystal structures were strongly depended on the growing temperature without any irradiation. The cubic type has a tendency to be grown with low temperature growth. At the first stage BP layers has a little bit inclined around 5 degrees to Si (100). The electrical conductivity of BP is widely variable with the gas ratio and the temperature.

As an electron devices GaN FET is possible to grow by using AlN as an insulating layers. The combined devices will be the fundamental OEIC. At the meeting, the device structures and the growth will be shown.

19:00 -

Light scattering topography of excimer grade CaF₂ crystal.

Yasunao Oyama¹⁾, Suzuka Nishimura²⁾, Kazutaka Terashima¹⁾

poster

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2) *Science and Engineering, Keio University, Hiyoshi Yokohama, Japan*

A-38

It is important to observe the optical property of crystal of calcium fluoride because it is possible to use for the lens material of the lithography apparatus of silicon device. We have measured the scattering substance of calcium fluoride used for the excimer-lithography grade materials by the new type of light scattering topography method. This method is thought to be effective for the observation method which is no need of cutting samples. Some scattered particles have been observed in the matrix of calcium fluoride. The two types of the particles are shown. One is a large spherical particle, which exists independently, and the other is a small gathered particle, which are formed like a line. The latter is speculated to accumulate along the dislocation of the crystal. Additionally the fog-like particles of calcium fluoride which are occurred by annealing at high temperature have been observed by the method. The particles are formed by the small scattering substances which coordinate along the structure of the crystal and the large substances which are disordered. The small one is thought to be related to the structure of crystal of calcium fluoride.

19:00 -

Conductivities and waveguide properties of ZnO films and multilayer structures

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poster

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A-39

A sol-gel and electron-beam deposition methods were used to obtain ZnO:X films doped with different elements (X=Al, Ga and Li). The developed technique allows to obtain both high-conductive transparent and dielectric doped ZnO films.

Influence of Li, Ga and Al impurities of various concentrations on electric and photoelectric properties of zinc oxide films has been investigated. The developed technique allows to obtain as high-conductive and dielectric films. Measurements of film conductivity were carried out in a wide frequency range (0-10¹⁰ Hz). The value of resistivity depends on impurity and changes from 10⁻⁴ up to 10⁶ Ωcm.

Photoelectric properties studies have shown that at Li doping it is possible to achieve essential increase of photoconductivity. This phenomenon can be used for development of solid-state photodetectors for UV range (290-340 nm).

Planar and channel waveguides were developed on the basis of ZnO films and multilayer structures with various impurity. About twenty waveguides were created on one substrate (thickness 0.5-1 μm , width 20 μm , length 25-30 mm, turning radius 2 mm).

Color centers in YAP:Pr crystals

P. Potera⁴⁾, A. Matkovskii^{2,4)}, Ya. Zhydachevskii²⁾, Larisa Grigorjeva¹⁾, Donats Millers¹⁾, T. Lukaszewicz³⁾, Z. Galazka³⁾

1) *Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia*

2) *Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine*

3) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*

4) *Institute of Physics, University of Rzeszow, Rzeszow, Poland*

19:00 -
poster

A-40

YAlO₃ (YAP) single crystals doped with praseodymium ions are of interest as perspective materials for solid-state lasers.

In the present work we show results of investigation of both transient and stable optical absorption induced in YAP:Pr crystals by ionizing radiation. The transient changes of absorption and relaxation kinetics were measured after excitation of the crystals with the pulsed electron beam ($E \sim 0.25$ MeV, pulse duration - 10 ns) at room temperature. The stable changes of absorption were measured after gamma-irradiation of the crystals up to 10^6 Gy absorbed dose and high temperature treatments in reducing and oxidizing atmosphere.

The gamma-irradiation of YAP:Pr leads to the crystal coloration (absorption maxima near 40000, 33000, 25000 and 20000 cm^{-1}) caused by recharging of intrinsic point defects of the host.

The transient changes of absorption measured just after electron beam pulse reveal the absorption maxima near 25000, 20000 and 13000 cm^{-1} . The centers responsible for the absorption bands at 25000 cm^{-1} and 20000 cm^{-1} decay after electron beam pulse, while part of them remain as stable ones. The centers responsible for the band near 13000 cm^{-1} are unstable at room temperature.

The results obtained for YAP:Pr crystals are compared with the corresponding ones for pure YAP and YAP:Ca,Pr crystals as well as results obtained previously for YAP:Nd crystals (see [1]). The origin of color centers induced in the crystals is discussed.

[1] D. Sugak et al., Phys. Stat. Sol. (a) 184 (2001) 239.

Sensitive InGaAs /InP (SI) magnetic field sensors

Tomasz R. Przesławski¹⁾, Andrzej Wolkenberg¹⁾, Kazimierz Regiński¹⁾, Janusz Kaniewski¹⁾

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19:00 -
poster

A-41

On the basis of experimental data, it has been shown that at room temperature undoped n-type MBE In_{0.53}Ga_{0.47}As/InP_{ins} as a film ($t=4$ μm) with Hall electron mobility $\mu_H=0.7$ m^2/Vs and carrier concentration $n_H=2.25 \cdot 10^{20}$ m^{-3} as well MOCVD In_{0.53}Ga_{0.47}As/InP_{ins} ($t=2.7$ μm) with $\mu_H=1.2$ m^2/Vs and $n_H=1.3 \cdot 10^{21}$ m^{-3} can be used both for the Hall sensors and magnetoresistive elements. The calculations were performed for the case, that length to width ratio of a sensor (l/w)=1.

For the MBE sample the absolute sensitivity γ_0 defining maximal output voltage of the Hall Sensor (HS) and the current-related sensitivity γ deduced from the measurement results are $\gamma_0=1.1$ VT^{-1} , $\gamma=5600$ ΩT^{-1} , respectively. We found magnetoresistor current sensitivity $S_I \sim 800$ ΩT^{-1} and voltage sensitivity $S_V \sim 0.5$ T^{-1} for the layer. For the MOCVD sample $\gamma_0=0.26$ VT^{-1} , $\gamma=1320$ ΩT^{-1} , respectively. We found magnetoresistor current sensitivity $S_I \sim 600$ ΩT^{-1} and voltage sensitivity $S_V \sim 0.5$ T^{-1} for the layer. Similarly for 1 μm thick $n_H=8.5 \cdot 10^{23}$ m^{-3} , $\mu_H=0.5$ m^2/Vs we obtained the values of the parameters $\gamma_0=0.003$ VT^{-1} , $\gamma=5.6$ ΩT^{-1} and $S_I \sim 1$. ΩT^{-1} , $S_V \sim 0.05$ T^{-1} .

Self-passivation mechanisms of N clusters in SiC

Riccardo Rurali^{1,2)}, Eduardo Hernández²⁾, Philippe Godignon¹⁾, José Rebollo¹⁾, Pablo Ordejón²⁾

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19:00 -
poster

A-42

Nitrogen (N) is one of the most interesting n-type dopant for wide band-gap semiconductors (diamond, silicon carbide) due to its low mass and to the easy way with which it can be chemically manipulated.

In device processing it is often interesting to achieve very high doping doses of the substrate and N has proven not to be the best choice, as its activation rate starts decreasing when a certain threshold in the dose is reached. Particularly, in silicon carbide has been shown that for doses of the order of 10^{20} cm^{-3} it is more convenient to implant phosphorus or, to a lesser extent, to co-implant nitrogen and phosphorus.

In this work we present a first-principles theoretical study of the most relevant mechanisms that lead to N clusters passivation in silicon carbide (SiC) to account for N poor activation rate. We will show that all the N complexes, dominant in the different doping condition of the host material, do self-passivate, although the mechanism is not

always the same. The role of the off-centre relaxation of substitutional N at a Si site is also discussed.

19:00 - **Electroluminescence of thin films prepared by photopolymerization**

poster **pedro I. saez¹⁾, carlos aguilar**

1) *Universidad de Concepcion, Chile 56, Chile*

A-43

The synthesis and characterization of new materials based at monomers directives which containing a fluorene core are described. They exhibit an efficient fluorescence and have liquid crystalline behavior. Thin films have been prepared by the in-situ photopolymerization technique order to produce polarized light emitting films and the formation of the polymer network enhances for electroluminescence. We have investigated their thermal behavior and optical properties of the polymeric material.

19:00 - **Reactive Pulsed Laser Deposition of Doped Zinc Oxide Films as Emitting Layers of Thin Film Electroluminescent Devices**

poster **Victor Savchuk²⁾, Bohdan Kotlyarchuk²⁾, Maciej Oszwaldowski¹⁾**

1) *Politechnika Poznanska, Instytut Fizyki (PUT), Nieszawska 13a, Poznan 60-965, Poland*

A-44

2) *Institute of Applied Problems of Mechanics and Mathematics (IAPMM), 3B Naukova Str., Lviv 79060, Ukraine*

In this communication we report on studies of the electroluminescent (EL), optical and electrical properties of In-doped Zinc oxide (ZnO:In) films depending on the deposition parameters. ZnO:In films were prepared by, developed in our laboratory, reactive pulsed laser deposition method.

The experiments were carried out using a laboratory technological unit based on Nd:YAG laser. We used quasi-closed reaction chamber, which was specially constructed for the reactive pulse laser sputtering in oxygen ambient. The ZnO:In thin films were deposited on various substrates in a wide range of temperatures at different values of oxygen pressure.

Analysis of experimental results shows that ZnO and ZnO:In thin films can be deposited in situ by pulsed laser sputtering of synthesized homogeneous targets or of pure metallic targets in high pressure of oxygen at suitable values of substrate temperatures.

We found that the characteristics of the grown films are strongly depend on substrate temperature. Moreover, it was established, that EL and optical properties of films, grown at optimal substrate temperature, can be improved by increasing the oxygen pressure. The performed studies showed that the molar fraction of doping atoms introduced into the host film material is the most important factor for achieving maximum intensity of luminescent emission. As a result, we prepared the ZnO:In films at optimal deposition conditions, which demonstrated excellent optical parameters in the visible region of the spectrum, good electrical characteristics and strong luminescent emission, with maximum intensity located at 520 nm. The films the optical transmittance higher than 85% and the electrical resistivity up to 10^4 - 10^5 Ohm*cm.

Finally, the correlation between the luminescent properties of ZnO:In films and the parameters of the deposition process were determined and compared with their electrical and optical characteristics as well as with their structural parameters.

19:00 - **Electroconductivity and thermo-stimulated conductivity of cubic boron nitride**

poster **Nikolai A. Shishonok¹⁾, Gennadii V. Gatalskii, Anatolii A. Leusenko, Aleksandr Y. Erosh, Viktor L. Semenov**

A-45

1) *The Institute of Solid State & Semiconductor Physics, National Academy of Science of Belarus, 17 P.Brovka, Minsk 220072, Belarus*

Due to cubic boron nitride (cBN) extraordinary properties like a high Debye temperature, wide band gap (6.4 eV), elasticity modulus, high thermo-conductivity and mechanical strength, thermal and irradiative resistance the material has been considered as the prospective one for solid state electronics. CBN semiconductor properties investigations were started many years ago, but systematic dates about them have been absent. Electro-conductivity, thermo-stimulated conductivity, current-voltage characteristics and electro-conductivity dependences on temperatures for polycrystalline cBN. are investigated in present work. Undoped polycrystals were synthesized from pyrolytic boron nitride under high pressure/high temperature conditions. CBN n-type was realized by sulfur and aluminium doping. The curves of electroconductivity - temperature dependences have typical activation character at low temperatures. However the electro-conductivity drastically increases (of 2-4 orders in magnitude) for all doped and weakly doped samples at temperature about 670K. The current-voltage characteristics in logarithmic scales investigated at room and low (up to 570K) temperatures consist from some linear plots. An electrical instability appears as current multiplication of 2-4 orders at temperatures beyond 670K. Such a "switch on" of low

resistance cBN state preferably appears in sulfur doped polycrystalline samples. The thermo-stimulated electro-conductivity investigations of cBN undoped samples allow to establish three local levels in energy region from 1.0 to 2.2 eV.

Optical, EPR and RBS studies in Tm implanted ZnO samples

T. Monteiro¹⁾, M. J. Soares¹⁾, A. Neves¹⁾, M. Oliveira¹⁾, E. Rita²⁾, U. Wahl²⁾, E. Alves²⁾

1) *University of Aveiro, Campus de Santiago, Aveiro 3810-193, Portugal*

2) *Instituto Tecnológico Nuclear (ITN), Sacavém 2686, Portugal*

19:00 -

poster

Due to its potential applications for electroluminescent devices the doping of wide band-gap materials, such as GaN and ZnO, with rare earth (RE) ions is actually an interesting field of study. Trivalent RE ions like Eu³⁺, Er³⁺ and Tm³⁺ are known to be suitable dopants for red, green, and blue emitters, respectively. Moreover Er³⁺ and Tm³⁺ emissions in the middle infrared have promising applications as fiber amplifiers and optical switches in optical fiber communication technology.

In this work Tm³⁺ ions with 150 keV energy were implanted with different fluences, from 4x10¹⁵ to 5x10¹⁶ ions/cm² on ZnO [0001] single crystals at room temperature and 450°C. RBS/channeling results indicate that after implantation the majority of the ions are incorporated into substitutional Zn sites. After annealing the substitutional fraction decreases and is accompanied by some recovery of the lattice damage produced by the implantation.

Dependence of intraionic luminescence on the fluence and implantation temperature was observed in as-implanted samples with above band gap excitation. After implantation and following the 30 min thermal annealing in air at 800°C the samples implanted with lower dose present well-structured intraionic emission lines in the near infrared region that could be observed up to 200K. From the differences observed in the luminescence spectra of different samples it is clear that changes in the ion environment occurs.

A model for the recombination centers is proposed based on the combined experimental results.

A-46

Micro-Raman study of laser damage in CdTe surfaces

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1) *University of Aveiro, Campus de Santiago, Aveiro 3810-193, Portugal*

19:00 -

poster

The development of electronic devices involves a variety of technologies including laser-assisted doping of materials. However, laser irradiation can induce severe damage of the electronic materials, changing the structure by phase segregation and promoting defect migration and aggregation. So it is very important to know the extent and relevance of such side effects.

CdTe is a wide band gap semiconductor largely used in photonic applications such as radiation detectors and solar cells. In this work we show that laser irradiation of CdTe induces significant structural modifications localized in a thin surface layer. The relationship between laser power density, local temperature and structural changes is discussed by monitoring the relative intensity of the Te modes in Raman spectra. Local temperature is measured through the anti-Stokes/Stokes ratio of the same modes. We also show that the surface decomposition occurs at temperatures well below the melting point.

A-47

Photo-ESR and optical studies of Cr photoionization transition in CdZnSe:Cr crystals

Krzysztof Swiatek¹⁾, Marek Godlewski^{1,3)}, Tanya Surkova²⁾

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2) *Institute of Metal Physics, Ural Division of Russian Academy of Sciences, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation*

3) *College of Science, Cardinal S. Wyszyński University, Warsaw, Poland*

19:00 -

poster

A-48

Excitation processes of radiative recombination of transition metal ions, which are doped in wide band-gap II-VI semiconductors, are of great interest because of possible application for novel optical devices. Recent studies have shown that the Cr²⁺ + hν → Cr¹⁺ + hν_B photoionization transitions (hν > 2.2 eV) is an efficient channel for excitation of the Cr²⁺ intra-ion infra-red emission and laser action in ZnSe:Cr. Since common used semiconductor diode lasers emit light in energy range of 1.9 - 2.0 eV, it is interesting to investigate the same problem in a host system with a bit smaller band gap energy than in case of ZnSe.

In this work we study 2+ → 1+ and 1+ → 2+ photoionization transitions of chromium ions in Cd_xZn_(1-x)Se:Cr bulk crystals (0 < x < 0.3) in wide temperature range (2 - 300 K). By monitoring changes of the Cr¹⁺ ESR (electron spin resonance) signal under external illumination we are able to determine energies of Cr²⁺ → Cr¹⁺ + hν_B and Cr¹⁺ → Cr²⁺ + e_{CB} transitions in different Cd_xZn_(1-x)Se host crystals. At low temperature we observe metastable population of photo-excited Cr¹⁺ centers. Free holes created in the valence band (VB) under the 2+ to 1+ ionization of chromium, are efficiently trapped by shallow acceptor centers. We observe also appropriate photoionization bands in low temperature absorption spectra.

Moreover, obtained by us values of the chromium PT energy in Cd_xZn_(1-x)Se agree well with predictions of the

universal-level-model and band offsets in CdZnSe/ZnSe heterostructures.

19:00 - **Cu(I) complexes with perfluorinated carboxylates as potential CVD precursors**

poster **Iwona Szymańska¹⁾, Edward Szytk¹⁾, Piotr Piszczek¹⁾, Lilianna Dobrzańska¹⁾**

A-49 *1) Toruń, Nicolaus Copernicus University, Faculty of Chemistry (WChUMk), Gagarina 7, Toruń 87-100, Poland*

Previous studies revealed that Au(I) and Ag(I) complexes with perfluorinated carboxylates and tertiary phosphines presented sufficient volatility for chemical vapour deposition (CVD), hence the purpose of this work was to prepare volatile precursors for CVD of copper metal or copper oxide thin films. Cu(I) complexes with trimethyl phosphite and aliphatic perfluorinated carboxylates of the type $[\text{Cu}_2\{\text{P}(\text{OMe})_3\}_2(\mu\text{-RCOO})_2]$ where $\text{R} = \text{CF}_3, \text{C}_2\text{F}_5, \text{C}_3\text{F}_7, \text{C}_6\text{F}_{13}, \text{C}_7\text{F}_{15}, \text{C}_8\text{F}_{17}, \text{C}_9\text{F}_{19}$ have been prepared as viscous liquids. The EI mass spectrometry, temperature-variable IR spectroscopy and thermal studies were undertaken to determine the type of species appeared in the gas phase and the thermal decomposition reaction mechanism. The thermal analysis data suggest, that compounds studied can be used in CVD of copper oxide films, however the decomposition product can be modify by conditions applied for the deposition technique. The MS and temperature-variable IR data analysis pointed to $[\text{Cu}_2\{\text{P}(\text{OMe})_3\}_2(\mu\text{-C}_6\text{F}_{13}\text{COO})_2]$ as the most suitable potential liquid CVD precursor from the complexes studied. CVD experiments were performed in the low-pressure horizontal CVD reactor using argon as the carrier gas. The obtained films were investigated by XPS. More than two forms of copper were existed on the sample surface and unfortunately some impurities of carbon, fluorine and phosphorous.

19:00 - **Temperature, pressure, strain and composition effects on band offsets in III-nitride heterostructures**

poster **Hilmi Unlu¹⁾**

A-50 *1) Istanbul Technical University (ITU), Maslak, Istanbul 80626, Turkey*

Reliable and precise knowledge about the strain and composition effects on the bandgaps and band offsets for the III-nitrides based heterostructures is crucial for the optimization of device structures. We present a nonorthogonal tight binding theory (H. Ünlü, Phys. Stat. Sol. (b), 235, pp 248-253, 2003) to investigate the interface misfit, thermal expansion gradient and pressure induced strain and composition effects on band offsets in pseudomorphic InGaN/GaN, AlGaN/GaN, GaAsN/GaAs, and AlGaAsN/GaAs heterostructures with zinc-blende structures. The model considers the nonorthogonality of hybrids of adjacent atoms and the interaction of bonding and antibonding states at high symmetry points (e.g., L and X) in calculating the valence band energies of semiconductors. The valence band offsets are then obtained from the difference between the valence band energies, screened by the optical dielectric constants of constituent semiconductors. Model predictions compare well with experiment (H. Morkoç et al., Semiconductors and Semimetals, 50, 193, Academic, 1998) for the band offsets of nitride systems. Simulations show that the valence band offset in AlGaN/GaN system is small but in the InGaN/GaN system is very large. Tensile strain in AlGaN leads to positive bowing of the conduction band offsets in AlGaN/GaN system and the compressive strain in InGaN leads to negative bowing of conduction band offsets in InGaN/GaN system as a function of composition.

19:00 - **Effect of deposition conditions and annealing on residual stress of ITO films magnetron sputtered on silica**

poster **Arnoldas Užupis³⁾, Renata Butkute¹⁾, Bonifacas Vengalis¹⁾, Vaclovas LISAUSKAS¹⁾, Sigitas Tamulevičius²⁾**

A-51 *1) Semiconductor Physics Institute, A. Gostauto 11, Vilnius LT-2600, Lithuania*

2) Institute of Physical Electronics, Kaunas University of Technology (KTU FEI), Savanoriu 271, Kaunas LT-3009, Lithuania

3) Lithuanian University of Ugriculture (LZUU), Studentu 11, Kaunas, Akademija Lt-4324, Lithuania

Thin films of indium-tin oxide (ITO) ($d \sim 1000$ nm) were deposited on heated ($T_d = 20-500$ °C) glass (silica) substrates by reactive DC-magnetron sputtering followed by post-deposition annealing at $T = (150-750)$ °C in oxygen and vacuum. Highly conductive (10^{-4} Ohm) and transparent for visible light ($\sim 95\%$) films were deposited under Ar:O₂ (1:1) pressure of about 1-3 Pa. Crystalline structure and surface quality of the films were studied by X-ray diffraction, scanning electron microscopy and atomic force microscopy. Optical interferometry was used to investigate residual stress in the films prepared under various deposition and annealing conditions. Influence of oxygen content and structural quality of the films on the residual stress has been defined.

19:00 - **Effect of ordering on the optical properties of III-nitrides alloys**

poster **Oleksandr Voznyy¹⁾, Vitaliy Deibuk¹⁾, Stepan Melnychuk¹⁾**

A-52 *1) Chernivtsi National University, Chernivtsi, Ukraine*

During the past few years remarkable progress has been made in the development of optical and electronic devices based on the group-III nitrides. Light emitting diodes, laser diodes, ultraviolet detectors, Bragg reflectors, high-power transistors, etc were fabricated employing III-nitrides and their ternary alloys layers.

In contrast to the cubic III-V arsenides and phosphides where ordering was previously observed, the III-nitrides have the hexagonal wurtzite structure. The first observation of atomic long range ordering in AlGa₂N and InGa₂N thin films grown by electron cyclotron resonance assisted molecular beam epitaxy on sapphire and 6H-SiC substrates was reported recently. The observed superlattice exists in a form of ideal, stoichiometric, perfectly ordered alloy.

The electronic band structure of ternary III-nitrides random alloys and ordered structures was investigated using model empirical pseudopotential dependent on local strain. 32-atom 2x2x2 wurtzite supercell with periodic boundary conditions was adopted to describe different atomic configurations and bond length relaxations.

Imaginary and real part of the dielectric function and reflectivity of the disordered and ordered III-nitride alloys were calculated. The effect of substrate was investigated using the biaxial strain. It is shown that the atomic long range order changes the band gap and energy positions of the main peaks of $\epsilon_2(h\nu)$ much more stronger than in conventional III-V ternary alloys.

Thermoelectric properties of amorphous AlInN thin films

Shigeo Yamaguchi^{1,2)}, Ryohei Izaki¹⁾, Yasuo Iwamura^{1,2)}, Atsushi Yamamoto²⁾

19:00 -

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poster

2) National Institute of Advanced Industrial Science and Technology, Energy Electronics Institute, AIST Tsukuba Central 2 Umezono 1-1-1, Tsukuba 305-8568, Japan

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Seebeck effect can cause solids to directly convert heat energy into electric energy without producing emissions such as carbon dioxide gas and radioactive substances. Moreover, using the reverse effect, Peltier effect, the simultaneous cooling and heating phenomena can be utilized to medical applications such as the local cooling or heating for the human body. There has recently been a great increase in the research and development of thermoelectric power generation systems that are designed to employ the vast resources of waste heat and environmentally sound cooling. The realization of practical applications associated with such systems requires the achievement of a high efficiency characterized by electric resistivity, thermoelectric Seebeck coefficient and thermal conductivity. At this stage, no binary compounds better than Bi₂Te₃, PbTe have been found. We have recently studied the thermoelectric properties of AlInN system in terms of substituting it for Be₂Te₃ and PbTe, which are considered to environmentally stress. AlInN thin films were prepared by reactive RF sputtering grown on SiO₂ glass substrates. They were of amorphous, judging from X-ray analysis. We evaluated the values of Seebeck coefficient and electrical resistivity. Between the temperatures from RT to 873K, with increasing In content in AlInN from 0.45 to 0.84, the absolute value of the Seebeck coefficient decreased. Moreover, with increasing temperature, the Seebeck coefficient increased monotonically to be 70 μ V/K at 873K. On the other hand, the resistivity decreased with increasing temperature, and it was about 10⁻⁵ Ω m. Considering both Seebeck coefficient and resistivity, we estimated the value of power factor, which is a landmark for the applicational potential, and we obtained the value of over 10⁻⁴ W/mK². This value is smaller only by a factor of 10 compared to those for Bi₂T₃ and PbTe. We believe that AlInN can take place of them in the near future.

Modelling of quantum wells with smooth boundaries

Adam J. Zakrzewski¹⁾

19:00 -

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

poster

In the past two decades properties of various low dimensional heterostructures received much theoretical and experimental attention. The nature of the shallow (hydrogenic) states in such systems and their modification due to external magnetic field was widely investigated. Such effects are routinely studied with variational method within effective mass theory.

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In this communication we focus on the influence of the smoothness of quantum well boundaries on donor states. Confining potential is taken in the form of Wood-Saxon potential. Such form of potential allows us to include the finite slope of the potential across quantum well boundaries. We also investigate the effects related to differences in effective masses and dielectric constants between well and barrier material and the influence of external magnetic field.

This research was supported by grant no. 5 P03B 007 20 of KBN for 2001-2003 years

Kinetics of the donor-acceptor and "pseudo-donor-acceptor" pairs in nitrides

Adam J. Zakrzewski¹⁾

19:00 -

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

poster

Radiative tunneling of electrons and holes localized on randomly distributed donors and acceptors is one of the dominant recombination mechanism in many semiconductors, including new laser and optoelectronic materials such

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as ZnSe [1] and GaN [2]. Recently, Morel et. al. introduced pseudo-donor-acceptor pairs model for the description of recombination processes in InGaN/GaN quantum wells [3]. In this case electrons and holes are localized due to potential fluctuations at interfaces and not on donors and acceptors. It was shown that summing all possible configurations of such localized carriers explains nonexponential decay of luminescence, quite similarly to what is observed for bulk system. However, the tunneling probability in former case is in the form of Gaussian function of the electron-hole distance and not exponential like in the latter.

In this communication we show, that both in bulk or low dimensional system kinetics of such processes can be calculated exactly and next we discuss how this observation simplifies analysis of experimental results.

This research was supported by grant no. 5 P03B 007 20 of KBN for 2001-2003 years

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[3]. A. Morel, P. Lefebvre, T. Taliercio, T. Bretagnon, B. Gil, N. Grandjean, B. Damilano, J. Massies, Physica E17, 64 (2003)

19:00 - High-resistance low-doped GaAs and AlGaAs thin films for high power electronics

poster **Dmytro M. Zayachuk²⁾, S I. Krukovsky³⁾, M. Kaniewska¹⁾, N Strukhlyak²⁾**

A-56 ¹⁾ *Institute of Electron Technology, al. Lotnikow 32/46, Warsaw 02-668, Poland*

²⁾ *Lviv National Polytechnic University, Lviv, Ukraine*

³⁾ *SPC "Karat", Lviv, Ukraine*

High-resistant low-doped GaAs and AlGaAs layers are the basis to create the fast-operating high-voltage diodes, thyristors, thermosensors and so on. The difficulties while obtaining pure III-V layers by LPE are due to the presence of the background impurities in the initial charge, especially of silicon and oxygen. Significant progress has been achieved in the controlling properties of the semiconductor materials under influence of the rare-earth elements (REE) for last time. The best conditions in this aspect taking into account high chemical activity of the REE can be potentially provided by LPE. At the same time, it is rather difficult to realise possible advantages of using rare-earth elements to control properties of the GaAs and AlGaAs layers by utilising present LPE based on the adjusting the vapour phase humidity and the using quartz equipment. Main reason for that is interaction between aluminium and REE on one side, and quartz equipment on another, which prevents creation p-i-n structures in the GaAs/AlGaAs system. However just these heterostructures attract significant practical interest due to their low back-voltage currents and higher breakdown voltages in comparison to the materials with smaller energy band gap. In the present work new approaches based on the complex doping with REE and isovalent element impurities to be applied to control electrophysical properties of the LPE grown GaAs layers are developed. Influence of co-doping with Yb and Al of the gallium melts during LPE growth of the GaAs epitaxial layers on their properties is investigated. It is shown, that both the electrophysical parameters and the emitting features of deep traps of the films did get changed under influence of the doping impurities applied. Obtained results are explained by simultaneous Yb gettering action with respect to the oxygen and silicon in the solution-melt, and also by lowering the concentration of the Si in the films due to the Al entering into the Ga sub-lattice.

19:00 - Thermoluminescence of doped YAlO₃ crystals

poster **Ya. Zhydachevskii^{1,2)}, A. Durygin¹⁾, A. Suchocki¹⁾, A. Matkovskii^{2,4)}, D. Sugak²⁾, Z. Frukacz³⁾, G. B. Loutts⁵⁾, M. A. Noginov⁵⁾**

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⁴⁾ *Institute of Physics, University of Rzeszow, Rzeszow, Poland*

⁵⁾ *Center for Materials Research, Norfolk State University, Norfolk, United States*

The work presents results of the thermally stimulated luminescence (TSL) studies of nominally pure YAlO₃ (YAP) crystals as well as the crystals doped with various dopants (among them Ho³⁺, Tm³⁺, Mn).

Measuring of thermal glow curves in the 10-600 K temperature range after preliminary exposition with Ar⁺-laser light (351 nm) of Xe-lamp (UV part of spectrum) shows the presence of similar glow peaks in all the crystals studied. Namely, the glow peaks near T=175 K (E=0.48±/0.06 eV, lns=27.8±/4.1 s⁻¹), T=195 K (E=0.55±/0.03 eV, lns=28.6±/1.9 s⁻¹), T=205 K (E=0.68±/0.03 eV, lns=34.5±/1.8 s⁻¹), T=255 K (E=0.80±/0.05 eV, lns=32.2±/2.4 s⁻¹), T=360 K (E=1.35±/0.05 eV, lns=39.1±/1.7 s⁻¹) and T=400 K (E=1.7±/0.1 eV, lns=44.9±/3.0 s⁻¹) were observed. Besides, two (or maybe more) feebly pronounced glow peaks at T<100 K as well as the peak near T=320 K were observed in some crystals.

It was shown that the transient optical absorption of YAP crystals near 15000 cm⁻¹ described in [1] correlates by activation energy with the low-temperature thermal glow peak at 255 K.

TSL spectra of YAP:Mn crystals correspond to intra-center luminescence of Mn⁴⁺ ions. TSL spectra of nominally pure YAP and YAP:Ho crystals also correspond to Mn⁴⁺ ions, which are present in the crystals as uncontrolled

impurity and obviously act as recombination centers. Whereas TSL spectra of YAP:Tm crystals in visible region correspond to intra-center luminescence of Tm^{3+} ions that testifies energy transfer processes between point defects and Tm^{3+} ions.

The effect of preliminary annealing ($T > 1000$ K) in reducing or oxidizing atmosphere on the TSL properties of the crystals has been studied.

On the basis of the obtained results a nature of trap centers and mechanisms of recombination are discussed.

[1] A. Matkovski, A. Durygin, A. Suchocki, et al. Optical Mater. 12 (1999) 75.

The luminescent convertors based on single crystalline films

Yurij V. Zorenko¹⁾

1) *Laboratory of Optoelectronic Materials Ivan Franko Lviv National University, Lviv, Ukraine*

It has been analyzed the possible applications of single crystalline films (SCF) of Al_2O_3 - Y_2O_3 - Lu_2O_3 :Ce oxide system as luminescent transformers of different types:

- * scintillators of α - and β -particles for radiation monitoring;
- * composition scintillators/detectors (CSD) containing SCF-phosphors deposited on substrate-phosphor for registration of the mixing ionizing flux components;
- * screens for electron-beam tubes with high spatial resolution for special applications;
- * screens for visualization of X-ray images for medical or biological monitoring;
- * phosphor-convertors for LED-diodes radiation (LUCOLED-system) for light devices.

It has been established the peculiarities of growth the SCF of Al_2O_3 - Y_2O_3 - Lu_2O_3 oxide system with garnet, perovskite and α - Al_2O_3 structures doped with Ce^{3+} or other rare-earth (RE) dopants by liquid phase epitaxy on Al_2O_3 , $YAlO_3$ and $Y_3Al_5O_{12}$ substrates.

It has been shown the possibility of "engineering" of emission and excitation spectra of SCF based on solid-solution $(Y-Lu)_3(Al-Sc-Ga)_5O_{12}:Ce$ which are correlated with the range of spectral sensitivity of Si-diodes and CCD-cameras or with the region of radiation of LED-diodes, by means of changes in the crystals field strength or co-doping with the different RE ions.

The main peculiarity of spectral-kinetic characteristics of SCF is determined by the extremely low concentration of emission and trap centers created by the antisite-type and vacancy-type defects. This fact determines the advantages of phosphors based on SCF in comparison with the bulk crystal analogues, which consist in the absence of the dissipation channels of excitation energy connected with such type centers. The above mentioned peculiarity make the SCF also as an useful model object for investigation of relaxation phenomenas of low-energy excitation, in particular excitons, in the many-sublattices oxides.

This work partly was supported by grant STSU2042.

19:00 -

poster

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Tuesday, September 16th

Morning session - Oliver Ambacher

Main Building, room 219

Wide band gap materials for optoelectronics I

11:00 - 12:30

Cathodoluminescence properties of Zinc Oxide nano-particles

Matthew R. Phillips¹⁾, **Olaf Gelhausen**¹⁾, **Ewa M. Goldys**²⁾

1) *University of Technology, Sydney (UTS), Broadway, Sydney 2007, Australia*

2) *Macquarie University, Sydney 2109, Australia*

11:00 - 11:45

invited oral

CL spectra from nano-granular and electronic grade bulk ZnO have been measured as a function of electron beam current, I_B , and specimen temperature (80K-300K). A power-law model ($I_{CL} = k \cdot I_B^m$) was used to investigate the influence of particle size on the excitation power dependence of each of the observed ZnO CL emission bands. Bulk ZnO displayed a near band edge (NBE) emission peak at 3.28eV with no other CL emission. The intensity of the bulk ZnO NBE exhibited a superlinear dependence on I_B with a power law exponent of $m = 2.2$ at 300K, while its peak position remains fixed with increasing I_B . Conversely, the NBE in the nano-granular ZnO displayed a strong red shift with increasing I_B at 300K. Its position, however, did not change with I_B at low temperature. Power exponents of $m = 0.9$ and $m = 1.4$ were measured for the NBE in the nano-granular ZnO at 300K and 80K, respectively. CL emission from defect-related centres positioned at 1.8eV and 2.3eV was observed at 300K in the ZnO nano-particles but not in the bulk ZnO. The nano-granular ZnO also exhibited an intense featureless emission which steadily increased up to the IR detection limit at 0.75eV. Its intensity displayed a supralinear dependence on I_B with $m > 9$. This emission was completely quenched by a slight decrease in specimen temperature, and was not observed in the bulk ZnO under all excitation conditions. The IR emission is attributed to black-body radiation where the low thermal conductance of the ZnO nano-particles leads to significant electron beam heating. The observed red shift of the NBE peak position in nano-granular ZnO is therefore attributed to its dependence on temperature rather than carrier density. Variation in the NBE power law exponents between bulk and nano-granular

ZnO is explained by the higher defect density of the ZnO nano-particles as well as their large surface-to-volume ratio.

11:45 - 12:30

Solid-state lighting

invited oral

Arturas Zukauskas¹⁾, Michael S. Shur²⁾, Remis Gaska³⁾

1) Institute of Materials Science and Applied Research, Vilnius University (IMSAR, VU), Sauletekio 9, building III, Vilnius LT-2040, Lithuania

2) Rensselaer Polytechnic Institute, Troy, NY, United States

3) Sensor Electronic Technology, Inc., Columbia, SC, United States

Recent breakthroughs in growth of novel wide-bandgap materials, and in the design of high brightness light-emitting diodes (LEDs) have resulted in the development of a new exciting solid-state lighting technology [1]. In addition to future application for general lighting, mass applications of solid-state lighting now include full-color video displays, signals, traffic lights, and local and decorative illumination. In addition, many niche applications in phototherapy, photosynthesis, and optical measurements have emerged. In 2020, solid-state lighting is expected to save up to 10% of the global electric power consumption and replace a significant percentage of ineffective incandescent bulbs and mercury-containing discharge tubes.

In our talk, we review the history of lighting, LED basics, extraction of light from semiconductor chips, white and UV LEDs, and new applications of solid-state lighting. We also describe our work on optimization of solid-state lighting sources and on the development of versatile white polychromatic lamps with digital feedback that are being tested for a more effective treatment of seasonal affective disorder. Novel application of high-power LEDs for Raman measurements and for water testing is also demonstrated.

[1] A. Zukauskas, M. S. Shur, and R. Gaska, Introduction to Solid-State Lighting (Wiley, New York, 2002).

12:30 - 14:00

Lunch break

14:00 - 15:30

Afternoon session - Arturas Zukauskas

Main Building, room 219

Wide band gap materials for optoelectronics II

14:00 - 14:45

Electronic structure of shallow impurities in GaN studied via bound exciton magnetooptics

invited oral

Roman Stepniewski^{1,2)}, Andrzej Wyszomolek¹⁾, Marek Potemski²⁾

1) Institute of Experimental Physics, Warsaw University (IEP UW), Hoza 69, Warsaw 00-681, Poland

2) Grenoble High Magnetic Field Laboratory, MPI/FKF-CNRS (GHMFL), 25, Avenue des Martyrs, Grenoble 38-042, France

There is a rekindled interest in wide gap compounds of which Gallium Nitride is a relevant example. The need for precise control of doping, which is essential for appropriate device design, continues to stimulate the basic studies of impurity centres in this semiconductor. A detailed knowledge of their electronic structure (energy and symmetry of the ground and excited states) allows probing their location in the lattice and assigning the specific properties with their chemical identification.

In this paper we present the photoluminescence experiments performed on high quality GaN samples in high magnetic fields. A detailed analysis of the recombination due to excitons bound to neutral donors and acceptors is presented. Special attention is focussed on transitions for which the impurity is left in the excited state (so called two electron satellites). Such experiments allow us to reproduce the rich energetic structure of the excited states of the impurity involved in such a recombination process. The magnetic field dependence of the energy structure of the shallow neutral donor and acceptor in GaN is then discussed.

The validity of the effective mass approximation for shallow impurities in wide gap semiconductors will be considered.

This work has been partially supported by State Committee for Research (Republic of Poland), Grant No. 2P03B 011 22, NATO Science Program, EC Project No. HPRI-CT-1999- 40013.

14:45 - 15:30

Wide band-gap light emitters with improved hole injection

invited oral

V A. Kochelap¹⁾

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The difficulties in achieving high hole concentrations in group-III nitrides originate from high values of activation energy of acceptors. The average hole concentration can be increased in a p-doped nitride superlattice (SL). However, most of the holes ionized from the acceptors are localized inside the quantum wells (QWs) and cannot

participate in vertical transport utilized in traditional light-emitting devices (LEDs). In this report we propose two novel solutions of the problem of hole injection enhancement in wide band-gap LEDs.

Low-intensity emitters: The conventional LEDs can be modified by introducing a two-terminal hole injector that consists of a p-doped SL-base with two contacts. A bias voltage applied between these contacts provides lateral hole acceleration and increases the effective hole temperature. This results in significant enhancement of overbarrier hot-hole concentration. The proposed LED can be thought of as a three terminal device, where the hot-hole SL-injector is placed on the top of heterostructure with an intrinsic i-layer, and an n-doped region. In the report, we discuss parameters of the nitride-based hot-hole injectors and characteristics of the three terminal UV-LEDs.

High-intensity lateral current pumped emitters: To achieve high-density electron-hole plasma (EHP) and interband population inversion in group-III nitrides, we propose a planar p-i-n structure created in selectively-doped SLs: a region doped with acceptors is followed in lateral direction by an i-region and, finally, by an n-region. Thermal activation of the dopants supplies carriers into the QW layers. The QW layers accumulate both types of free carriers and a lateral p-i-n structure is formed.

Coffee break

15:30 - 15:50

Afternoon session - continued - Roman Stepniewski

15:50 - 17:10

Main Building, room 219

Oral session I

Studies of symmetry of erbium centers in GaN.

15:50 - 16:10

Vasyl Glukhanyuk¹⁾, A Kozanecki¹⁾, H. Przybylinska¹⁾*1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

oral

Er-doped semiconductors are promising materials for optoelectronic devices operating on the intra 4f-shell transitions of the rare earth impurity. GaN:Er is currently studied very intensively, because of its importance for modern optoelectronics. In spite of many efforts little is known, however, about Er energy structure, as generally Er creates complexes with another dopants, mainly with oxygen, which makes analysis difficult. To omit this Er should be introduced into substrates with low oxygen content and by ion implantation.

In this work we use site selective excitation of Er-centres to study their symmetry in cubic and hexagonal GaN. Photoluminescence excitation (PLE) spectroscopy at an energy range corresponding to the 4I15/2 - 4I9/2 absorption is applied to study splitting of energy levels of Er³⁺. The Stark split energy levels as determined from the PLE spectra serve then as a basis for model calculations. By means of point charge approximations we calculated the splitting of 4I9/2 free Er ion level in weak crystal field of the host material and we got good agreement with experimental results. Because the nature of this splitting directly depends on the symmetry of the crystal field, we can investigate the symmetry of Er centers and their position in host in such way. From our results one can make a conclusion that in cubic GaN sample there is a small distortion in [001] direction of crystal growth and Er is in interstitial position. The symmetry of Er centers is C_{3v} in hexagonal GaN samples.

Luminescent properties of wide bandgap materials at room temperature

16:10 - 16:30

Aleksandra Sokolowska³⁾, A Werbowy⁴⁾, A Barcz¹⁾, Marek Godlewski^{1,2)}, Jan Szmidi⁴⁾, A. Olszyna⁵⁾*1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland**2) College of Science, Cardinal S. Wyszyński University, Warsaw, Poland**3) Institute of Mat. Sci. Eng., Technical Univ. of Lodz, Lodz, Poland**4) Institute of Microelectronics and Optoelectronics, Warsaw Technical Univ., Warsaw, Poland**5) Faculty of Mat. Sci. Eng., Warsaw Technical Univ., Warsaw, Poland*

oral

Investigated nanocrystalline (grain size < 100 nm) submicron thick GaN, AlN and BN layers were produced on Si substrates by means of reactive crystallization from impulse plasma (RPP CVD method), while diamond-like carbon (DLC) films were obtained in the course of radio frequency (13.56 MHz) chemical vapor deposition (RF CVD) process. In our previous works (e.g. [1-3]) we presented results of the studies of their optical (refractive index value, absorption edge value), electronic (resistivity, dielectric constant, state of the substrate/layer interface) as well as low-temperature photoluminescent properties [4]. Attempts of correlating structural, optical and electronic properties of discussed films with their photoluminescent characteristics were also undertaken.

In the present study we investigated photoluminescence (PL) of the layers excited by He-Cd laser (325 nm) beam at the room temperature. All materials, except BN, demonstrated PL in UV and violet light (fairly homogenous in plane in the case of GaN and AlN). Relatively the most intensive emission was observed for DLC and AlN films. Moreover, the subject of investigations was also cathodoluminescence (CL) of GaN and AlN layers. Both showed very broad-band emission, fairly homogenous in plane. An attempt of correlating luminescent properties of studied materials at room temperature with their bandgap widths and nanocrystallinity is presented as well.

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[3] A. Werbowy, P. Firek, J. Szmidt, A. Olszyna, M. Galazka, *J. Wide Bandgap Mater.*, 9(2002)169.

[4] J. Siwiec, A. Sokolowska, A. Olszyna, R. Dwilinski, M. Kaminska, J. Konwerska-Hrabowska, *Nanostructured Mater.*, 10(1998)625.

16:30 - 16:50

oral

Luminescence transients in highly excited GaN grown by hydride vapor-phase epitaxy

Saulius Jursenas¹⁾, Saulius Miasojedovas¹⁾, G. Kurilcik¹⁾, Arturas Zukauskas¹⁾, P.R. Hageman²⁾

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Formation of high-quality bulk-like GaN layers is of great importance since they are used as substrates for further epitaxy of light emitting structures and can facilitate efficient light extraction from light emitting diodes. The best-quality GaN epilayers are obtained by using homoepitaxial growth over bulk GaN crystals. However at present, the size and growth rate of the high-pressure crystals are limited. Hydride vapor phase epitaxy (HVPE) offers large growth rates with relatively good quality of crystals. Optical properties under high excitation conditions, which are important for establishment of laser and high-power LED operation regime, are not well investigated in HVPE grown GaN.

We have studied carrier recombination dynamics in HVPE grown GaN at room temperature by means of transient photoluminescence under high photoexcitation conditions that are close to stimulated emission regime. The obtained luminescence transient was shown to be in a good agreement with a model of saturated centers of non-radiative recombination with the trap density of $\sim 10^{17} \text{ cm}^{-3}$ and carrier recombination coefficients of $\sim 10^{-8} \text{ cm}^3/\text{s}$. In such a regime, the lifetimes of electrons and holes have a common value of 410 ps. This value is smaller than that measured in homoepitaxially grown samples but it is considerably higher than in high quality MOCVD grown epilayers on sapphire. This suggests high potential of HVPE grown GaN for short-wavelength optoelectronics applications.

16:50 - 17:10

oral

Simulation of localized exciton hopping in quaternary AlInGaN

Karolis Kazlauskas⁴⁾, G. Tamulaitis⁴⁾, Arturas Zukauskas⁴⁾, M. A. Khan¹⁾, J. W. Yang¹⁾, J. Zhang¹⁾, G. Simin¹⁾, M. S. Shur²⁾, R. Gaska³⁾

1) *Department of EE, University of South Carolina, Columbia, United States*

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AlInGaN materials have found applications in the fabrication of high efficiency blue, white, and UV light emitters. Indium is believed to significantly improve the efficiency of semiconductor lasers and light emitting diodes due to carrier localization in the potential fluctuations caused by In clustering in the alloy. Such localization prevents non-radiative carrier recombination. At low temperatures, exciton transport in In-containing nitrides is only possible by phonon-assisted hopping. Observed "anomalous" S- and W-shaped temperature dependences of the photoluminescence (PL) band peak and width, respectively, serve as a signature of exciton hopping.

We report on Monte Carlo simulation of the localized exciton hopping in quaternary AlInGaN. The simulation results enabled a qualitative and quantitative interpretation of the measured temperature behaviour of the PL band peak position and width. Our calculations are based on a model of clusters with different In content, where excitons are hopping through the states dispersed due to potential fluctuations. The model successfully accounts for the inhomogeneous PL band broadening caused by the size fluctuations of individual clusters, i.e. by fluctuations of average localization energy. It provides a quantitative description of the temperature dependence of the PL band peak and width in the temperature range from 8 to 150 K. The simulation results for quaternary AlInGaN containing 1% of In yield 16 meV for the roughness of the potential profile in individual clusters and 42 meV for their distribution in average localization energy.

Wednesday, September 17th

11:00 - 12:30

Morning session - Lauri Niinisto

Main Building, room 219

Oxides I

ZnO and its low dimensional systems**Bernard Gil¹⁾**

11:00 - 11:45

1) Groupe d'Etude des Semiconducteurs, CNRS-UMR 5650, Universite de Montpellier 2, cc074, 12 Place Eugene Bataillon, 34095 Montpellier, France, Montpellier, France

invited oral

In the first part of the talk, we discuss the specificities of the bandgap properties of zinc oxide.

Second, we review the last results available which concern the optical properties of quantum wells, quantum dots and microcavities based on it. A systematic comparison is made with GaN.

Third, we discuss the polariton laser issue and we show the potentialities of oxides compared to nitrides.

Last we give some words concerning the realization of single photon sources using ZnO spherical quantum dots surface-passivated by MgO, and we compare this system with its challenger, namely CdS capped by ZnS.

ZnO - a promising semiconductor for application**Axel Hoffmann¹⁾, U. Haboeck, M. Strassburg, A. Rodina, M. Dworzak, A. Zeuner²⁾, D.M. Hofmann, H.R. Alves³⁾, B.K. Meyer³⁾**

11:45 - 12:30

invited oral

*1) Technical University Berlin, Berlin, Germany**2) I. Physics Institute, Justus-Liebig-University Giessen, Giessen, Germany**3) I. Physics Institute, Justus-Liebig-University Giessen, Giessen, Germany*

There is rising interest in investigating the properties of ZnO epitaxial films due to its superior properties such as high exciton binding energy combined with low lasing threshold density and good resistance to bombardment with high-energy particles. The material is a potential competitor for GaN-based light-emitting devices in the ultraviolet and blue spectral range. Like for other wide-band gap semiconductors as GaN and ZnSe controlled p-type doping is difficult. However, there have been reports on the synthesis of p-conducting ZnO doped with As and a Ga/N codoping as well as the fabrication of a p-n-junction by excimer-laser doping. Magnetic field dependent optical absorption and photoluminescence (PL) experiments were performed to study shallow bound exciton complexes in Na-, N-, Ga- and In- doped and nominally undoped ZnO. These data were interpreted using theoretical modeling calculations of the ordering of the valence bands, Zeeman splitting, selection rules for optical transitions and thermalization behavior for each of the investigated specimens. Both the angular-dependent magneto-PL results and the theoretical calculations indicate that the top of the valence band in ZnO possesses the Γ_7 symmetry. The sign and the value of the effective g factor of the holes involved in all bound exciton transitions were determined by analyzing the polarization properties and fitting the angular dependence of the transition energies. The incorporation of nitrogen into ZnO was studied by Raman-scattering experiments. The Raman spectra revealed vibrational modes at 275, 510, 582, 643 and 856 cm^{-1} in addition to the host phonons of ZnO. The intensity of these additional modes correlates linearly with the nitrogen concentration and can be used as a quantitative measure of nitrogen in ZnO. These modes are interpreted as local vibrational modes. Furthermore, SIMS showed a correlation between the concentration of incorporated nitrogen and unintentional hydrogen.

Lunch break

12:30 - 14:00

Afternoon session - Luis Arizmendi

14:00 - 15:30

Main Building, room 219

Wide band gap materials: Optical properties

Optical performance of Yb^{3+} in LiNbO_3 laser crystals**Luisa E. Bausá¹⁾, María O. Ramírez¹⁾**

14:00 - 14:45

1) Universidad Autonoma de Madrid (UAM), Cantoblanco, Madrid 28034, Spain

invited oral

Yb^{3+} doped materials are recently attracting much attention as potential solid state laser materials due to the particular characteristics provided by the energy level scheme of this ion. Among others, its special electronic configuration makes the 4f electrons less shielded than in other ions of the lanthanide series, showing a higher tendency to interact with the lattice and neighbour ions. Thus, laser action from Yb^{3+} doped systems shows several interesting properties such as the possibility of a certain tunability range.

The most relevant results obtained in the $\text{Yb}^{3+}:\text{LiNbO}_3$ system will be shown.

On one part, stable laser action in the near infrared region and coherent green radiation by self-frequency doubling have been obtained simultaneously. The results have been obtained for single domain and PPLN crystals.

On the other hand, and from the fundamental viewpoint, under near infrared excitation cooperative green emission is obtained from Yb^{3+} ions in this system due to a relatively strong (dipole-dipole, dipole-quadrupole) interaction among 4f electrons of two neighbour Yb^{3+} ions. Their simultaneous de-excitation produces one visible photon of double the energy of the infrared one. By using the cooperative emission as a probe it is possible to determine the distribution of Yb^{3+} ions in the host matrix and predict the presence of Yb^{3+} pairs in the system.

Additionally, intrinsic optical bistability has been observed in materials exhibiting cooperative luminescence. Accordingly, preliminary results on the hysteretic behaviour of the luminescence from Yb^{3+} (in the near infrared

and in the green region of the spectrum) as a function of the excitation intensity will be also presented for Yb³⁺ activated LiNbO₃ crystals.

14:45 - 15:30

Wide bandgap materials for detection of ionizing radiation

Andrzej J. Wojtowicz¹⁾

invited oral

1) Nicolaus Copernicus University, Institute of Physics, Grudziądzka 5/7, Torun 87-100, Poland

In the last 10-15 years there has been a significant effort toward development and studies of new, more efficient and faster materials for detection of ionizing radiation. A growing demand for better scintillator crystals for detection of 511 keV gamma rays has been due mostly to recent advances in modern imaging systems employing positron emitting radionuclides for medical diagnostics in neurology, oncology and cardiology. While older imaging systems were almost exclusively based on BGO and NaI:Tl crystals the new systems, e.g. ECAT Accel, developed by Siemens/CTI, are based on recently discovered and developed LSO (Lu₂SiO₅:Ce, Ce-activated lutetium oxyorthosilicate) crystals. Interestingly, despite very good properties of LSO, there still is a strong drive toward development of new scintillator crystals that would show even better performance and characteristics.

In this presentation we shall review research conducted in the 90ties by a group led by Alex Lempicki in Boston, USA and then continued by a group in Torun, Poland. In particular we shall present spectroscopic and scintillator characterization of BaF₂:Ce, YAP (YAlO₃:Ce, cerium activated yttrium aluminate perovskite), and two relatively new complex oxide materials, namely LSO and LuAP (LuAlO₃, lutetium aluminate perovskite) activated with Ce. We shall present and discuss results of measurements of scintillation light yields, scintillation time profiles, VUV spectroscopy and low temperature thermoluminescence performed on these crystals. We shall demonstrate that all these experiments can be consistently interpreted in the frame of a scintillation model that assumes radiative recombination of electron-hole pairs via Ce ions as a prime scintillation mechanism.

15:30 - 15:50

Coffee break

15:50 - 17:10

Afternoon session - continued - Andrzej J. Wojtowicz

Main Building, room 219

Oral session II

15:50 - 16:10

Magnetotransport studies of Ga(Mn,Fe)N bulk crystals

C. Jastrzebski³⁾, **W. Gebicki**³⁾, **M. Bockowski**¹⁾, **B. Strojek**²⁾, **T. Szyszko**²⁾, **S. Podsiadlo**²⁾

oral

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GaN based DMS are considered the very promising wide band gap materials for spintronic applications. Electric properties (carrier concentration, mobility) of the materials are crucial for understanding the nature of magnetism in these materials and in consequence for use in possible applications. It seems undoubtedly that electric and magnetic properties of the materials are tightly coupled.

In our work we studied two types of samples GaMnN and GaFeN. The Mn doped samples were grown by high pressure method and chemical transport method and GaFeN samples were grown only by chemical transport method. Some of the GaN:Fe samples were co doped with Si atoms.

Van der Pauw resistance measurements and Hall resistance measurements were applied for the first samples and five contacts bar type method for the second ones. Measurements were performed in the range of temperature from 4.2 to 250K and at magnetic field up to 7 T. We obtained positive magnetoresistance for GaMnN but some GaFeN samples (especially the ones doped with Si atoms) indicates negative magnetoresistance.

Carrier concentration was extracted from the linear slope of the Hall resistance versus magnetic field. Electrons concentration of about 10e19 cm⁻³ was determined for GaMnN samples and it didn't depend from growth method but for GaFeN samples concentration exceeded 10e21 cm⁻³ indicating metallic type of the samples. At low temperature deviation from linearity of Hall resistance versus magnetic field was observed for small values of B and will be further discussed.

Magnetic properties of the same GaFeN samples were studied elsewhere and the magnetic measurements show coexistence of para- and ferromagnetic phases in the same sample. The effect was also visible in GaMnN samples but the ferromagnetic phase was significant weaker in this case.

Temperature dependence of resistance and Hall resistance of the samples was measured and will be discussed.

Partially supported by KBN grant PBZ-KBN-044/P03/2001.

16:10 - 16:30

Optical Detection of 2DEG in GaN/AlGaIn Structures - High Magnetic Field Studies

Barbara Chwalisz²⁾, **A Wysmolek**²⁾, **Roman Stepniewski**²⁾, **Marek Potemski**³⁾, **W Knap**⁴⁾,

oral

Jacek Baranowski²⁾, Nicolas Grandjean⁵⁾, J Massies⁵⁾, P Prystawko¹⁾, I Grzegory¹⁾

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5) CRHEA-CNRS, rue B. Gregory, Sophia Antipolis, 06560 Valbonne, France, France

Photoluminescence (PL) studies of the GaN/AlGaIn structure are presented. PL spectra measured in magnetic fields up to 28T show periodic intensity oscillations, what is interpreted as a fingerprint of 2DEG confined at GaN/AlGaIn interface.

The investigated structure was grown by molecular beam epitaxy (MBE) using highpressure bulk GaN as a substrate. The complete structure consisted of 2nm GaN quantum well (QW) embedded in the Al_{0.08}Ga_{0.92}N barriers grown on top of a thick GaN buffer layer.

Low temperature PL spectrum of the structure is dominated by the emission from the QW observed at 3.53 eV followed by sharp luminescence lines due to free and bound excitons originating from the GaN buffer layer.

In addition, in the energy range 3.40 - 3.45 eV of the PL spectrum, pronounced but relatively broad emission structure is observed. It shows typical temperature and excitation power dependencies as expected for 2DEG at GaN/AlGaIn interface [1-3]. This identification is confirmed by the observation of intensity oscillations in magnetic field. They are direct consequence of 2DEG Landau quantization and the Fermi level oscillations. This observation allows us to determine the electron concentration present at the GaN/AlGaIn interface. The obtained result is in good agreement with the value expected for this structure [4].

Surprisingly, it was observed that magneto-PL intensity oscillates also in other energy regions of the spectra. Appearance of these oscillations is discussed in terms of magnetic field induced potential redistribution and interactions between different layers existing in the structure.

[1] J. P. Bergman, et al. Appl. Phys. Lett. 69 (23), 3456 (1996)

[2] B. Shen, et al. Appl. Phys. Lett. 76 (6), 679 (2000)

[3] G. Martinez-Criado, et al. Mat. & Science Eng. B82, 200 (2001)

[4] O. Ambacher, et al. J. Phys. Cond. Matter 14 (13), 3399 (2002)

Sub-micron InGaIn Ring Structures for High-efficiency LEDs

Hoi Wai Choi¹⁾, Paul R. Edwards²⁾, Chan-wook Jeon¹⁾, Robert W. Martin²⁾, Martin D. Dawson¹⁾, Sudhiranjan Tripathy³⁾, Soo Jin Chua³⁾

1) Institute of Photonics, University of Strathclyde (IOP), Wolfson Centre, 106 Rottenrow, Glasgow G4 0NW, United Kingdom

2) Department of Physics, University of Strathclyde, Glasgow, United Kingdom

3) Institute of Materials Research and Engineering, Singapore, Singapore

Various forms of the InGaIn micro-LED concept have been reported in recent years, all of which indicate that an enhanced efficiency can be achieved with such microstructures. The overall external quantum efficiency of an InGaIn LED is heavily dependent on the internal quantum efficiency (which is limited by the build-in piezoelectric field) and the extraction efficiency (due to the high refractive index of nitride materials). A significant increase in extraction efficiency is certainly achieved in the current generation of micro-LEDs, where the dimension of an individual element of typically larger than 4µm. However, in order to achieve a reduction of the piezoelectric field in the InGaIn quantum wells, the dimension of the micro-LED should be further reduced. In fact, Demangeot et al observed significant strain relaxation in InGaIn microstructures of less than 1 µm through micro-Raman scattering, suggesting that micro-LEDs should be scaled down to sub-micron dimensions in order to benefit from an increased in internal quantum efficiency.

We report on the fabrication of sub-micron InGaIn ring microstructures using standard micro-fabrication techniques. 1mm disk structures are patterned using photoresist as a masking material and standard photolithography. The ring structures are spontaneously formed after photoresist development. The exact mechanism for the spontaneous ring formation is unknown at this time. The ring structures, as shown in the SEM image of Figure 1, have an external diameter of 1.5 µm with a wall width of 750nm. Preliminary evidence of strain relaxation in these structures is provided by the spectroscopic results shown in Figure 2. From the normalized cathodoluminescence spectra, a consistent blue shift is observed as the dimension of the InGaIn structure is scaled down. High-efficiency InGaIn micro-LEDs can potentially be fabricated from these sub-micron ring structures.

Surprisingly low built-in electric fields in quaternary InAlGaIn heterostructures

S. Anceau^{1,2)}, Pierre LEFEBVRE²⁾, L. Kończewicz²⁾, T. Suski¹⁾, S. P. Lepkowski¹⁾, H. Teisseyre¹⁾, Agata Kamińska³⁾, Andrzej Suchocki³⁾, H. Hirayama⁴⁾, Y. Aoyagi⁴⁾

16:30 - 16:50

oral

16:50 - 17:10

oral

- 1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland
 2) Universitete Montpellier2, XXXX, Montpellier F-34095, France
 3) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland
 4) Institute of Physical and Chemical Research (RIKEN), 2-1 Hirosawa, Wako-Shi, Saitama 351-0198, Japan

In ternary InGaN/GaN or GaN/AlGaIn quantum wells (QWs) grown along the (0001) direction, presence of large internal strain results in a piezoelectric polarization and thus in a built-in electric field in the QW layers. Moreover, spontaneous polarization characteristic for nitrides crystallizing in wurtzite structures causes an increase of the built-in electric field in both InGaN/GaN and GaN/AlGaIn QWs. The large internal electric fields induce in turn the separation of the electron and hole wavefunctions in the QW and a reduction of the optical transition matrix element. This effect is known as the Quantum Confined Stark Effect (QCSE). On the contrary, for the case of quaternary InAlGaIn based QWs one may consider cases where the QWs are strained (with compressive or tensile strain) or unstrained. Particularly, one may expect that, for properly chosen compositions of barriers and QWs, the internal electric field is negligible since a term coming from the spontaneous polarization cancels a term from the piezoelectric effect. In such a case no reduction of the optical transition probability due to QCSE occurs.

In this work we analyze the pressure behavior of light emission and the photoluminescence decay time, τ , for structures consisting of InAlGaIn based QWs. Both effects are very sensitive to the presence of the internal electric field in the QW systems. τ and the pressure coefficient of the light emission, dE_E/dp show drastic variation with the QW width in the QW system with strong built-in electric field in wurtzite InGaN/GaN and GaN/AlGaIn QWs. On the contrary, τ and dE_E/dp remain almost independent of the QW width when the internal electric field in QWs is negligible. It particularly applies to cubic nitride QWs.

We have found surprisingly low magnitude of the built-in electric field in the studied structures. This result is not confirmed by calculations based on available data concerning nonlinear piezoelectric properties of the used nitride alloys.

19:00 - 21:00

Poster session

Thursday, September 18th

09:00 - 10:30

Morning session

Main Building, room 219
 Lithium niobate A. Suchocki

9:00 - 9:45

Photonic applications of lithium niobate

Luis Arizmendi¹⁾

invited oral

1) Universidad Autonoma de Madrid (UAM), Cantoblanco, Madrid 28034, Spain

In this contribution the most important properties of lithium niobate crystals for photonic applications are reviewed. We will summarize how acting on the stoichiometry, sample structure, doping and domain structure of the crystals, these properties are governed and can be tailored for each specific application. Finally we present a review of photonic applications in a wide spectrum of fields as lasers and non-linear optics, optical communications, optical memories, and diffractive optics.

9:45 - 10:30

Phase relations in the growth of stoichiometric LiNbO₃

Katalin Polgar¹⁾, Ágnes Péter¹⁾, Michel Ferriol²⁾

invited oral

1) Research Institute for Solid State Physics and Optics of Hungarian Academy of Sciences (SZFKI), Konkoly Thege M. út 29-33, BUDAPEST H-1121, Hungary
 2) Metz University, Victor Demange, Saint-Avold 57500, France

LiNbO₃ is one of the most common perovskite-type crystals for optical applications. This a typical non stoichiometric material melts congruently at 48.6 mol % Li₂O content and can be grown easily by Czochralski method from its melt. The congruent composition is a serious drawback causing a defect structure with about 1% anti-site Nb⁵⁺ ions in the lattice. Therefore stoichiometric crystals (50 mol % Li₂O) are expected to show improved performance for a number of applications. In this structure the amount of anti-site Nb⁵⁺ is below 0.001% which results in the blue shift of the UV absorption edge, the narrowing of several spectral lines (EPR, NMR, Raman, IR OH-vibration lines, Cr³⁺ luminescent line of the doped crystal.) An important reduction of the electric field (200 V instead of 2kV) required for ferroelectric domain inversion in the stoichiometric crystal makes easier the production of periodically polarized structures for quasi-phase matching.

Several methods exist for the preparation of stoichiometric LiNbO₃ single crystals. The one which yields the composition closest to 50% Li₂O is the high temperature top seeded solution growth method from the K₂O- Li₂O-Nb₂O₅

ternary mixture. Part of this system has been investigated by several authors who concentrated on the formation of the tetragonal tungsten bronzes. So far no systematic research was done on the formation of LiNbO_3 . Our aim is to explore the limiting chemical and thermal conditions of the growth of stoichiometric LiNbO_3 single crystals from this three component solution.

For the construction of the phase diagram, besides the conventional thermal analytical and X-ray phase analysis, we used experimental crystal growth as well, for the identification of the consecutively crystallizing phases. With the combination of these methods we are able to determine the lay-out of the isotherms and to localize the singular points of the thermal surface representing the phase relations.

Morning session - continued - Axel Hoffmann

11:00 - 12:30

Main Building, room 219

Wide band gap materials: Technology and Applications I

Novel substrates for heteroepitaxy by lateral overgrowth technology

11:00 - 11:45

Zbigniew R. Zytewicz¹⁾

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

invited oral

Modern micro- and optoelectronic semiconductor devices consist of thin multilayers grown epitaxially on a substrate. These layers must be of high crystallographic quality. Otherwise, defects present in the structure deteriorate parameters of devices and lead to their fast degradation. Very often however, there are no suitable substrates for lattice-matched epitaxy and defects are generated at the epilayer/substrate interface. To prevent their propagation to the next-grown layers of the structure the lateral overgrowth technique has been elaborated. The breakthrough in development of long lifetime cw GaN/InGaN blue lasers, being due to high efficiency of defects filtration during lateral overgrowth, is the most spectacular recent achievement of the technique.

The talk will provide a general review of the epitaxial lateral overgrowth (ELO) technology and of application of ELO layers as substrates with adjustable lattice parameter. In particular, the issues of ELO growth mechanism, defect filtration during ELO procedure and strain in ELO layers will be addressed. Recent literature data on MOVPE ELO growth of GaN on sapphire or SiC and our results on lateral overgrowth of III-V compound semiconductors (GaAs/Si, GaSb/GaAs, etc.) by liquid phase epitaxy (LPE) will be used as examples. Finally, other lateral overgrowth techniques will be presented and compared with the conventional ELO technique.

Lasers and medicine

11:45 - 12:30

Przemyslaw Deren¹⁾

1) Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Okolna 2, Wrocław 50-422, Poland

invited oral

Light has always been a symbol of goodness and purity. Ancient Egyptians disinfected surgeon tools in flame; cured leukoderma using rays of the Sun. Herodotus in his papers also mentioned positive influence of Sun light on human health.

Old good methods have been forgotten for ages. Beginning of the XXth century brings certain interest in light therapy when sunbath was introduced to cure some dermatosis. The situation changed, however drastically when physicists constructed first LASER. At that moment new possibilities for diagnosis and therapy in medicine appeared, because laser-light had properties that had been never observed before. Neither natural source of light like Sun, nor artificial one like electric bulb produces light so monochromatic, directed and coherent.

Water, hemoglobin, melanin; three main tissue components have their specific absorption. Since laser-light is monochromatic selective impact on tissue components is possible. Because laser light is parallel it is possible to introduce all power of light into a fiberoptic and deliver it to the desired place in the body. Coherency of laser-light may be significant in wound treatment by weak laser radiation.

Physical bases of laser-light impact on human tissue are presented. Laser diagnosis and laser therapy, in particular:

- proper selection of laser for cutting of tissue,
- Low Level Laser Therapy (bio-stimulation) to improve wounds healing,
- Photodynamic Therapy (PDT) a new method for diagnosis and treatment of tumours and,
- emission properties of sick and health tissue

are reviewed in this lecture.

Lunch break

12:30 - 14:00

Afternoon session - Bernard Gil

14:00 - 15:30

Main Building, room 219

Wide band gap materials: Technology and Applications II

14:00 - 14:45
invited oral

SiC - Present Status and Material Challenges
Peder Bergman, **Liutauras Storasta**, **Carlsson Fredrik**, **Magnusson Björn**, **Jakobsson Henrik**, **Hallin Christer**, **Janzen Erik**

SiC is a material with properties of interest for high power and high frequency applications. The material quality including understanding and control of defects is however currently a limiting factor for many device applications. We will in this presentation discuss the current state-of-art of SiC for different applications, and the different material challenges in the form of material quality and reduction of defects.

The critical material issues are either related to general crystal quality, residual impurities, intrinsic defects, process related defects, or maybe most importantly to the influence of structural defects, such as dislocations, on the electrical properties of the material. The latter will be specially discussed and exemplified by the formation and properties of stacking faults in bipolar diodes.

14:45 - 15:30
invited oral

Applications of dislocation loop engineering to silicon
Kevin Homewood¹⁾, **M A. Lourenco**¹⁾, **R M. Gwilliam**¹⁾, **S A. Siddiqui**¹⁾, **M Milosavljevic**¹⁾, **Gousheng Shao**²⁾

1) *School of Electronics and Physical Sciences, University of Surrey, Guildford Surrey GU2 7XH, United Kingdom*

2) *School of Engineering, University of Surrey, Guildford Surrey GU2 7XH, United Kingdom*

The development of efficient light emitting devices in silicon would lead to major advances in the microelectronics industry and suggest a radically different approach to the development of optical communications as well as other key applications in the future.

There has been much recent progress in the field of silicon-based light emitting devices with several key reports of silicon-based devices that operate at room temperature with practical efficiencies. We show here developments of one of the most promising technologies - Dislocation Engineering. A key benefit of this technology, difficult to over emphasize, given the huge "tool-up" costs in the microelectronics industry, is that all the process steps are completely compatible with ULSI technology. Conventional implantation technology, using the standard dopant species are used to make light emitting diodes (LEDs) in crystalline silicon that operate efficiently at room temperature under forward bias.

Dislocation loop arrays, formed by ion implantation, are used to produce a strain field that can be made to modify the band gap of the silicon in such a way that the silicon itself can be used to provide spatial confinement of carriers. This enables the non-radiative recombination in the bulk and at the surface to be decoupled allowing efficient radiative recombination to occur in the active region of the device. Boron implantation has been used to form both the dislocation loop array and the p-type dopant to form a p-n junction in n-type silicon. The device operates conventionally under forward bias. Unpackaged LEDs are achieving external power efficiencies, at room temperature, of up to 0.1%, comparable, to that achievable in conventional III-V infra-red LEDs. Here we will also discuss methods of tuning the emission wavelength of the device over the important 1.1 to 1.7 μ m range.

15:30 - 15:50

Coffee break

15:50 - 17:50

Afternoon session - continued - Peder Bergman
Main Building, room 219
Oral session III

15:50 - 16:10
oral

Phonon band population inversion in GaN
Romuald BRAZIS¹⁾, **Romas RAGUOTIS**¹⁾, **Daniel NAUSEVIC**¹⁾
1) *Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania*

Gallium nitride crystals with their comparatively high-energy phonons, and strong electron-phonon coupling, seem to be promising for the phonon band population inversion affecting the two-phonon-difference absorption of infrared radiation. We devote this work to the theory of phonon generation by electrons in n-type GaN crystals at the lattice temperature T between 300 and 10 K, including deformation- and piezo-potentials, as well as ionized impurity contributions to the scattering rate of electrons. We determine the non-equilibrium phonon numbers using our calculated Monte Carlo data on phonon generation rate and literature data on phonon lifetimes. We conclude the inelastic process of optic phonon emission by electrons to be dominating in the electric field range from 1 to 10 kV/cm, and the optic phonon band population to be much in excess compared to the acoustic one at the same wave vector values. We give a trial of the phonon-photon coupling constant values for the two-phonon-difference process together with the non-equilibrium phonon number values, including photon re-absorption by free carriers, to calculate the resulting absorption coefficient. It turns out to be highly sensitive to electric fields in the range from

1 to 10 kV/cm, in the photon frequency range of 15-20 THz.

Novelties about cubic boron nitride luminescence properties

Elena M. Shishonok²⁾, J. W. Steeds¹⁾

1) *University of Bristol, Tyndall Avenue, Bristol BS8 1TL, United Kingdom*

2) *The Institute of Solid State & Semiconductor Physics, Belarus NAS (ISSSP), P. Brovka, Minsk 220072, Belarus*

16:10 - 16:30

oral

As superhard material cubic boron nitride (cBN) has been widely used in industrial application its usage as wide bandgap semiconductor is extremely being held back by the lack of basic knowledge of its properties. A wide band gap of 6.4 eV of cubic boron nitride (cBN) has made the material to be very attractive for its application in semiconductor and optical devices. As closest analogues of diamond cBN suppresses the latter in band gap, thermal, electrical and radioactive resistance, in possibility to emit secondary electrons. It can be easily transformed in n- and p-type and has a high electrical breakdown strength making a promising material for high-power transistor application. The most promising application of cBN is the use as field emitting device. Current applications are primarily based on silicon technology and operable up to 2000C only. cBN seems to be the only one that could cover the most demanding application range beyond 6000C. cBN has been considered as a model material for A3B5 group compounds at last. The real synthesis achievements under high pressure/high temperatures conditions of p- and n-type cBN doped by appropriate impurities have showed promising prospects for practical application and fabrication of p-n junction and ultraviolet light emitting diodes based on the material were realized. The perfect technology of n- and p-type cBN single crystals (or films) fabrication is very important for cBN application as semiconductor. No less than important is a development of different control methods of impurities and intrinsic defects in the material. In present work new dates are reviewed about recent photoluminescence (PhL) investigations of cBN single crystals irradiated by low energy electrons and doped by successful acceptor. PhL spectra of the near-threshold irradiated cBN (radiation damage centres RC1-RC4 and PF center) are analyzed in details. New fundamental characteristics of cBN are delivered

Defects dynamics in P+ implanted 6H-SiC, studied by AFM and positron annihilation

Grzegorz P. Karwaz^{1,2)}, Riccardo Rurali³⁾, Giovanni Consolati⁴⁾, Philippe Godignon³⁾

1) *Dipartimento di Fisica, Universita' di Trento, via Sommarive, 14, Trento 38050, Italy*

2) *Institute of Physics Pedagogical University of Stupsk (IF PAP), Arciszewskiego 22B, Stupsk 76-200, Poland*

3) *Centre Nacional de Microelectrónica (CNM) - CSIC, Barcelona 08193, Spain*

4) *Dipartimento di Fisica, Politecnico di Milano, Piazza L. da Vinci, 32, Milano I-20133, Italy*

16:30 - 16:50

oral

6H-SiC 0.6 mm thick monocrystals from Cree Inc. have been implanted by phosphorus ions with 10^{14}cm^{-2} dose at 280 keV energy. TRIM modelling yields the P^+ ions profile of 200 nm FWHM centred at about 380 nm. Samples were studied by positron annihilation technique, using a depth resolved, variable energy (0.1-25 keV) slow positron beam at Trento University. Doppler-broadening of the 511 keV annihilation line, indicating annihilation with low-momentum electrons was monitored. Positron-diffusion numerical packet VEPFIT was used to analyse data. For un-implanted samples present results agree well with data of Ohshima et al. (1998) - a slow fall of the annihilation S-parameter from the surface (0.493) to the bulk (0.465) value, indicating a long (190 nm) diffusion length, i.e. absence of positron-trapping defects. This result is also confirmed by positron-lifetime measurements performed at Politecnico di Milano, yielding essentially a lifetime of 148 ps. Ohshima et al. implanted samples with at a lower fluence - 10^{13}cm^{-2} and lower energy (200 keV) P^+ ions; their "as-implanted" samples show a similar dependence as our - with a wide flat region of slightly risen defectiveness (S-value of 0.497). However, in order to reproduce reasonably well the depth of the damaged layer, one has to assume presence of strong (10 kV/cm) electric fields in the implanted region. Otherwise, VEPFIT program would, as an artefact, indicate the implanted depth down to as much as 1100 nm. With annealing, S-curves show a rising maximum, moving towards surface - probably, in as-implanted samples the defects are decorated by P^+ ions and do not trap positrons in effective way. After the highest temperature annealing (1400°C for Ohshima, 1550°C for us), our results differ from those of Ohshima et al. In their samples, SiC recovers totally and no defects at any depth are seen by positrons; in our samples a highly defected region (S=0.524) still extends for the first 50 nm depth.

XPS and AFM investigations of buffer layer between Si(100) and 3C-SiC, created by interaction with C_2H_2

Andriy Goryachko¹⁾, Yevgen Yeromenko¹⁾, Karsten Henkel¹⁾, Yevgen Burkov¹⁾, Jürgen Wollweber²⁾, Dieter Schmeißer¹⁾

1) *Brandenburg Technical University, Department of Applied Physics/Sensorics (BTU), Erich Weinert Str. 1, Cottbus 03044, Germany*

16:50 - 17:10

oral

2) *Institute for Crystall Growth, Department of Vapour Growth (IKZ), Max Born Str. 2, Berlin 12489, Germany*

An epitaxial 3C-SiC/Si(100) system is of special interest for future microelectronics technology, since top performance SiC-based devices are created in a few-micrometer SiC film, simultaneously avoiding high costs of SiC wafer production. The buffer layer is an important aspect of epilayer growth, damping the lattice mismatch between two materials. We have chosen the Si(100) carbonisation technique of H. Nagasawa and K. Yagi /Phys. Stat. Sol. (b) 202 (1997) 335/ for buffer layer formation, due to its highest potential for commercial application. The silicon surface was carbonised by C₂H₂ diluted in H₂ (ratio 1:10) under the pressure of $\sim 10^{-1}$ Pa and in the temperature range from 700°C to 1100°C. The XPS investigation shows the onset of bulk SiC growth starting from 1000°C. This is an undesired scenario, because such process is accompanied by Si outdiffusion from the substrate and leads to a strained 3C-SiC/Si(100) interface with voids and micropipes /R. Scholz et al, Appl. Phys. A 64 (1997) 115/, thus producing rough morphologies of the buffer layer and subsequently of the 3C-SiC film itself. On the contrary, we observe a mild carbonisation in the range of 800°C to 900°C, where the SiC thickness is of the order of 1 monolayer (ML) - as judged from the XPS data. This scenario corresponds to an ideal buffer layer, namely where C atoms penetrate several ML deep into the Si substrate, while a completed ML of SiC is formed on its surface. This would be an ideal template for growing an epitaxial 3C-SiC film, with a smooth transition between two different lattice constants. We detect a small amount of O impurities in our buffer layers, coming from residual atmosphere of the growth chamber and from the contact with the environment. Their influence on the main 3C-SiC film, determined by AFM investigation of the surface topography, is determined by correlating the O concentration with the quality of the films grown on the corresponding buffer layer.

17:10 - 17:30

Multi-Phonon process in C6v4 (P63mc) hexagonal semiconductors: GaN, ZnO, 2H - SiC, 6H - SiC, ZnS, CdS

oral **Herbert Willi Kunert¹⁾**

1) *Department of Physics. University of Pretoria., University str., PRETORIA 0084, South Africa*

Based on the irrps of the C6v4 space group the selection rules for Raman active multi-phonon processes are derived. We used the following group-theoretical procedure:

- 1) Decomposing previously derived displacement representation (DR) onto irrps of all high symmetry points and lines, we obtained all possible modes and their symmetries. For example at Gamma (k^0), there are eight symmetry allowed modes. The frequencies of phonons can be measured by means of time-of-flight neutron spectroscopy. In this method, the neutrons are scattered by phonons with the wave vectors from the entire Brillouin zone. However, the accuracy of the measured long-wavelength phonons at k^0 is rather pure. In this case, the conventional back-scattering Raman spectroscopy yields the exact energies of long wavelength phonons at k^0 .
- 2) In order to determine the Raman active modes (one-phonon processes) we decompose the Kronecker Product (KP) of the Vector Representation (VR) onto irrps. This procedure yields at k^0 six Raman allowed modes. These modes are frequently observed. These are also present on our spectra from GaN, ZnO, and 6H-SiC.
- 3) Two-phonon processes. Overtones and combinations. The symmetry allowed overtones result from the complete reduction of the symmetrized squares (Sym.Sq) of the normal modes spanned by DR at critical points and high symmetry lines. Combinations are obtained from decomposition of the KP of two different species (symmetry allowed one-phonon processes).
- 4) Similarly, three-phonon overtones result from symmetrized cubes and the combinations from the complete reduction of the KPs of three different species. The derived selection rules at critical points are applicable to all wurtzite compounds with the space group C6v4. In order to verify our multi-phonon processes selection rules we have performed the inelastic light experiment on GaN, ZnO, and 6H-SiC. Some two- and three phonon processes are present on our spectra.

17:30 - 17:50

Temporal response of CVD diamond detectors to modulated low energy x-ray beams

oral **Gennaro CONTE¹⁾, Maria C. Rossi, Stefano Salvatori, Fabio Spaziani, Paolo Ascarelli²⁾, Daniele Trucchi**

1) *DIE University Roma Tre (DIE), Via Vasca Navale, 84, Rome 00146, Italy*

2) *CNR-IMIP, Viaalaria km 39,200, Rome, Italy*

Natural diamond is considered an optimum electronic material due to the uniqueness of its properties (i.e. Johnson figure of merit, breakdown voltage, maximum allowable frequency, thermal conductivity, etc.). However, due to the limitation of geological diamond, electronic devices based on this material have found only restricted usage. On the contrary, polycrystalline diamond deposited by CVD techniques is considered a new "wide bandgap material" which can be tailored to fulfill the requirements of the industry for the development of electronic devices. Therefore, temporal response of thin film polycrystalline materials often differ significantly from those of single crystalline bulk materials because of grain boundary effects. We have used a modulated 8.06 keV X-ray beam to study the photoconductive response of films deposited by a CVD technique. The collection efficiency of charge carriers under irradiation both in DC and AC conditions has been studied to infer the transport mechanisms as a function of the modulation frequency and film morphology. The evolution of the device sensitivity to the ionising radiation

has been analysed and a correlation with the mobility-lifetime product is proposed. Finally, achieved results will be correlated with sub bandgap photoconductivity spectra able to evaluate the amount of grain boundary related defects.

Friday, September 19th

Morning session - Matthew R. Phillips

Main Building, room 219
Oxides II

09:00 - 10:30

Advanced electronic and optoelectronic materials by Atomic Layer Deposition: An overview with special emphasis on recent progress in processing of high-k dielectrics

Lauri Niinistö¹⁾, Minna Nieminen¹⁾, Jaakko Niinistö¹⁾, Matti Putkonen¹⁾, Jani Päiväsaari¹⁾

1) Laboratory of Inorganic and Analytical Chemistry, Helsinki University of Technology, P.O. Box 6100, FIN-02015 Espoo, Helsinki, Finland

9:00 - 9:45

invited oral

Atomic layer deposition (ALD) was originally developed in the 1970s to process wide band-gap semiconducting II-VI sulfides as well as insulating oxides for thin film electroluminescent (TFEL) displays. ALD is now a mature technique which has found other applications as well, e.g. in producing catalysts and sensors. Currently, ALD is experiencing a breakthrough as a method-of-choice in the semiconductor industry where so-called high-k insulating oxides can be processed by ALD as very thin overlayers with precise thickness control.

The dielectric constants for conventional dielectrics such as SiO₂, silicon nitride and alumina are 3.9, 7, and 9, respectively, but much higher k-values (in the range of 15 to 30) can be obtained with the oxides of Y, La, Hf and Zr. We have recently demonstrated that all these high-k oxides can be processed in a surface-controlled ALD mode exploiting various precursor approaches each having its own advantages. Thus, for instance, completely oxidized and high density films can be obtained by the use of ozone, while low processing temperatures and high growth rates are characteristic for the use of true organometallics, viz. cyclopentadienyl compounds.

When choosing the precursor chemistry, the impurities of the films and reactions at the silicon interface need also to be considered requiring advanced analytical characterization by XPS, TOF-ERDA, X-TEM and other surface-sensitive and high-resolution techniques. Finally, electrical measurements are used to evaluate the dielectric properties.

Atomic Layer Deposition of Oxide Dielectric Films for Microelectronics

Markku Leskela¹⁾, Kaupo Kukli, Mikko Ritala

1) Department of Chemistry, University of Helsinki, A. I. Virtasen aukio 1, Helsinki FIN-00014, Finland

9:45 - 10:30

invited oral

The special features of atomic layer deposition (ALD): easy control of layer thickness also in ultra thin films and conformal coating with extreme aspect ratios can be utilized in microelectronics in MOSFET gate oxides and trench capacitors in DRAM technology, respectively. In both application dielectrics with permittivities greater than that of SiO₂ are required to continue downward dimensional scaling of the devices. In gate dielectrics ZrO₂ and HfO₂ are the most often studied alternative materials whereas in DRAM capacitors very high k-materials or ferroelectrics like BST (barium strontium titanate) and SBT (strontium bismuth tantalate) are examined. In this presentation the deposition of these materials by ALD is described.

In ALD the choice of precursors is extremely important. Most of the ALD studies on ZrO₂ and HfO₂ films have been dealing with one process, tetrachloride plus water. We have studied several alternative metal precursors such as iodides, alkoxides, aminoalkoxides, alkylamides and hydroxylamides. All precursors have their own impact to the electric properties of the ZrO₂ and HfO₂ films. The deposition of ternary oxide films by ALD is not very straightforward and totally new processes had to be developed. These processes are based on metalorganic compounds as metal source and water as oxygen source. In the presentation the growth processes will be discussed in more detail.

Coffee break

10:30 - 11:00

Morning session - continued - Kevin Homewood

Main Building, room 219

11:00 - 13:00

Oral session IV

11:00 - 11:20

oral

An Atomistic view of the oxidation mechanism of silicon clusters on (0001) SiC 3x3 reconstructed surface**Kian Ping Loh¹⁾, Jia Mei Soon²⁾, Ida Ma³⁾***1) National University of Singapore (NUS), Singapore, Singapore**2) National University of Singapore (NUS), Singapore, Singapore**3) Institute of High Performance Computing (iHPC), Singapore, Singapore*

Silicon carbide (SiC) is a promising material for applications in high-power and high-temperature electronic devices. Besides these highly desirable properties, SiC is the only compound semiconductor that can be thermally oxidized to form SiO₂. However, SiC exhibits worse electrical passivation characteristics compared to Si due to the presence of silicon oxycarbide species that produce interfacial states at the oxide/SiC interface. For this reason, studies of the oxidation mechanism and chemical composition of thermally grown oxides on SiC have attracted a lot of attention recently.

The oxidation of Si-enriched SiC surfaces present interesting fundamental problems in terms of the sites where oxygen will favor an attack, and the possibility of forming well-ordered silicon oxide nanoclusters. Using first principles calculations, we studied the problem of oxygen insertion into well-ordered 3x3 reconstructed Si-enriched SiC nanoclusters.

The various configurations of physisorbed and chemisorbed oxygen have been identified and relaxed theoretically using the Gaussian98 suite. Transition states of the all the oxidation products have been ascertained and the activation energy for each oxygen-addition step has been calculated. The knowledge of activation energy for each oxygen-addition step allows quantitative and qualitative assessment of the most stable oxidation states. With this knowledge, we mapped out the potential energy diagram for the oxidation mechanism of Si-enriched SiC. This information, coupled with scanning tunneling microscopy (STM) images of the oxidation process, allowed us to identify the most stable oxidative products of the 3x3 reconstructed Si-enriched SiC surface.

11:20 - 11:40

oral

Mechanisms of optical control of domain structures in ferroelectrics**Armen R. Poghosyan¹⁾***1) Institute for Physical Research (IPR), IPR-2, Ashtarak-2 378410, Armenia*

Intensive studies on fabrication, characterization and performance of second harmonic generation devices on periodically inverted ferroelectric domain lithium tantalate crystals are taking place, but studies on basic material properties of crystals in the domain inversion process are lacking.

Several methods have been applied to obtain the crystals with regular domain structures. The most common is the use of patterned electric field poling, but the quality of resultant periodically poled material can depend on a number of factors and periodicities below ~3 μm are difficult to produce however, particularly with simultaneously large aspect ratios.

On the other hand all post-growth techniques for achieving periodic domain profiles require either specialist clean-room photolithographic fabrication of patterned electrodes, or lengthy thermal or electron-beam scanning processes. Recently the experiments demonstrating the advantages of direct optical control of ferroelectric domain profiles have been carried out. Here we present our findings and detailed analysis on time dependence of the ferroelectric coercive field after domain inversion of the LiTaO₃ and stoichiometric LiNbO₃ crystals and report a simple room temperature technique for patterning the domain profile by the simultaneous application of combined electrical and optical fields: the electric field is applied via planar electrodes, while light is used to define those regions where domain inversion should occur. The optical poling route therefore offers a potentially simpler method, effectively eliminating the photolithographic patterning steps.

We have investigated the mechanisms of optical control of domain structures in ferroelectrics, in particular the roles of the various internal field components, their origins and dynamic behavior following domain reversal. Our experiments have shown the possibility of direct optical control of domain patterning in ferroelectrics.

11:40 - 12:00

oral

Cr³⁺ IONS IN HYDROGENATED AND PROTON EXCHANGED LiNbO₃ CRYSTALS**Agata Kaminska¹⁾, Luis Arizmendi²⁾, Andrzej Suchocki¹⁾***1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland**2) Universidad Autonoma de Madrid (UAM), Cantoblanco, Madrid 28034, Spain*

Lithium niobate (LN) crystals remain of the interest for applications as well as for fundamental studies. The Cr³⁺ ions turned out to be a very good probe of that structure since they substitute for both Li or Nb ions in LN host. Several studies with use of EPR and ENDOR techniques have been performed that helped to identify several Cr³⁺ centers both in Li (Cr_{Li}) and Nb (Cr_{Nb}) sites. Also high-pressure low-temperature optical spectroscopy was very successful in identification of optically active Cr³⁺ centers. High-pressure application transforms low-strength

crystal field of Cr^{3+} centers into high-strength crystal field, what increases spectral resolution of the measurements and allows for their easier characterization.

Doping of lithium niobate with trivalent ions requires some charge compensation. Recently on the basis of EPR and ENDOR it has been suggested that interstitial hydrogen ions or additional Li^+ ions in structural vacancies in LiNbO_3 can provide required charge compensation for chromium ions in niobium sites (Cr_{Nb}). It is known that LN can accommodate large amount of OH^- molecules and their concentration can be controlled either by annealing crystals in water vapor or by proton-exchange in relatively thin surface layer of the crystal.

We studied influence of both annealing in water vapor and proton exchange on high-pressure low-temperature luminescence spectra of near stoichiometric $\text{LN}:\text{Cr},\text{MgO}$ crystals, containing both Cr_{Li} and Cr_{Nb} centers by using diamond-anvil cell. The observed changes of the spectra of Cr^{3+} ions are associated with the larger inhomogeneous broadening and increase of the splitting of the ${}^2\text{E}$ level in one of the Cr_{Li} center, so called center γ . This shows that neither increase of hydrogen concentration in LN by almost two orders of magnitude (from 10^{18} to almost 10^{20} cm^{-3}) nor proton-exchange procedure do not create any new optically active Cr^{3+} centers in those crystals.

Elementary energy bands in band structure calculations of some wide bandgap crystals

12:00 - 12:20

Malgorzata Sznajder¹⁾, D. M. Bercha^{1,2)}, K. Z. Rushchanskii²⁾

oral

1) Institute of Physics, University of Rzeszow, Rzeszow, Poland

2) Uzhgorod, National Uzhgorod University, Ukraine, Ukraine

In this report we shall discuss a method which allows to predict and to verify in an easily way numerical calculation results of the band structure of semiconducting and dielectric crystals. The method presented is based on the Zak's concept of the elementary energy bands and it is particularly useful for crystals with a large number of atoms in a unit cell. We demonstrate that the elementary energy bands create the crystal band structure, moreover, they can be obtained starting from the empty-lattice approximation completed by the general data concerning a semiconducting crystal (the existence of the forbidden gap, lattice constants, number of valence electrons in the unit cell and space symmetry group). It should be emphasized that the presented method does not require information about positions of atoms in the unit cell and gives initial information about electron density distribution and chemical bonding. Our method has been applied to the wide bandgap YAlO_3 dielectric crystals, to the acoustooptic family of Tl_3AsS_4 crystals and, for comparison, to narrow band crystals. It was confirmed by ab initio numerical calculations of the band structure of some of these crystals. The obtained results are in a good agreement with experimental data.

Field Emission from Si nanoclusters embedded into insulating SiO_2 films

12:20 - 12:40

Anatoli A. Evtukh²⁾, Ivan Z. Indutnyy²⁾, Igor P. Lisovskyy²⁾, Vladimir G. Litovchenko²⁾, Denis O. Mazunov²⁾, Hans Hartnagel¹⁾, Oktay Yilmazoglu¹⁾

oral

1) Technische Universitat Darmstadt, Darmstadt, Germany

2) Institute of Semiconductor Physics (ISP), Nauki pr., Kiev 03028, Ukraine

Efficient electron field emission from silicon flat cathode coated with SiO_x film ($x=0.3-0.5$) was observed both before and after thermal (1000C) annealing with subsequent etching in HF solution. Oxide films were produced by silicon thermal evaporation in vacuum (10-5Torr.). Using optical spectroscopy in visible light and in infrared ranges, and AFM technique the structural features of these films were investigated. It was shown, that initial SiO_x film may be represented as $\text{SiO}_x(\text{Si})$ composite ($x=1.3$). Thermal annealing causes further phase segregation in the film material and it is transformed into $\text{SiO}_2(\text{Si})$ composite. During such a process silicon grains size decreases and their density increases. The model of electron field emission from the surface of such films was proposed. It was supposed that limitation process of the current flow under high electric fields is connected with Fowler-Nordheim tunneling through barriers $\text{Si-SiO}_x+\text{vacuum}$ or Si-vacuum . Current peaks on emission I-V characteristics were explained in the framework of resonance tunneling mechanism.

Investigated structures seems to be perspective for application as flat field cathodes in vacuum electronic devices and in flat-paneling field emission displays.

Properties of ultrathin SiO_2 films grown on hydrogenated silicon

12:40 - 13:00

Igor Lisovskyy¹⁾, Vladimir G. Litovchenko¹⁾, D A. Mazunov¹⁾, A Szekeres²⁾, S Alexandrova²⁾

oral

1) Institute of Semiconductor Physics NAS Ukraine, Kiev, Ukraine

2) Institute of Solid State Physics, Sofia, Bulgaria

The optical and electrical properties of ultrathin (8-12 nm) SiO_2 layers thermally grown at 850 °C on rf-hydrogen plasma-cleaned Si studied and are compared with those of oxides grown on standard RCA-cleaned Si. The structural characterization was performed using spectral ellipsometry and IR techniques. IR data was used for computer simulation of oxide structure. Characterization of electrically active defects has been performed by frequency dependent C-V technique. From the analysis of the pseudo-dielectric function of Si the oxidation-induced stress was evaluated. Its level was in the order of 10^8 N/m^2 and was substantially smaller in plasma treated samples. An analysis of the IR spectra has shown that the structure of SiO_2 grown on hydrogenated silicon possesses a contribution of 4- and 6-fold SiO_4 tetrahedra rings. The structure of the standard oxides contains an additional

contribution of SiO_2Si_2 molecular complexes. Their presence leads to increase of the distances between neighbouring Si atoms and to increase of stresses in SiO_2 lattice. Electrical measurements of such structures have also revealed larger densities of interface defects (fixed oxide charge and interface traps). This is in correlation with content of SiO_2Si_2 clusters, which are known to contribute to the fixed oxide charge.

13:00 - 13:10

Closing address

Symposium B

Development of Methods for Characterizing the Microstructure of Novel Materials

Introduction

I have great pleasure in welcoming you to Symposium B: Development of methods for characterizing the microstructure of novel materials of the E-MRS 2003 Fall Meeting. The Symposium is co-organized by the Institute of Physics of the Polish Academy of Sciences; as part of its activities as a Centre of Excellence, CELDIS; and the Polish Synchrotron Radiation Society (PSRS).

The increasing interest in fabrication of nanometer-sized structures creates new challenges for the methods used for materials characterization on the atomic scale. Knowing and controlling the nano-scale microstructure is considered to be the key factor for the development of advanced materials. Excellent tools for this purpose have been provided by the rapid development of synchrotron radiation sources, electron microscopy and spectroscopy, and secondary ion mass spectroscopy. We have done our best to bring together scientists exploring the opportunities offered by new instruments and those involved in the fabrication technology of novel materials, and we hope that this will allow recognition of the needs and actual limits of the methods used for materials characterisation.

As a demand of technology, numerous techniques have been developed to investigate the structure of matter on an atomic scale. This has enabled physicists to describe, with increasing precision, the fundamental interactions in matter. These techniques are mainly based on radiation and particle interactions with matter. During recent years scientists have described precisely, using advanced mathematics and numerical simulation, the propagation and absorption phenomena of X-rays, electrons and particles. Nowadays, these techniques are commonly used in medicine, pharmacology, biology, archeology, physics, chemistry and materials science.

I would like to express my deep gratitude to all lecturers for acceptance of our invitation to make a presentation at Symposium B and to the participants choosing the Program of Symposium B. Finally, I would like also to thank the Scientific and Organizing Committee for their joint efforts in organizing the scientific programme of the Meeting.

I wish all of you enjoyable and fruitful stay during the E-MRS Fall Meeting

Krystyna Ławniczak-Jabłońska, Organizer of Symposium B

Welcome address by the President of Polish Synchrotron Radiation Society

You are cordially welcome to Symposium B: Development of methods for characterizing the microstructure of novel materials of the E-MRS 2003 Fall Meeting. It is my pleasure to represent the Polish Synchrotron Radiation Society (PSRS) as co-organizer of the Symposium.

It was a dream of many scientists to have a continuous source of high intensity light with a wide spectrum range e.g. source of light which covers the optical and X-ray range of photon energies from 1 eV to 100 000 eV. This dream came true when the electron emitting radiation (source of light) was accelerated to the velocity close to the speed of emitted light. It appears in a circular vacuum tube (storage ring) where the electrons, guided by powerful magnets are accelerated by high frequency microwaves. The spectrum spans from microwave, through infrared,

visible, ultraviolet, vacuum ultraviolet, soft X-ray and hard X-ray radiation up to 100 000 eV. The intensity of radiation is several orders of magnitude higher than standard ultraviolet lamps or X-ray tubes and it is polarized, collimated, and pulsed electromagnetic radiation. New generations of these magnificent sources of radiation were built worldwide and all of them were surrounded by excellent experimental equipment for effective application in optical and X-ray experiments. Modern SYNCHROTRONS with high resolution monochromators provide monochromatic radiation in any range of SYNCHROTRON RADIATION. Modern experimental equipment based around the synchrotron, create an excellent laboratory for optical and X-ray experiments. New scientific techniques are being developed in areas of atomic, molecular, solid state physics, physical chemistry, biophysics and medicine. Synchrotrons have become the center of interdisciplinary laboratories applying modern optical and X-ray radiation sources. As an example, the precise measurement of X-ray atomic absorption edges EXAFS (Extended X-ray Absorption Fine Structure) or XANES (X-ray Absorption Near Edge Structure) have been performed. The anomaly strong Fano-type resonance correlated to the highly localized transitions 4d-4f in rear earth or 3p-3d in transition metal atoms were observed for the first time and applied for the study of the electronic structure of these atoms and these localized electrons contribution to the valence band electronic structures, e.g. to the semimagnetic semiconductors valence band structure.

The Polish Synchrotron Radiation Society (created in 1991), consists of a group of about 100 scientists to organize annual meetings and edit the Bulletin on "Synchrotron Radiation in Natural Science". A main goal of the Society is to: - exchange and disseminate information about possible experiments to be performed with synchrotron radiation and sponsoring it in different countries, - lecturing, teaching and popularizing knowledge on synchrotron radiation and publicizing the newest results obtained by its application. Polish scientists actively participate in writing proposals and undertaking for important and interesting experiments. I cordially invite participants to join our society and actively, and with success, apply synchrotron radiation to solve research problems associated with new materials.

Prof. dr. hab. Bronisław Orłowski President of the Polish Synchrotron Radiation Society

Organisers

- **Krystyna Lawniczak-Jablonska**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Rajmund Bacewicz**, Warsaw University of Technology, Department of Physics, Poland
- **Vaclav Holy**, Masaryk University, Faculty of Sciences, Brno, Czech Republic
- **Ake Kvick**, European Synchrotron Radiation Facility, Grenoble, France
- **Sergei Malyshev**, National Academy of Science of Belarus

Proceedings

Papers will be published in Journal of Alloys and Compounds.

Monday, September 15th

Opening address - Krystyna Lawniczak-Jablonska, Bronislaw A. Orlowski 13:50 - 14:00

Main Building, room 134

Afternoon session - Maria Lefeld-Sosnowska 14:00 - 15:30

Main Building, room 134

X-ray imaging of semiconductor micro- and nanostructures in real and reciprocal space 14:00 - 14:30

Tilo Baumbach¹⁾ invited oral

1) Fraunhofer Institute for Nondestructive Testing (EADQ), Kruegerstrasse 22, Dresden 01326, Germany

Quality inspection and defect analysis starting for semiconductor wafers, including layers, nanostructures up to the complete electronic devices are crucial for improvements of the fabrication technology, based on correlation of device properties with the different technological processes.

The talk demonstrates new capabilities of X-ray imaging techniques with conventional and synchrotron radiation sources for detailed quality inspection concerning structural perfection. It covers real space imaging of microstructures and reciprocal space imaging of nanostructures employed to characterize grown-in and process-induced defects as well as stresses.

Considering real space imaging, it includes results for different materials (SiC, GaAs, InP, GaPO₄, GaN and ultrathin silicon) and devices (wafers, thin layers up to completely mounted opto-electronic devices under operation) as well as different fabrication technologies. Examples are: surface damage in thick and ultra-thin silicon wafers, μm -resolved lattice misorientation and dislocation density maps, μm -resolved imaging of GaN layers fabricated by epitaxial lateral overgrowth, degradation in semiconductor laser devices.

Regarding reciprocal space imaging, it overviews high-resolution X-ray scattering techniques for the characterization of periodic nanostructures (e.g. gratings, etched quantum wires and quantum dots, self assembled patterning in strained superlattices).

Examples are: the evolution of residual lattice strain in buried patterned structures during of planar growth, patterning and embedding, periodic interface strain in ultra-thin twist bonded samples, interface morphology, lateral correlation and vertical replication properties in the case of strain-driven self-assembled patterning.

In-Situ High Energy Ultra-Fast X-Ray Micro-Tomography 14:30 - 15:00

Marco Di Michiel¹⁾, David Fernandez¹⁾, Veijo Honkimaeki¹⁾, Thomas Buslaps¹⁾, Thierry Martin¹⁾, Peter Falus²⁾, Luc Salvo³⁾, Olivier Ludwig³⁾, Daniel Bellet³⁾, Olivier Lame³⁾, Didier Bouvard³⁾ invited oral

1) European Synchrotron Radiation Facility (ESRF), Grenoble BP220-38043, France

2) Massachusetts Institute of Technology (MIT), Cambridge 02139, United States

3) Laboratoire Génie Physique et Mécanique des Matériaux (GPM2), Saint Martin d'Herès 38042, France

X-ray computed micro-tomography (CMT) is a non-destructive technique for three-dimensional (3D) imaging from projection data. For many years it has been extensively used at synchrotron radiation sources to characterize the microstructure of materials. By recording high-resolution x-ray transmission images (projections) at different angular positions CMT allows to reconstruct a 3D map of the attenuation coefficient of an object. CMT is a relatively slow technique because the collection of an entire dataset requires several minutes. For this reason it is used for characterizing static systems. "In-situ" experiments have been performed only in a few limited cases. Moreover CMT is mainly used to characterize low Z materials because high Z materials, like metals or ceramics, require a high-energy x-ray beam and special optics that do not suffer of radiation damage. Here we show how it is possible to overcome these limitations. Combining the high flux and high energy of the ID15 white x-ray beam with a new ultra-fast imaging detector and a new mirror optics, it is possible to record a full high-resolution 3D dataset in 10 seconds. This is two orders of magnitude faster than what was achieved before at third generation synchrotron sources. The very high speed allows for the first time to perform "in-situ" studies of systems evolving in the timescale of few seconds. Applications are the study of the micro structural evolution in powder samples during sintering process, partial re-melting in semisolid phases and solidification of alloys, grain growth and morphological evolution, diffusion etc. We present some preliminary results obtained on the solidification process of a binary alloy (Figure 1) and on the sintering of metallic powders.

15:00 - 15:30

invited oral

High resolution x-ray scattering from self-organized nanostructures**Julian Stangl²⁾, Vaclav Holy¹⁾, Anke Hesse²⁾, Rainer T. Lechner²⁾, Günther Bauer²⁾***1) Masaryk University, Faculty of Sciences, Kotlarska 2, Brno 61137, Czech Republic**2) Institut für Halbleiterphysik Universität Linz, Altenberger Str. 69, Linz A-4040, Austria*

Semiconductor nanostructures receive increasing interest due to their potential for new device concepts, as well as from a scientific point of view. In structures smaller than the DeBroglie wavelength of electrons or holes, quantum confinement effects determine the electronic and optical properties. In typical semiconductor materials, these dimensions are in the order of several nm. Self-assembly is a powerful mechanism to fabricate a large number of nanostructures directly during epitaxial growth.

The properties of self-assembled structures depend sensitively on growth conditions such as substrate temperature, adatom flux, surface miscut etc. For the understanding and finally control of growth in order to fabricate structures with designed properties, structural investigation, i.e., the determination of size, shape, chemical composition and strain state is mandatory. X-ray diffraction is a powerful technique for this purpose. In particular, the strain fields within nanostructures as well as in the surrounding matrix can be determined with high precision. As strain fields depend sensitively on other properties such as size and chemical composition, the latter become accessible, too. With synchrotron radiation, even the distribution of chemical composition within objects with typically several nm height and 10 to 100 nm width can be established. Furthermore, with x-ray diffraction, the non-destructive investigation also of buried structures is possible. The latter is important, as for applications buried structures are needed, and during capping the structural properties may change considerably.

This talk will present scattering techniques sensitive to shape, composition and strain of nanostructures. Results will be presented on the shape and positional correlation of nanostructures in superlattices, and on the composition and strain profile in self-assembled islands on a sample surface. The effects of capping on such islands will be addressed as well.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duža aula)

15:50 - 17:00

Afternoon session - continued - Czesław Kapusta

Main Building, room 134

15:50 - 16:05

oral

Characterization of defects in protein crystals by means of synchrotron multiple-crystal topography and reciprocal space mapping**Bernard Capelle^{2,3)}, Yves Epelboin^{2,3)}, Jürgen Härtwig¹⁾, Anna B. Moraleda⁴⁾, Fermin Otálora⁴⁾, Vivian Stojanoff⁵⁾***1) European Synchrotron Radiation Facility (ESRF), Grenoble BP220-38043, France**2) Université P.M. Curie, case 115, Paris 75252, France**3) LMCP, UMR 7590 CNRS, France**4) LEC (IACT), Campus Fuentenueva (Fac. Ciencias), Granada 18002, Spain**5) Brookhaven National Laboratory, National Synchrotron Light Source, Upton, NY 11973, United States*

X-ray diffraction topography (XRT) and reciprocal space mapping (RSM) are used to study the perfection of single crystals. They allow in a complementary way obtaining local and integral information about their defects and defect structures, or their mosaic spread. Modern synchrotron radiation sources in combination with modern detectors, this lead among others to higher resolution, shorter adjustment and exposure times, and to the possibility to record weak reflections or to work under weak beam conditions. Dedicated instruments permit combining several experimental methods within one experimental set-up - e.g. XRT, with RSM, and data collection for structure analysis. This allows relating the defect structure of crystals with a) growth conditions and processes as well as the effects of crystal treatment, and b) the resulting properties (diffraction conditions, physical properties). This will be demonstrated characterizing the perfection of protein crystals. Hen egg white lysozyme was chosen as model protein owing to its high resistance to radiation damage, feasibility of growing large rather perfect crystals and easy handling. The crystals were oriented and its reflections were indexed using a MAR image plate detector. In that way we could work with selected and well-indexed reflections. A comparison of results of double and triple crystal topography, high-resolution diffraction and reciprocal space mapping allowed obtaining complementary information about the defect structure of the crystals. The crystals revealed a number of features quite well known in solution or hydrothermal grown inorganic crystals: dislocations, growth bands and growth sector boundaries. Dislocations in the $\langle 110 \rangle$ sectors have been characterised as edge dislocations with Burgers vector parallel to the c axis. They were distinguishable only under weak beam conditions. The existence of spiral growth steps (observed by surface techniques) is consistent with the presence of edge dislocations.

16:05 - 16:20

oral

Multifractal analysis in diagnostics of nanostructures**Natalia M. Shmidt¹⁾, S. V. Ivanov¹⁾, A. V. Kamanin¹⁾, A. G. Kolmakov²⁾, A. V. Loskutov²⁾,**

A. N. Titkov¹⁾

1) *Ioffe Physico-Technical Institute (Ioffe), Polytechnicheskaya, 26, St.-Petersburg 194021, Russian Federation*

2) *Institute of Metallurgy, Moscow 117911, Russian Federation*

Materials produced by modern nanotechnology, as a rule, have a complicated structure. It should be noticed that most of diagnostic techniques characterize the materials either integrally or locally, while the methods revealing, for example, topographic peculiarities of the complicated systems do not always allow the different structures to be compared quantitatively. Moreover, traditional techniques allow no internal bindings to be revealed in complicated systems, while these bindings determine many system properties.

In the present paper a new approach to characterization of both III-nitrides and nanostructures based on III-V and II-VI compounds is presented. The approach uses the multifractal analysis to process the Atomic-Force Microscopy data, the mapping data of electrical and optical properties of these systems. As a result, such characteristics of the complicated systems as the level of self-organization, the degree of order, the degree of local symmetry disruption could be quantitatively obtained.

The degree of order of a III-nitride mosaic structure has been established to characterize the structural properties more exactly than surface roughness. The direct correlation between mobility, optical properties and degree of order of the III-nitride mosaic structure has been established.

For the first time, the level of self-organization of a quantum dot system based on II-VI and III-V semiconductor compounds have been quantitatively determined.

Modelling of indium rich clusters in MOCVD InGaN/GaN multilayers
Grzegorz Jurczak²⁾, Grzegorz Maciejewski²⁾, Sławomir Kret¹⁾, Paweł Dłużewski²⁾, Pierre Ruterana³⁾

16:20 - 16:35

oral

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In this approach the study of a 1-5 nm diameter indium cluster in MOCVD InGaN/GaN quantum well is presented. The quantitative High Resolution Transmission Electron Microscopy (HRTEM) coupled with image simulation and Finite Element Method (FEM) thin foil relaxation modeling are used. Results obtained by means of those two technics are compared.

The measurement of tetragonal distortion from experimental images is the powerful tool for the determination of chemical composition in heterostructures. Transmission Electron Microscopy samples used in high resolution mode have 5-15 nm thick for 11-20 zone axis. So, the foil thickness is bigger than the size of studied nanocluster. For the correct interpretation of the measured lattice distortion on HRTEM images one needs to take into account the strain averaging across TEM sample and inhomogeneous relaxation of the sample. As a function of a chemical composition, size and position of In cluster relative to foil surface the 3D FEM modeling of such relaxation phenomena have been performed. Obtained 3D displacement distribution were used to simulate the HRTEM images. The results show that such modeling, which takes into account the atomic column bending, gives well matching of simulated images with the experimentally observed. Additionally the most probable size, position in quantum well and indium concentration are determined with small error bar.

Structure characterisation of MBE-grown ZnSe:Cr layers
Michel Jouanne³⁾, Jean-Francois Morhange³⁾, Elzbieta Dynowska¹⁾, Elzbieta Lusakowska¹⁾, Wojciech Szuszkiewicz¹⁾, Laurens W. Molenkamp²⁾, Grzegorz Karczewski^{1,2)}

16:35 - 16:50

oral

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The stability of the ferromagnetic state in Cr-based diluted magnetic semiconductors (DMS) has been recently predicted. A few years ago, the low-temperature MBE technique resulted in the successful growth of II-VI-based DMS incorporating a relatively large Cr concentration, i.e., (Zn,Cr)Se epilayers with Cr concentration up to 2.1% were obtained [1]. Evidence of a strong ferromagnetic coupling between Cr ions was given in Ref.[1]. However, the possible domination of some magnetic properties of investigated layers by the presence of small grains of ZnCr₂Se₄ spinel was also suggested (direct proof of even partial substitution of Zn by Cr in ZnSe has not been found).

The goal of the present work is to look for efficient methods of characterisation of the structure of such layers and to check the possible presence of mixed (Zn,Cr)Se crystals in the zinc-blende phase. MBE-grown, Cr-doped ZnSe layers were deposited on (001) GaAs substrates (which, in some cases, were also covered with a pure ZnSe buffer layers). The structure of layers was investigated by means of the high-resolution X-ray diffraction, atomic force microscopy and Raman scattering. Presence of Cr at substitutional positions is demonstrated, and details of

(Zn,Cr)Se/GaAs interface as well as that of the layer structure are discussed.

This work was supported in part within European Community program G1MA-CT-2002-4017 (Centre of Excellence CEPHEUS) and by SPINOSA project (IST-2001-33334).

[1] G. Karczewski, M. Sawicki, V. Ivanov et al., J. Superconductivity: Incorporating Novel Magnetism 16, 55 (2003).

19:00 - 21:00

Poster session

Main Building, Main Hall (Duża aula)

19:00 - poster B-1

Transmission Electron Microscopy of Iridium Silicide Contact to MOSFET Structures

Adam Laszcz¹, Jerzy Kątczi¹, Jacek Ratajczak¹, Guilhem Larrieu², Emmanuel Dubois²

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The IrSi ohmic contacts has been used in modern Accumulated Low Schottky Barrier MOSFET on SOI. An IrSi layer has been formed as a result of metallurgical reaction between metal and semiconductor during annealing. The process of iridium silicide formation in the Ir/Si/SiO₂/Si structure has been studied by means of cross-sectional transmission electron microscopy (XTEM). The influence of annealing temperatures (300, 600, 900°C) on the Ir/Si interface was analysed. The changes of the Ir/Si interface after annealing at 300°C were not observed. However, in the Ir/Si/SiO₂/Si structure annealed at 600°C the Ir atoms penetrated the Si layer, causing disturbance of the Ir/Si interface. IrSi grains were formed in Si after annealing at 900°C. The expansion of the IrSi grains in the Si layer was observed. TEM results were correlated with XPS and electrical measurements.

19:00 - poster B-2

Nanostructure of laser annealed Ge-implanted near-surface Si layers

Julian Auleytner¹, Dorota Klinger¹, Sławomir Kret¹, Maria Lefeld-Sosnowska², Elżbieta Lusakowska¹, Danuta Żymierska¹

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

2) *University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland*

Short high-power laser pulses have been used for annealing of semiconducting layers, amorphised by ion implantation. For ion-implanted samples, laser annealing can be used for reconstruction and correction of lattice distortions and for inducing a redistribution of dopants. The structure of a laser-irradiated sample of a-Si amorphised by ion implantation depends on its initial structure and on the laser-pulse energy density [1-3]. Damage in Si caused by the implantation and annealing processes was studied for different kinds of implanted ions. Implantation with elements of IVA group is especially interesting, in view of the possible formation of Si-Ge and Si-Sn layers.

The presented paper reports in detail the defect structure induced by the nanosecond laser pulse with different energy density in Ge implanted silicon crystal. We observe changes of the annealed layer in dependence on the value of the energy density of laser beam. The redistribution of the dopant and its influence on the recrystallisation process of thin near-surface layer is discussed. To study the annealed areas we applied the high-resolution electron microscopy, X-ray section topography, atomic force microscopy and optical microscopy. Experimental findings are taken to determine more details of defects formed in Ge implanted Si crystal and to full characterization of the structural changes caused by the LPA.

References

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[3] R.F. Wood, G.E. Gellison, Semicond. Semimet. 23 (1984) 115.

19:00 - poster B-3

Structural and optical properties of high temperature and high pressure treated Si:H,D

Jadwiga Bak-Misiuk¹, Andrzej Misiuk², Artem Shalimov¹, A. Wnuk², Barbara Surma^{2,4}, I.V Antonova³, V.P Popov³, Jadwiga Trela¹

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2) *Institute of Electron Technology, al. Lotnikow 32/46, Warsaw 02-668, Poland*

3) *Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation*

4) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*

Single crystalline silicon implanted with hydrogen /deuterium (Si:H,D) indicates (in the implanted areas) interesting properties in respect its photoluminescence properties and of getting the harmful impurities by the implantation disturbed areas. Structural perfection and other properties of Si:H,D have been reported to be strongly affected by temperature.

The effect of high temperature and pressure (HT-HP) treatment on the structural perfection of Si:H,D prepared by Si implantation with high hydrogen (deuterium) doses was investigated in presented work. The 001 oriented Czochalski grown Si single crystals (Cz-Si) were implanted with H⁺ or D⁺ at doses (d) higher than $1.7 \times 10^{17} \text{ cm}^{-2}$. For example, Cz:Si:H was prepared by hydrogen implantation with energy, $E = 24 \text{ keV}$ and $d = 2.710^{17} \text{ cm}^{-2}$ using the ion plasma source. After implantation, the Cz-Si:H,D samples were (HT-HP) treated at HT up to 1000°C under HP up to 1.1 GPa for up to 10 h. Structural properties of Cz-Si:H,D were investigated by high-resolution X-ray diffraction. Measurements of the rocking curve (RC) shape and reciprocal space mapping (RSM) were performed for the symmetrical 004 reflection. The photoluminescence (PL) spectra of HT and HP treated Cz-Si:H were determined at 6 K (excitation with Ar laser, $\lambda = 488 \text{ nm}$).

Enhanced hydrostatic pressure of ambient gas at annealing of Cz-Si:H (D) prepared by high dose implantation results in the changed defect structure dependent on HP, HT and treatment time. For the (HT-HP) treated samples, apparent changes of defect structure were observed through the changed diffuse scattering intensity as well as the presence and position of defect-related PL peaks. Our results can be interpreted as indicating on enhanced creation of small defects in the Si surface layers, more pronounced after the treatments under the highest applied pressure.

CBN-Ti/TiN composites: hardness and chemical equilibrium as function of temperature

Ewa Benko^{3,4}, Piotr Klimczyk⁴, Krystyna Lawniczak-Jablonska¹, Edyta Piskorska¹, Tery L. Barr⁵, Sudipta Seal⁶, Vladimir S. Urbanovich²

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5) University of Wisconsin - Milwaukee, Milwaukee, United States

6) University of Central Florida, Orlando, United States

BN and TiN are widely used in industrial application because of their wear resistance. Ti and TiN can be used as binding phase and as layered phase in cBN composites. The cBN-TiN composites were pressed using high pressure and high temperature. Ti and TiN films were grown by the arc plasma deposition method. Some of samples were heat treatment in temperature 1000 and 1400 °C. The various phases formed in different temperature have been studied using element selective techniques. X-ray absorption near edge structure (XANES) at K and L -edges were measured with synchrotron radiation and X-ray photoelectron spectra (XPS) using conventional X-ray tube. The shape of the element's absorption edge is a fingerprint for a chemical bonding of an element. When the investigated element is present in two phases in the sample, the X-ray absorption spectral shapes are a linear combination of the spectra from each phase. The chemical shift of the XPS line was analyzed by curve fitting procedure. The possibility of formation of the metallic Ti, TiB₂, TiC and TiN phase's inclusions in the investigated samples was examined. The formation of new phases in cBN-Ti/TiN composites are shown to be determining factors for the microhardness. The hardness was measured by Vickers method using an indentation load of 9.81 N. The hardness of the cBN-TiN composites decreases after heat treatment during the formation of nonstoichiometric phases. The hardness of cBN samples layered with Ti and TiN are increase after annealing process due to the formation of stoichiometric phases.

Quantum Chemistry Analysis of Electron Density in Cyclophosphazenes and in their Supramolecular Complexes

Mikhail F. Bobrov¹, Vladimir G. Tsirelson, Galina V. Popova

1) D.I.Mendeleyev University of Chemical Technology of Russia (MUCTR), Miusskaya Sq., Moscow 125047, Russian Federation

The electron density, molecular electrostatic potential, Laplacian of the electron density and the electron localization function have been used to elucidate the features of the chemical bond in cyclophosphazenes P_nN_nH_{2n} (n = 2, 3, 4, 5, 6) and their derivatives. It was demonstrated that mutual arrangement of negative and positive regions in molecular electrostatic potential, topology of the Laplacian and presence of monosynaptic ELF attractors near N atoms explain all together the details of the selfassembling the supramolecular complexes of cyclophosphazenes and their derivatives. Analysis shows that stacking-interaction between phosphazene cycles is realized by P-N close-shell contacts.

The study was supported by Russian Ministry for High Education (grant E 02-5.0-161).

X-Ray diffraction studies of radiation defects in CdTe single crystals and

epitaxial layers

B-6 **Oleksandr Y. Bonchuk²⁾, Andrij P. Vlasov³⁾, Igor M. Fodchuk¹⁾, Ruslan M. Zaplitnyy¹⁾, Zbigniew T. Swiatek⁴⁾**

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4) Institute of Metallurgy and Materials Sciences, Polish Academy of Sciences, Reymonta 25 St, Krakow, Poland

The report investigates the influence of radiation defects, formed in near-surface regions of CdTe after As ion implantation, on the high temperature doping and ISOVPE growth of Cd_xHg_{1-x}Te epitaxial layers. For experiments, the undoped CdTe single crystals of (111) orientation and epitaxial layers on their basis with the surface source of dopant produced by As ion implantation were used. The structure of both CdTe ion implanted surfaces after high temperature annealing and Cd_xHg_{1-x}Te epitaxial layers grown on those substrates has been studied. The X-ray spectrometry single and double-crystal techniques in symmetric, asymmetric and skew-asymmetric geometry of sample placement have been used. Structural changes in both rocking curves and X-ray topograms obtained at different angles of azimuth scanning have been revealed. On the basis of a semi-kinematic and dynamical theory of X-ray diffraction the computer simulations of strain and distortion distributions in material after applied technological processes were performed. For a given distribution of deformations the rocking curve were calculated and compared with the experimental one.

19:00 - **XANES and X-MCD study of Nd₂TM₁₇A_x (TM=Fe, Co; A=N,H) compounds**
 poster **Marta Borowiec¹⁾, Czesław Kapusta¹⁾, Marcin Sikora¹⁾, Dariusz A. Zajac¹⁾, Damian Rybicki¹⁾, Bartosz Sobanek¹⁾, Colin J. Oates¹⁾, Peter Fischer²⁾, Gisela Schütz²⁾**

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2) Max-Planck-Institute for Metals Research (MPI), Stuttgart 70569, Germany

B-7

We report on the X-ray absorption near edge structure (XANES) and X-ray magnetic circular dichroism (X-MCD) study of Nd₂TM₁₇A_x (TM=Co,Fe, A=N,H) materials for novel permanent magnets. Introducing N or H atoms onto interstitial sites increases the Curie temperature of iron compounds and a large increase of magnetocrystalline anisotropy can be achieved with N uptake. In order to study the influence of light elements on the electronic properties of individual elements, an X-MCD study on the Fe,Co:K edge as well as on the Nd:L_{2,3} edges has been carried out. The XANES spectra on the Fe,Co:K edges show a smoothening of the middle-edge kink with H and N uptake. The effect is much stronger for the nitrides than for the hydrides and is attributed to a higher degree of localisation of Fe,Co 4p band, which is the final state of the K edge transition. The XANES spectra at the Nd:L_{2,3} edges show a narrowing of the white line with H,N uptake. The effect is much stronger for the nitrides than for the hydrides and denotes a higher degree of localisation of the Nd 5d band which is the final state of the L_{2,3} transitions. The normalised area of the white line for the nitrides is larger than in other compounds, which is attributed to a lower 5d band filling in the nitrides due to an electron transfer to adjacent nitrogen atoms. A decrease of magnitude of the negative K-edge X-MCD peak with H and N substitution in the cobalt compound is attributed to a decrease of polarization of the 4p band. A lowering of the positive X-MCD signal at the Nd:L₂ edge with N uptake denotes a lower spin polarization of 5d^{3/2} band in Nd₂Co₁₇N_x. In iron-based compounds, the X-MCD spectra at Fe:K and Nd:L_{2,3} edges do not show considerable changes with H or N addition. This can be interpreted as a weak influence of H and N atoms on the 4p and 5d electron polarization, possibly due to larger interatomic distances in the Fe compounds than in the Co compounds.

19:00 - **Characteristics of the interface dependent silicide formation for Gd on Si substrate**

poster **Kwun-Bum Chung^{1,2)}, Y. K. Choi¹⁾, M. H. Jang¹⁾, SeongJun Kang¹⁾, H. J. Kim¹⁾, M. Noh¹⁾, C. N. Whang¹⁾**

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B-8

Silicides are widely used in silicon integrated circuits as contact and interconnection. The metal-Si interface is of fundamental interest in Si-based semiconductor research and technology. Among the various silicides, rare-earth(RE) silicides have attracted interests because of their low Schottky barrier heights on n-type silicon(0.3~0.4eV). During post annealing, the solid-state reaction between epitaxially grown Gd film and Si substrate typically result in pitted rough surface. These pits increase sheet resistance and enhance leakage current such that they are detrimental to the electronic properties of diodes using Gd-silicide. Therefore, understanding and controlling of the silicide formation in Gd on Si system are important issue. We report our result of investigation on Gd-silicide formation for the samples with Gd layer deposited on Si substrates, where each Si surface was treated differently.

19:00 -
poster**Investigation of Ge quantum dots buried in silicon matrix by x-ray absorption**
Iraida N. Demchenko¹⁾, Krystyna Lawniczak-Jablonska¹⁾, Konstantin S. Zhuravlev²⁾, Edyta Piskorska¹⁾

B-9

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*2) *Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation*

The physical properties of quantum confined semiconductor heterostructures have attracted wide attention both from the experimental and theoretical point of view because of their potential applications in microelectronics and development of Si-based optoelectronics. Particularly, there is a considerable interest in the improvement of the electron-hole recombination efficiency of indirect-gap semiconductors using quantum low-dimension effects. Investigation of changes in the local atomic arrangement of formed germanium quantum dots (QD) in Si/Ge/Si structures with different thickness of germanium monolayers (ML) and capped Si layer will be reported. The presence of Si covering layer on the Ge ML induces additional stresses and can, considerably, change the shape and composition of formed structures. It can also introduce local distortion of the symmetry: changes in interatomic distances and bond angles between neighbouring atoms. To get inside into structure of buried Ge the element selective technique is necessary. Knowing that the X-ray absorption spectra are very sensitive to the local order in formed structures and are sources of unique information about morphology of matter we studied the K-edge of Ge. The measurements were performed for two angles (20° and 75°) between incidence beam and samples surface. The analysis was done taking into account linear polarization of synchrotron radiation and with assumption of the solid solution $\text{Ge}_x\text{Si}_{1-x}$ inside QD. The existence of the interface between QD and Si matrix introduces a chemical disorder, which consequently increases of the Debye-Waller factor value in analyzed spectra. The comparison of investigated structures with reference $\text{Ge}_x\text{Si}_{1-x}$ solid solution grown on the Si (001) substrate will be discussed. This work was supported, in part by grant of the State Committee for Scientific Research (Republic of Poland) No. SPUB-M/DESY/P-03/ DZ-213/2000.

Lattice parameters changes of GaMnAs layers induced by annealing
Jaroslav Domagala¹⁾, Jadwiga Bak-Misiuk¹⁾, Janusz Sadowski^{1,2)}, Artem Shalimov¹⁾, Jadwiga Trela¹⁾19:00 -
poster1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*2) *University of Copenhagen, Universitetsparken 5, Copenhagen DK-2100, Denmark*

B-10

A lot of efforts have been recently concentrated on investigation of ferromagnetic semiconductors due to their potential application for spintronic devices [1]. Ferromagnetic GaMnAs is one of materials most interesting for these purposes. The lattice parameter of $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ (a_L) increases linearly with the Mn content up to x about 0.1. However, different scientific groups (e.g. [2]) presented rather non-consistent data concerning the a_L value dependence on the Mn content. It has been reported, that these discrepancies are due to the presence of different defects, namely of manganese interstitials and arsenic antisites in GaMnAs [3].

The aim of present work was to determine the influence of substitutional Mn and both mentioned kinds of defects on the lattice parameter value. The concentration of defects was tuned in this work by post-growth annealing of GaMnAs in different temperatures (240°C, 260°C, 280°C). The total Mn concentration in the samples, determined by Secondary Ions Mass Spectrometry (SIMS), remained unchanged after GaMnAs post-annealing. The concentration of substitutional Mn in the GaAs lattice, before and after post-annealing, was determined from the lattice parameter of low temperature (LT) grown GaAs and GaMnAs, subjected to annealing at the same conditions. Based on our results the influence of the substitutional Mn and antisites defects on the lattice parameter of GaMnAs will be discussed.

This work was supported in part within European Community program G1MA-CT-2002-4017 (Centre of Excellence CEPHEUS).

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High-pressure phase transition and compressibility of zinc-blende HgZnSe mixed crystals19:00 -
poster**Elzbieta Dynowska¹⁾, Wojciech Szuszkiewicz¹⁾, Jaroslav Domagala¹⁾, Witold Daniel Dobrowolski¹⁾, Christian Lathe²⁾**1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*2) *GeoForschungsZentrum Potsdam,, Telegrafenberg A17, Potsdam D-14473, Germany*

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The aim of the present work was to determine the compressibility and to characterize briefly the room-temperature phase transition from the sphalerite to the cinnabar structure under high-pressure conditions for various $\text{Hg}_{1-x}\text{Zn}_x\text{Se}$ mixed crystals. High quality $\text{Hg}_{1-x}\text{Zn}_x\text{Se}$ crystals were grown at the Institute of Physics of the Polish Academy of Sciences in Warsaw using the modified Bridgman method. The phase analysis and compositions of these samples were determined by the X-ray diffraction using conventional X-ray powder diffractometer. In analysed mixed

crystals from a few to about 20% of Hg atoms were replaced by Zn atoms.

Energy-dispersive X-ray powder diffraction experiments were carried out at the F2.1 synchrotron beamline at DESY - HASYLAB. The beam diameter was equal to 100 μm . High-pressures were obtained at a cubic-anvil X-ray diffraction device, MAX80. The measurements were performed in the pressure range from 1 bar to 35 kbar at the ambient temperature.

The mixed crystal composition-dependent shift of the phase transition was clearly observed. The zinc-blende phase existed in relatively low-pressure range (up to ~ 7.5 kbar in the case of pure HgSe and up to ~ 20 kbar in the case of the highest mixed crystal composition under investigation). A comparison of the properties of magnetic and nonmagnetic mixed crystals grown on the basis of HgSe was discussed.

This work was partially supported by the IHP-Contract HPRI-CT-1999-00040/2001-00140 of the European Commission and by the grant 72/E-67/SPUB-M/DESY/P03/DZ-213/2000 from the State Committee for Scientific Research (Poland).

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poster
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A set of equations of stress-mediated evolution of the nonequilibrium dopant-defect system in semiconductor crystals

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The principal trend in modern electronics is decrease in the dimensions of semiconductor devices and usage of diverse multilayer structures to achieve the required parameters of devices. It means that interfaces exert a significant effect on the evolution of dopant-defect system and hence on the electrophysical parameters of advanced electronic devices. This influence has a multifactor character, but changes in defect generation and annihilation due to elastic stresses in the vicinity of interface comes into particular prominence.

For analysis of the above-mentioned phenomena, a set of the equations for stress-mediated evolution of the nonequilibrium dopant-defect system is proposed. This set includes the equation of the dopant atoms diffusion in a field of elastic stress written for the two-stream diffusion mechanism by pairs dopant atom - self-interstitial and dopant atom - vacancy; continuity equation for the dopant atoms incorporated into clusters, diffusion equation of point defects under stress conditions written for interstitials and vacancies; continuity equation for the self-interstitials which are included in the clusters of dopant atoms and extended defects, expression for the built-in electric field strength distribution and expression for the effective drift velocities of dopant atoms, vacancies and self-interstitials in the field of elastic stress. Analysis of the system of equations obtained shows that it allows one to describe the most of features of doping processes investigated experimentally, such as the phenomena of uphill diffusion and the formation of a maximum of dopant concentration in the vicinity of interface during thermal treatments of ion-implanted layers as well as the effect of vacancy and self-interstitials separation. Using the formulated equations, we have investigated the processes of stress-mediated evolution of the nonequilibrium dopant-defect system and compared these calculations with experimental data.

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Simulation of oxygen-contained complexes in silicon-silicon interface in cluster approximation

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Grain boundaries (GBs) are very essential type of semiconductor-semiconductor interfaces having a great technological importance for solar cells or electronic devices fabrication and offering low-dimensional electric properties. Electronic states associated with GBs contamination by oxygen atoms strongly affect electronic properties of the whole material. In accordance with some known experimental and theoretical research, the oxygen atoms built-in the GB core can generate both electrically active and inactive centers. Their activity is mostly connected with the character of Si-O configurations which can form thermal donors, silicon oxide precipitates, etc. Since Si-O complexes are local defects it is better to use a cluster approach to their calculations. In this work, we investigated a set of clusters reproducing local atomic structure of O containing complexes configurations (with number of O atoms between 1 and 4 on a cluster) at silicon tilt boundaries using MO LCAO method in PM3 approximation. For all clusters full optimization of oxygen-containing complex geometry, calculations of atomic and electronic structure and electronic density distribution were conducted.

These simulating experiments have shown that mechanism of creation of oxygen complexes at GB core lies in original formation of 'Si₂O' and 'Si₄O' structures corresponding to arrangement of oxygen atoms in tetrahedron of silicon oxide. Moreover, as has been exhibited, structures involving oxygen atoms that are formed near the GB core can contain not only 3 or

more threefold O atoms but also only 1 and 2 atoms of oxygen. It has been also studied the influence of variations in the above-mentioned 'Si_xO' configurations on the changes in electronic structures of clusters, localization of energy states near the band edges and redistribution of valence electron densities in the neighbourhood of GB core.

Studies of Interface Carbon Layer Formation on the Cleavage Surfaces of In₄Se₃ Layered Crystals by X-ray Photoelectron and Auger Electron Spectroscopy

19:00 -

poster

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XPS spectra were obtained for interface formation on the cleavage surfaces of In₄Se₃ layered semiconductor crystals that have been exposed in the air. Those exposed for 2-15 min are called fresh cleavages and those exposed for more than 24 hours are called the old cleavages.

The presence of the carbon on the fresh and old cleavage surfaces was shown using XPS analysis. The most intense XPS lines, viz. Se 3d, In 3d, C 1s, O 1s and the sharpest Auger lines were recorded in an expanded binding energy scale for the purpose of atomic concentration calculation and the charge transfer estimation. The exact peak position of these intense lines have been subsequently determined from the corresponding profiles, recorded on an expanded binding energy scale for Se 3d and In 3d. In each case Gaussian line shape analysis has been done for the XPS expanded profiles after making the background correction using Shirley's method to determine the exact peak positions and peak areas. The chemical shifts have been calculated from the binding energy values of the XPS lines to Se, In, C and O, corrected with reference to vacuum level.

The strong decrease of the (O/C) and (N/C) ratios is observed in the case of the fresh, in comparison with the same ones for the old cleavages. This indicates, that in UHV chamber the adsorbed nitrogen is absent and carbon monoxide is the main adsorbate on the In₄Se₃ surface, and it forms In₄Se₃-C interface.

The presence of non-simple interaction in the In₄Se₃-C interfaces of old and fresh surfaces can be also confirmed by a closer inspection of the C 1s core level spectra. For the fresh cleavages of In₄Se₃ surface interface, the deconvoluted C 1s peak shows the presence of two types of carbon. They could be attributed to C-C (284.0 eV) and C-Se (284.39 eV). For the old In₄Se₃ surface interface, the presence of another carbon is detected at 284.39 eV attributed to C-C interaction in the "graphite phase".

X-ray study of quartz single crystals implanted with fast Ar ions

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19:00 -

poster

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The aim of the research was to determine structural changes induced by heavy ion implantation in single crystals. Previously it was shown that the direction of radiation flow is changed in the ion shot-through layer at the depth corresponding to the ion mean range. In order to perform new experiments of ion implantation, we planned and constructed the special irradiation chamber for the heavy ion multiple implantation, which allows one to irradiate different places on the surface of a studied crystal without necessity of opening it. The device is equipped with the special tantalum diaphragm, which suffers implantation to be performed in the strictly determined regions of sample. Such construction makes possible to repeat the implantation process many times without renewal pumping, which effects in large saving of time and work.

As an example of the use of the chamber the implantation of quartz single crystal with 2.0 MeV/amu Ar ions at three different doses is presented. The X-ray study results are discussed.

Transmission electron microscopy and X-ray diffraction study of α'-Al₂CO crystals

19:00 -

poster

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B-16

Sapphire crystal is widely used as a substrate materials for semiconductor, superconductor and metallic layers. Incorporation of carbon atoms into cationic or anionic sites may modify its

properties. In the Al-C-O system two ternary compounds are known: Al₂CO and Al₄O₄C. For Al₂CO two polymorphs: α and α' have been reported. The polymorph α -Al₂CO crystallizes in wurtzite structure with $a=3.170$ Å and $c=5.078$ Å the structure of α' -Al₂CO is not fully understood yet. In this paper we suggest crystal structure for α' -Al₂CO obtained by use of different optimisation methods. The proposed structure is consistent with X-ray diffraction and transmission electron microscopy results.

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poster
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Sub-surface Investigation of Open-volume Defects induced by Ion Implantation in Zirconia Single Crystals

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The knowledge of defect type and distribution induced during ion implantation in oxide materials is of special technological interest in microelectronics processing. In this work, using Slow Positron Implantation Spectroscopy, we tentatively characterize the nature of possible open-volume defects resulting from such irradiation in cubic zirconia (fully-stabilized with 9.5 mole % of yttria). In particular, in order to check which empty substitutional sites are effective in trapping positrons, we implanted self-ions (e.g. : oxygen) at low energy (projected range \sim 200 nm). The trapping dependence on both the irradiation dose and the temperature annealing up to 1100 °C is compared with previous results obtained after foreign ion implantation (noble gas ions for instance).

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poster
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Examination of the atomic Pair Distribution Function (PDF) of SiC nanocrystals by in-situ high pressure diffraction

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Key properties of nanocrystals are determined by their real atomic structure, therefore a reasonable understanding and meaningful interpretation of their properties requires a realistic model of the structure. In this paper we present an evidence of a complex response of the lattice distances to external pressure indicating a presence of a complex structure of SiC nanopowders. The synchrotron and neutron powder diffraction experiments were performed on nanocrystalline SiC subjected to hydrostatic or isostatic pressure. Elastic properties of the samples were examined based on X-ray diffraction data using a Diamond Anvil Cell (DAC) in HASYLAB at DESY. The dependence of the lattice parameters and of the Bragg reflections width with pressure exhibits a dual nature of the properties (compressibilities) of the powders and indicates a complex structure of the grains. We interpret this behaviour as originating from different elastic properties of the grain interior and surface. Analysis of the dependence of individual interatomic distances on pressure was based on in-situ neutron diffraction measurements done with HIPD diffractometer at LANSCE in Los Alamos National Laboratory with the Paris-Edinburgh cell under pressures up to 8 GPa ($Q_{\max} = 26$ Å⁻¹). Interatomic distances were obtained by PDF analysis using the PDFgetN program. We have found that the interatomic distances undergo a complex, non-monotonic changes. Even under substantial pressures a considerable relaxation of the lattice may take place: some interatomic distances increase with an increase in pressure. We relate this phenomenon to (i), changes of the microstructure of the densified material, in particular breaking of its fractal chain structure and, (ii), its complex structure resembling that of a material composed of two phases, each with its distinct elastic properties.

19:00 -
poster
B-19

Separation of vibrational and static disorder in amorphous In-Se films by EXAFS

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Configurational disorder in amorphous materials is due to thermal vibration of atoms and their static displacements from equilibrium positions. These two components can be separated by means of two EXAFS experiments performed at room and low temperatures. In the present work semiconducting indium selenide amorphous films with Se content at 50, 60 and 66 at. % has been studied. Such a separation has fundamental importance for understanding of electronic and optical properties of amorphous semiconductors. The BM29 and BM32 beam lines at ESRF

(Grenoble) were used to record the EXAFS spectra at room temperature and at 20K at the In and Se K-edge. The generalized Debye-Waller factors at both absorption edges for all investigated samples were derived from the EXAFS oscillations using standard processing procedures and the Marquard-Levenberg least squares method. At 20K the thermal vibrations are practically frozen out and the Debye-Waller factor is related only to static disorder, which is a source of amorphous nature. The correlations thermal-static disorder as a function of the chemical composition is dissonsed.

Polymeric flocculation of ceramic slurry obtained by the Direct Coagulation Casting

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The role of enzymes in Direct Coagulation Casting forming method consists in the decomposition of an appropriately selected substance, which results in slow release over the whole volume of a substance changing the pH of the system modifying the double electric layer. In the case of applying a powder of a suitable composition, profiles of high density and relatively high mechanical strength in the green state enabling the removal of a green product from the form without destruction can be obtained by this method.

The assurance of a respective mechanical strength of a green samples is a technological problem when forming products of a complicated shape. This requires the introduction of a certain amount of a polymeric binder, often of high molecular weight. Such binders cause a considerable increase in the viscosity of ceramic casting slips, which in turn precludes the exact filling of the form.

This problem can be partly minimized by introducing a binder simultaneously playing the role of a flocculant of the ceramic casting slip. If the flocculation is carried out with the participation of enzymes, then at a relatively small rate of such a reaction appropriate fitting of the ceramic powder particles is possible, which should lead to an improvement in the uniformity of thickening in the ceramic product formed.

The slow decomposition of a respective starting material by an enzyme should cause a change in the character of interactions between particular bundles of the polymer chains and change their conformation to a dense net of entanglements. This should result also in the transfer of the ceramic casting slip from the state of small viscosity to that of a stiff material, additionally strengthened by a bond between the individual particles of the ceramic powder and the binder.

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poster

B-20

Processing of the ceramic materials by the gelcasting method

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Gelcasting is an attractive novel ceramic forming process for making high-quality, complex-shaped ceramic parts. The idea of this method is based on a slurry made from ceramic powder and water based monomer solutions is poured into a mold. The ceramic slurry consists of a ceramic powder, organic or inorganic solvent, organic monomer and free radical initiator. Water is a preferred solvent. At appropriate conditions is possible to start the polymerization in the ceramic slurry.

The general aim of research is looking for new water-soluble low toxic monomers. The aqueous monomer solution provides a low-viscosity vehicle that is combined with ceramic powder and appropriate dispersants to form a highly fluid slurry. That is why in our research we use only water-soluble organic monomers, for example 2-hydroxyethyl acrylate.

The choice of organic monomer and initiator system depends on several factors just like gel strength, wet and dry strength of ceramic sample, reactivity.

The ceramic slurry was based on the alumina powder. Gelcasting is very close to the traditional ceramic processing. In this process we can use the same dispersants as are normally used for slip casting; no special molding machines or presses are needed. We use standard mixing equipment such as ball mills for slurry preparation in the gelcasting process.

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poster

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Characteristics of the optimized pentacene thin film transistor via OTS treatment of gate insulator and thermal treatment of pentacene layer

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Pentacene thin film transistor is a promising competitor of amorphous Si-based semiconductor devices though further study is necessary to improve mobility. In our experiment, preferentially oriented high quality pentacene thin film was obtained via thermal evaporation in ultra-high vacuum. To investigate the effect of substrate surface on the pentacene layer formation, pentacene film was prepared on either octadecyltrichlorosilane (OTS) treated SiO₂ substrate or untreated SiO₂ substrate. The grain size of the pentacene layer and the organic-metal interface roughness were modified through the thermal treatment of the sample evaporated on either substrate surface.

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Both the OTS treatment and the thermal treatment increase the structural ordering of pentacene molecules in the pentacene layer. The crystallinity of the pentacene film was investigated via the high-resolution synchrotron x-ray diffractometer at the PLS. The roughness of the film was studied by using atomic force microscopy. We obtained the best device performance at the pentacene thin film transistor that was OTS treated as well as annealed. Our investigation results of the electrical properties on the pentacene thin film transistor will be presented.

- 19:00 -
poster
B-23
- XANES and X-MCD study of $\text{Nd}_2\text{TM}_{17}\text{A}_x$ (TM=Fe, Co; A=N,H) compounds**
**Marta Borowiec¹, Czesław Kapusta¹, Marcin Sikora¹, Dariusz A. Zajac¹, Damian Rybicki¹,
 Bartosz Sobanek¹, Colin J. Oates¹, Peter Fischer², Gisela Schütz²**
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We report on the X-ray absorption near edge structure (XANES) and X-ray magnetic circular dichroism (X-MCD) study of $\text{Nd}_2\text{TM}_{17}\text{A}_x$ (TM=Co,Fe, A=N,H) materials for novel permanent magnets. Introducing N or H atoms onto interstitial sites increases the Curie temperature of iron compounds and a large increase of magnetocrystalline anisotropy can be achieved with N uptake. In order to study the influence of light elements on the electronic properties of individual elements, an X-MCD study on the Fe,Co:K edge as well as on the Nd:L_{2,3} edges has been carried out. The XANES spectra on the Fe,Co:K edges show a smoothening of the middle-edge kink with H and N uptake. The effect is much stronger for the nitrides than for the hydrides and is attributed to a higher degree of localisation of Fe,Co 4p band, which is the final state of the K edge transition. The XANES spectra at the Nd:L_{2,3} edges show a narrowing of the white line with H,N uptake. The effect is much stronger for the nitrides than for the hydrides and denotes a higher degree of localisation of the Nd 5d band which is the final state of the L_{2,3} transitions. The normalised area of the white line for the nitrides is larger than in other compounds, which is attributed to a lower 5d band filling in the nitrides due to an electron transfer to adjacent nitrogen atoms. A decrease of magnitude of the negative K-edge X-MCD peak with H and N substitution in the cobalt compound is attributed to a decrease of polarization of the 4p band. A lowering of the positive X-MCD signal at the Nd:L₂ edge with N uptake denotes a lower spin polarization of 5d^{3/2} band in $\text{Nd}_2\text{Co}_{17}\text{N}_x$. In iron-based compounds, the X-MCD spectra at Fe:K and Nd:L_{2,3} edges do not show considerable changes with H or N addition. This can be interpreted as a weak influence of H and N atoms on the 4p and 5d electron polarization, possibly due to larger interatomic distances in the Fe compounds than in the Co compounds.

- 19:00 -
poster
B-24
- Positron-annihilation monitoring of reduction processes in conducting glasses**
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Bismuth and lead doped silicon and germanium-oxide glasses are used, among others, for electron continuous-dynode, electron (and photon) amplifiers. To make them conducting, reduction procedures in hydrogen atmosphere are applied. The depth of the reduced layer must be thoroughly chosen, in order to assure a high secondary-electrons yield, and to assure a low surface conductivity, on the other hand.

We show, how a beam of low-energy positons can be used to monitor in a detailed way the dynamics of metal precipitates in near-to-surface layers. S-parameter defining annihilation with low momentum and W-parameter - with high momentum electrons are monitored. In particular for silica glass samples, lead and bismuth-doped one notes that long ducton times (91 h at 300°C and 21 h at 400°C, respectively) cause "over reduction" of the samples - it means that reduced layer thickness is bigger than apparatus capabilities (about 2 micrometers). Samples of the second series, were annealed in slightly higher temperature (340°C) but for shorter times (up to 5 h), and therefore was possible to observe the whole reduced layer, extending down to 700 nm. For bismuth doped samples, positron annihilation shows two reduced layers - one for the first few tens of nm, and a deeper, extending down to 500 nm, which merge for high reduction times. Existence of the near-to-surface reduced layer is less clear in germanium-based glasses. Additionally, the fact that W/S ratio for glasses with different proportions are at the same straight lines shows that annihilation occurs in homogenous "glass phase", independently of the reduction rate, and not in separate, two separated oxide phases.

- 19:00 -
poster
B-25
- Pr^{+3} and Tm^{+3} containing transparent glass ceramic in the $\text{GeO}_2\text{-PbO-PbF}_2\text{-LnF}_3$ system**
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Oxyfluoride glasses with composition $50\text{GeO}_2-(50-x)\text{PbO}-x\text{PbF}_2$ ($x = 5, 10$ and 15 mol%) containing praseodymium or thulium were melted from commercial raw materials in normal atmosphere. Based on results of differential thermal analysis (DTA), glass transition temperatures and oxide glass crystallisation temperatures have been derived. Then the glass samples were thermally treated in order to achieve controlled precipitation of PbF_2 . Process of formation of glass-ceramics has been monitored with the aid of the DTA and X-ray measurements. The control acoustic emission (AE) measurements confirm the DTA results. It has been found that after heat treatment the crystallisation temperatures of investigated materials have been changed due to the PbF_2 content and kind doped ions. The influence of the kind of doped ions on the glass stability was observed. The effect of thermal treatment on the absorption spectra and kinetics of the fluorescent levels of Pr^{3+} and Tm^{3+} was studied. In the case of Tm^{3+} doped systems it was found that this effect on the relaxation of the $^1\text{G}_4$ level was weak, whereas, the decay of the $^3\text{H}_4$ level was slower by a factor of three roughly with respect to that recorded with as-melted glass. Relaxation rates of the $^3\text{P}_0$ and $^1\text{D}_2$ levels of Pr^{3+} showed marked dependence on both the PbF_2 and Pr^{3+} content in as-melted glass and in glass-ceramics.

This work was partially supported by Polish Committee for Scientific Research under grant no 7 T08D 020 21.

Defect structure of Sn-implanted Si crystal annealed by nanosecond laser pulse

19:00 -

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poster

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B-26

A surface layer of disordered structure is created in Si crystal as a result of implantation with ions of a respectively high dose. Different types of defects have been observed depending on the implant species, the implant dose and the annealing temperature [1, 2]. Recently, one takes interest in an influence of the implanted ions mass from group IV: Si, Ge, Sn on the type of structural defects created in the silicon matrix [3-7]. For crystal-lattice reconstruction of the disturbed near-surface layer pulsed laser annealing can be used, as an efficient, fast and well-controlled technique [8]. Laser annealing induces migrations of defects created by implantation process and initiates dopant diffusion.

In the present paper the influence of implanted Sn ions on the defect structure in laser annealed thin near-surface layer is considered. Regions irradiated with different energy densities of the laser beam are compared. A XeCl laser with the pulse duration of 30 ns (FWHM) generates laser light pulses with the wavelength of 308 nm. Evolution of defect structure during nanosecond pulse annealing has been characterized by reflection high-energy electron diffraction, interference polarizing microscopy and atomic force microscopy.

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Microstructure of Fe_3O_4 film grown on Si(100) substrate, investigated by TEM and X-ray methods

19:00 -

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poster

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B-27

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Epitaxial growth of metal oxide films on semiconducting substrates plays an important role in microelectronic monolithic integrated circuits. Different techniques like MBE, laser ablation, chemical vapour deposition and sputtering are used for production of the layers. The main difficulties arise from lattice mismatch and chemical reactions between semiconductor substrate and oxide layer. This has been observed for Fe_3O_4 film deposited by laser ablation on Si substrate.

Microstructure of Fe_3O_4 film deposited on (100) oriented Si substrate by sputtering was investigated by TEM and X-ray methods. Fe_3O_4 film was polycrystalline with preferential orientation of (111) planes parallel to Si substrate, but without preferential in plane direction. An amorphous 3-4 nm thick layer was observed between the substrate

and Fe₃O₄ film. Occurrence of crystallites, oriented in the same manner like substrate suggest, that even in presence of an amorphous layer, the pseudoepitaxial growth of Fe₃O₄ film is possible.

- 19:00 -
poster
B-28
- Use of element selective methods for characterization of thin films**
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 1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*
 2) *Laboratory of Materials Science, Department of Physics, University of Turku,, Turku FIN-20014, Finland*
 3) *Institute of Electronics, National Academy of Science of Belarus, Minsk, Belarus*

To control the technological processes it is necessary to characterize the grown films using advanced methods of samples characterizations. We report on the results of depth profile studies of the contents and chemical bonding of Dy, O and Si in DyxOy films grown on n-Si (100) substrate at different conditions. The investigations were performed by x-ray photoelectron spectroscopy and Auger scanning microscopy. The studies of the morphology of crystalline structure were done by atomic force microscopy and synchrotron radiation grazing incidence diffraction. New generation of electronic, basing on nano-structures, brings new challenges for technology. One of examples is the need for new dielectrics with high dielectric constant k. Among the high-k dielectrics being studied, DyxOy appears promising due to its relatively high dielectric constant (k=12) as compared to SiO₂ (k=3.9). The dysprosium oxide is characterized as chemical and thermal stable, with high breakdown electric field, low magnitude of dielectric losses and conductivity. Studies of the electrical properties of DyxOy films in the silicon MIS structures lead to promising results and suggestion to apply it as a gate insulator. The homogeneity of the gate dielectric composition and thickness and crystalline structure control are very important for reliability of the MISFET constructed with high-k dielectric. This work was supported in part within the E.C. program G1MA-CT-2002-4017 (Centre of Excellence CEPHEUS) and by the State Committee for Scientific Research (Poland) No. 72/E-67/SPB/5.PR UE/2003-2005.

- 19:00 -
poster
B-29
- Conventional and SR reflection topography of GdCa₄O(BO₃)₃ crystals**
Maria Lefeld-Sosnowska²⁾, E. Olszyńska^{2,3)}, W. Wierzchowski³⁾, K. Wieteska⁴⁾, W. Gräff¹⁾, A. Pajczkowska³⁾, A. Kłos³⁾
 1) *Hamburger Synchrotronstrahlungslabor HASYLAB, Notkestr. 85, Hamburg D-22603, Germany*
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 3) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*
 4) *Institute of Atomic Energy, Warsaw/Otwock-Świerk 05-400, Poland*

Highly efficient visible and UV light laser sources are of great importance for many applications. Visible emission can be obtained by frequency doubling in solid state lasers, using non-linear optical crystals. A particularly valuable recently developed non-linear optical crystals is GdCa₄O(BO₃)₃ (GdCOB), grown by Czochralski technique, which is a non hygroscopic, easy to polish and of high hardness. In order to achieve high efficiency of second harmonic generation the crystals of high structural quality are needed. Crystal lattice defects are the source of lattice strains, which change the crystal optical properties, mainly the refractive indices.

In the present work the GdCOB crystals are studied with the X-ray diffraction topography in reflection geometry. The conventional Lang topography and Synchrotron Radiation (SR) Plane Wave Reflection Topography were used. The investigated samples were cut out perpendicularly to the growth axis at top, middle and end part of the crystal grown by Czochralski technique.

The topographs taken for symmetrical (0 16 0) reflection for different angle between the [100] crystallographic direction and the projection of the direction of primary beam on the crystal surface show the dependence of the diffraction contrast of the dislocations on this angle. The highest resolution was achieved in asymmetric reflection geometry, at the small angles of incidence of the primary beam.

The synchrotron radiation plane wave (SRPWT) topographs were recorded for different azimuths in different regions of the rocking curve. The characteristic changes of the dislocation contrast were observed in different topographs. Both plane wave and Lang topographs did not reveal any segregation fringes proving high homogeneity of the chemical composition of the examined crystal.

This work was partly supported by Polish Committee for Scientific research under the project No T 11 B 054 22.

- 19:00 -
poster
B-30
- Structure and Properties of dynamically compressed Al99,5 and AlCuZr alloy.**
Maria Richert^{1,2)}, Beata Leszczyńska²⁾
 1) *University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland*
 2) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland*

The dynamically compressed AlCuZr alloy and technically pure aluminum Al99,5 have been investigated. The jump of microhardness has been found in both materials deformed in the compression test by special hammer. In the cases of AlCuZr alloy it appeared at the true strain of about 0,45 and in the case of Al99,5 at the true strain of about 0,3. In both cases the considerably increase of microhardness has been found.

The observation performed by light microscope revealed band microstructure in Al99,5 and AlCu4Zr from the beginning of deformation. With the increase of deformation the density of bands considerably increased. There were not found the substantial differences between the samples before and after the jump of microhardness. However thin foil observations showed the changes of microstructure above the microhardness transient. Above the microhardness jump, the mutually crossing microbands were found in contrast to samples below this point, in which only one family of microbands has observed.

The electrodeposition of amorphous Ni-P coatings containing TiO₂ and Ti powders

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1) Silesian University, Institute of Physics and Chemistry of Metals, Bankowa 12, Katowice 40-007, Poland

19:00 -

poster

B-31

In this paper, an environmentally friendly electroplating process of amorphous Ni-P+TiO₂+Ti coatings was developed. Co-deposition of inert or/and metallic powder particles in an amorphous nickel matrix enables the production of a large range of materials with unique properties. Composite Ni-P+TiO₂+Ti coatings were prepared by simultaneous electrodeposition of nickel with titanium dioxide and titanium powder from a solution in which TiO₂ and Ti micro-particles were suspended by stirring. The electrodeposition was carried out using a rotating disk electrode on a steel substrate under galvanostatic conditions at room temperature. Obtained coatings exhibit a structure of Ni-P matrix in which the crystalline TiO₂ and Ti are embedded. Using AES method, the presence of the non-stoichiometric oxide, Ti₁₀O₁₉, and intermetallic compounds, NiTi, formed during the electrodeposition, were additionally observed on the Ni-P+TiO₂+Ti coating surface. Present studies on examination of the effects of the variation of electrodeposition conditions has produced some understanding of Ni-P+TiO₂+Ti coatings electrodeposition mechanism.

Determination of vanadium valence in hydrated compounds

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19:00 -

poster

B-32

Physical properties of vanadium pentoxide based compounds are strongly affected by the presence of vanadium ions of reduced valence. Thus, the identification of V⁴⁺ ions in mixed valence compounds and the determination of the reduction ratio $RR = V^{4+} / (V^{5+} + V^{4+})$ is an actual task.

Quantitative information of the relative amount of element species may be obtained from XPS data. The knowledge of BE and FWHM for individual species is required for the accurate deconvolution of the experimental spectra. Unfortunately, the known values of BE and FWHM show large discrepancy and cannot be directly used in fitting procedure.

The aim of this work is to study the main XPS peak parameters (such as BE and FWHM) of the minor V⁴⁺ ions diluted in the host V₂O₅ matrix. For this purpose the XPS spectra of the samples with different V⁴⁺ ions concentrations and the identical crystal structure were analyzed. In this case, the crystal field is the same for various valence ions, and only the vanadium ions charge affect on the chemical shift and FWHM.

Polyvanadium acid xerogel - H₂V₁₂O₃₁·nH₂O was used as a reference material. The spectra differentiation technique indicate that the replacing of (H₂)²⁺ by a (VO)²⁺ cations allows to increase the RR of about 7%. Additional annealing of (VO)V₁₂O₃₁·nH₂O xerogel increases the of reduced valence V⁴⁺ - ions concentration. The spectra comparison technique was used to determine chemical shift and FWHM of V⁴⁺ cations in the vanadium pentoxide matrix. It was found that binding energy of V⁴⁺ ions shifts to the lower energy side of about 1.3 eV in respect to the main V⁵⁺ ions in the host matrix, while for pure main valence VO₂ and V₂O₅ this difference is about 0.6 eV. FWHM in V₂O₅ based compound is actually the same for both minor V⁴⁺ and major V⁵⁺ ions in contrast to the main valence vanadium oxides.

Photoemission study of the LT-GaAs

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3) Chalmers University of Technology, Goeteborg S-412 96, Sweden

4) MAX-lab, Lund University, Lund SE-221 00, Sweden

19:00 -

poster

B-33

Low - temperature (LT) molecular beam epitaxy-grown (MBE) GaAs have found many applications in ultrafast, integrated optoelectronic photoswitches and photodetectors. Such layers of GaAs grown by MBE at LT conditions under the arsenic rich flux exhibit unique properties, such as ultrashort carrier lifetimes and high resistivity after annealing.

The object of this study - LT-MBE grown GaAs (100) at one temperature and at different As flux conditions by means of angle-resolved photoemission spectroscopy. The experiment was performed on the toroidal grating monochromator beamline (BL 41) at the MAX I storage ring. A dedicated MBE system is connected to the electron spectrometer, allowing the between the units under ultrahigh vacuum conditions. The valence band spectra for samples prepared at different As flux conditions show very similar features. The peak related with interband transition shifts toward low binding energies, when As flux increase. That can be relate with the change of lattice constant in the LT-GaAs due to increasing of the density of As_{Ga} antisites with increasing As flux. To identify surface and bulk contribution the Ga 3d and As 3d core level spectra were taken at different photon energies and take off angles. In Ga 3d spectra appear a new component in compare with HT-GaAs depends on As flux ratio and is bulk derived. This component origin is bonded with high density of As_{Ga} antisite point defects in LT-GaAs [1].

Acknowledgment: This work was supported by the European Community-Access to Research Infrastructure of the Improving Human Potential Programme.

[1]. H. Asklund, L. Ilver, J. Kanski, J. Sadowski, M. Karlsteen, Phys. Rev. B 65, 115335 (2002).

19:00 - **Geometrical description of the X-ray capillary and total reflection mirror surfaces with assumed reflection features**

poster **Robert Mroczka¹⁾, Grzegorz Zukociński, Andrzej Kuczumow**

B-34 *1) Catholic University of Lublin, Department of Chemistry (KUL), Al. Kraśnicka 102, Lublin 20-718, Poland*

The geometrical description of the capillary systems adjusted for guiding X-rays is presented. Simple relationships between the reflection angle, the angle of the capillary wall inclination towards the capillary's main axis and the angle of the ray inclination in relation to the main axis are proved. Once established, the relationships help derive differential equations describing the profile of the capillary wall, in the small angle approximation. The equations can be made more general, by the deliberate assumption about the variability of the reflection angle along the reflecting surface. The way of calculating the forces necessary to draw capillaries of desired shape is then demonstrated. Finally, the full assembling of capillary shape with the variability of the reflection coefficient is suggested. Many solutions given in the paper apply to the case of total reflection X-ray mirrors.

19:00 - **Electrolytic production and heat-treatment of Ni-based composite layers containing intermetallic phases**

poster **Iwona Napłoszek-Bilnik¹⁾, Antoni Budniok, Eugeniusz Łągiewka**

B-35 *1) Silesian University, Institute of Physics and Chemistry of Metals, Bankowa 12, Katowice 40-007, Poland*

Composite Ni+Al layers were prepared by co-deposition of nickel and aluminium particles on a steel substrate from a nickel plating bath in which Al powders were held in suspension. The electrodeposition was carried out under galvanostatic conditions at a temperature of 323 K. These layers are characterized by the presence of Al micro-particles embedded into the nanocrystalline nickel matrix. The embedding mechanism of solid particles into the layers structure was explained basing on Ni^{2+} adsorption/desorption processes on the metal powder. Obtained layers could be applied as semi-finished products to obtain Ni-based layers containing the intermetallic phases as well as novel electrode materials in electrochemistry. The influence of Al content in the electroplating bath on the chemical composition of obtained layers was investigated. It was found that Al content in the layers was an increasing function of metal powders amount in the bath. The maximum Al content was 44 wt% for the Ni+Al layers, respectively. The size of the nickel crystallites has been determined from the diffraction peaks half-width (27 nm). Basing on phase diagrams for Ni+Al systems. The heat-treatment in argon atmosphere of deposited layers under proposed conditions leads to obtain different intermetallic phases of Ni with Al.

19:00 - **Phase transitions in the spinel $Li_xMn_{3-x}O_4$ intercalation compounds. Synchrotron X-ray diffraction studies.**

poster **Waldemar Nowicki¹⁾, Emilia Wolska¹⁾, Jolanta Darul¹⁾, Paweł Piszora¹⁾**

B-36 *1) Laboratory of Magnetochemistry, Faculty of Chemistry, Adam Mickiewicz University, Poznań, Poland, Grunwaldzka 6, Poznań 60780, Poland*

We have undertaken a detailed investigation of the lithium-for-manganese substitution effect of lithium manganese oxide, in the system $Li_xMn_{3-x}O_4$, for $0.95 < x < 1.05$, that is for the nearly stoichiometric lithium content. The samples have been obtained by solid state reaction of Li_2CO_3 with the manganese oxide precursor, $\alpha-Mn_2O_3$, at $750^\circ C$ and $800^\circ C$. The lithium deficient samples were quenched in the solid CO_2 . High-resolution synchrotron X-ray measurements have been performed in the temperature range 20K-300K. The diffraction experiments were carried out at the DESY/HASYLAB powder diffractometer (beamline B2), equipped with a closed-cycle He cryostat. Very small changes in the lithium content influence clearly the low-temperature crystal structure of $Li_xMn_{3-x}O_4$ spinels

and the nature of phase transitions. It was found that for $x=0.95$ the sample remains tetragonal in the whole 20K-300K temperature range. The stoichiometric LiMn_2O_4 transforms from cubic to orthorhombic at about 280K. For $x=1.0125$ the temperature of phase transition from cubic to orthorhombic decreases down to about 260K, whereas for $x=1.025$ the transformation goes from cubic directly to tetragonal, at the temperature 220K. No phase transition has been observed for the cubic sample with $x=1.0375$. Our results partly explain the divergences in recent reports on the low-temperature structure and phase transformations of lithium manganese oxides.

Mn doped ZnTe (110) (1x1) surface in Resonant Photoemission study

Bronislaw A. Orlowski²⁾, Sigitas Mickevicius¹⁾, B. J. Kowalski²⁾, I. A. Kowalik²⁾, K. Kopalko¹⁾, A. Mycielski²⁾, R. L. Johnson³⁾

19:00 -

poster

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2) *Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland*

3) *Institute for Experimental Physics, University of Hamburg, Hamburg, Germany*

B-37

Diluted magnetic semiconductors (DMS) are most interesting materials for spintronic application [1] due to s-p-d interaction of 3d electrons with the s-p valence electrons of the volume (3D) and surface (2D) of the crystal. Photoemission study of the valence band electronic structure for clean, freshly cleaved (110) relaxed 1x1 surface of ZnTe crystals and for these surface after sequentially deposited on it small and controlled amount of Mn atoms were performed. The crystalline samples of ZnTe was grown in the Institute of Physics, PAS in Warsaw and oriented as parallelepiped ($10 \times 5 \times 5 \text{ mm}^3$) with the cleavage plane parallel to the 5×5 plane. The Flipper II beam line of the synchrotron radiation in HASYLAB was used. The clean surface of ZnTe (110) was obtained by the cleavage and LEED study shows the (1x1) patterns. The analysis of: constant initial state, energy distribution curves, Zn 3d and Te 4d core level spectra showed that at very low Mn coverage ($< 0.15 \text{ nm}$) manganese interaction with ZnTe results in the creation of the $\text{Zn}_{1-x}\text{Mn}_x\text{Te}$ (110) surface ternary alloy. Further deposition of Mn atoms leads to the formation of the metallic manganese on the surface. The successive annealing leads to the diffusion of Mn atoms and to the formation of DMS at the surface region. The experimental results were compared to the calculated electronic band structure of $\text{Zn}_{1-x}\text{Mn}_x\text{Te}$ crystals.

This work was supported in part within: European Commission IHP-Contract HPRI-CT-1999-00040/2001-00140; and KBN SPUB-M/DESYP/P-03/DZ-213/2000; and European Community program G1MA-CT-2002-4017 (Center of Excellence CEPHEUS).

[1]. T. Dietl, Physica E 10, 120 (2001)

Differential Reflectivity and Photoemission study of ZnTe and CdTe(110) surface

19:00 -

Bronislaw A. Orlowski¹⁾, I. A. Kowalik¹⁾, B. J. Kowalski¹⁾, M. Suffczynski¹⁾, A. Mycielski, S. Colonna²⁾, C. Ottaviani²⁾, F. Ronci²⁾, A. Cricenti²⁾

poster

1) *Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland*

2) *Istituto di Struttura della Materia del CNR, Rome, Italy*

B-38

The surface differential reflectivity (SDR) and photoemission methods were used to study surface electronic structure of CdTe and ZnTe (110) surface. The changes of the reflectivity coefficient were caused by the changes of electric field (band bending) induced at the surface region by high power laser illumination of the sample. The obtained results are interpreted as corresponding to the Franz-Keldish effect [1]. The CdTe and ZnTe samples were grown by modified Bridgeman method in the Institute of Physics PAS in Warsaw [2]. The SDR spectra were measured for the (110) surface illuminated by the Argon laser (radiation in range between 457 and 514.5 nm, power 500 mW). The SDR spectra were obtained as a function of laser power illumination and the linear increase of the measured amplitude of SDR spectra was observed. The shift of the surface potential was estimated on photoemission spectra as a shift of the Cd 4d level position caused by the laser illumination of the surface. The exponential time decay of the amplitude of SDR spectra were measured after turning of the laser illumination. The decay time constant was estimated on 1300 sec for clean surface of CdTe. The attempts were done to discuss obtained results in the frame of the Franz-Keldish effect description.

This work was supported within: Eur. Comm. IHP-Cont. HPRICT199900040/2001-00140; and KBN SPUB-M/DESYP/P-03/DZ-213/2000; and European Community Prog. G1MA-CT-2002-4017 (Center of Excellence, CEPHEUS).

1. L.V. Keldish, Zh. Eksp. Teor. Fiz. 34, 1138 (1958), and W. Franz, Z. Naturforsch. A13, 484 (1958)

2. A. Mycielski et al. J. Cryst. Growth, 197, 423 (1999)

Thermo- and galvanomagnetic characterisation of heterophase structures with variable configuration of inclusions

19:00 -

Sergey V. Ovsyannikov¹⁾, Grigoriy V. Vorontsov¹⁾, Vladimir V. Shchennikov¹⁾

poster

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B-39

For describing of properties of heterophase structures the model of orientated inclusions with variable configuration of phase inclusions have been developed and the calculations of electrical and thermoelectric properties have been performed. The results agreed with the experimental data obtained for a range of semiconductors at high pressure

near phase transition points. Recently the novel technique for experimental investigations of thermomagnetic properties of semiconductor micro-samples have been developed at ultrahigh pressure up to 30 GPa.

In the present work the general equations for thermomagnetic (longitudinal and transverse Nernst-Ettingshausen effects) and galvanomagnetic (magnetoresistance, Hall effect) properties of heterophase systems on concentration and configuration of phase inclusions have been derived in the model of orientated phase inclusions. For the most important particular cases of inclusions: spherical, needle-shaped, and disk shapes the simplified equations have been obtained. The relation between thermomagnetic effects and their galvanomagnetic analogues (Hall effect, magnetoresistance) have been analysed. The expressions for thermoelectric effectiveness and figure of merit (Q-factor) of heterophase systems have been derived. The results of calculations are compared with experimental data for semiconductors of VI and IV-VI Groups obtained in the vicinity of phase transitions under pressure. The approach developed is a perspective one for using in micro-device technology for quality control, characterisation, etc. and for advanced manufacturing of semiconductor micro-devices.

The work was supported by Russian Foundation for Basic Research Gr.No.01-02-17203.

19:00 -
poster
B-40

Investigation of PbSe single crystals under the neutron irradiation

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In the present work the electric and galvanomagnetic properties of p-PbSe single crystals were investigated under the action of fast neutrons. Samples of PbSe were irradiated by the fast neutrons at $T = 320$ K and after that they were annealed up to 390 K with 10 K step (20 min). The present work was devoted to the experimental investigations of influence of irradiation

-induced defects on the electron structure and electronic properties of PbSe.

The semiconductor-metal phase transition induced by the neutron irradiation have been established by the jump of electrical resistance in several orders of magnitude and by the inverse of the temperature coefficients of resistivity. The temperature and magnetic field dependencies of electrical resistance, magnetoresistance and Hall coefficients of p-PbSe have been obtained for a wide interval of temperatures $T=1.5-370$ K and magnetic fields $B=0-13.6$ Tesla. The Hall effect conserved the positive value under the irradiation and consequent annealing. The above technique allows to change reversibly the defect concentration and, therefore, to vary the electron structure of the crystals. The increasing of density of states in the defect level under the neutron irradiation may be responsible for the transferring of electrons from valence band to this level and for the corresponding enlarging of resistivity observed. This work was partly supported by RFBR Gr.No.(01-02-17203), Ministry of Industry, Science and Technologies of the Russian Federation (Contracts No. 40.012.1.1.1166 (Project No 6/03), Special Federal Program of Basic Research at Russian Academy of Sciences "Quantum macrophysics" and Program of government support to leading scientific schools of Russia (Project No. SS. 639.2003.2)

19:00 -
poster
B-41

X-Ray Study of Lattice Parameters of GaN in a Broad Temperature Range

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III-V nitrides (AlN, GaN and InN) form one of the most intensively studied families of semiconductors, due to their potential wide applications in short-wave optoelectronics. The lattice mismatch and differences in thermal expansion coefficient values of the layer and of the substrate result in the occurrence of strain in the layers. As the physical properties of the layer are strain-dependent, the knowledge of the thermal expansion of the IIIN layer material is of high importance. Detailed studies of the a and c lattice parameters in a broad temperature range are expected to be useful in development of short-wave optoelectronic devices.

Thermal expansion measurements require collecting of a diffraction patterns at a large number of temperature points. This task can be performed with the use of synchrotron radiation. The X-ray diffraction measurements using Debye-Scherrer geometry were carried out at a powder diffractometer (B2 beamline, HASYLAB/DESY) equipped with a He-closed-cycle cryostat and a furnace. In the low temperature experiments, the temperature was determined by a silicon diode while for high temperatures a NaCl internal standard was applied.

The dependence of a and c on temperature was studied in a broad range $11 < T < 1173$ K. The results are consistent with previously collected (for limited T intervals) data [1-4]. The c/a ratio shows a weak tendency to decrease with temperature. This behavior is similar to that for InN reported in [5]. The free positional parameter, u, is found to be practically independent on T.

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Electron backscatter diffraction as a useful method for alloys microstructure characterization

Leszek Klimek¹⁾, Bożena Pietrzyk¹⁾

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Microstructure examination of cast Co-Cr-Mo alloy is presented in this paper. The surface morphology and chemical composition of alloy were investigated by scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX), respectively. The alloy phases identification was carried out by electron backscatter diffraction (EBSD). The different kinds of precipitates in non-homogeneous metallic matrix were found. They were identified as MC and M₂₃C₆ type of carbides in Co-lattice solid solution. The advantages and limits of the EBSD method are described. It is presented that EBSD, as excellent tool for phase identification is valuable, supplementary method for materials research.

19:00 -
poster
B-42

X-ray powder diffraction investigation of lithium manganese spinel oxides with cubic, tetragonal and orthorhombic structure.

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Lithium-ion rechargeable batteries find wide application in modern portable electronic devices. LiMn₂O₄ spinel is the most attractive cathode material for lithium-ion batteries due to its low cost, low toxicity and high energy density. An important problem prohibiting it from wider use as a cathode material is capacity fading during cycling. Jahn-Teller distortion and spinel solution are two main causes for the capacity loss. The charge ordering of Mn⁴⁺ and Mn³⁺ ions and Jahn-Teller distortion induces a phase transformation near the room temperature. The partial substitution of Li⁺ ions for manganese reduces the concentration of Mn³⁺ ions, suppresses the Jahn-Teller effect, and the cubic spinel phase can be retained at low temperature.

Small deviations in the preparation process can cause distinct structural changes. A slight lithium ions deficiency causes the tetragonal phase formation provided that the samples obtained at high temperature are rapidly quenched structure in the solid CO₂. On the contrary, very small amount of excess lithium can influence the cubic to orthorhombic phase transition observed for stoichiometric LiMn₂O₄. Structure refinement by the Rietveld profile analysis, based on the laboratory and synchrotron radiation X-ray diffraction data, has been performed on the stoichiometric, Li-excess and Li-deficient lithium-manganese spinel oxide samples.

19:00 -
poster
B-43

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GISAXS study of hydrogen implanted silicon

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2) *Sinchrone Trieste, Trieste, Italy*

3) *Universita di Modena and Reggio Emilia, Modena, Italy*

The grazing incidence small angle X-ray scattering (SAXS) technique was used to study monocrystalline silicon samples implanted with H₂ ions at energy of 32 keV and to the dose of 1E16 ions/cm². Samples were annealed isochronally at different temperatures in the range from 100 to 900 C.

Although the H depth distribution was expected to be smooth initially, nanosized features, like agglomerates of defects have been detected (minor correlation peak observed in implanted but not annealed sample). Annealing destroys this feature due to the relaxation of defects structures, i.e. redistribution of vacancies and hydrogen. Above 300 C a well defined film with highly correlated borders is formed on the edge of the layer rich in defects, whose thickness is slowly decreasing from 17 to 12 nm with increasing annealing temperature. Moreover, defects as well as hydrogen are migrating towards the surface with increasing annealing temperature, as indicated by the increase in surface roughness. We will present a model for the film structure changes obtained by data evaluation based on the distorted wave Born approximation.

19:00 -
poster
B-44

Twin structure of LSGMO crystals studied by Laue method

19:00 -
poster

B-45 **Dmytro I. Savytskii³⁾, Dmytro Trots³⁾, A. Matkovskii³⁾, Carsten Paulmann²⁾, Ulrich Bismayer²⁾, Marek Berkowski¹⁾**

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2) *Mineralogisch-Pertographisches Institut, University of Hamburg, Hamburg, Germany*

3) *Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine*

Twin domains and their boundaries are the dominant microstructural features in many perovskites. Increasing interest in perovskite-like compounds for advanced energy applications has led to intensive research on real structures since it substantially influence the physical properties. The "white" character of synchrotron radiation re-actualized the old Laue method: it is now possible to record thousands of diffraction spots from very small crystals in a very short time using a charge-coupled device (CCD) area detector.

An investigation of real structure in $\text{La}_{0.95}\text{Sr}_{0.05}\text{Ga}_{0.9}\text{Mg}_{0.1}\text{O}_{2.92}$ (LSGMO) perovskite-type crystals has been undertaken using the technique of white beam X-ray diffraction in 300-1000 K temperature range. Experiments have been carried out using the Kappa-diffractometer at HASYLAB beamline F1 equipped with a SMART CCD system. $\text{La}_{0.95}\text{Sr}_{0.05}\text{Ga}_{0.9}\text{Mg}_{0.1}\text{O}_{2.92}$ twinned crystals were grown using the Czochralski technique.

Characteristic twin structure is formed by the intersection of walls $(010)_p$ and $(01-1)_p$ in orthorhombic phase at room temperature. The same domain walls were observed in high temperature trigonal phase. Temperature cycling via the phase transition demonstrated that the domain structure is reversible in small crystal and is composed entirely of mutually permissible twin walls, and thus allows a stress-free intergrowth of all observed domain states in both phases. The Laue technique gave the orientation of observed domains and provided important information for the study of anisotropy of observed diffuse scattering and short vacancy ordering in oxygen conductor $(\text{La,Sr})(\text{Ga,Mg})\text{O}_{3-x}$.

19:00 - **Thermal expansion of NdGaO_3 perovskite**

poster **Anatoliy Senyshyn³⁾, Leonid Vasylechko³⁾, Carsten Bähz²⁾, Ulrich Bismayer¹⁾, A. Matkovskii³⁾**

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B-46

Thermal behaviour of the NdGaO_3 crystal structure has been precisely investigated by means of the high-resolution powder diffraction technique using synchrotron radiation in the wide temperature range 12-1223 K. The diffraction experiments were carried out at the powder diffractometer at B2 line (HASYLAB/DESY). It was shown that NdGaO_3 possesses orthorhombically distorted GdFeO_3 type of structure (sp. group Pbnm, Z=4) in the whole temperature range investigated. Temperature variations of the lattice parameters and cell volume were found to be anisotropically and nonlinearly increasing with the temperature.

Thermal expansion was calculated by two independent theoretical methods-quasiharmonic lattice dynamics using GULP software and Debye model. Deviations between calculated and observed thermal expansion at low temperatures (50-200 K) [1] can be explained by the partially non-applicability of the Debye approximation for perovskites and by the influence of phonon-phonon interaction. Observed deviations at high temperatures might be associated with the increasing of anharmonicity contribution and with the possible processes of the vacancies formation.

[1] D. Savytskii, L. Vasylechko, A. Senyshyn et al., Low-temperature structural and Raman studies on rare-earth gallates, Physical Review B (accepted for publication)

Acknowledgement. This work was supported by WTZ (UKR-012-97), Ukrainian Ministry of Science (Project "Ion"). We would like to thank Dr. A. Oganov for assistance with the analysis of the data.

19:00 - **Influence of substrate miscut angle on dislocation density in GaAs/Si heterostructures obtained by HRXRD**

poster **Artem Shalimov²⁾, J Bak-Misiuk²⁾, Andrzej Misiuk¹⁾, Maria M. Calamitoutou³⁾, A Georgilakilas⁴⁾**

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4) *Physics Department, FORTH/IESL and University of Crete, Greece, Greece*

B-47

The dislocation density of a GaAs layer grown on a vicinal (001) Si substrate has been studied as a function of Si miscut angle by X-ray diffraction technique. The high-resolution X-ray diffractometer equipped with Bartels monochromator in double and triple crystal modes was used.

Using the kinematical theory of X-ray scattering by a crystal with dislocations developed by Hordon and Averbach [1], the dislocation content was investigated by analysing the diffraction peak profiles, the reciprocal lattice maps and the Williamson-Hall plots for selected peaks.

The results were described by a model assuming that, as a result of the 3-D layer growth and GaAs cell deformation character (caused by difference of thermal expansion coefficient), subgrains have dislocations inside and at the boundaries. In this case, dislocations at the subgrain boundaries and surrounding strain cause the planes tilt effect. The broadening of diffracted peaks caused by strain inside subgrains and by tilt from dislocations at the boundaries is independent of each other [2]. This model explains the decrease of dislocation density in the layer

after high pressure-high temperature treatment.

Our results show that the increase of substrate miscut angle causes a decrease of mismatch strain and dislocation density. The data obtained from X-ray measurements and other experimental methods are compared.

1. M.J.Hordon, B.L.Averbach. (1961) Acta Metallurgica v.9, 273-246
2. C.Ferrari et al. (1996) Applied Physics Letters. 69 (27), 4233-4235

Time-resolved SAXS data analysis of a lamellar two-phase polymer system by means of the correlation function

Czesław Slusarczyk¹⁾

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Melting and crystallization of linear polyethylene (HDPE, high-density polyethylene) and its blends with poly(ethylene- α -olefin) copolymers has been the subject of many studies in recent years. The final properties of such polymers depend on the volume fraction of the crystalline domains as well as on their size and structure. During crystallization from quiescent melt these polymers usually form thin lamellar crystals. These lamellae are arranged in stacks, with layers of amorphous material being inserted between the crystalline lamellae. Because amorphous and crystalline layers in lamellar stacks exhibit different electron densities, the small-angle X-ray scattering (SAXS) patterns and the derived correlation functions are commonly used in polymer morphology characterization. These functions allow for determination the values of the long period (L_P), the crystalline and amorphous layer thickness (l_C and l_A respectively) and the local volume fraction crystallinity (Φ_L). Φ_L can be obtained from the correlation functions in two different ways. Both methods have their own prerequisites and limitations. Moreover, application of the correlation functions requires subtraction of contribution due to electron density fluctuations and realization a number of corrections of measured intensities.

The present paper shows all steps necessary in the evolution of the correlation function and the extraction of structural data from this function. In the application part of this study the correlation functions derived from time-resolved SAXS experiments using synchrotron radiation are used to describe the structural changes of blends of HDPE and a homogeneous copolymer of ethene-1-octene. The structural changes were observed during cooling at a rate 10° C per minute from the melt to room temperature and during subsequent heating at the same rate from room temperature to melting temperature.

19:00 -

poster

B-48

Moessbauer spectroscopy, interlayer coupling and magnetoresistance of irradiated Fe/Cr multilayers

Feliks Stobiecki²⁾, Michal Kopcewicz³⁾, Jacek Jagielski^{1,3)}, Bogdan Szymański²⁾, Marek Schmidt²⁾, Janusz Dubowik²⁾, Maciej Urbaniak²⁾, Justyna Kalinowska³⁾

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3) *Institute of Electronic Materials Technology (ITME), Wólczyńska, Warsaw 01-919, Poland*

The influence of 200 keV Ar-ion irradiation on the interlayer coupling in Fe/Cr multilayers exhibiting the giant magnetoresistance effect (GMR) is studied by the conversion electron Mössbauer spectroscopy (CEMS), magnetic hysteresis loops, magnetoresistivity and electric resistivity measurements and supplemented by the small angle X-ray diffraction (SAXRD).

An increase of interface roughness of Fe/Cr multilayers caused by irradiation with 200 keV Ar ions and doses exceeding 5×10^{12} Ar/cm² is clearly seen in CEMS measurements, while, the SAXRD technique, even at higher ion doses, hardly detects such changes in microstructure. On the other hand, a subtle modification of the microstructure induces distinct changes in magnetization reversal (increase of the remanence magnetization) and strongly decreases GMR effect with increasing irradiation dose. The most prominent changes are observed for the samples with a small thickness of Cr layers.

The increasing immunity of GMR effect to ion irradiation with increasing thickness of Cr layers as well as correlation between changes in GMR and antiferromagnetically coupled fraction suggest that the main effect responsible for the decrease of GMR is caused by the pinholes creation. Characteristic changes in the temperature dependence of the remanence magnetization measured for the as-deposited and irradiated samples results from an increase of pinholes density and their sizes during irradiation process.

This project was financially supported by Polish Committee for Scientific Research (KBN), grant number: PBZ/KBN-013/T08/23

19:00 -

poster

B-49

XPS investigations of NdGaO3 wafers

Ewa Talik¹⁾, Magdalena Kruczek¹⁾, Halina Sakowska²⁾

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19:00 -

poster

B-50

X-ray photoelectron spectroscopy was used to examine the electronic structure of neodymium gallium perovskite single crystals. They are a pseudo - cubic materials being used as a substrate for epitaxial growth of high temperature superconductors and for group III nitrides (such as GaN). The NdGaO₃ single crystals were grown by the Czochralski method. In order to prepare specimens for XPS analysis, crystals were cut into wafers (0.7 mm thickness), lapped and polished (0.1 m SiO₂). Before the XPS analysis substrates were chemically etched in H₃PO₄ at 65°C for 10 min.

The electronic structure measurements of the unpolished, polished and etched (011) wafer planes were performed. For a comparison one wafer was broken perpendicular to the (011) plane, to obtain a fresh surface, without contamination with adsorbed carbon and oxygen. The XPS spectra in the wide energy range 0 - 1400 eV show a presence, for all measured crystals, a contamination with carbon. In the treated crystals a calcium contamination was found. The etched crystal surface was additionally contaminated with phosphorus. In the broken crystal composition is in agreement with a nominal value. For the other crystals a decrease of the gallium concentration was observed. The Nd, Ga and O line widths change. They are widest for unpolished crystal. The the XPS measurements show that etching with H₃PO₄ did not cause surface damage.

Moreover XPS measurements of the (011) wafer planes annealed in hydrogen atmosphere at 850-1200°C were performed. There is a requirement of stability of substrate at the temperature of the growth of epitaxial layers. It was found that during the annealing processes the wafer's surfaces were covered with a white powder. The XPS measurements and chemical composition calculations for the annealed wafers show a vaporising process of Ga.

The Hg_{1-x}Cd_xTe Epilayers Investigation and Characterization

Zinoviya F. Tsybriy-Ivasiv¹⁾, Larysa O. Darchuk¹⁾, Fedor F. Sizov¹⁾

1) Institute of Semiconductor Physics NAS Ukraine, Kiev, Ukraine

19:00 -
poster

B-51

Most modern IR photodetectors based on Hg_{1-x}Cd_xTe narrow-gap semiconductors are manufactured from LPE- and MBE-films grown on CdTe and Cd_{1-y}Zn_yTe substrates. The energy gap of these ternary compounds is a function of the cadmium atomic concentration x. Precise determination of the composition grading is extremely essential for monitoring the material quality and predicting the photoelectrical properties of Hg_{1-x}Cd_xTe-based infrared detectors.

The Hg_{1-x}Cd_xTe epilayers (12-25 mkm thickness) with different Cd concentrations x, p- and n-type conductivity were investigated. The thickness of the layers was determined, using high-resolution light microscope and interference optical methods. The temperature dependence of the minority carrier lifetime were measured. To determine the value of x, the composition profile x and the calculation of the cutoff wavelength at 80 K were carried out the experimental and theoretical studying of the optical transmission spectra at room temperature by two different methods. In the case of MBE-layers the composition grading is absent, but in LPE-layers case there is non-linear composition grading. This data were confirmed by independent SIMS - measurements of the LPE and MBE films. The photocurrent investigations of the photoresistors prepared on the Hg_{1-x}Cd_xTe epilayers confirmed that cutoff wavelength corresponds to the composition, measured by the optical methods.

Crystal structure and thermal expansion of PrGaO₃ in the temperature range 10-1253 K

Leonid Vasylechko⁴⁾, Anatoliy Senyshyn⁴⁾, Michael Knapp³⁾, Marek Berkowski¹⁾, Carsten Paulmann²⁾, Ulrich Bismayer²⁾, A. Matkovskii⁴⁾

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4) Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine

19:00 -
poster

B-52

Crystal structure and thermal expansion of PrGaO₃ have been investigated in the temperature range 10-1253 K by means of single crystal and high-resolution powder diffraction techniques using synchrotron radiation. Single crystal and powder diffraction experiments were carried out at the beamlines F1 and B2 at HASYLAB/DESY. PrGaO₃ possesses an orthorhombically distorted GdFeO₃ type structure in the whole temperature range investigated. The changes in lattice parameters and cell volume are anisotropic and nonlinear with increasing temperature. Negative thermal expansion in [010]- and [100]-directions were detected in the temperature ranges 10-180 K and 10-50 K, respectively. Negative thermal volume expansion is observed in the temperature range 10-50 K. Due to pronounced anisotropy of the thermal expansion, the normalized values of the lattice parameters b_p and c_p become equal at 1200 K. For the average interatomic distances (PrO)₈-(PrO)₁₂, (GaO)₆ and (OO)₈ a linear increase with temperature is observed. For the temperature dependencies of the (PrPr)₆, (PrGa)₈ and (GaGa)₆ average distances a change of slope occurs at 200-300 K.

A Reduction of the bond-length distortion in the corresponding polyhedra with temperature and an increase of the values of the observed tolerance factors indicate a decrease of the perovskite structure deformation in PrGaO₃. From extrapolation of the experimental values of the interatomic distances and their ratios, the temperature of the predicted orthorhombic-to-rhombohedral phase transition could be estimated to 1840-1875 K.

Acknowledgement. This work was supported by WtZ (UKR-012-97), Ukrainian Ministry of Science (Project No. "Ion") and Polish Committee for Scientific Research (Grant N 7 T08A 00520).

Characterisation of Permalloy based thin films and magnetic multilayers using the magnetoresistance and Hall effect measurements

19:00 -

Marius Volmer¹⁾, Jenica Neamtu²⁾

poster

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2) *Advanced Research Institute for Electrical Engineering, Splaiul Unirii 313, Bucharest 74204, Romania*

B-53

Correlation between the Hall effect and magnetization enables the use of this technique for the study of magnetic properties in Permalloy (Py) thin films and Py/NM/Py multilayers. NM denotes Cu, Mo and Al₂O₃ layers. We made the measurements at room temperature in a setup which allows us to perform both Hall effect and magnetoresistance measurements. The Hall effect measurements were performed for different values of the angle between the magnetic field and the normal to the film plane from 0 to 90 degrees. The measured voltages present hysteresis loops at low magnetic field. Because at low magnetic fields the shape anisotropy forces the magnetization to rotate in the film plane what we have is a Planar Hall Effect (PHE) signal which can be used as probe of magnetization reversal. Basically, PHE is an Anisotropic Magnetoresistance Effect (AMR). The PHE measurements were employed to evaluate the magnetization reversal process and the coupling between the magnetic layers. We explain the hysteretic behaviour of the Hall voltage using a simple phenomenological model in which the extraordinary Hall coefficient is expressed as the sum of two terms: a field-independent extraordinary Hall coefficient and a coefficient corresponding to the magnetically disordered state which is related to the AMR and GMR effects. We made also measurements of the Hall voltage as a function of the applied field orientation θ (from 0 to 360 degrees) in order to estimate the perpendicular anisotropy. We complete this discussion using a micromagnetic simulator which can show us the spin distribution for different values of the applied magnetic field and can calculate the hysteresis loop of the sample. The results are related with the samples microstructure. The saturation fields obtained when the magnetic field is applied normal to the film plane are less than the values predicted from the shape anisotropy known for flat surfaces, but are consistent with the film surfaces topography.

Synchrotron X-ray Diffraction studies of silicon implanted with high energy Ar ions after thermal annealing

19:00 -

Wojciech Wierzchowski¹⁾, Julian Auleytner²⁾, Krzysztof Wieteska³⁾, Walter Graeff⁴⁾, Danuta Zymirska⁵⁾

poster

1) *Institute of Electronic Materials Technology (ITME), Wolczynska 133, Warsaw 01919, Poland*

2) *Institute of Physics PAS (IF PAN), Warsaw, Poland*

3) *Institute of Atomic Energy (IEA), Otwock=Swierk, Poland*

4) *HASYLAB at DESY (HASYLAB), Hamburg, Germany*

5) *Institute of Physics PAS (IF PAN), Warsaw, Poland*

B-54

A slab formed sample cut out from low doped Czochralski-grown silicon crystal was implanted with 3 MeV Ar ions to the dose $5 \cdot 10^{14}$ cm⁻² and thermally annealed in two step cycle at 400° C and 700° C. The crystal was characterized with a number of X-Ray diffraction methods exploring synchrotron source of radiation. The methods included white beam Bragg-case section and projection topography, plane wave topography and studying of local rocking curves with a small probe beam.

It was found that the performed annealing did not quite remove the lattice strain induced by the implantation. The residual strain caused the series of interference maxima on the low angle side of substrate maximum in the rocking curve. The rocking curves were different in different regions of the implanted area and together we observed characteristic fringes in plane wave topographs. These effects were most probably caused by unhomogeneity of the ion beam. The analysis of rocking curves including the numerical simulation points the existence of local strain profile maximum situated below the shot-through layer with a relatively small deformation. The Bragg-case section pattern of the implanted area revealed the presence of tiny defects situated at the depth corresponding to maximum of ion range distribution, which may be eventually interpreted as small dislocation loops formed from point defect clusters. The topographs revealed however a concentration of defects in the whole volume of the crystal that most probably are the oxygen precipitates formed during thermal annealing.

New water thinnable acrylic - allyl dispersions in die pressing of alumina

19:00 -

Paweł Wiśniewski, Mikołaj Szafran, Gabriel Rokicki

poster

The die pressing of ceramic powder samples is a high packing process of small grains in stiff forms under the influence of uniaxial action of external pressure.

The disadvantages of pressing are trying to be minimized by selecting suitable materials for making molds, favorable pressing parameters, and by optimizing the rheological properties of the molded ceramic powders. Polymeric organic binders provide the pressed powders with appropriate thickening capabilities and the molded ceramic samples with mechanical strength. These binders should, at their minimal quantity, provide such rheological properties which

B-55

would cause a decrease in the friction forces between the powder particles and matrix, simultaneously providing the green samples with high mechanical strength. These binders are added most often in a 0.5-3 wt. % amount with respect to the ceramic powder.

Water soluble polymeric binders such as poly(vinyl alcohol), methylcellulose and poly(oxyethylene) glycol are most often used in the pressing of ceramic powders. A disadvantage of this type of binders is the small mechanical strength of the samples obtained with them, which precludes the processing of samples in the green state. Various types of polymers, often of hydrophobic properties, prepared in the form of water dispersions, are more and more often used in recent years. Polyacrylic, polyurethane and polyvinyl emulsions are most often used. Studies on the application of water thinnable binders comprising inbuilt amphiphilic macromonomers playing the role of hydrophilizing agents and internal plasticizers intended for alumina molding are presented. The results of studies on the application in the alumina pressing of a new group of acrylic-allyl binders and the effect of water soluble carboxyalumoxanes as coupling agents and polyurethane dispersions applied in molding are also presented.

This work was supported by the Polish State Committee for Scientific Research 4/T09/130/24.

19:00 -
poster
B-56

Surface morphology, nanomechanical and tribological behaviours of ultrathin nitride superlattice films.

HANNA WRZESIŃSKA¹⁾, M. Ekwieńska²⁾, M. Misiak²⁾, Z. Rymuza²⁾, S.A. Chizhik³⁾

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3) *Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus*

Superlattice films made of TiN, CrN, NbN nitrides deposited on n-type single crystal silicon without or with interlayers of silicon dioxide or silicon nitride were studied. The superlattice films with thickness below 200 nm were deposited by PVD technique.

The Atomic Force Microscopy was used to study surface topography and tribological (friction and wear) behaviours and Hysitron's TriboIndenter for nanomechanical tests by indentation technique.

The effect of the design of the superlattice film on its surface morphology and mechanical/tribological behaviours was identified.

The studied films seems to be very good candidates to be applied on rubbing elements of Micro Electro Mechanical Systems (MEMS) or AFM cantilever tips working in contact mode to prevent them against wear.

19:00 -
poster
B-57

X-ray diffractometric study of micro-precipitates created by fast nitrogen ions in GaAs single crystal

Danuta Zymierska¹⁾, Krzysztof Godwod¹⁾, Julian Auleytner¹⁾, Jarosław Choiński²⁾

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2) *Heavy Ion Laboratory of University of Warsaw (HIL), Pasteura 5a, Warsaw 02-093, Poland*

The problem of the influence of fast nitrogen ion implantation on the structure of GaAs is considered. GaAs single crystal was irradiated with a medium dose of 2.85 MeV/n (total energy equal to 40 MeV) nitrogen ions. Previously we showed that a buried layer was created at a depth of the mean ion range. The determination of the structure of this layer is difficult because it is only a small fraction of the whole volume of the sample studied. For this purpose we proposed the experimental strategy for the X-ray diffractometric research of very small polycrystalline precipitates within a single crystal matrix. It was shown that it was possible to reveal the diffraction lines from an additional polycrystalline phase of concentration much below 0.5%, being the limit of standard powder diffraction method. The recording of these lines was possible due to the lowering of the diffractometer background by means of the set consisting of the precise bent quartz monochromator of diffracted beam and a Soler slit, as well as by the rotation of a single crystal sample out of Bragg condition by the angle large enough. In the present paper we apply this diffractometric method to investigation of the buried layer consisting of small precipitates of an additional phase, different from the GaAs single crystal matrix.

19:00 -
poster
B-58

New Technique for Magnetic Phase Transitions Diagnostics

Tim G. Zagurenko¹⁾, Aleksei N. Lachinov¹⁾, Michael Matlak²⁾, Victor M. Kornilov¹⁾

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2) *University of Silesia, Katowice, Poland*

It is commonly known that solids undergo phase transitions with changing temperature, characterized by a critical temperature. However the temperature behaviour of real solids is very often much more complicated. And here

two main problems arise: how to detect mentioned characteristic temperatures and find out what really happens in solids at these temperatures.

This work presents a new technique for the diagnostic of the structural transformation in metals - the surface potential barrier method. The method is based on the measurement of the current flowing through the potential barrier formed on the metal - electroactive polymer interface. The current changes within the temperature range of the structural-phase transitions in metals may be big enough for their simple registration.

In present work Cr, Gd, GdCo₂ samples demonstrating complex magnetic phase transitions were investigated.

The technique allowed both reproducing the well-known results with high certainty and obtaining a lot of complementary information about fine details of these transformations. For the Cr samples the Neel temperature (315 K), the fine structure of the antiferromagnetic-paramagnetic transition and the their dependence on the sample structure were detected. The measurements on the Gd sample revealed not only the Curie temperature (290 K), but also the characteristic temperature of the transition from a spiral to a ferromagnetic spin structure (~230 K). In case of the measurements on the GdCo₂ sample the Curie (410 K) and characteristic temperatures associated with displacements of the Gd and Co ions and shift of the grain boundaries were detected.

Novel technique can be used to study fine structure of various structural transformations in metals and alloys, influence of external fields on the metals, etc. Earlier this method was employed successfully for the study of nanostructured materials.

Metastable state in Hg_{1-x}Cd_xTe crystals

Oleksandr Zakharyash¹⁾

1) National University "Lvivska Polytechnika", Bendera St. 12, Lviv UA-79013, Ukraine

19:00 -

poster

B-59

Measurements of electrical and thermal parameters of Hg_{1-x}Cd_xTe crystals in situ at the melting and crystallization processes and also measurements of inner friction at their deformation process were performed.

Effects of heal releasing have been shown on temperature dependencies of Hg_{1-x}Cd_xTe, CdTe and HgTe crystals thermal capacities.

These effects manifested themselves in the absolute thermal capacity values decreasing for the crystal specimens and ingots after the thermal treatments of the corresponding regimes.

Investigation results unequivocally indicate the thermal instability of the crystals.

Additional investigations with application of the inner friction method confirm conclusions about the predominate influence of nanodefekt complexes and intergranular boundaries on the mechanical and thermal material parameters made on the base of the heat capacity investigation.

Anomaly high absorption of the elastic energy at the temperature 600K was shown. It decreased essentially at the specimen cooling.

Such inner friction spectra change can testify the chemical composition change due to the processes on the intergranular boundaries.

The segregation of Arsenic and Boron in the interface of a polysilicon on silicon during rapid thermal annealing

Abdelali Merabet¹⁾

1) Laboratoire Physique et Mecanique des Materiaues, Faculte des Sciences de l'Ingenieur, University de Setif, Setif 19000, Algeria

19:00 -

poster

B-60

In this work, we have used wafers achieved in the CNET CNS of Meylan, according to a manufacturing process used for the bipolar compatible technologie CMOS (Complementary Metal-Oxide-Semiconductor). These wafers of type P, doped with boron. Before the deposit of polysilicon, the oxide film has been removed with HF, followed with a cleanning permits to minimize effects of diffusion barrier in the interface and to increase the gain. Afterwards, the polysilicon recevies either a single implantation with arsenic or boron, or double implantation starting with arsenic and followed by boron. The arsenic play the role of a doping emitter, the boron that the extrinsic base. The samples were then annealed at various temperature between 1000 and 1150°C, for 20 s, to permit an electrical activation of the ions and their fast redistribution. The samples are then analysed by a secondary ion mass spectroscopy (SIMS) CAMECA IMS4F of Lyon, France.

The test of values permits to note that the percentage of the dose distributed in the monosilicium is weaker than the one of the dose segreated in the interface. A remarkable point is the discontinuity of the profiles of diffusion of the boron. This indicates the absence of the segregation peak in the interface. We notice the continuity of arsenic profiles extrapolated in the interface, that shows the absence of the barrier of diffusion. It appears that about 11% and 10% of arsenic atoms implanted are segreated in the interface in the case of the diffusion and the codiffusion respectively.

The comparison of diffused arsenic dose in the monosilicium in the case of the diffusion and the one of the codiffusion, shows a reduction of about 50% of the arsenic dose in codiffusion relatively to the one in monodiffusion. Where as for the boron, the difference is big which confirms the stopping of the diffusion of the boron in the presence of the arsenic.

Auger Electron Spectroscopy studies of the microstructure and diffusion pro-

19:00 -

poster

cesses at interfaces of multilayer platings used in laser diode technology**Valentina V. Arbenina¹⁾, R H. Akchurin¹⁾, A A. Marmalyuk²⁾, O I. Govorkov³⁾**

B-61

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In modern technology of the laser diode fabrication, based on heterostructures AlGaAs/GaAs, metallic multilayer plating for contact platform is used. Each of formed layers with the thickness of the order 500-1000 Å executes the certain function.

It is important to know the microstructure of the thin layers of plating and phase composition on boundaries for choice contacting metals, and to estimate correlation of the thicknesses of the layers, as well as condition of their fabrication.

Formation of the microstructure of metallic layers is accompanied by the development of the internal tensions. The diffusion processes on boundaries in AlGaAs/GaAs/Ti/Pt/Au and AlGaAs/GaAs/Ti/Pt/Ti/Au systems were studied using Auger Electron Spectroscopy (AES) and Electron Imaging Microscopy (EIM).

High-resolution grazing-sputter-angle AES was also used for concentration depth profile investigation.

A broadening and shift of the peaks in Auger-spectra of the surface after etching, allows to conclude about chemical bond between elements and possible phases of the intermediate composition, which are formed as a result of "reactive" diffusions on Ti/Au, Ti/Pt interfaces during the deposition and impulse annealing of contact .

Breakdown of passivity in Al-Ta and Al-Nb amorphous alloys**Artur Jaśkiewicz²⁾, Maria Janik-Czachor³⁾, Piotr Kędzierzawski³⁾, Zbigniew Werner¹⁾**

19:00 -

poster

*1) The Andrzej Soltan Institute for Nuclear Studies (IPJ), Świerk, Otwock 05-400, Poland**2) Department of Materials Science, Warsaw Institute of Technology, Warsaw, Poland**3) Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland*

B-62

The effect of Cl⁻ (minus) ions (0.1M NaCl) on the anodic behaviour of two series of sputter-deposited Al-Ta (11-46 at %) and Al-Nb (13-46 at %) amorphous alloys was investigated in neutral electrolytes by using electrochemical and in-situ microscopic methods.

The samples were characterized by AES; a thin alumina film was found to cover the surface of the substrate, whereas transition metal was not detected therein. In solutions containing Cl⁻ (minus) the alloys broke down at and above the pit nucleation potential E_{np} . The alloys exhibited a stable passivity within a much wider ($\Delta E > 1V$) potential range than crystalline Al. In-situ microscopic investigations revealed that for Al-Ta and Al-Nb amorphous alloys the local current density within the pits was $\sim 10^6$ higher than the c.d. from the remaining passive surface. The results are compared with those for Al-Mo and Al-W alloys and the mechanism of restrained passivity breakdown is discussed.

This work was supported by the State Committee for Scientific Research under grant 7T08C0 1220

Photoemission studies of very thin (<10nm) silicon oxynitride (SiO_xN_y) layers formed by PECVD**Patrick Hoffmann¹⁾, Romuald B. Beck²⁾, M. Cuch²⁾, M. Giedz²⁾, A. Jakubowski²⁾, Dieter Schmeisser¹⁾**

19:00 -

poster

*1) Brandenburg Technical University, Department of Applied Physics/Sensorics (BTU), Erich Weinert Str. 1, Cottbus 03044, Germany**2) Institute of Microelectronics and Optoelectronics, Warsaw Technical Univ., Warsaw, Poland*

B-63

The "ITRS Roadmap" suggests the necessity of working out the processing methods allowing formation of ultrathin dielectric layers with higher than for silicon dioxide dielectric permittivity value. The silicon oxynitride layers (SiO_xN_y) seem to be the most natural compromise. But still none of high temperature methods used for its formation can be seriously considered as final solution for future ULSI-CMOS ICs production due to the inevitable formation of nitride monolayers just at the silicon-insulator interface. The main scope of this investigation is to check if this is true.

The oxynitride layers were produced by PECVD method. The process has already been optimised in order to allow repeatable and reliable formation of ultrathin layers (<10 nm). These layers were investigated by photoelectron spectroscopy (PES) using variable excitation energy. This results in a variable escape depth of the photoelectrons from were the depth structure of the sample can be concluded. Due to the combination of chemical information and depth information this method is a unique tool for investigating the hidden nitride layers. In this work we present a comparing study of oxynitride layers with different oxygen-to-nitrogen ratios and different post-deposition annealing temperatures investigated by the above described method.

Tautomeric preferences in solution and crystalline state of new ylidemalonodinitrile, fungicidal species precursor.

19:00 -

Jacek M. Grochowski¹⁾, Paweł Serda¹⁾, Michał Markiewicz¹⁾, Bartłomiej Kozik²⁾, Janusz J. Sepioł²⁾

poster

1) Regional Laboratory of Physicochemical Analysis and Structural Research, Jagiellonian University (REG LAB UJ), Ingadena 3, Kraków 30-060, Poland

B-64

2) Department of Organic Chemistry, Jagiellonian University (Org.Chem, UJ), Ingadena 3, Krakow 30-060, Poland

Some aromatic, vicinal aminonitriles exhibit strong fungicidal properties against selected tested fungi [1]. This encouraged us to search for easily synthesized carbocyclic, aromatic aminonitriles. Using the Knoevenagel condensation of 2,2-bis(4-chlorophenyl)acetaldehyde with malonodinitrile and chromatographic purification, a slowly solidifying oily product was obtained. NMR spectra recorded in CDCl₃ revealed presence of two tautomeric forms which arose through the migration of the carbon-carbon double bond. The ratio of the expected tautomer (3,3-bis(4-chlorophenyl)-1-propene-1,1-dicarbonitrile) and the second tautomer (3,3-bis(4-chlorophenyl)-2-propene-1,1-dicarbonitrile) was estimated from ¹H NMR spectrum as 3.8:1. The former is a precursor for 1-amino-4-(4-chlorophenyl)-naphthalene-2-carbonitrile which belongs to a group of compounds with proven fungicidal activity. Analytical sample of ylidemalonodinitrile was recrystallized from petroleum ether (bp: 60-90°C) affording yellowish crystals, mp 101.5-103.5°C, monoclinic system, space group P2₁/c, lattice parameters a=13.8905(9), b=7.7323(4), c=14.335(1) Å, β=93.846(2)°. The structure was solved by direct methods and refined using full-matrix LSQ procedure. All hydrogen atoms were located on the difference Fourier map of electron density.

Both the hydrogen atom position bonded to the carbon coordinating two chlorophenyl groups and the double bond position indicated univocally the presence of solely tautomer 1 in the crystalline phase.

The energy calculations for both tautomers were carried out in order to find the most stable conformer for each tautomer and then compare their energy values to prove which tautomer is energetically preferred. The resulting energy differences are rather low indicating that both tautomers are energetically equivalent in vacuum.

[1] J. Wilamowski, E. Kulig, J.J. Sepiol, Z.J. Burgiel, Pest Manag. Sci. 57 (2001) 625.

Towards In-Situ Pulsed Laser Deposition

19:00 -

Vedran Vonk¹⁾, Sybolt Harkema²⁾, Guus Rijnders²⁾, Heinz Graafsma¹⁾

poster

1) European Synchrotron Radiation Facility (ESRF), 6, Jules Horowitz, Grenoble 38000, France

2) Low Temperature Division and MESA+ Research Institute, Faculty of Science and Technology, University of Twente, Postbus 217, Enschede 7500 AE, Netherlands

B-65

Pulsed Laser Deposition (PLD) has become a widely used method to grow single crystal thin films of high quality. Since PLD can take place under controlled oxygen atmosphere, it is especially suited for the growth of thin-film high-Tc superconductors (HTSC's), whose properties are highly sensitive to their oxygen content. Although the PLD process is commonly used, the growth and nucleation mechanisms are still not completely understood. In order to gain more insight into these processes, a miniature PLD chamber is being constructed for the purpose of following with x-ray diffraction (XRD) the initial stages of growth of YBa₂Cu₃O_{7-δ} (YBCO) on SrTiO₃ (STO) substrates, and the subsequent annealing of the film.

A small furnace is constructed to study the structural properties of YBCO films on STO substrates under controlled environment. In the case of thin films grown on substrates, strain is built up in the film due to the lattice mismatch between the former and the latter. Since the lattice mismatch is a function of temperature, it is of interest to study the strain-state of the film at different temperatures.

Here we present the results of in-situ XRD measurements of YBCO thin films at elevated temperatures in controlled oxygen atmosphere, thereby imitating the annealing step as carried out in the PLD process. From the splitting of the XRD reflections due to twinning, the spontaneous strain is determined. This is directly related to the difference in lattice parameters a and b. In 1 bar of oxygen, a gradual transition is seen around 250°C, indicating strain relaxation.

Structure modifications in materials irradiated by ultra-short pulses of VUV free electron laser

19:00 -

Jerzy B. Pełka¹⁾, Andrzej Andrejczuk⁵⁾, Henryk Reniewicz⁵⁾, Norbert Schell⁴⁾, Jacek Krzywiński^{1,3)}, Ryszard Sobierajski²⁾, Andrzej Wawro¹⁾, Zbigniew R. Zytewicz¹⁾, Dorota Klinger¹⁾

poster

1) Institute of Physics, Polish Academy of Sciences, al. Lotników 32/46, Warsaw 02-668, Poland

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2) Warsaw University of Technology, Department of Physics, Koszykowa 75, Warsaw 00-662, Poland

3) HASYLAB at DESY, Notkestrasse 85, Hamburg Germany, Germany

4) ROBL-CRG at the ESRF, B.P. 220, Grenoble F-38043, France

5) University of Białystok, Institute of Experimental Physics, Poland

The VUV TESLA FEL delivers radiation of peak power in the order of 1 GW in a short pulse of only 50-100 fsec. Such power levels are typical for laser ablation using conventional pulsed lasers. Theoretical predictions and very few experiments indicate that the behaviour of the solid matter under such conditions can differ substantially from that observed when the irradiation pulse durations are in the picosecond or nanosecond region. Investigation of the structure and microstructure of solids exposed to this type of irradiation is crucial to understand damage mechanisms in materials, and can imply also promising applications in various areas of nanotechnology.

The irradiation of various solids - insulators, semiconductors and metals has been carried-out with the quantum energy centered around 14 eV, in short pulses of only 80 fs and of peak power up to 1 GW. The FEL beam was focused on sample surfaces to microspots of size 10-100 μ m. The energy density in the spots varied from below the ablation threshold ("annealing mode") up to far above this threshold ("damage mode"). The structural modifications induced with the irradiation was studied by a set of microscopic methods, the micro-Raman, as well as by the X-ray diffraction. AFM and Nomarski pictures illustrating variety of morphological structures created in the damaged areas will be shown. Especially promising seems to be a technique of recording the x-ray diffracted intensity distribution maps. We will show examples of the maps recorded on Si wafers around chosen spots. The maps determine the damage distribution range and intensity around the spots. The observed features are related to the FEL irradiation fluencies applied.

19:00 - **SAXS and WAXD real time studies on nanostructures of selected polymer materials**

poster **Jaroslav Janicki¹⁾**

B-67 *1) University of Bielsko-Biala, Institute of Textile Engineering and Polymer Materials, Willowa 2, Bielsko-Biala 43-309, Poland*

The results of the nanostructure investigations of selected polymers in non-isothermal conditions are presented. The main interest is focused on the formation and changes of the supermolecular structure during dynamic melting and crystallisation processes. The wide group of polymers including polyethylene, homogeneous ethylene copolymers, isotactic polypropylene (iPP), liquid crystal oligoester (LCO) and LCO-iPP blends were taken as the material for investigation.

These were chosen in view of the fact that both homogenous ethylene-1-alkene copolymers as well as liquid crystal oligoester are new polymers of great significance for industrial practice. The new type of polymer obtained from liquid crystal oligoester and isotactic polypropylene is of particular importance for materials science; this is one of the first such materials obtained from a thermoplastic polymer and a liquid crystal polymer with a low melting temperature, which enables it to be processed from a melt while exploiting the unique properties of the liquid crystal state.

The research carried out shows that the nanostructure of the polymers investigated is complex and depends on thermal conditions. This in turn means that both the qualitative and especially the quantitative description of the nanostructure require using of complementary research methods. The methodology, which is of particular significance in this respect, is that which combines small-angle X-ray scattering (SAXS) with wide-angle X-ray diffraction (WAXD) and differential scanning calorimetry (DSC). Real-time dynamic SAXS and WAXD measurements were performed using synchrotron radiation with very high beam intensity and very fast position sensitive detectors. This meant that distributions of intensity of radiation scattered over both angle ranges could be simultaneously recorded at very short time (for each sample 156 SAXS and WAXD curves were recorded).

This paper was supported by KBN - Grant No 7 T08E 027 20

19:00 - **Effect of cathodic hydrogen charging on catalytic activity of Cu-Hf amorphous alloys**

poster **Marcin Pisarek¹⁾, Maria Janik-Czachor²⁾, Annett Gebert⁴⁾, Piotr Kędzierzawski³⁾, Bulcsú Rác⁵⁾**

B-68 *1) Department of Materials Science, Warsaw Institute of Technology, Warsaw, Poland*

2) Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland

3) Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland

4) Institut fuer Metallische Werkstoffe, IFW, Dresden, Germany

5) Department of Organic Chemistry, University of Szeged, Szeged, Hungary

Hydrogen is one of the most efficient embrittling atomic species introducing both structural and chemical changes in solid materials and eventually, causing their degradation. This highly detrimental effect - when controlled - may, however, be used in benefit of e.g. enhancing catalytic activity of materials by increasing their specific surface area and modifying surface chemistry. This method was used to activate Cu-Hf (Cu₆₅Hf₃₅, Cu₆₁Hf₃₉) amorphous alloys for catalytic purposes.

Hydrogen charging at $i=1$ mA/cm² in alkaline solution (0.1M NaOH) was used to study the effect of hydrogenation on morphology and crystallization processes. A similar procedure was applied in acid solution (0.1M H₂SO₄) to introduce more hydrogen and more pronounced morphological and structural changes. The samples were then catalytically tested for dehydrogenation of 2-propanol. The efficiency increased up to 50% at selectivities close to 100%. The structural, chemical and morphological changes were followed with: XRD, AFM, SEM, X-ray electron

probe microanalysis (EDS). H/Me ratio was determined by elemental analysis.

This work was supported by the State Committee for Scientific Research under grant 7T08C02524

X-ray diffraction studies of thermal properties of bulk- and surface-atoms of nanocrystalline SiC

Svetlana Stelmakh²⁾, Stanisław Gierlotka²⁾, Ewa Grzanka^{1,2)}, Bogdan F. Palosz²⁾

1) University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland

2) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

19:00 -

poster

B-69

Until recently no definite experimental evidence of a presence of strain and/or of reconstruction of the surface structure of nanocrystals has been presented. Using powder diffraction techniques we were able to determine the atomic structure of nanocrystalline grains. In a series of powder diffraction experiments, using synchrotron and neutron sources, we showed that powder diffraction techniques can be effectively applied for studies on nanopowders with crystallite dimensions below 20 nm and can differentiate between the structure of the grain interior and its surface. A meaningful and unique interpretation of such diffraction experiments requires that the data are collected in a large diffraction vector range, 150 nm⁻¹ or more. We evaluated the structure of nanocrystals of SiC, diamond, and GaN using the following characteristics: (a), the type of strains at the surface (compressive or tensile), (b), the profile of the strain field, (c), thickness of the surface layer and, (d), a response of the lattice of the grain interior to the stress exerted by the surface atoms. In this paper we report preliminary results of examination of thermal properties of SiC nanocrystals aiming at, (1), determination of the thermal expansion coefficient and, (2) the Debye temperature, assuming that thermal vibrations are different in the grain core than at its surface. The experiments were performed at SNBL station, ESRF, Grenoble (wavelength of 0.05 nm) in the temperature range of 130 - 920 K. We examined 15 nm SiC powders: (i), as synthesized, (ii), sintered under high-pressure high-temperature conditions, and (iii), embedded in a nanocomposite with (Zn + Al) matrix. The elaboration of the experimental data is based on the theoretical diffraction patterns calculated for nanocrystals with different thermal vibrations modes.

Tuesday, September 16th

Morning session - Jürgen Härtwig

Main Building, room 134

11:00 - 12:30

Quantitative TEM analysis of quantum structures

Wolfgang Neumann¹⁾, Holm Kirmse¹⁾, Ines Häusler¹⁾, Reinhard Otto¹⁾, Irmela Hähner¹⁾

1) Humboldt University of Berlin, Institute of Physics, Newton Street 15, Berlin 12489, Germany

11:00 - 11:30

invited oral

Nowadays semiconductor structures can be generated of low dimensions that charge carriers are confined to a space of only some nm³ and thus quantum-physical phenomena become important. The properties of so-called quantum structures essentially depend on the perfection of their structure, size, arrangement, morphology, and on their chemical composition.

The potential of combined use of conventional transmission electron microscopy (CTEM), high-resolution imaging (HRTEM) and digital image analysis is applied to study quantum dot (QD) structures in various semiconductor materials. The classical diffraction contrast method is applied to visualize the strain field in the surrounding of the QDs. Dark-field imaging allows a qualitative analysis of chemical composition using chemically sensitive reflections. Furthermore, it will be presented how the techniques of quantitative HRTEM (qHRTEM) can be used to determine the local strain, chemical composition and the structural peculiarities on atomic scale.

In detail, respective results gained from the following quantum structures will be presented:

- (i) Ga(Sb,As) QDs grown by metalorganic chemical vapour deposition (MOCVD) on GaAs substrates,
- (ii) (Si,Ge) islands grown by liquid phase epitaxy (LPE) on Si substrates

The possibilities and limitations of different program packages for the qHRTEM will be demonstrated.

Electron diffraction and microscopy investigations of nanocrystals envelope

Piotr A. Dłużewski¹⁾, Elzbieta Czerwos^{2,3)}

11:30 - 12:00

invited oral

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

2) *University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland*

3) *Industrial Institute of Electronics (PIE), Długa, Warsaw 00-241, Poland*

Metallic nanocrystals are interesting subject for investigations due to their application in catalysis, cold emitters, self-assembled systems etc. Nanocrystals are difficult for studies with classical crystallographic methods. Transmission electron microscopy and diffraction are widely used to study structure of such objects.

Metallic nanocrystals of size of few or few tens of nanometers give characteristic electron diffraction patterns. In the case of Ni and Pt nanoparticles 10-20 nm in size electron diffraction pattern rings characteristic for fcc structure as well as additional weak and sharp rings, which can be indexed as forbidden reflections are observed. These rings can originate from outer shell formed around metallic core. Computer simulations of electron diffraction patterns for different structural model of the core and oxide or carbide envelope are presented. Comparison of experimental and calculated results will be performed. Discussion of detail differences for various structural models concerning core and shell construction will be also done.

12:00 - 12:30

Soft X-Ray Spectroscopy of Clusters, Surfaces, and Interfaces

Wilfried Wurth¹⁾

invited oral

1) *Institute for Experimental Physics, University of Hamburg, Hamburg, Germany*

Modern, third generation, synchrotron radiation sources are superior in flux and brilliance compared to second generation sources by many orders of magnitude. Furthermore, they provide soft-x-ray radiation with variable polarization. Hence, new opportunities for soft x-ray spectroscopy have emerged in recent years.

In my talk, I will present some examples from surface and cluster physics where this is illustrated for low-dimensional nanoscale systems. Those examples will include the investigation of magnetic properties of small supported metal clusters, the electron dynamics at surfaces and the early stages of interface formation at semiconductor surfaces.

Finally, I will discuss the new perspectives for soft x-ray spectroscopy resulting from the development of free electron laser sources

12:30 - 14:00

Lunch break

14:00 - 15:30

Afternoon session - Jacek M. Grochowski

Main Building, room 134

14:00 - 14:30

Materials characterization using high flux diffraction.

Wroblewski Thomas¹⁾

invited oral

1) *Hamburger Synchrotronstrahlungslabor HASYLAB, Notkestr. 85, Hamburg D-22603, Germany*

Position resolved structural information requires precise definition of the diffracting volume. One approach to achieve this is the use of micro beams from sources of high brilliance like undulator stations at third generation storage rings. An alternative approach requiring only high flux was realized by the novel MAXIM (Materials X-ray Imaging) method in which the diffracting volume is not defined by the primary beam but from the diffracted radiation. It applies a micro channel plate (MCP) as collimator array between sample and a position sensitive detector (PSD) to suppress crossfire of rays diffracted at different sample locations. This micro diffraction on the secondary side permits the illumination of a large specimen area requiring no high brilliance but only flux. Experiments can, therefore, easily be performed on first and second generation synchrotron radiation sources using radiation from bending magnets. Certain experiments can even be done with sealed X-ray tubes. A modified setup using boron silicate capillaries as collimator array has been used with neutrons.

In contrast to conventional diffraction experiments yielding one diffraction pattern in a scan, a series of images is obtained by a MAXIM scan. These data can be rearranged such that up to one million diffraction patterns are obtained, one for each element of the PSD. Powerful algorithms have been developed to reduce this huge amount of data to few diagrams for the characteristic sample features (composition, strain, texture, etc.) and maps showing their spatial distribution. Methods from other disciplines yielding similar data structure (e.g. remote sensing) have been successfully adapted. The MAXIM method allows all kind of investigations that could be performed with conventional methods and even kinetic experiments like the observation of (re-)crystallization processes.

14:30 - 15:00

Application of positron annihilation techniques in semiconductor studies

Grzegorz P. Karwasz^{1,2)}, **Bogdan Kusz**³⁾, **Roberto S. Brusa**¹⁾, **Antonio Zecca**¹⁾

invited oral

1) *Dipartimento di Fisica, Universita' di Trento, via Sommarive, 14, Trento 38050, Italy*

2) *Institute of Physics Pedagogical University of Stupsk (IF PAP), Arciszewskiego 22B, Stupsk 76-200, Poland*

3) *Instytut Fizyki, Politechnika Gdanska, Narutowicza 11/12, Gdansk 80952, Poland*

New, high intensity, narrow-spot positron beams open new possibilities for solid state spectroscopy. Positron annihilation techniques, being non-destructive, allowing depth profiling down to a few micrometers and detecting open-volume defects (vacancies, dislocations etc.) at single ppm concentrations constitute a valuable and complementary method, compared to other solid-state-physics studies.

We give examples of investigation in the field of semiconductors, performed with experimental techniques available at Trento University: 1) Doppler broadening of the 511 keV annihilation line method studied with a slow-positron beam facility, 2) positron life-time techniques with about 160 ps resolution, 3) Doppler-coincidence method in bulk. Examples of applications are shown with the aim:

- i) to follow the interstitial oxygen atoms dynamics and oxygen precipitates in Czochralski-type silicon at different stages of annealing using Doppler coincidence;
- ii) to monitor the reduced layers and metal nanocrystals in semiconducting glasses
- iii) to follow ageing processes in high-porosity, novel dielectric materials.

In particular:

- i) Lowering of the coincidence parameter in Si samples annealed at 450°C indicates migration of oxygen atoms from defect sites to interstitial positions. This type of annealing is a standard recipe of semiconductor industries, used for "as-grown" samples, in order to remove the electrical activity of oxygen atoms. The recipe was purely phenomenological and positron annihilation studies allow to explain it.
- ii) Tracing the "valence" annihilation S-parameter allows to determine optimum thermal treatments for the desired reduced depth. Glasses are used for electron-multipliers and image-intensifiers.
- iii) Ageing of high-porosity, low- ϵ materials, due to filling of pores by atmospheric gases. The lowering of 3- γ annihilation events indicated reductions of free-volumes inside the material. This processes is reversible.

Diffraction studies of the thickening transition in ideal polymers

Ann E. Terry²⁾, Jamie K. Hobbs¹⁾

15:00 - 15:30

1) *University of Bristol, Tyndall Avenue, Bristol BS8 1TL, United Kingdom*

2) *Dept. of Zoology, University of Oxford, Oxford, United Kingdom*

invited oral

A time resolved X-ray diffraction study following the thickening transition in ultra-long, strictly mono-disperse alkanes - a model polymer system - will be presented. Long chain alkanes crystallize into different integer folded forms, all of which are unstable with respect to the extended chain crystal. It will be shown that the re-organisation that accompanies thickening from one folded form to another results in a contraction of the crystal lattice - crystals with different numbers of folds have a subtly different structure. Prior to this contraction there is an increase in disorder, revealed by an increase in the peak width. In addition, by applying a sinusoidal temperature modulation with varying amplitude and period, on top of the linear heating ramp, the X-ray equivalent of a TMDSC experiment can be performed, utilising the high brilliance, high resolution capabilities of ID11 at the ESRF. This provides direct access to a wide range of additional information, and promises to be a technique of general applicability to polymer processes and transitions. Data relating to the reversible and irreversible parts of the transition between different folded forms will be presented, providing conclusive evidence of a reversible ordering and disordering within each folded crystal phase.

Coffee break

Main Building, Main Hall (Duża aula)

15:30 - 15:50

Afternoon session - continued - Elisabeth Holub-Krappe

Main Building, room 134

15:50 - 17:10

Precise determination of full Grain Size Distribution from diffraction peak profile as a result of kinematical theory of diffraction for polydisperse nano-materials

Roman Pielaszek¹⁾, Witold Lojkowski¹⁾

15:50 - 16:20

oral

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

Size of crystallites strongly influences diffraction peak profiles, especially in range of nanometers. In present work we show that classical methods of determination of the average crystallite size from peak width (Scherrer method) or shape (Warren-Averbach method) are accurate only for monodisperse powders. Systematical deviations of measured grain size in polydisperse (real) case ranges between few tens percent and infinity.

In general case of a polycrystals having (wide) Grain Size Distribution (GSD), traditional powder diffraction theory based on interference equation of kinematical theory of diffraction is imprecise (contains a number of approximations). We show new approach (based on Debye equation as a starting point) that allowed derivation of exact

formula for a peak profile for polycrystalline powder with Grain Size Distribution. The formula is entirely analytical and mathematically precise.

This formula was then transformed and gave simple method of determination of full Grain Size Distribution of a crystalline powder. To determine GSD one has to measure peak widths at 1/5 and 4/5 of its maximum (FW1/5M and FW4/5M). Simple analytical expression evaluates directly average $\langle R \rangle$ and dispersion σ of the Grain Size Distribution as a function of measured peak widths FW1/5M and FW4/5M.

Examples of evaluation of diffraction data using FW1/5;4/5M method are given.

16:20 - 16:35

oral

Diamond as X-ray Wavelength Standard for Thermal-Expansion Studies Using Synchrotron Sources

Wojciech Paszkowicz⁶⁾, Pawel Piszora³⁾, Roman Minikayev⁴⁾, Jianzhong Jiang²⁾, Michael Knapp¹⁾, Carsten Baetz¹⁾, Rajmund Bacewicz⁵⁾

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2) *Department of Physics, Technical University of Denmark, Building 307, Lyngby DK-2800, Denmark*

3) *Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, Poznań 60-780, Poland*

4) *Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland*

5) *Warsaw University of Technology, Department of Physics, Koszykowa 75, Warsaw 00-662, Poland*

6) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

Synchrotron radiation has an important advantage of high flux on even small samples. In particular, the in-situ or ex-situ high-pressure studies are performed almost entirely at synchrotron beamlines. Users of synchrotron beams are aware of possible wavelength instabilities which may arise during the measurements (illustrated e.g. in [1]). The instabilities are due largely to thermomechanical phenomena at primary-beam monochromators. For many purposes, the instabilities can be neglected. The stability requirements are particularly severe in thermal expansion measurements, as the changes of the lattice constants with temperature are extremely small. In such studies we have noticed that different $a(T)$ runs of the given material differ slightly. Using an internal wavelength standard was expected to reduce this effect. The diamond standard was chosen because of its known small thermal expansivity making that the possible effect, due to impurities or to the defect structure of the standard, on the final result is also small. We used a 99.9% pure diamond powder with single crystal grains of about one micrometer size. The low-temperature XRD measurements were carried out at a powder diffractometer (for construction details see [2]) at the B2 bending-magnet beamline using Debye-Scherrer geometry. The closed-circuit He-cryostat ensured good temperature stability and accuracy. The results of thermal expansion coefficient measured for several materials in the 10-300 K range using the diamond standard are found to be reproducible. Moreover, they exhibit a good agreement with the first-principles theoretical calculations.

References:

1. Cernik, R. J. and Louer, D., *J. Appl. Crystallogr.* 26 (1993) 277.
2. J. Ihringer and A. Koester, *J. Appl. Crystallogr.* 26 (1993) 135.

16:35 - 16:50

oral

Computation of powder diffraction patterns for carbon nanotubes.

Jacek Kołoczek¹⁾, Andrzej Burian¹⁾

1) *University of Silesia, Department of Biophysics and Molecular Physics, Uniwersytecka 4, Katowice 40-007, Poland*

Structural information about carbon nanotubes comes mainly from electron microscopy (EM) and electron diffraction (ED). These techniques proved to be an efficient tool and present knowledge about atomic arrangement in nanotubes, their symmetry and chirality are based on EM and ED studies. In fact EM and ED are the local probes of the structure. On the other hand there is need for characterization of spatial correlation in nanotubes as a whole. Therefore it is desirable to develop a numerical procedure for computation of powder X-ray or neutron diffraction patterns. The results of such modeling could be then compared with the experimental data and agreement between them would be a criterion for rejection or acceptance of the model. Quantitative theory of the kinematical diffraction based on the Cochran, Crick and Vand [1-3] approach developed for the chiral DNA molecules has been adopted to compute the diffraction patterns of carbon nanotubes.

In the present work we develop the direct method, which uses the Debye equation. The Cartesian coordinates of atoms building up the nanotube of given length, diameter and chirality and generated converting a planar graphene sheet into a seamless cylinder. Then all interatomic coordinates are calculated and finally the powder X-ray or neutron diffraction patterns are obtained. The results of such simulations for the zig-zag, armchair and chiral nanotubes of various size are compared with the experimental data collected using synchrotron radiation. The influence of the size on the diffraction patterns is discussed.

[1] X.-D. Fan, L.A. Bursill, *Phil.Mag.* A 72 (1995) 139

[2] A.A. Lucas, V. Bruyninckx, Ph. Lambin, Europhys. Let. 35 (1996) 1471

[3] Ph. Lambin, A.A. Lucas, Phys.Rev.B 56 (1997) 3571

Characterization of the c- BN/ TiC, Ti₃SiC₂, TiN systems by element selective spectroscopy

16:50 - 17:05

Edyta Piskorska³⁾, Krystyna Lawniczak-Jablonska³⁾, Ewa Benko^{1,2)}, Iraida N. Demchenko³⁾, Piotr Klimczyk²⁾, Roman Minikayev³⁾

oral

1) University of Bielsko-Biala, Willowa 2, Bielsko-Biala 43-309, Poland

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Composites based on the cubic boron nitride (c- BN) have recently attracted increasing interest due to the possibility of the synthesis of materials with unique properties such as super-hardness, combined high hardness and low friction, high corrosion resistance, attractive thermal and electrical properties. There is an increasing number of composites based on the c- BN matrix with addition of Ti compounds such as TiC, Ti₃SiC₂, TiN. In order to understand the origin of the exceptional properties of these materials it is necessary to have a good comprehension of the multiphase composition and microstructure of these materials. The lack of this knowledge is result of difficulties in characterization of the multiphase materials using conventional methods.

In the presented paper we used element selective spectroscopies for estimation of the content of different compounds of Ti formed during the technological processes in composites. The X-Ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES), and X-Ray Absorption Spectroscopy (XAS) was chosen to estimate the amount and kind of formed compound. Additionally the X-Ray diffraction measurements were performed on the same samples. Due to the existence of many compounds with not very different crystal structure the quantitative phase analysis of diffraction pattern was not possible. The analyses of element selective spectroscopies data proves the existence of several Ti compounds (TiC, TiB₂, TiSi₂, TiN) and allowed to evaluate the percent of Ti bonded in each of compound, therefore are the perfect tools for the investigation of micro-chemical mechanism taking place in composites.

Acknowledgements

This work was supported in part within European Community program ICA1-CT-2000-70018 (Centre of Excellence CELDIS)

Wednesday, September 17th

Morning session conducted by A. Kvick

11:00 - 12:30

Main Building, room 134

Neutron and synchrotron radiation non-destructive methods for the characterisation of materials for different applications

11:00 - 11:30

FRANCO RUSTICHELLI¹⁾

invited oral

1) Univ. Politecnica delle Marche, Istituto di Scienze Fisiche (UNIVPM), Via P. Ranieri 65, Ancona I-60131, Italy

Neutron and synchrotron radiation techniques are very powerful non-destructive methods for the characterisation of a wide variety of materials.

In particular, neutron and synchrotron radiation diffraction is nowadays widely used for the evaluation of residual stresses induced by thermal and mechanical treatments in materials and components for industrial applications.

By Small-Angle Neutron Scattering (SANS), microstructural features induced by thermomechanical treatments, such as precipitation and cavitation, can be investigated from a quantitative point of view (determination of size distributions, volume fraction and so on).

Novel high-energy X-ray imaging, such as phase-contrast radiography, allows to obtain information on structural details of biological matter and other kind of materials, with much better definition than ordinary absorption radiography.

In this work the techniques mentioned above will be described, and some applications to materials for technological applications will be presented.

Microstructure of high temperature - pressure treated nitrogen doped Si determined by TEM, PL and X-Ray methods

11:30 - 11:45

Deren Yang³⁾, Jin Xu³⁾, Andrzej Misiuk²⁾, Barbara Surma^{2,4)}, J Bak-Misiuk¹⁾

oral

- 1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*
 2) *Institute of Electron Technology, al. Lotnikow 32/46, Warsaw 02-668, Poland*
 3) *Zhejiang University, State Key Lab of Silicon Materials (ZJU), Zhe Da Lu 38, Hangzhou 310027, China*
 4) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*

Oxygen as the main impurity has the multiple influence on silicon crystals. It has been confirmed that nitrogen can enhance the formation of oxygen precipitates while it suppress the formation of voids. It has been reported that external stress influences the formation of oxygen precipitates. But few results related to oxygen precipitates generated under high pressures (HP) at high temperatures (HT) were reported.

In this paper, we investigate microstructure of nitrogen doped Czochralski silicon (NCZ) treated under 1.2 GPa at 1000 - 1570 K by means of PL, TEM, FTIR and X-Ray methods.

In NCZ and CZ-Si treated at 1230 K under 1 GPa, the strong D1 dislocation-related PL peak at about 0.81eV was observed. After the treatment at 1400K under 1 GPa, the D1 as well as D4 lines were detected in the NCZ silicon, of the higher intensity if compared with the case of CZ-Si treated at the same conditions. PL at 1.1 eV related to interband transition was of higher intensity in NCZ.

11:45 - 12:00

oral

Complete description of structure features of textured metal materials by use of modified X-ray methods and computer data treatment

Yuriy Perlovich¹⁾, Margarita Isaenkova¹⁾, Vladimir Fesenko¹⁾

1) *Moscow Engineering Physics Institute (MEPhI), Kashirskoe shosse, Moscow 115409, Russian Federation*

In the course of technological treatment most metal materials acquire a crystallographic texture, accompanied by the sharp substructure nonuniformity. Grains with different orientations differ in their prehistories, including operated plastic deformation mechanisms and resulting trajectories in the orientational space. The quantitative characterization of inhomogeneous materials ought to base on the distributions of structure parameters instead of their solitary values, relating to grains only with definite orientations. Combining of the diffractometric texture measurement with the registration of profile for the same X-ray line by all successive positions of the sample allows to obtain separate data on the structure of grains with all possible orientations. This results in a multiple increase of accessible information on the structure of studied material and became possible due to the automation of X-ray measurements and computer data treatment. The necessary techniques were developed in details as applied to various metal materials. For grains of each orientation the measured diffraction parameters and calculated substructure parameters are determined. Distributions of these parameters in the stereographic projection (Generalized Pole Figures) are constructed. Besides, distributions of volume fractions of grains, characterized by different values of diffraction and substructure parameters, are constructed as well. The presented experimental results give the most complete, statistically significant picture of substructure conditions in studied material. All substructure parameters of material, calculated on the basis of X-ray data, vary within wide limits, but at the same time obey the quite definite regularities and show the evident mutual correlation. Several general regularities, controlling the development of nonuniform substructure in textured materials, were found for the first time.

12:00 - 12:15

oral

The modern techniques used in the Institute of Nonferrous Metals for manufacturing of metallic materials of specific structure and properties.

Mieczysław Woch¹⁾, Ludwik Ciura¹⁾, Wiesław Kazana¹⁾, Stanisław Księżarek¹⁾, Roman Kolano¹⁾, Juliusz Senderski¹⁾, Tomasz Stuczyński¹⁾, Bartłomiej Płonka¹⁾

1) *Institute of Nonferrous Metals, Sowińskiego 5, Gliwice 44-100, Poland*

The experiences, characteristics and use of such processes and techniques as rapid solidification, thixocasting, equal channel angular pressing (ECAP), drawing and protection of clad wires have been presented in the aspect of forming of metallic materials characterised by specific structure and properties. The equipment for the mentioned above techniques was designed and made in the Institute of Nonferrous Metals (INM) and allows the Institute to develop its research into the new materials of high usability for industrial application.

Rapid solidification method and melt spinning process are used in INM for production of amorphous and nanocrystalline soft magnetic Fe-based and Co-based ribbons. During fully automated melt spinning process molten alloy hits a fast rotating copper wheel and almost instantaneously releases continuous metal ribbons at the room temperature. They are excellent semi-product for magnetic cores production.

Thixocasting is based on ability of an alloy to regain its viscosity when stirring during solidification is used and when formation of nondendritic structure in an ingot takes place. This technology is used in INM for obtaining Al-based alloys as the semi-product for forging of car engine elements.

ECAP is one of the effective severe plastic deformation techniques, which enables to form laminar, fibrous and fine-grained structure. This method is developed in INM for Al-based alloys for automotive industry. The angular channel is mounted on the 60 T press. Multiple extrusion makes possible to reach grain size below 1 micrometer. Drawing of clad wires is a special technology in which the steel core is drawn together with the Cu outer layer, leading to formation of bimetallic wire applicable for conductive connectors of electronic elements inside the glass and for telecommunication networks. The pilot production line includes also the equipment for modification and protection of the wire surface by oxidation, borax or nickel coating.

Industrial Research and Development with Synchrotron Radiation
Wroblewski Thomas¹⁾, Servicegroup Industry

12:15 - 12:30

1) Hamburger Synchrotronstrahlungslabor HASYLAB, Notkestr. 85, Hamburg D-22603, Germany

oral

The outstanding properties of synchrotron radiation (SR) allow manifold investigations of materials and processes which are not possible with conventional X-ray sources. The high intensity of SR allows extremely precise or time/spatial resolved measurements. In combination with its high collimation extremely high angular resolution is achievable. Typical applications of these properties are high resolution powder diffraction, small angle scattering and the investigation of surfaces.

The white spectrum of SR allows free choice or tuning of the X-ray energy. This property is used in X-ray absorption spectroscopy (XAS) for the determination of the short range atomic order allowing also the structural investigation of amorphous or nanocrystalline substances. Free choice of the wavelength further allows contrast enhancement by anomalous scattering yielding element specific information. The low absorption of high energy SR enables the investigation of bulk properties.

Typical examples of applied research with SR are the (in situ) investigation of catalysts by XAS, small angle scattering and diffraction and the determination of strain and texture in materials arising during their production, processing and use.

Lunch break

12:30 - 14:00

Afternoon session conducted by J. Ghijsen

14:00 - 15:45

 Main Building, room 134

Microstructure Development in 3D studied by Synchrotron X Rays
Larry Margulies^{1,2)}, Henning F. Poulsen²⁾, Erik M. Lauridsen²⁾, Soeren S. Schmidt²⁾, Grethe Winther²⁾, Dorte Juul Jensen²⁾

14:00 - 14:30

invited oral

1) European Synchrotron Radiation Facility (ESRF), Grenoble BP220-38043, France
2) Riso National Laboratory, Materials Research Department, Roskilde, Denmark

The 3-Dimensional X-Ray Diffraction (3DXRD) Microscope at beamline ID11 of the ESRF is a unique instrument for studies of polycrystalline materials. Combining the use of high energy x-rays with a tomographic approach to acquisition of diffraction data, it enables a 3D structural characterization within mm-cm size specimens and on a scale of 0.3-5 microns. The individual grains and sub-grains can be characterized with respect to their position, shape, phase, crystallographic orientation, elastic and plastic strain.

The study of the dynamics of microstructure development during the deformation and recrystallization of polycrystalline materials has been severely hindered by the general inability to non-destructively collect information on individual interior grains during the deformation and annealing processes. Although theoretical models exist which successfully predict overall properties such as the average texture development and flow stress evolution, there has been to date no method for experimentally verifying the model predictions on the level of an individual grain. Data sets on the order of hundreds of grains have been collected for both the dynamics of grain rotation during plastic deformation and the kinetics of nucleation and growth during recrystallization. In both cases it has been clearly demonstrated that the behavior of individual grains does not follow classical models for average grains. The basic principle of the 3DXRD methodology will be presented, and results in the area of plastic deformation and recrystallization will be presented.

Gallium nitride surface formation and modification by Mn deposition - photoemission studies with use of synchrotron radiation

14:30 - 15:00

Bogdan J. Kowalski¹⁾, Iwona A. Kowalik¹⁾, Ryszard J. Iwanowski¹⁾, Bronislaw A. Orlowski¹⁾, Elzbieta Lusakowska¹⁾, Janusz Sadowski^{1,4,5)}, Robert L. Johnson³⁾, Izabella Grzegory²⁾, Sylwester Porowski²⁾

invited oral

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland
2) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland
3) Institute for Experimental Physics, University of Hamburg, Hamburg, Germany
4) University of Copenhagen, Universitetsparken 5, Copenhagen DK-2100, Denmark
5) Chalmers University of Technology, Goeteborg S-412 96, Sweden

Gallium nitride (apart from its optoelectronic applications) is a system particularly interesting from the point of view of surface physics research. Its surfaces may reconstruct in various ways depending on formation conditions (surface polarity, preparation method, and presence of impurities). Moreover, doping with transition metals may transform GaN into a ferromagnetic semiconductor. Annealing of Mn/GaN is considered as a technique suitable

for fabrication of this material.

In this paper we present the results of photoemission studies of electronic structure of a GaN surface, clean and modified by deposition of manganese. Advantages of two experimental techniques based on application of synchrotron radiation manifest themselves in this report.

Details of electronic band structure of clean (000-1)-(1x1) surface of GaN bulk crystal were studied by means of angle-resolved photoelectron spectroscopy. The acquired results indicated that some part of the surface corresponds to the GaN (000-1):Ga configuration, covered with additional layer of Ga atoms bound at the "on top" positions above N atoms. However, substantially large regions have the relaxed clean GaN (000-1) configuration with a characteristic, almost dispersionless surface band occurring at the energy of the valence band maximum.

Mn/GaN interface formation on GaN(000-1)-(1x1) surface was investigated by resonant photoemission spectroscopy, as a function of Mn-coverage. A contribution of Mn 3d states to the valence band of the system was derived from photoemission spectra taken for photon energies near to Mn 3p-3d excitation. Interaction between Mn and GaN was also monitored by spectroscopy of Ga 3d core level. The results show that Mn on GaN forms a reactive interface. Moreover, annealing of Mn/GaN at 400°C makes the Mn 3d states distribution similar to that characteristic of Mn built into tetrahedrally coordinated semimagnetic semiconductors. Possible Mn atom environment will be discussed.

15:00 - 15:15

oral

Quantitative transmission electron microscopy investigation of localised stress in heterostructures

Slawomir Kret¹⁾, Paweł Dłużewski²⁾, Grzegorz Maciejewski²⁾, Grzegorz Jurczak²⁾, Pierre Ruterana³⁾, Jun Chen³⁾, Piotr Dłużewski¹⁾, Elżbieta Janik¹⁾

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3) *Laboratoire CRISMAT - UMR 6508, ISMRA et Universite de Caen, 6 Boulevard de Marechal JUIN, 14050 Caen Cedex, France*

We present methods and results of investigation of the local strain and stress at atomic scale in highly stressed GaN/Sapphire and GaAs/CdTe buffer layers.

Grow of the layer with big mismatch up to 15 % of lattice parameter generate huge density of defects. Understanding of the relaxation processes are important for optimisation of technology of growth of the buffer layers used as substrate for quantum structures.

The defect density can be reduced by technological parameters directed to amplification of different relaxation processes occurred at atomic scale as dislocation annihilation, bending of dislocation line to heterointerface, creation of misfit dislocation network at this interface etc. All this mechanisms are dynamic and depends on the dislocation movement in stress field generated by other defects.

So, for understanding and for creation a theoretical description of such processes, it is necessary to obtain quantitative information about 3d distribution of stress fields around isolated and interacted defects.

We propose an approach, which starts from high resolution transmission electron microscopy images taken in zone axis, where defect's Burgers vector components are not zero. The two dimensional strain fields are extracted by image processing using Geometric Phase method, which allows precisely measurement of lattice fringes shift in deformed zone in relation to no deformed lattice.

The experimental lattice distortion data are read by finite element (FE) program into nodes of the three dimensional mesh as initial solution. Boundary condition corresponding to bulk as well as to TEM specimen geometry was applied. After several FE iterations the stress field around dislocation core was determined in atomic scale in three dimensions. In FE calculations the anisotropy and non-linear elasticity were taken into account.

The validity of our procedure was checked by treatment of simulated images obtained from model of dislocation with known distortions.

15:15 - 15:30

oral

Study of GdSi_{2-x} formation in the presence of an interfacial SiO₂ layer

Kwun-Bum Chung^{1,2)}, Y. K. Choi¹⁾, M. H. Jang¹⁾, SeongJun Kang¹⁾, D. S. Park¹⁾, M. Noh¹⁾, C. N. Whang¹⁾

1) *Yonsei University (Yonsei UNIV.), Sinchon-dong, Sudaemoon-ku, Seoul 120-749, Korea, Republic of*

2) *Atomic-scale Surface Science Research Center (ASSRC), Sinchon-dong, Sudaemoon-ku, Seoul 120-749, Korea, Republic of*

Gadolinium silicide films were grown in ultrahigh vacuum (UHV) on a Si(111) substrate through an interfacial SiO₂ layer. In order to examine the role of an interfacial SiO₂ layer during the silicide formation, we used in-situ reflection of high energy electron diffraction, x-ray diffraction, x-ray photoelectron spectroscopy, and high resolution transmission electron microscopy. The structural transformation was observed from the initial GdSi_{1.7} hexagonal phase to GdSi₂ orthorhombic phase as the post-annealing temperature increase. Our experimental results implied that an interfacial SiO₂ layer decomposed and the reaction between Gd and Si was enhanced. We conclude that the competitive reaction among Gd, Si, and its oxides determine the reaction pathway in gadolinium silicide formation.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duża aula)

Poster session

19:00 - 21:00

Thursday, September 18th

Morning session - Piotr Dłużewski

09:00 - 10:30

Main Building, room 134

Surfaces and interfaces characterisation by neutron reflectometry.

9:00 - 9:30

Frederic OTT¹⁾

1) Lab. Léon Brillouin CEA/CNRS (LLB), CEA Saclay, Gif sur Yvette 91191, France, Metropolitan

invited oral

Neutron reflectivity allows to characterise surfaces and interfaces of ultra thin film layered systems down to a nanometric scale (~2 nm). It is especially a powerful tool for the study of magnetic and polymer thin film structures. The neutron magnetic interaction is very large and makes polarised neutron reflectivity a very sensitive tool for probing magnetic multilayers. It allows the determination of magnetic ordering and coupling in new artificial magnetic multi-layers (either metallic, semi-conducting or oxides). Examples of studies in the field of GMR (Giant Magneto Resistive) sensors used in hard drive and tape read heads will be given. The search for efficient spin-injection materials which could be used in spin electronics is also a growing field of activity. Several material candidates are presently evaluated. Recent studies on such materials will be presented : oxide materials (Fe₂O₃ - Fe₃O₄) epitaxial thin films ; multilayer systems involving semiconducting materials ([Fe/Si]_n, [GaMnAs/GaAs]). Besides the high sensitivity of neutrons to magnetism, the possibility of isotopic labelling (H/D substitution) offers a way to probe polymer and protein thin film structures with great details : polymer interdiffusion or polymer grafting can be studied at the substrate/polymer or liquid/polymer interfaces. A model study of PMMA polymer grafting will be presented.

Magnetic and crystalline microstructure of nanocrystalline metals studied by neutron and synchrotron radiation scattering

9:30 - 10:00

Radosław Przeniosło¹⁾, Rolf Hempelmann²⁾, Izabela Sosnowska¹⁾

invited oral

1) Warsaw University, Institute of Experimental Physics, Hoża 69, Warsaw, Poland

2) Institute of Physical Chemistry, University of Saarbruecken, Saarbruecken 66123, Germany

The crystalline and magnetic microstructure of nanocrystalline Ni (n-Ni), Co (n-Co) and Cr (n-Cr) have been studied by means of X-ray and neutron diffraction as well as small angle neutron scattering (SANS) and small angle X-ray scattering (SAXS). The nanocrystalline samples obtained by electrodeposition have crystallite sizes between 10 nm and 60 nm and relative densities of the order of 95%. SANS and SAXS studies show that the crystalline microstructure has fractal-like density correlations with coherence lengths exceeding the limitations of the SAS techniques, i.e. above 100 nm. The magnetic ordering of n-Ni and n-Co at RT is made of ferromagnetic domains with sizes exceeding a single crystallite. The magnetic ordering of n-Cr and polycrystalline Cr (poly-Cr) has been studied with powder neutron diffraction at temperatures between 2 K and 300 K. The magnetic ordering of n-Cr is antiferromagnetic between RT and 240 K. At 240 K the magnetic ordering changes and a part of the material adopts a transverse spin density wave (TSDW) ordering. The spin-flip transition from the TSDW to the longitudinal LDSW ordering is suppressed and about 40 % of the volume has the TSDW ordering stabilized down to 2 K. The thermal lattice expansion of the material becomes negative around the magnetic phase transition near 240 K. The values of the magnetic moments of the simple antiferromagnetic and TSDW orderings observed at 2K are in agreement with the magnetic moments observed in single crystals and poly-Cr.

Atomic structure of nanoparticles: X-ray diffraction and XAFS studies

10:00 - 10:30

Yuri L. Slovokhotov¹⁾

1) Nesmeyanov Institute of Organoelement Compounds RAS (INEOS), 28 Vavilov, Moscow 119991, Russian Federation

invited oral

Structural studies of nanocluster materials recently carried out in INEOS, are reviewed. Metal nanoclusters in polymer matrices (Co, Pt, Pt/Pd, etc.), as well as Pd blacks formed by oxidative aggregation of stoichiometric Pd clusters in organic media and binary nanoparticles (MoS₂ composites obtained via monolayer dispersions, coated nanocrystallites of TiO₂ anatase) were characterized. Laboratory and synchrotron X-ray diffraction data, together with local structural parameters taken from XAFS spectroscopy and the results of XRD simulation using model

atomic arrangements, revealed type and distortions of atomic packing. Published single crystal X-ray structural data for big stoichiometric clusters extracted from Cambridge Structure Database (CSD) were represented as new 2D polar histograms and used as references.

Finite size, ligand shell, and electronic effects can modify geometric and electronic structure of nanometric atomic aggregates. Structural monitoring of chemical transformations discussed in the talk include distortion of MoS_2^{x-} hexagonal layers in intercalated nanoparticles due to partial retention of a negative charge, as well as air oxidation of small stoichiometric palladium clusters in toluene and large mixed-metal stoichiometric cluster $[\text{Ni}_{132}\text{Au}_6(\text{CO})_{44}]^{6-}$ in acetonitrile. Larger (ca. 13 nm), and chemically stable TiO_2 nanocrystallites show local distortions of Ti coordination due to coating by photosensibilizing agents, which may facilitate electron transfer in solar cell prototypes. Structural transformations in chemical processed that produce nanoparticles open a way to nanocluster materials with designed bulk properties.

10:30 - 11:00

Coffee break

Main Building, Main Hall (Duža aula)

11:00 - 12:45

Morning session - continued - Bogdan J. Kowalski

Main Building, room 134

11:00 - 11:30

**Spectroscopic investigation of buried interfaces and liquids with soft x-rays
Clemens Heske¹⁾**

invited oral

1) Experimentelle Physik II, Universitaet Wuerzburg, Am Hubland, Wuerzburg 97074, Germany

When, in general, two entities interact, they do it by forming an interface. The properties of such interfaces are determined not only by the properties of the two interface partners, but also to a large degree by the peculiarities of the interface formation process itself. This is of particular importance in solid state devices composed of two or more different materials. Unfortunately, the investigation of such interfaces is very difficult for two reasons. First they are, by their nature, buried. Secondly, interfaces generally form a thin layer within a larger ensemble and thus give very weak signals. Nevertheless, a few experimental techniques are available to study such buried interfaces. It will be the purpose of this presentation to demonstrate that a combination of soft x-ray spectroscopies (x-ray emission, photoemission, and x-ray absorption) is extremely well suited for this task. As examples, the electronic and chemical properties of several interface systems will be discussed, including II-VI-semiconductors, $\text{Cu}(\text{In,Ga})(\text{S,Se})_2$ thin film solar cells, organic thin films, and liquid-solid interfaces.

11:30 - 11:45

The transition to perpendicular magnetization in an ultrathin Co film studied by XMCD and SXRR based magnetometry

oral

Elisabeth Holub-Krappe³⁾, Anders Hahlin¹⁾, Hansjörg Maletta³⁾, Cecilia Andersson¹⁾, Olof Karis¹⁾, Jonathan Hunter Dunn²⁾, Dimitri Arvanitis¹⁾*1) Uppsala University (UU), Regementsvägen 1, Uppsala SE-751 21, Sweden**2) MAX-lab, Lund University, Lund SE-221 00, Sweden**3) Hahn-Meitner-Institut, Berlin (HMI), Glienicke Str. 100, Berlin D - 14 109, Germany*

Ultrathin magnetic films are often used as a model system for investigating low dimensional and interfacial effects. Both, surface and volume anisotropy can favor in-plane or out-of-plane magnetization. The easy axis of magnetization can change from in-plane to out-of-plane when decreasing the thickness of the magnetic layer at a fixed temperature, or upon decreasing the temperature at a fixed layer thickness. A prominent example for such a Spin Reorientation Transition (SRT) is an ultrathin Co film, sandwiched between Au layers. Our previous work showed the SRT at the room temperature with decreasing the Au-cap thickness, linked with an 38% enhancement of the Co orbital moment and the hybridization of the Co 3d and Au 5d bands, resulting in a charge transfer from Co to Au.

Present study shows the evolution of the SRT with decreasing temperature, giving evidence for the influence of the Au/Co interface versus the interior of the Co film in the SRT process. X-ray magnetic circular dichroism (XMCD) and soft x-ray resonant reflectivity (SXRR) measurements were performed at MAX-lab, Lund on a Au/Co/Au trilayer. XMCD experiments provide quantitatively both the in-plane and out-of-plane magnetic response across the SRT temperature, and assess the functional onset of the magnetization reorientation versus temperature. The temperature dependent spin and orbital moments were determined by means of the magneto-optical sum rules. A set of element-specific hysteresis loops based on the SXRR signal allows us to quantify the onset of the SRT at the photon penetration depth. On the other hand, the XMCD spectral areas are used to enhance the interface contrast. Comparison of the data from both methods indicates for the first time that with decreasing temperature from the in-plane phase the Co at the Co/Au interface exhibits the spin-reorientation at higher temperatures than the interior of the Co film, i.e. the SRT starts at the interface.

11:45 - 12:00

NMR study of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ compounds

oral

Czesław Kapusta²⁾, Dariusz A. Zajac²⁾, Peter C. Riedi³⁾, Damian Rybicki²⁾, Marcin Sikora²⁾, Colin J. Oates²⁾, Jose M. De Teresa¹⁾, Clara Marquina¹⁾, Ricardo M. Ibarra¹⁾

1) *Universidad de Zaragoza-CSIC, Facultad de Ciencias, Pedro Cerbuna 12, Zaragoza 50009, Spain*

2) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

3) *School of Physics and Astronomy, University of St. Andrews, St. Andrews KY16 9SS, United Kingdom*

We report on the nuclear magnetic resonance (NMR) study of "colossal magnetoresistive" manganites $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$. The temperature measurement of zero field ^{55}Mn and ^{139}La spin-echo spectra were carried out on samples with doping $x=0.125$ and 0.15 . Previous works on these compounds suggest that the ground state of both compounds is a ferromagnetic conductor below T_C and orbitally ordered ferromagnetic insulator below $T_{OO} < T_C$. Neutron diffraction, magnetization, resistivity and linear thermal expansion measurements on our polycrystalline samples indicate T_C around 205 K and 240 K, for $x=0.125$ and $x=0.15$, respectively. The temperature of orbital ordering, T_{OO} , is 150 K and 180 K for $x=0.125$ and $x=0.15$, respectively. The ^{55}Mn spectra of both compounds exhibits Mn^{3+} and Mn^{4+} as well as double exchange (DE) lines with different relative intensities. The DE line corresponds to ferromagnetic metallic clusters (FMM) and the Mn^{3+} and Mn^{4+} lines are due to charge localised (CL) ferromagnetic insulating (FMI) regions. The DE line for $x=0.125$ is observed up to temperatures around or slightly above bulk T_C , revealing the presence of long lived FMM clusters. The intensity of the Mn^{4+} line decreases strongly with increasing temperature and the signal is not observed above 150 K due to the melting of FMI regions. The ^{139}La spectra of both compounds at 4.2K exhibit two lines, which are attributed to FMM and FMI regions and the ratio of their intensities is similar to that of the respective FMM and FMI signals in the ^{55}Mn spectra. This ratio shows that there are fewer FMM clusters in $x=0.125$ than in $x=0.15$. Measurements on the ^{55}Mn DE line at 4.2 K, for different pulse spacing, shows no effect of the Suhl-Nakamura interaction for $x=0.125$, in contrast to $x=0.15$. This feature indicates that the FMM clusters for $x=0.125$ are of nanometer size, whereas for $x=0.15$ they are much larger.

SAXS studies on materials inhomogeneous in nanoscale

Bożena Bierska¹⁾, Eugeniusz Lagiewka¹⁾, Lucjan Pajak¹⁾

12:00 - 12:15

1) *Silesian University, Institute of Physics and Chemistry of Metals, Bankowa 12, Katowice 40-007, Poland*

oral

Small Angle X-Ray Scattering (SAXS) method appeared to be very useful in the structure characterisation of materials which are inhomogeneous in nanoscale. Some possible applications of this method are shown. When the scattering regions (particles) are approximately of the same shape but different in size (like spherical Guinier-Preston zones in Al-Ag alloy) then size distribution of scattering particles can be determined. On the other hand the structure of most porous materials is quite complex and disordered. When the structure of scattering regions has the property of self-similarity then fractal geometry can be applied for the description of this structure. Such cases were observed for different type of dry gels (aerogels, xerogels), carbonaceous materials, electrodeposited materials. This work is financially supported by State Committee for Scientific Research (grant PBZ/KBN 013/T08/10)

RF-Mossbauer study of the magnetic properties of nanocrystalline alloys

Michał Kopcewicz¹⁾

12:15 - 12:30

1) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*

oral

The current status of the Mossbauer investigations of the soft magnetic nanocrystalline alloys produced by utilizing the first step of crystallization of amorphous alloys is discussed. Conventional Mossbauer measurements allow the identification of phases formed due to annealing of amorphous precursor and the evaluation of their relative content. Unconventional rf-Mossbauer studies utilizing radio-frequency (rf) fields provide information on the soft magnetic nature of the alloys by observing the degree of rf-induced collapse of the hyperfine fields. The Mossbauer experiment in which the rf collapse and rf sidebands effects are used allows the soft nanocrystalline bcc phase to be distinguished from magnetically harder microcrystalline ones.

Qualitative information concerning the distribution of anisotropy fields in the nanocrystalline grains can be inferred from the dependence of the rf collapsed spectra on the rf field intensity. The rf-sidebands effect reveals changes of magnetostriction due to the formation of the nanocrystalline phase. The rf-Mossbauer technique provides a unique opportunity to study the microstructure and magnetic properties of each phase formed in the amorphous precursor. The principles of the rf-Mossbauer technique and examples of its application will be discussed for FeZrB(Cu) and FeNiZrB nanocrystalline alloys.

Correlation between the dielectric constant and porosity of nanoporous silica based thin films

12:30 - 12:45

Ilanit Fisher¹⁾, Wayne D. Kaplan²⁾, Moshe Eizenberg³⁾

oral

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Low dielectric constant silica based films which incorporate a large amount of nanometer sized pores are attractive candidates as interlayer dielectrics in future gigascale integrated circuits chip technology.

Nanoporous silica based films were deposited by surfactant templated self-assembly spin-on deposition (SOD). Other low-k materials with relatively low density silica based films were deposited by plasma enhanced chemical vapor deposition (PECVD), and some silica films were deposited by a CVD process. The SOD films showed higher porosity compared with the PECVD and CVD films, as measured by x-ray reflectivity, Rutherford back scattering, and ellipsometry measurements. These SOD films had lower dielectric constant compared with the PECVD and CVD films, as derived from electrical and optical measurements.

Most commonly, a porous silica thin film is modeled as a parallel or serial connection of two capacitors, one with air and the other with SiO₂ as the dielectric medium. Our results of the correlation between the dielectric constant and the porosity for the SOD films fit well the Lorentz-Lorenz model, which is close to the lower limit serial model. For the PECVD and CVD films they agree with the Rayleigh model, which is close to the upper limit parallel model. These results suggest that the dielectric constant of the nonhomogeneous two phase (silica and air) nanoporous silica based films deposited by SOD technique is significantly lowered by forming closed air voids. The silica based films deposited by PECVD and CVD, consist of a homogeneous low density loose structure originating from the bonding nature, i.e., Si-CH₃ bonds and the six-fold rings of the Si-O bond, and therefore their dielectric constant is lowered to a smaller extent.

12:45 - 13:00

Closing remarks - Jacek M. GrochowskiMain Building, room 134

13:00 - 14:30

Lunch break

14:30 - 15:30

Afternoon session - Bronislaw A. Orlowski

Main Building, room 134

General Meeting of Polish Synchrotron Radiation Society

15:30 - 15:50

Coffee break

15:50 - 17:00

Afternoon session - continued - Bronislaw A. Orlowski

Main Building, room 134

General Meeting of Polish Synchrotron Radiation Society

Symposium C

5th International Workshop on Molecular Beam Epitaxy and Vapour Phase Epitaxy Growth Physics and Technology

Organisers

- **Jacek Kossut** - Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Detlef Hommel** - University of Bremen, Bremen, Germany
- **Zbigniew R. Zytkiewicz** - Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

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- Center of Excellence CEPHONA (European Commission Contract G5MA-CT-2002-04061)

Proceedings

Papers will be published in Physica Status Solidi.

Monday, September 15th

13:55 - 14:00

Opening address - J. Kossut

Main Building, room 213

14:00 - 15:30

Afternoon session

Main Building, room 213

14:00 - 14:45

MBE growth of ZnO layers and their physical properties

Christian Morhain¹⁾, Stéphane Vézian, Christiane Deparis, Monique Teisseire, Philippe Lorenzini, Phillipe Vénnegues, Frédéric Raymond

invited oral

1) CRHEA-CNRS, Park de Sophia Antipolis, Valbonne 06560, France

ZnO based heterostructures have potential interests both in the field of optoelectronics, where the very large binding energy of the excitons in ZnO should give rise to excitonic phenomena preserved well above room temperature, as well as in the field of spin electronics, where n- and p-type magnetic alloys are expected to present a ferromagnetic phase with a high curie temperature.

Since the first reports of MBE growth of single-crystalline ZnO epilayers in 1997, strong improvements in terms of crystal quality and morphology have been made. This first step make the intrinsic and extrinsic properties of this semiconductor more accessible than they used to be. Investigations on quantum heterostructures, alloys and doping are being carried out since the last 2-3 years.

In this talk, I will review these various achievements, underlining the following key issues : (i) strategy used for improved crystal and optical qualities, (ii) control of the epilayer polarity by using GaN buffer layers, (iii) residual and p-type doping and (iv) growth and properties of non magnetic and magnetic alloys.

14:45 - 15:30

Quantum dot formation induced by surface energy change of a strained two-dimensional layer

Henri Marriete¹⁾

invited oral

1) CEA-CNRS group Nanophysique et Semiconducteurs Laboratoire de Spectrometrie Physique, Universite J. Fourier-Grenoble I, and departement de Recherche Fondamentale sur la Matiere Condensee/SP2M, CEA-Grenoble, 17 avenue des Martyrs, France, France

Some combinations of lattice-mismatched semiconductors can exhibit, under specific growth conditions, a sharp transition from a layer-by-layer growth to the formation of islands. This is the Stranski-Krastanow (SK) growth mode which allows the relaxation of highly strained 2D layers through the free surfaces of 3D islands instead of generating misfit dislocations. These islands are expected to be dislocation free and thus of high structural quality. Usually their typical sizes are on the scale of a few nanometers, so that these self-assembled quantum dots (QDs) are attractive nanostructures for both fundamental physics (interplays of quantum confinement effects, Coulomb blockade effects, etc...) and device applications (QD lasers with higher gain, temperature-insensitive laser threshold, etc...).

To account for the occurrence (or not) of this SK transition during the heteroepitaxial growth, we present a simple equilibrium model taking into account not only the lattice mismatch, but also the dislocation formation energy and the surface energy [1]. It demonstrates the importance of these parameters especially for II-VI systems such as CdTe/ZnTe and CdSe/ZnSe. For II-VIs indeed, as misfit dislocations are easier to form than in III-Vs (such as InAs/GaAs and GaN/AlN) or IV-IV systems (Ge/Si), the 3D elastic transition is short-circuited by the plastic one. Nevertheless, by increasing the 2D surface energy, telluride and selenide quantum dots can also be grown as predicted by our model and as evidenced experimentally by both reflection high-energy electron diffraction (RHEED) and optical measurements[2]. The surface energy variation required to induce the morphology transition is obtained by exposing a given amount of CdTe (CdSe) to an excess of Te (Se) after the growth.

Finally this model has been applied to the low mismatched system GaN/AlN ($\Delta a/a^o = 2.4\%$). In this case the variation of surface energy has been calculated [3] when the GaN growth front is covered (or not) by a Ga-bilayer. It allows us to understand why, when the GaN is grown under an excess of Ga, the SK transition is completely inhibited, whereas in the opposite case, for a N-rich surface, the formation of SK-coherent QDs is the most favorable growth mode [4]

- [1] F. Tinjod, I.-C. Robin, R. Andre, K. Kheng, H. Mariette, invited paper at E-MRS Zakopane 2002. *J. of Alloys and Compounds* (2003)
- [2] F. Tinjod, B. Gilles, S. Moehl, K. Kheng, H. Mariette, *Appl. Phys. Lett.* 82 (2003) 4340 ; *J. Cryst. Growth* 237-239 (2002) 227.
- [3] C.G. Van de Walle et J. Neugebauer, *Phys. Rev. Lett.* 88 (2002) 066103.
- [4] C. Adelman, J. Brault, D. Jalabert, P. Gentil, H. Mariette, G. Mula, B. Daudin, *J. Appl. Phys.* 91 (2002) 9638 ; C. Adelman et al. *Appl. Phys. Lett.* 81 (2002) 3064.

Coffee break

15:30 - 15:50

Afternoon session - continued

15:50 - 17:20

Main Building, room 213

Controlling the growth of InAs/GaAs quantum dots for long wavelength applications

15:50 - 16:35

Tim S. Jones¹⁾

invited oral

1) Centre for Electronic Materials and Devices, Imperial College London, London, SW7 2AZ, UK, United Kingdom

InAs/GaAs self-assembled quantum dots (QDs) grown by molecular beam epitaxy are showing considerable promise for the development of GaAs-based optoelectronic devices for application in the 1300-1550 nm range [1-4]. However the growth of these structures is complex since many parameters influence the emission properties of the structures. In this talk I will review our recent progress in controlling the growth and properties of multilayer QD structures in which the individual QD layers are separated by a spacer layer of well-defined thickness [4-6]. In particular I will show how the spacer layer thickness, strain relaxation and control of the In/Ga intermixing process all play a crucial role in extending the emission to long wavelengths. Scanning tunnelling microscopy (STM), atomic force microscopy (AFM) and transmission electron microscopy (TEM) have all been used to provide quantitative information regarding the QD characteristics (density, size and uniformity) and results will be presented for structures that demonstrate emission up to 1500 nm [4]. Finally I will show how the choice of capping material also plays a strong role in determining the emission properties of the QDs and promising results will be presented for both InGaAs and GaInNAs capping layers.

References

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- [2] P.B. Joyce, T.J. Krzyzewski, G.R. Bell, T.S. Jones, E.C. Le Ru, R. Murray, *Phys. Rev. B* 64 (2001) 235317.
- [3] E.C. Le Ru, U. Marchioni, A. Bennett, P.B. Joyce, T.S. Jones, R. Murray; *Mat. Sci. and Engineering B* 88 (2002) 164.
- [4] E.C. Le Ru, P. Howe, R. Murray, T.S. Jones, *Phys. Rev. B* 67 (2003) 16530.
- [5] P.B. Joyce, T.J. Krzyzewski, P.H. Steans, G.R. Bell, J.H. Neave, T.S. Jones, *J. Cryst. Growth* 244 (2002) 39.
- [6] P.B. Joyce, E.C. Le Ru, T.J. Krzyzewski, G.R. Bell, R. Murray, T.S. Jones, *Phys. Rev. B* 66 (2002) 75316.

MBE growth of HgCdTe layers and their properties in the context of IR applications

16:35 - 17:20

Sergei Dvoretzky¹⁾

invited oral

1) Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation

Today molecular beam epitaxy (MBE) is the modern powerful epitaxial technique for growth of HgCdTe layers and complex heterostructures growth for routine and perspective infrared detectors (IRD's). At present time there are the essential successes in developing and production of MBE equipment, investigations of growth process, mechanisms of defects formation, studying of external doping. There are reported the numerical architectures of IRD's (including unique FPA's of 2042*2042 elements format and multicolor detectors) which fabricated on the basis of HgCdTe layers grown on different substrates (CdZnTe, GaAs, Si ect.) by MBE and operated in wide wavelength range.

We present the original development and production of unique MBE equipment including ultra fast ellipsometer for controlled industrially oriented technology of HgCdTe heterostructures on 4 inches in diameter substrates. The detailed investigations of growth processes and defects formation of buffer CdZnTe layer on GaAs substrate and HgCdTe on CdZnTe/GaAs substrates allow to find the optimal growth condition and substrate orientation for production high quality undoped large areas uniform material basically for IR photoconductors and photodiodes. As-grown HgCdTe layers are low carrier concentration (10^{14} cm⁻³) n-type conductivity. P-type conductivity HgCdTe layers are fabricated by thermal annealing of as-grown materials in inert atmosphere. There are constructed HgCdTe layers with gradient layers at the interface and at the surface to improve material properties for IRD. We demonstrate high quality modern photovoltaic IRD's of 1x574 and 4x288 elements ($\lambda_c=11.0 = 12.0 \mu\text{m}$

at 77K), which fabricated on the basis of HgCdTe/CdZnTe/GaAs heterostructures. The thermal image of 576x768 elements format on the basis of developed FPA's were done.

17:20 - 17:35

Break

17:35 - 18:35

Afternoon session - continued

Main Building, room 213

17:35 - 17:50

oral

The optimisation of the GaN and GaN/AlGaN heterojunctions on bulk crystals using plasma-assisted molecular beam epitaxy

Czesław Skierbiszewski¹⁾, M. Siekacz¹⁾, A. Feduniewicz¹⁾, K. Dybko²⁾, B. Wasilewski³⁾, I. Grzegory¹⁾, M. Leszczynski¹⁾, B. Pastuszka¹⁾

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3) Institute for Microstructural Sciences, National Research Council, Ottawa, Canada, Canada

In this work comparison of the MBE growth of GaN and AlGaN on dislocation free bulk substrates and GaN/sapphire MOCVD templates is presented. The RF plasma source is used as a source of active Nitrogen. In order to achieve 2D growth, the optimisation of the Ga to N ratio was performed. The step flow growth mode was obtained for Ga rich conditions (high Ga/N ratio) [1]. We found that for good quality GaN layers, the total Ga flux consists of two components: a) proportional to the growth rate (equal to the Nitrogen flux), b) constant flux which is desorbed from the surface during growth. The latter one strongly depends on the growth temperature. We will discuss the growth diagram and the conditions where smooth step flow growth mode without formation of the Ga droplets on the surface is achieved.

The AFM and photoluminescence data on MBE grown GaN samples confirms high layer quality. In 1 μm GaN layers grown on bulk substrates we observe very narrow donor bound exciton lines (D^0X) with the half-width of 0.3-0.5 meV. Our results are comparable to the best PL data from GaN layers grown on bulk crystals by Grandjean et al [2] using ammonia MBE. However, in contrast to their work we do not observe acceptor bound exciton (A^0X) in our spectra. This result indicates considerably lower acceptor concentrations in our layers.

We succeed in the growth of high quality of GaN/AlGaN heterojunctions. The growth on the semi-insulating bulk substrates results in high mobilities of 2D electron gas: $\mu=25000 \text{ cm}^2/\text{Vs}$ for high electron density $n_{2d}=1 \times 10^{13} \text{ cm}^{-2}$ at $T=4.2\text{K}$. Moreover at room temperature we found that electron mobility is higher than $2300 \text{ cm}^2/\text{Vs}$ for $n=3 \times 10^{13} \text{ cm}^{-2}$ which is very promising for device performance based on the dislocation free substrates. This suggest also that mobility limits at room temperature is not only govern by optical phonon scattering but also depends on scattering by dislocations. We will discuss the dependence of AlGaN spacer width and Al content on the transport properties of 2D gas. The role of polarization effects on the formation of 2D gas will be pointed out.

References.

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[2] N. Grandjean, B. Damilano, J. Massies in "Low -dimensional nitride semiconductors" edited by B. Gil, p.121, (2002), Clarendon Press, Oxford

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17:50 - 18:05

oral

Kinetic Monte Carlo simulation of SiC nucleation on Si(111)

A. A. Schmidt²⁾, Yu. V. Trushin²⁾, P. Weih¹⁾, Oliver Ambacher¹⁾, J. Pezoldt¹⁾, V. Cimalla¹⁾

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2) Physico-Technical Institute, Politekhnikeskaya 26, 194021 St. Petersburg, Russian Federation, Russian Federation

Nanosized SiC-Si heterostructures offers applications as tunnelling barriers and anti-dot structures with their specific applications in nano- and optoelectronics. Solid source molecular beam epitaxy is an effective technique for the formation and the investigation of the growth laws. The easiest way to form nanosized SiC nuclei with well defined distribution and forms is the interaction of carbon with silicon surfaces. The large lattice mismatch between the SiC and Si forces three dimensional nucleation. The nuclei distribution can be designed by using proper substrate temperature, carbon fluxes and process times [1]. Nevertheless, up to now the very early stages of the carbon silicon interaction are not well understood. To have an deeper insight into the early nucleation stages the kinetic Monte Carlo method was applied. The following basic physical processes were included in the model: deposition of carbon

atoms, diffusion, attachment to and detachment from the clusters, creation of the SiC nuclei on top of the existing two-dimensional clusters. The scaling of the simulated cluster size distribution is shown (fig. 1). The simulations allowed to estimate the range of the surface diffusion activation energy for the carbon atoms on silicon and predicts the cluster concentration and cluster size distribution of SiC on Si. In the present work in situ reflection high-energy electron diffraction and atomic force microscopy was used to compare the theoretical predictions with the growth experiments.

[1] F. Scharmann, P. Maslarski, Th. Stauden, W. Attenberger, J.K.N. Lindner, B. Stritzker, J
[ABSTRACT TRUNCATED TO 2000 LETTERS]

The role of Ge predeposition temperature in the epitaxy of SiC on Silicon
F. M. Morales²⁾, Ch. Zgheib^{1,3)}, S. I. Molina²⁾, D. Araújo²⁾, R. García²⁾, C. Fernández⁴⁾, A. Sanz-Hervás⁵⁾, P. Masri³⁾, P. Weih¹⁾, Th. Stauden¹⁾, V. Cimalla¹⁾, Oliver Ambacher¹⁾, J. Pezoldt¹⁾

18:05 - 18:20

oral

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Silicon carbide on silicon is a promising heteroepitaxial system for the integration of wide band gap semiconductors into silicon technology. The main problem for the fabrication of devices based on that heteroepitaxial system is its large lattice and thermal expansion mismatch. In this work we present an alternative method for stress relaxation in the SiC/Si heteroepitaxial system based on a theoretical approach, which consists in the incorporation of a group IV element into the interface between SiC and Si. According to this, 1 ML germanium was predeposited on the silicon surface at different substrate temperatures prior to the carbonization of the Si substrate by molecular beam epitaxy. After the carbonization, a 120 nm-thick 3C-SiC layer was grown by solid source epitaxy. The resulting structures were investigated by transmission electron microscopy (TEM), x-ray diffraction, secondary ion mass spectroscopy (SIMS) and Fourier transform infrared spectroscopy (FTIR). TEM and SIMS results revealed a strong segregation of Ge at the interface leading to an increased stress relaxation and improved crystalline quality, in agreement with theoretical predictions. The improved crystalline quality of the grown 3C-SiC layer is shown by a decreased lattice distortion and smaller damping constants of the TO phonon. Furthermore, the incorporation of Ge at the interface suppresses the outdiffusion of Si from the substrate to the surface of the growing SiC layer and, therefore, impedes the formation of voids at the SiC/Si interface.

Problems with cracking of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layers

Ewa Dumiszewska^{1,2)}, Dariusz Lenkiewicz^{1,2)}, Włodzimierz Strupiński²⁾, Agata Jasik^{1,2)}, Rafal S. Jakiela^{2,3)}, Marek Wesołowski²⁾

18:20 - 18:35

oral

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' $\text{Al}_x\text{Ga}_{1-x}\text{N}$ ' is a wide gap material, which can be used for preparing of UV detectors. Unfortunately there are problems with cracking of that layers above some critical thickness, which is a little smaller from the one used for detectors (about $1\mu\text{m}$). Our investigations concentrated on the reason of forming of the cracking. To avoid that we used so called special AlN nucleation layer, which was to stop the relaxation. We received the strained layer free of cracking, but with very big number of dislocations. We compared dislocation density of strained and relaxed ' $\text{Al}_x\text{Ga}_{1-x}\text{N}$ ' layers. The first one characterized higher dislocation density than the second one.

We also investigated the problem of cracking of ' $\text{Al}_x\text{Ga}_{1-x}\text{N}$ ' epitaxial layers during the doping. There was the problem with controlling the process. The relaxation of the layers started for very low impurities density and went on when we increased the amount of the dopant.

Poster session

19:00 - 21:00

Optimization of Zn profiles in MOCVD grown AlGaAs/GaAs QW heterostructures.

19:00 -

R. Akchurin²⁾, A. Y. Andreev²⁾, P. V. Bulaev¹⁾, A. A. Marmalyuk¹⁾, D. B. Nikitin¹⁾, A. A. Padalitsa¹⁾, I. D. Zalevsky¹⁾

poster

C-1

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2) *Moscow State Academy of Fine Chemical Technology, 86 Vernadskogo Avenue, 119571 Moscow, Russia, Russian Federation*

The Zn doping of AlGaAs/GaAs QW heterostructures for laser diodes during MOCVD growth was examined. Fast diffusion of this dopant result in problems of sharp concentration profiles attainment. Experimental concentration profiles of heterostructures were analyzed to obtain more specific information because of wide scattering of published Zn diffusivity data. Electrochemical profiling was used to determine the electrically active Zn concentration and diffusion depth. Effective Zn diffusivity $\sim 6 \cdot 10^{-14} \text{ cm}^2/\text{s}$ for used epitaxial growth temperature 770C was estimated. Numerical simulation of Zn profiles for different growth conditions was carried out using this value. Calculation model provided for controllable variation of assigned doping profile ("profile doping") and considering the growth surface movement was employed. Results of simulation allowed to optimize doping regimes and achieve well-controlled p-n junction depths.

19:00 -

poster

C-2

Structural characterization of original 3D gallium structures grown by LP-MOCVD.

Luc IMHOFF¹⁾, Marco SACIOTTI²⁾, Claudie JOSSE COURTY¹⁾, Marie MESNIER¹⁾, Carmen MARCO DE LUCAS¹⁾, Sylvie BOURGEOIS¹⁾

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2) *Couches Minces et Nanostructures, LPUB, CNRS-Université de Bourgogne, 9 avenue Alain Savary - BP 47870, DIJON 21078, France*

The elaboration of metallic three dimensions (3D) structures with dimensions in the micro or nanometer range presents substantial interest in numerous research fields. From device applications point of view, the fabrication of patterned 3D structures with quantum properties is an attractive technological challenge for electronic or optoelectronic for instance. Unfortunately, it remains difficult to obtain such devices without several fabrication processing steps.

This study presents the growth and characterization of original metallic gallium 3D structures, obtained with a single growth step, by the LP-MOCVD technique on various substrates. Commercial organometallic is used for gallium precursor with nitrogen like carrier gas. The growth temperature and the reactor pressure are ranking between 400 and 700 °C, and between 150 and 700 torr, respectively.

Depending on the elaboration conditions, different 3D structures are obtained such as droplets, cauliflowers, big compact piles or thin stems, with micrometer sizes. Moreover, selective growth is also observed on patterned substrates, depending on the substrate chemical composition. The morphologies, substrate surface densities and thermal stability are studied by optical microscopy and scanning electron microscopy. At last, X-ray microanalysis and X-ray diffraction are carried out in order to determine the elemental composition and the crystallographic structure of these 3D objects.

19:00 -

poster

C-3

Optical quality improvement of InGaAs/AlAs/AlAsSb coupled double quantum wells grown by molecular beam epitaxy

Jun-ichi Kasai¹⁾, Teruo Mozume¹⁾, Haruhiko Yoshida¹⁾, Takasi Simoyama¹⁾, Achanta V. Gopal¹⁾, Hiroshi Ishikawa¹⁾

1) *Femtosecond Technology Research Association (FESTA), 5-5 Tokodai, Tsukuba 300-2635, Japan*

Intersubband transitions (ISBT) in quantum wells are attractive for high-speed all-optical switches. We previously reported that low absorption saturation intensities (I_s) of ISBTs were achieved in an InGaAs/AlAsSb system on InP. The I_s of a multiple quantum well sample finally reduced to $3 \text{ fJ}/\mu\text{m}^2$ when AlAs diffusion-stopping layers were inserted into the well-barrier interfaces. Our most recent study indicates that intersubband-absorption response time was greatly improved, decreasing it to 690 fs, by using coupled double quantum wells (C-DQWs). This accomplishment has led to the expectation that the application of diffusion-stopping layers to C-DQWs will improve the ISBT properties in both I_s and response time. We therefore tried to grow C-DQWs with AlAs diffusion-stopping layers by molecular beam epitaxy. The InGaAs wells were doped to $1 \times 10^{19} \text{ cm}^{-3}$ with Si. The I_s and response time in the present sample were measured using an optical parametric amplifier. The results showed that the I_s was extremely low, down to $34 \text{ fJ}/\mu\text{m}^2$ at the optical communication wavelength of $1.62 \mu\text{m}$, while ultrafast response times of 600 fs were maintained. This demonstrates that AlAs stopping layers can also be effectively used with C-DQWs. Although the optical measurements of the sample indicated its high quality, there are some problems that need to be solved. An optical microscope observation indicated that the sample had many cross-hatched lines, suggesting that the sample may not be of the highest quality. Preliminary results to optimize the thickness of AlAs layers, however, indicate that cross-hatched lines can be reduced. Further optimization of the growth parameters promises to enhance the quality of C-DQW structures, thereby reducing the switching energy of ultrafast ISBT switches. This work was performed under the management of Femtosecond Technology Research Association supported by the New Energy and Industrial Technology Development Organization.

Some aspects to the RHEED behaviour of LT GaAs growth**A. Nemcsics**

19:00 -

poster

Recently, (low temperature) LT growth of GaAs has become an increasingly important method since it provides highly insulating films and contributes to the synthesis of magnetic semiconductors. We will examine the (reflection high-energy electron diffraction) RHEED behaviour during (molecular-beam-epitaxy) MBE growth on GaAs (001) surface at LT growth condition in our work. The RHEED and its intensity oscillation of LT GaAs growth has some particular behaviour. The intensity, phase and decay of oscillation depend on the (beam equivalent pressure) BEP ratio and substrate temperature. We will give here an explanation for the phase change dependence on the deposition temperature. Furthermore an examination of the intensity dependence of RHEED behaviour on BEP ratio and substrate temperature will given here.

C-4

Growth of InSb Thin Films on GaAs(100) substates by Flash Evaporation Epitaxy**Maciej Oszwaldowski¹⁾, T. Berus¹⁾, A. Borowska¹⁾, R. Czajka¹⁾, M. Zimniak¹⁾**

19:00 -

poster

1) Instytut Fizyki, Politechnika Poznańska, ul. Nieszawska 13a, 60-965 Poznań, Poland

The technology of obtaining high quality epitaxial InSb thin films is important for applications in the Hall sensors and the IR detectors. The most available substrate material for that purpose is the semi-insulating GaAs. Unfortunately, there is a 15% misfit in the lattice parameter between InSb and GaAs, which makes the epitaxial growth difficult.

C-5

In the present contribution we report on a novel flash evaporation method for obtaining high quality epitaxial InSb films. We show that to obtain a well crystallographically oriented, in the (001) plane, InSb films with mirror smooth surface, a two stage evaporation process has to be applied. The first stage is a low temperature epitaxy (performed at 250°C) and the second stage is a high temperature epitaxy (performed at 470°C). In the first stage powdered InSb (the ration of Sb/In = 1) is evaporated, whereas in the second stage a mixture of powders of InSb and Sb, giving the ratio of Sb/In = 1.5, is evaporated. If the first stage is omitted, the obtained films have a rough and milky surface, and their XRD spectra can contain a small admixture of additional peaks from the (111) and (311) planes.

We have investigated the dependences of the electron mobility and the electron concentration of the films on the thickness of the underlayer obtained at 250°C in the thickness range (0 - 80) nm. The films themselves were about 2 μm thick. It is found that the electron concentration is in the range (2 - 20)*10¹⁶cm⁻³, and the maximum of the room temperature mobility of 25000 cm²/V*s is reached for the underlayer thickness of (10 -20) nm. The temperature dependence of the mobility is investigated in the temperature range from 77 K to 500 K and discussed. The early stages of the underlayer growth are investigated with AFM. Those investigations performed on underlayers having thickness smaller than 15 nm suggest Stranysky - Krastanov mode of growth.

MBE growth and characterization of InAs/GaAs for infrared detectors**Kazimierz Regiński¹⁾, Janusz Kaniewski¹⁾, Kamil Kosiel¹⁾, Tomasz R. Przesławski¹⁾, Jadwiga Bąk-Misiuk²⁾**

19:00 -

poster

*1) Institute of Electron Technology, Warsaw, Poland, Poland**2) Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland, Poland*

The growth of InAs epilayers on GaAs substrates has been a subject of intense studies over the past few years, due to important device applications of such layers in optoelectronics. In the field of high-speed infrared detectors, InAs is especially attractive as an absorption layer, due to its narrow band gap and high electron mobility. On the other hand, a semi-insulating GaAs is very convenient as a substrate, due to its transparency. However, the large lattice mismatch (7.3%) between InAs and GaAs causes major problems in obtaining high quality material.

C-6

In this work we concentrate on the problem of optimization of the MBE growth of thick (3.5 - 7 μm) InAs epilayers. Three types of layers were grown: undoped, Si-doped, and Be-doped. For such layers the optimum growth conditions comprising the substrate temperature, the flux ratio, and the growth rate have been found.

The grown layers are strongly nonhomogeneous as regards morphology, and, as a result, they exhibit some spatial distribution of carrier concentration and mobility. Therefore, we analyzed the dependence of transport properties of the layers on their thickness. Special attention has been devoted to the problem of low doping with Be to obtain the p-type material of low concentration of acceptors. Such a material is very promising as an absorption layer in infrared detectors. It has appeared that in the case of InAs slightly doped with Be, the transport properties dramatically depend on the thickness of the layer.

The transport properties of the studied layers have been correlated with their structural quality studied by X-ray diffractometry.

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Evidence of threading and misfit dislocations in partially relaxed InGaAs/GaAs heterostructures

19:00 -

poster

C-7 **O. Yastrubchak²⁾, E. Lusakowska²⁾, A. Morawski²⁾, O. Demchuk¹⁾, T. Wosinski²⁾**1) *Institute of Organic Chemistry, Polish Academy of Sciences, Kasprzaka, Warsaw, Poland*2) *Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland, Poland*

We have investigated MBE-grown InGaAs/GaAs heterostructures with a small lattice-mismatch that contained a net of well-resolved 60° misfit dislocations at the (001) interface. The misfit dislocations are accompanied by threading dislocations, which propagate into the epitaxial layer up to the surface. Atomic force microscopy (AFM) was used to examine an undulating and cross-hatched surface morphology of the heterostructures associated with the threading and misfit dislocations. The dislocations under investigation differ in their core structures. In the predominated glide set configuration the two dislocation types, referred to as α and β , are aligned along two orthogonal $[-110]$ and $[110]$ directions, respectively, at the interface. In order to distinguish between the nonequivalent $\langle 110 \rangle$ crystallographic directions on the (001) face of heterostructures we have utilized wet etching of the heterostructures in $\text{H}_2\text{SO}_4:\text{HF}:\text{H}_2\text{O}_2$ solution. Additionally, an ultrasonic-vibration aided etching in $\text{CrO}_3\text{-HF}$ aqueous solution was used to reveal the terminations of threading dislocations at the surfaces of the structures.

From analysis of the cross-hatched surface morphology of the structures we revealed an asymmetry in the formation of the two types of underlying misfit dislocations resulting from the difference between their glide velocities. Moreover, the obtained results allowed to draw the conclusion that relaxation of epitaxial layers in heterostructures with a small lattice-mismatch is realized in the model of Matthews and Blakeslee who considered the development of interfacial misfit dislocations from pre-existing threading segments driven by the misfit stress against their line tension.

Resistivity and mobility in ordered InGaP grown by MOVPE19:00 - **Stanislav Hasenöhr¹⁾, Julius Betko¹⁾, Marian Morvic¹⁾, Jozef Novák¹⁾, Ján Fedor¹⁾**

poster

1) *Institute of Electrical Engineering, Slovak Academy of Sciences (IEE SAS), Dubravska cesta 9, Bratislava 841 04, Slovakia (Slovak Republic)*

C-8

The MOVPE grown InGaP layers are often used in device structures, like heterojunction bipolar transistors, high electron mobility transistors, solar cells, light-emitting and laser diodes. Typical for this ternary prepared by MOVPE or MBE is that the group III atoms are not arranged randomly but they tend to create an ordered CuPt-B type structure. The extent of group III atoms rearrangement is significantly influenced by the growth conditions and layers with various degree of order are usually prepared. Ordering lowers the crystal symmetry what leads to anisotropic behaviour of material parameters.

Parameters of complete device structures, particularly of those where the carrier transport proceeds parallel to substrate-structure interface, are significantly influenced by transport properties in InGaP layer. Anisotropy of device parameters was reported e.g. for laser diodes, but it is expected to occur also in HEMTs.

Very little was reported on transport properties in epitaxial InGaP layers. We have investigated this problem in detail and we found that ordered layers exhibit resistivity anisotropy in $[011]$ and $[0-11]$ crystallographic directions. Moreover, we found the strong influence of misfit strain in mismatched layers on transport parameters, which is demonstrated e.g. by enhancement of mobility in slightly mismatched layers. In our contribution we will report on results from evaluation of electrical properties using temperature dependent Hall measurements on square samples (van der Pauw method) in confrontation with results from temperature dependent magnetoresistance measurements on stripe 4-point probes.

(Ga,Gd)As thin film growth by mass-analyzed low energy IBE19:00 - **Song S. Lin¹⁾, Chen N. Fu, Zhou J. Ping, Yang S. Yan, Liu Z. Kai**

poster

1) *Song Shulin, Semiconductor research institute of Chinese Academy of Sciences, Beijing 100083, China*

C-9

Ferromagnetic semiconductors can make full use of the spin degree of electron and provide bench for new ideas and new concepts. (Ga,Gd)As thin film is fabricated by mass-analyzed low energy ion beam epitaxy technique with Gd^+ ion can provide much bigger magnetic momentum. There is no new phase found by X-ray diffraction (XRD) while the main substrate peaks broaden by Double-crystal X-ray diffraction (DC-XRD).

Light induced contrast in Kelvin Force Microscopy of GaN epilayers19:00 - **Rafał Bożek¹⁾, Krzysztof Pakuła¹⁾, Jacek Baranowski¹⁾**

poster

1) *University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland*

C-10

Scanning Kelvin Force Microscopy (KFM) [1] is a technique based on Atomic Force Microscopy (AFM) for simultaneous recording of topography and surface potential. KFM applied for GaN epi-layers [2] revealed that surface potential in n-type material is related to the threading dislocations due to their charge states and the contrast was dependent on type and level of doping i.e. position of the Fermi level [3].

We report on the fluctuations in the surface potential, that appear during illumination close to the energy gap of GaN. The investigated samples were high quality undoped GaN layers grown on sapphire by Metal-Organic Vapour Phase Epitaxy. The AFM allows to find that the density of dislocations is of the order 10^8 cm^{-2} and distinguish between edge and screw type dislocations. The surface potential measured in dark is almost flat and featureless. Two types of potential fluctuations can be observed. The first one, "short range" ($\sim 200\text{nm}$) shows a clear correlation with screw type dislocations, i.e. the potential peak usually surrounds a dislocation pit. This is explained as an

effect of negative charging of dislocations. However in some samples the potential peaks are more numerous than pits observed on the surface. This can be understood as an image of dislocations below the surface of the sample. The "long range" (~5 μm) fluctuations are visible in the 25x25 μm images and can be assigned to heterogeneous distribution of impurities due to strain or mosaic structure of the epilayer. The observed fluctuations of the potential are strongly dependent on the intensity of the illumination. Very intense light flattens the potential distribution and removes observed contrast.

References

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Mg diffusion in GaN:Mg grown by metalorganic vapor-phase epitaxy MOVPE

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1) Warsaw University of Technology, Koszykowa 75, Warsaw 00-662, Poland

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19:00 -
poster

C-11

The diffusion characteristics of Mg dopant in MOVPE-grown GaN was studied. Cp₂Mg, TMGa and NH₃ were used as precursors. The Al₂O₃ was applied as a substrate. The temperatures range from 1000 C to 1300 C ensured conditions for device quality layers growth. Applying these temperatures, the back diffusion of magnesium were observed. Similar phenomenon was measured in Zn doped GaAs and InP.

Dopant profiles were investigated by Secondary Ions Mass Spectrometry (SIMS). We studied the effect of Cp₂Mg partial pressure and growth temperature on diffusion profile and on diffusion coefficient of Mg atoms.

Abrupt diffusion profile was observed when Cp₂Mg partial pressure was increased. Mg diffusion coefficient dependency on concentration was obtained from Boltzman-Matano analysis of dopant profile. Applying this method it was shown that diffusion coefficient was increasing by more than one order of magnitude within the concentration range: $5 \times 10^{17} - 1 \times 10^{20} \text{ cm}^{-3}$.

Increasing growth temperature of Mg doped GaN caused shift of diffusion profiles towards layer-substrate interface. Activation energy E_A and independent temperature factor D_0 were calculated by applying the profiles shift.

Optical properties of nitride diluted (Ga, In)(As, Sb) layers and quantum wells: character of optical transitions and carrier localization effect

R. Kudrawiec¹⁾, G. Sjk¹⁾, J. Misiewicz¹⁾, L. H. Li²⁾, J. C. Harmand²⁾

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2) Laboratoire de Photonique et de Nanostructures, CNRS Route de Nozay 91460 Marcoussis, France, France

19:00 -
poster

C-12

GaAsN, GaInAsN, and GaAsSbN compounds and their quantum wells (QWs) were grown by molecular beam epitaxy (MBE) on GaAs substrate. Such structures are very promising materials for optoelectronic devices operated at 1.3 and 1.55 μm , however their optical properties are still controversial and not fully understood. In this work, optical properties of the compounds and QWs have been studied by photoluminescence (PL) and photoreflectance (PR) spectroscopies. The character of optical transitions has been investigated systematically from 10 to 300 K. Many interesting features have been found for these structures. We have observed both well known (like e.g. carrier localisation effect, Stokes shift, annealing induced blue-shift detected in PL) and not reported so far experimental facts (a decrease of the modulation efficiency with a decrease of temperature, an increase of PR transition broadening with a decrease of temperature, annealing induced blue-shift detected in PR). In this work we have focused on the carrier localisation effect and their influence on the character of optical transitions. The strong localisation effect can be attributed to alloy fluctuation and different nitrogen nearest-neighbour environment. It has been observed that a rapid thermal annealing (RTA) essentially reduces the carrier localisation. In addition, the RTA induces a blue-shift of energy band gap. The nature of the shift and emission bands are discussed in this work.

Growth kinetics of (0001)GaN from Ga and NH₃ fluxes

Vladimir Mansurov¹⁾, Yurii G. Galitsyn, Valerii V. Preobrazhenskii, Konstantin S. Zhuravlev

1) Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation

19:00 -
poster

C-13

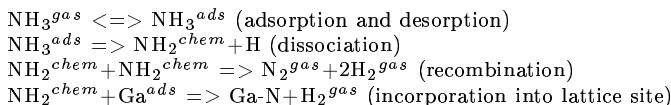
Recently the growth of high quality III-nitrides attracted a great deal of interest because of its usefulness for modern optoelectronics as well as high temperature and power devices. The growth of GaN by molecular beam epitaxy (MBE) using Ga and NH₃ is a sequence of elementary steps: adsorption-desorption, dissociation of NH₃, recombination of NH₂ radicals, incorporation of Ga and N atoms into lattice sites. This means that without the knowledge of the parameters of the microscopic surface processes it is not clear how to choose the optimum growth conditions.

In the present work the kinetics of GaN growth under Ga-rich conditions was investigated by reflection high energy electron diffraction (RHEED). Specular beam (SB) intensity evolution with time during successive supplying of the growth components onto a GaN surface was monitored. The strictly determined amount of Ga was deposited onto

GaN surface without NH₃ flux first, then the surface was exposed to NH₃. The transformation time (τ) of the adsorbed Ga into GaN was measured as a function of the NH₃ flux and substrate temperature.

An increase of the growth rate with increasing substrate temperature was demonstrated. It was revealed also that growth rate (V^{-1}/τ) depended on NH₃ pressure as $V^{-1}P^n$, where n increased with increasing substrate temperature from 0.5 (below 760 degrees C) to 1 (above 800 degrees C).

In order to explain the experimental data a kinetic model was developed in the frame of the mean field kinetic equations:



An analytical expression for the GaN growth rate was developed as a function of the elementary kinetic constants and NH₃ pressure. This kinetic model is in good agreement with the experimental data.

19:00 -
poster
C-14

Comparative analysis of (0001)GaN and (001)GaAs growth kinetics

Vladimir Mansurov¹⁾, Yurii G. Galitsyn, Konstantin S. Zhuravlev

1) Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation

It is well known that the bimolecular reaction of dissociation of As₄ molecules on the surface ($\text{As}_4 + \text{As}_4 \Rightarrow 2\text{As}_2 + \text{As}_4^{gas}$) plays an important role in the molecular beam epitaxy (MBE) of GaAs using Ga and As₄ fluxes. This reaction provides the formation of As₂ active intermediate complexes. This is the necessary stage in the chain of successive steps of surface reactions for incorporation of the V-group atoms into lattice sites.

On the other hand, the analysis of the growth kinetics of GaN by MBE using Ga and NH₃ fluxes under Ga rich conditions reveals the importance of the recombination of NH₂ radicals: $\text{NH}_2 + \text{NH}_2 \Rightarrow \text{N}_2^{gas} + 2\text{H}_2^{gas}$. But in contrast to GaAs in this case the bimolecular reaction decreases the concentration of the active species (NH₂ radicals) on the surface.

We have developed kinetic models for the growth of GaN and GaAs in the frame of the mean field kinetics equations. Analytical expressions for the growth rate were derived. For the correct comparison of the GaN and GaAs growth rates the normalized characteristic functions $f(\lambda)$ and $g(\lambda)$ were introduced.

$$V^{GaN} = V_{max}^{GaN} \times f(\lambda), \quad V^{GaAs} = V_{max}^{GaAs} \times g(\lambda),$$

For both materials the parameter λ depends on the substrate temperature and pressure (of NH₃ or As₄) in a similar way. Namely, it decreases with increasing substrate temperature and increases with increasing NH₃ (As₄) pressure. However, the characteristic functions $f(\lambda)$ and $g(\lambda)$ are drastically different: $g(\lambda) > 0$, $f(\lambda) > 1$ when λ tends to zero, and vice versa $g(\lambda) > 1$, $f(\lambda) > 0$ when λ tends to infinity.

Hence, the growth rate should increase with temperature for GaN (normal kinetics) and decrease for GaAs (anomalous kinetics), which is in good agreement with experimental data.

The authors are grateful to the RFBR financial support (grant 02-03-32307).

19:00 -
poster
C-15

Correlation between nitrogen incorporation and optical quality in MOCVD-grown Ga(In)NAs investigated by photoluminescence and modulation spectroscopy.

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Semiconductor structures based on Ga(In)NAs has been recently thoroughly investigated. Unique properties of such structures come from an unusual behaviour of nitrogen band and make them perfectly suitable for telecommunication devices for 1.3 or even 1.55 micron.

Technology of epitaxial growth of nitrogen-diluted structures is currently being optimised. It is difficult to incorporate more than small fractions of percent of nitrogen and at the same time to maintain high optical quality of samples.

In this work there has been studied GaNAs bulk layers and GaInNAs/GaAs Quantum Wells grown by MOCVD, with different TBAs flow. Room temperature photorefectance and contactless electroreflectance measurements have been carried out in order to determine the energies of optical transitions. Numerical calculations in envelope function formalism has been used to ascribe the measured energies to band structure and thus to verify the nitrogen content of the well layer. Low temperature PR and PL spectra have been compared and the value of Stokes shift, as an indicator of optical quality, has been correlated with nitrogen content of the samples. Also the line broadening of PR resonances and FWHM of PL peaks has been analysed to further confirm the relation between the amount of nitrogen in the structure and its optical quality.

19:00 -
poster

Predictive Use of Ab Initio MO Methods In PDEC-Based Approach to

Low-Temperature Epitaxy of Stoichiometric Group-III Nitrides

Keiji Hayashi¹⁾, Takuo Kanayama, Noriyoshi Omote

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C-16

In order to further develop integrated quantum functional devices and nano-electromechanical systems it has become indispensable in recent years to ingeniously utilize selective surface reactions of labile neutral chemical species such as free radicals and organometallic compounds for the device processing. The problem encountered in the experimental study of a chemical reaction between a neutral free radical and a well-characterized material surface is how to sufficiently supply only the desired free radical species onto the surface. We have proposed several experimental methods to produce a steady-flux purified beam of neutral free radicals¹⁾. One of them is the method of photo-dissociation of energetic compound beams (PDECB) where a beam of desired neutral free radical species is efficiently produced from a molecular beam of a purified unimolecular metastable dye by wavelength-selective photolysis using a near-UV CW laser²⁾.

With the help of ab initio molecular orbital (MO) methods, we have been developing a novel approach to the growth of defect-free epitaxial films of group-III nitrides¹⁾. Use of a molecular beam of organic group-III nitrene characterized from the theoretical point of view not only by the biradicaloid nature, that is to say, two unpaired electrons almost localized at the nitrogen atom but also by the covalently bound pair of one group-III and one nitrogen atoms is advantageous to the low-temperature growth of stoichiometric group-III nitride. The beam of organic group-III nitrene is obtainable by the PDECB method using organic group-III azide as source material.

This work is supported by the Ministry of Education, Science, Sports and Culture, Japan.

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CdTe thin films properties fabricated by hot wall epitaxy

Yevgen O. Bilevych¹⁾, Andriy I. Boka¹⁾, Joanna V. Gumenjuk-Sichevska¹⁾, Fedor F. Sizov¹⁾

1) *Institute of Semiconductor Physics NAS Ukraine, Kiev, Ukraine*

19:00 -
poster

CdTe thin films are widely used in semiconductor technology for many purposes: near-infrared detectors, optoelectronic devices and others. That is why great attention is paid to improve the existing methods of CdTe thin films fabricating to obtain films with repeatable thickness, morphology as well as electrical and optical properties.

CdTe thin films where prepared by hot wall epitaxy (HWE). The main feature of this method is the growth conditions that are rather close to the thermodynamic equilibrium. Such conditions are provided by quasi-close growing area and by the appropriate choice of the three temperature parameters: T_{source} , T_{wall} , $T_{substrate}$.

The thin films were grown on different substrates, such as BaF₂, Si, SiO₂, (110)CdTe. To investigate the fabricated films properties as a passivating material for IR technology they were also grown on narrow-gap Hg_{1-x}Cd_xTe. The influence of the substrate materials on properties of epitaxial films were investigated. The best results were obtained for the following temperature parameters: $T_{source} = 653$ K; $T_{wall} = 673$ K; $T_{substrate} = 393$ K. The growth rate was estimated to be of the order of $\sim 0,02 - 0,03$ mkm min⁻¹.

The Schottki barriers were prepared by thermal and chemical deposition of Au contacts on the grown CdTe layers. The current-voltage and capacitance-voltage investigations were performed to analyze their electrical properties. The samples exhibited the characteristics typical for the Schottky diodes.

Optical and photoelectrical properties were investigated by measurements of optical transmission spectra as well as the photoresponse and photoconductivity. For thin CdTe films transmission spectra are found to be similar to those ones obtained for thick films and bulk crystals. In the IR spectral region 2-15 mkm the optical transmission was ~ 60 %. As a test of the film quality, elastic strains were calculated from the shift of the absorption edge in the the transmission spectra

C-17

Properties of MBE Cd_xHg_{1-x}Te/GaAs structures modified by ion-beam milling

I. I. Izhnin²⁾, V. V. Bogoboyashchyy³⁾, Sergei Dvoretzky¹⁾, N. N. Mikhailov³⁾, Yu. G. Sidorov³⁾, V. S. Varavin³⁾, V. A. Yudenkov²⁾

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2) *R&D Institute for Materials SRC "Carat", Lviv, Ukraine, Ukraine*

3) *Kremenchuk State Polytechnical University, Kremenchuk, Ukraine, Ukraine*

19:00 -
poster

C-18

Despite the fact that ion beam milling (IBM) is a promising method for Cd_xHg_{1-x}Te photodiode arrays production only a little investigation has been made for MBE grown epitaxial layers. So the main aim of this paper was to investigate the modification of electrical properties under IBM in MBE Cd_xHg_{1-x}Te structures with variable band gap layers (1.5 μm thickness with gradually increasing Cd content up to x=0.4) near the both active layer interfaces. Both n-type (as-grown) and p-type (obtained by thermal annealing in He atmosphere) MBE structures grown on (310) GaAs substrates with 4 μm thickness CdTe buffer layer were studied.

It was revealed that IBM results in creation of n-type layer in p-Cd_xHg_{1-x}Te with thickness that determined by initial acceptor concentration. But the value of the converted layers thickness were much more smaller than it will be predicted by theory for the uniform Cd_xHg_{1-x}Te samples with the same acceptor concentration and IBM

conditions. These distinctions are due to the presence of the wide gap graded surface layer, which results in decrease of the conversion rate. The character of carrier concentration distribution over the structure depth for both n-type and p-type samples was the same as for the uniform $\text{Cd}_x\text{Hg}_{1-x}\text{Te}$ samples. Namely, for the p-type samples the electron concentration in thin subsurface damage layer (2-3 μm) with high electron concentration and low mobility is linearly decreased from surface to sample depth and then - (the main part of n-type layer with low electron concentration and high mobility) electron concentration has constant value that corresponds to the residual donor concentration. The hole concentration and mobility in unconverted region of formed p-n structures are the same as in the samples before IBM.

19:00 - **Investigation of Si-Ge whisker growth by CVD**

Anatoly A. Druzhinin¹⁾, Igor P. Ostrovskii¹⁾

poster

1) Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine

C-19

The present paper deals with studies of Si-Ge whisker growth in sealed Si-Br and Si-Ge-Br systems by CVD method. The whiskers were grown in a tube loaded in a furnace with temperature gradient. The temperatures of evaporation and crystallization zones were 1300-1500 K and 1000-1400 K, respectively. As a result of growth process the whiskers with diameters 0.1-50 μm were created. Ge content as well as the whisker homogeneity were controlled by microprobe analysis. Modeling of whisker homogeneity based on calculation of vapour phase content and analysis of such thermodynamical parameter as oversaturation was carried out. Good agreement between calculated data and experiment was obtained.

19:00 - **Stress analysis of strained superlattices**

Halyna Khlyap¹⁾, R. Peleshchak¹⁾

poster

1) State Pedagogical University, 24 Franko str., Drohobych, 82100, Ukraine, Ukraine

C-20

The latest successful development of smart technologies, in particular, molecular-beam epitaxy technique and pulse-laser deposition method, made it possible to manufacture optoelectronic active elements based on semiconductor materials with sufficient mismatch of the lattice parameters. This problem is of special interest for preparing photosensitive devices with strained superlattices. The abstract focuses on the numerical analysis and computational modeling of energy spectra of charge carriers in mechanically strained superlattices based on semiconductor materials from A2B6 and A4B6 (ZnSe, ZnTe and PbS) playing an important part in the optoelectronics design. Computational modeling is settled on the solution of one-dimensional Schroedinger equation and Poisson equation. The simple numerical algorithm is proposed for simulation of the superlattice energy structure in dependence of doping impurity concentration, thickness of the grown layers and their crystallographic orientation.

19:00 - **Medium scale modeling of the CdTe/ZnTe islands the empirical potential and finite element approach**

Piotr Traczykowski²⁾, Sławomir Kret¹⁾, Piotr Dłużewski¹⁾, Paweł Dłużewski²⁾, Sebastian Maćkowski¹⁾, G Karczewski¹⁾

poster

C-21

1) Institute of Physics PAS (IF PAN), Warsaw, Poland

2) Institute of Fundamental Technological Research, PAS (IPPT PAN), Świątokrzyska 21, Warsaw 00-049, Poland

Precise determination of the size, shape and composition of nanosize coherent islands obtained by MBE growth of CdTe/ZnTe heterostructures is important to understand the formation mechanisms and optical properties of such objects.

The Stillinger-Weber interatomic potential have been used to perform medium scale computer simulation of the island structure, which is in good agreement with experimental high resolution transmission electron microscopy (HRTEM) cross-sectional and planar view images

We use Finite Element solver to calculate atoms position of elliptical shaped island in the box containing up to 100000 atoms. In this approach we describe bonds as pseudoelements and restrict calculations to two types of them: 2-node, which model interaction between 2(1 bond) atoms and 3-node, which model interaction between 3-atoms (2 bonds). Novel mesh generator was developed for creation of the pseudoelements compatible with standard Finite Element routines.

The boundary conditions coming from geometry of TEM specimen were applied. The generated mesh was used to Finite Element Solver and equilibrium atoms positions were obtained in the sense of molecular static. Such atoms positions were used for simulation of the HRTEM images.

In this paper we give the initial model of CdTe/ZnTe, which match qualitatively with HRTEM observation. More precise results can be obtained by feed back approach with quantitative comparison of the simulated and experimental images.

This calculation method can be extended to large scale calculation to simulate growth of quantum dots stack or initiation of dislocations between epitaxial layers.

Optical and structural properties of films based on CdHgTe**I. S. Virt²⁾, I. O. Rudyi³⁾, I. V. Kurilo³⁾, O. I. Vlasenko¹⁾, I. V. Petrovych³⁾***1) Institute for Physics of Semiconductors of NAS of Ukraine, 45 Prospekt Nauki, Kyiv 03028, Ukraine**2) Drohobych State Pedagogical University, Grushevski 117-1, Drohobych 82106, Ukraine**3) Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine*

19:00 -

poster

C-22

CdHgTe epitaxial films (EF) used in IR-detectors have to be quenched after annealing in order to control the p-type carrier concentrations. Structural, physical, optical and mechanical properties of CdHgTe/CdTe grown by ISOVPE after different cooling modes were investigated (in air, water, oil, and liquid nitrogen). Thin poly-crystal layer (~3 nm) including amorphous structure forms on the surface during high cooling rates. Thin polycrystalline layer are formed at cooling in air at low rates. The oil-quenching in thin nearsurface layer leads to the block structure. The water-quenching of the films leads to the surface cracks appearance. The dependence of mechanical properties from a mode of cooling is revealed. The reflection spectra (77 K) in the range of 2-15 μm for four groups of EF samples with different cooling routine have similar EF structure with a peaks near 0.27, 0.24, 0.22, 0.20, 0.18, 0.16, 0.14 and 0.12 eV. Defects concentration in undersurfacing layers of EF increase in order of cooling in air, oil, and liquid nitrogen. Decreasing of reflection coefficient and some smoothing of features of spectrum take place in the same order. The revealed changes of structure and properties of EF are connected with generation and diffusion of defects in undersurfacing layers.

Photoemission study of manganese thin films deposited on layered semiconductors surfaces**Frederic Mirabella¹⁾, Robert L. Johnson²⁾, Jacques Ghijsen¹⁾***1) Laboratoire Interdisciplinaire de Spectroscopie Electronique (LISE), 61 rue de Bruxelles, Namur 5000, Belgium**2) Institute for Experimental Physics, University of Hamburg, Hamburg, Germany*

19:00 -

poster

C-23

Germanium sulphide (GeS) and tin diselenide (SnSe₂) present anisotropic properties due to their layered crystal structures. In fact, these crystals are composed of atomic layers interacting with each other only by van der Waals forces. Recently these materials have been used to prepare new diluted magnetic semiconductors (DMS) by substituting some of Ge or Sn atoms by manganese in crystal lattices forming Ge_{1-x}Mn_xS and Sn_{1-x}Mn_xSe₂. Aiming at a comparison of Mn atoms behaviour with the layered crystal, Mn/GeS and Mn/SnSe₂ thin films have been grown and investigated by X-ray and synchrotron radiation induced photoelectron spectroscopies.

Magnetic properties of V atoms in ultra-thin epitaxial Gd/V bilayers**Piotr Pankowski¹⁾, Lech T. Baczewski¹⁾, A. Wawro¹⁾, K. Mergia²⁾, S. Messoloras²⁾, F. Ott³⁾***1) Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland**2) Institute of Nuclear Technology & Radiation Protection, National Centre for Scientific Research "Demokritos" in Athens, Athens, Poland**3) Laboratory of Leon Brillouin CEA, Saclay, Poland*

19:00 -

poster

C-24

The properties of artificial layered structures are in many cases revolutionary, revealing many phenomena that are not found previously in bulk materials. We present the study of magnetic properties of MBE grown epitaxial vanadium (V) / gadolinium (Gd) bilayers. The samples were grown in UHV conditions using standard MBE system. Configuration of the samples is the following: substrate Al₂O₃[11-20]/Mo 200 Å buffer layer/V layer with thickness 4, 6, 8 or 10 monolayers (ML)/Gd layer 65 Å. All samples were oxidation protected with Al cover layer. Quality of the subsequent layers and interfaces were investigated by RHEED during evaporation. To characterise structural properties and determine a detailed structure of the samples the X-ray reflectivity measurements were performed using synchrotron radiation. The magnetic properties of ultra thin gadolinium / vanadium bilayers were studied as a function of vanadium layer thickness (from 4 to 10 ML). To determine magnetic properties of the samples two methods were applied: VSM measurements for the hysteresis loops determination and Polarised Neutron Reflectivity (PNR) experiment was performed in order to evaluate separately the magnetic moments of V and Gd. The simulations of neutron reflectivity curves treated with Parratt algorithm clearly show a strong magnetic moment induced in V layers, which decreases with V layer thickness. We found approximately half of its bulk magnetic moment value for Gd atoms, this fact we connect to the roughness of Gd layers and its reduced dimensionality.

Acknowledgments: This work was partly supported by EU RTD project SPINOSA, contract no. IST-2001-33334.

Development of Indium Tin Oxide (ITO) films for the Bragg reflectors application**HANNA WRZESIŃSKA¹⁾, Lidia Lilka¹⁾, Dorota Wawer¹⁾, Krzysztof Hejduk¹⁾, Andrzej Kudła¹⁾***1) Institute of Electron Technology, al. Lotnikow 32/46, Warsaw 02-668, Poland*

19:00 -

poster

C-25

The ITO films are used in wide range of optoelectronic applications and are of growing importance in the electronic device fabrication. One of the possible applications of ITO films is in the newly designed vertical cavity surface

emitting lasers (VCSEL) [1].

This research were focused on the deposition technology of ITO films, which are intended to be used in VCSEL lasers emitting in 1000 nm band fabrication technology. The ITO films should have high transmission (above 85%), low resistivity (about 10^{-4} Ωcm) and smooth surface to allow for deposition of dielectric Bragg reflectors.

In this work there was examined the influence of parameters of deposition process on optical and electrical parameters of ITO films. The films were deposited using the dc magnetron sputtering system. With the ITO film thickness 200 nm the subsequent annealing steps were done in the air atmosphere in 500°C. Spectral characteristics were measured in the 400-1100 nm range. Optical constants n , k were determined with the spectroscopic ellipsometer and surface smoothness was tested with the AFM use.

As deposited ITO transmission for the 1000 nm wavelength was in the 60% - 93% range. After annealing the ITO transmission increased to 84-100%. It was observed that the deposition step parameters strongly affect electrical resistivity of the ITO films. After annealing of the ITO films the resistivities of all sample decreased to tens $\mu\Omega\text{cm}$. The refractive index of ITO films measured for 1000 nm wave does not depend on the partial pressure of argon and oxygen, but depends on the deposition rate.

In conclusion, we have elaborated stable technological conditions of the ITO films with satisfactory optical and electrical properties for optoelectronic applications.

1. A. Szerling, D. Wawer, K. Hejduk, T. Piwonski, A. Wojcik, B. Mroziewicz, M. Bugajski: Reflectance study of $\text{SiO}_2/\text{Si}_3\text{N}_4$ dielectric Bragg reflectors. *Optica Applicata*, Vol. XXXII, No. 3, 2002

19:00 -

poster

C-26

Optical investigations of two dimensional electron gas in the AlGaIn/GaN heterostructures

R. Kudrawiec¹⁾, M. Motyka¹⁾, M. Syperek¹⁾, J. Misiewicz¹⁾, R. Paszkiewicz²⁾, B. Paszkiewicz²⁾, M. Tlaczala²⁾, Włodzimierz Strupiński³⁾

1) *Institute of Physics, Wrocław University of Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland, Poland*

2) *The Faculty of Microsystem Electronics and Photonic, Wrocław University of Technology Janiszewskiego 11/17, 50-372 Wrocław, Poland, Poland*

3) *Institute of Electronic Materials Technology, Wolczynska 118, 01-919 Warszawa, Poland, Poland*

AlGaIn/GaN heterostructures were grown by metalorganic vapour phase epitaxy (MOVPE) on the c-plane sapphire substrate. The sample consists of low temperature GaN buffer layer, high temperature 1.5 μm GaN layer, and 30 nm thick $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layer. All these layers are nominally undoped. For the set of samples the Al content changes from 20 % to 35 %. The electron concentration in the structure was determined by the impedance spectroscopy method. We have found that for investigated structures the two dimensional electron gas (2DEG) concentration at the interface was in the 2.0-3.0 10^{12} cm^{-2} range. Optical properties of AlGaIn/GaN heterostructures and a presence of the 2DEG have been investigated by both absorption-like (photorefectance PR) and emission-like (photoluminescence PL) experimental techniques. In photorefectance, spectral features associated with AlGaIn barrier, the 2DEG at the interface, and bulk GaN were observed. PL spectra were measured at two combinations: first was excitation through AlGaIn layer, and second was excitation through GaN layer. In these two combinations different PL spectra have been observed. In the first case PL spectrum was dominated by transitions from AlGaIn layer, and in the second case features associated with GaN layer and 2DEG are observed. All PL and PR features change with the increase of Al content. The nature of absorption and emission transitions and their temperature dependence was studied and discussed in this work.

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Tuesday, September 16th

14:00 - 15:30

Afternoon session

Main Building, room 213

14:00 - 14:45

invited oral

Magnetotransport investigations of AlGaIn/GaN heterostructures grown on bulk GaN, SiC, and sapphire substrates

Wojciech Knap¹⁾

1) *GES UMR5650 CNRS and Universite Montpellier 2, France, France*

We will present the results of the experimental studies of transport properties of two-dimensional electron gas (2DEG) and three-dimensional electrons (that might be responsible for a parallel conduction) in AlGaIn/GaN heterostructures grown over high-pressure bulk GaN, sapphire, and insulating SiC substrates. The experimental

results include the low field Hall measurements [1], cyclotron resonance measurements, and cryogenic temperature Quantum Hall Effect studies [2]. The room temperature high field measurements allow us to clearly separate contributions of a parasitic parallel conduction from 2DEG conduction in all investigated heterostructures.

The Quantum Hall Effect measurements are performed in the magnetic fields up to 30 Tesla and temperatures between 2K-80K. This high magnetic field in combination with very high mobilities [3] in the samples on the bulk GaN substrates allow us to observe very interesting features related both to cyclotron resonance and spin splitting. The temperature dependence of these splittings determines the spin and cyclotron resonance energy gaps and, in combination with cyclotron resonance results, allows us to determine a complete set of 2DEG transport parameters.

1) Acoustic phonon scattering of two-dimensional electrons in GaN/AlGaIn heterostructures, W. Knap et al. Appl. Phys. Lett. 80, 1228 (2002)

2) Cyclotron resonance and quantum Hall effect studies of the two-dimensional electron gas confined at the GaN/AlGaIn interface, W. Knap et al. Appl. Phys. Lett. 70, 2123 (1997)

3) High electron mobility in AlGaIn/GaN heterostructures grown on bulk GaN substrates, E. Frayssinet et al. Appl. Phys. Lett. 77, 2551 (2000)

Homoepitaxy of GaN-based blue and UV lasers

Piotr Perlin¹⁾

14:45 - 15:30

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

invited oral

GaN and related compounds are nowadays the key materials for so called 'blue optoelectronics'. Thanks to their large, direct gap, they are able to emit the light in the very broad spectral range. The use of nitrides semiconductors made possible the realization of first 'blue light' emitting laser diodes. The biggest obstacle in the development of these devices is the lack of GaN substrate crystals for epitaxy. So far commercial techniques base on sapphire substrates. Sapphire is one of very few materials stable enough to withstand the GaN growth conditions. However incompatibility of crystalline structures of GaN and sapphire leads to the very serious degradation of GaN overlayers quality. In spite of the sophistication of the growth method, the density of dislocations is much too high, specially for the construction of broad-stripe, high-power lasers.

In this presentation I will focus on the application of high quality, high-pressure grown bulk GaN crystals for MBE and MOVPE epitaxy of laser structures, showing the advantages of this technology in particular for manufacturing of high-power laser diodes and UV light emitting devices. I would like to stress that in case of homoepitaxy both MBE and MOVPE methods can give excellent and very comparable results.

Coffee break

15:30 - 15:50

Afternoon session - continued

15:50 - 17:20

Main Building, room 213

Multiscale modelling of group-III nitride growth

Joerg Neugebauer²⁾, **Liverios Lymperakis**²⁾, **Chris G. Van de Walle**³⁾, **John E. Northrup**³⁾, **Randy Feenstra**¹⁾

15:50 - 16:35

1) *Carnegie Mellon University, Pittsburgh 15213, United States*

invited oral

2) *Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, Berlin D-14195, Germany*

3) *Palo Alto Research Center (PARC), Coyote Hill Road 3333, Palo Alto 94304, United States*

A first step towards controlling and improving the quality of semiconductor devices is a deeper understanding of the fundamental mechanisms during doping and growth. For example, the morphology and structure of the surface controls the sharpness and thus the electronic characteristics of the interfaces, the incorporation of dopants or the formation of nanostructure such as quantum dots.

A challenge in performing simulations addressing these questions is the large range of relevant length and time scales. While eventually we are interested in a description on a mesoscopic scale (the size of typical quantum dots or surface features is in the order of 10-100 nm and the growth time is in the order of seconds) the mechanisms leading to these structures (adatom adsorption, diffusion, desorption, island nucleation) require a resolution in the length scale of 10^{-1} nm and in the time scale of 10^{-13} s⁻¹. Therefore, common approaches to simulate growth have been restricted on specific properties (on the mesoscopic scale) and included microscopic information only indirectly by empirical/adjustable parameters. In the present talk it will be discussed how by combining density-functional theory with concepts of thermodynamics, continuum elastic theory, and/or statistical physics simulations can be performed which allow to bridge between microscopic and mesoscopic scales. To discuss the application but also the present limits of this approach we will focus on various issues regarding doping and growth of group-III nitrides (GaN, AlN, InN and their alloys).

16:35 - 17:20

Control of the polarity and crystal face of GaN epilayers grown by MBE**S. Pezzagna**, **Nicolas Grandjean**¹⁾, **A. Dussaigne**, **P. Vennegues**

invited oral

¹⁾ CRHEA-CNRS, rue B. Gregory, Sophia Antipolis, 06560 Valbonne, France, France

GaN epilayers are grown by molecular beam epitaxy using NH₃ as nitrogen precursor on sapphire substrates. When a low temperature buffer layer is used, the GaN films grow with the Ga polarity. However, the polarity can be changed from Ga to N by the deposition of a Mg monolayer during growth interruption. Such a polarity inversion may also occur when a large Mg flux is supplied during growth. Secondary ion mass spectroscopy shows that the critical Mg concentration for the polarity inversion must be larger than 3x10²⁰ cm⁻³. The interface between the two polarities has been characterized by transmission electron microscopy (TEM). It demonstrates a faceted morphology. The polarity inversion is perfectly controlled over a 2 inch wafer and no dislocation is introduced. Actually, the quality of the N-polarity GaN layer replicates that of the Ga-polarity underlayer. Despite some indications about the role of the Mg, the actual mechanism responsible for the polarity inversion is still unclear. Furthermore, if the growth proceeds on the N-polarity with a large Mg flux, the GaN layer undergoes a crystal phase transition from hexagonal to cubic. Surprisingly, this is achieved for standard growth temperatures (800°C) for which the cubic phase should be unstable. Moreover, high-resolution TEM images show a perfectly smooth interface between h- and c-GaN.

On a wafer with a single Ga/N polarity inversion, stripes have been etched up to the Ga polarity. At this stage the sample exhibits an in-plane Ga/N periodic polarity modulation. Then, GaN is overgrown in order to get thick periodic polarity layers, which are dedicated to non-linear optics. Indeed, the lateral modulation of the GaN polarity opens the way for quasi-phase matching structures. The verticality of the polarity domain boundaries has been checked by TEM as a function of the stripe orientation. Smooth (10-10) planes are obtained for properly chosen stripe orientation. This is confirmed by selective wet etching, which reveals only the Ga polarity material. We will show that this approach allows the fabrication of high-quality three-dimensional structures without plasma etching step.

17:20 - 17:35

Break

17:35 - 18:35

Afternoon session - continued

Main Building, room 213

17:35 - 17:50

Growth and Characterization of GaN Epilayers on Si Substrates**Olga Kryliouk**⁵⁾, **Michael Mastro**⁴⁾, **Sang Won Kang**¹⁾, **Kee Chan Kim**²⁾, **Tim Anderson**³⁾

oral

¹⁾ University of Florida (UF), 227 Chemical Engineering Bldg., Gainesville 32611, United States²⁾ University of Florida (UF), 227 Chemical Engineering. Bldg., Gainesville 32611, United States³⁾ University of Florida (UF), 227 Chemical Engineering Bldg., Gainesville 32611, United States⁴⁾ University of Florida (UF), 227 Chemical Engineering Bldg., Gainesville 32611, United States⁵⁾ University of Florida (UF), 227 Chemical Engineering Bldg., Gainesville 32611, United States

The group III nitrides are wide band-gap semiconductors that have demonstrated considerable promise for various optoelectronic, high-temperature and high-power device applications. The development of GaN epitaxy on Si substrates would decrease substrate cost compared to sapphire and SiC and would allow integration with existing Si processing technology.

A technique was developed to deposit single crystal GaN on Si (111) directly using an oxynitride SiO_xN_{1-x} compliant interface to relieve stress at the substrate-film interface. A thermodynamic assessment of the Ga-N-O-Si system was performed using the ThermoCalc software package to understand the chemistry at the Si/GaN interface. An equilibrium phase-diagram was generated. It was shown experimentally that low-temperature growth prevented the formation of the detrimental Si₃N₄ phase. Our direct process uses 2 steps: a thin MOCVD layer followed by a HVPE growth in the same reactor. A capping GaN layer was grown in an alternate reactor that is a traditional low-pressure cold-wall MOVPE system. Crack-free, single-crystal GaN have been demonstrated using the sequence of a low temperature MOCVD layer (560°C) followed by a HVPE GaN layer (560 to 900°C). Single crystal GaN was demonstrated by MOCVD even at a growth temperature of 900°C. Additional parameters such as growth temperature, V/III ratio, III/Cl ratio, substrate preparation and pre-treatment were optimized to provide the best crystalline quality as judged by XRD, TEM and SEM.

We have investigated the use of GaN, AlN and ZnO buffer layers on the quality of GaN films grown on Si(111) and Si(100) substrates.

Different characterization techniques, such as PL, ESCA, SIMS, AES were used for films and substrates characterizations.

17:50 - 18:05

Raman scattering study of InGaAs/AlAsSb and InGaAs/AlAs/AlAsSb heterostructures

oral

TERUO MOZUME¹⁾, Nikolai Georgiev²⁾, Jun-ichi Kasai¹⁾*1) Femtosecond Technology Research Association (FESTA), 5-5 Tokodai, Tsukuba 300-2635, Japan**2) Forschungszentrum Rossendorf (FZR), Dresden 01314, Germany*

InGaAs/AlAsSb quantum wells (QWs) grown on InP substrates are recently attracting much attention for optical and electrical devices. We have reported the near-infrared intersubband transitions from this material system. We have also reported the ultra-fast absorption recovery of 0.69 ps and shown that this material system is suited for the ultra-high speed optical devices used in the optical communication network. However, the growth of this system is pretty difficult. We have already reported that As-interface termination procedure improve the QW property. The PL spectra of Sb-terminated QWs are broadened and red-shifted than that of the equivalent As-terminated version. In order to further improve the interface quality, we have introduced several mono-layers (MLs) of AlAs layer between InGaAs well and AlAsSb barrier.

We report here a detailed Raman scattering study of AlAsSb/InGaAs superlattices (SLs) and quantum-wells (QWs) grown by molecular beam epitaxy (MBE). The interface termination procedure and AlAs interface control layer are shown to influence a lot the Raman spectra: (1) the Sb-terminated SLs show clear InSb-related interface phonon at around 195 cm⁻¹ and a weak broad peak at around 169 cm⁻¹, while As-terminated SLs only show a weak broad peak, (2) when 4-MLs of AlAs layer is introduced, no features were observed below 200 cm⁻¹, indicating well controlled interfaces, and (3) by inserting AlAs layer, AlAsSb band changed from 2-mode to single mode behavior. These results suggest that a Raman scattering is a sensitive microscopic probe of local disorder and clearly show that high quality interface is achieved in sample with AlAs layer.

This work was supported by New Energy and Industrial Technology Development Organization (NEDO) in the framework of Femtosecond Technology Project.

Defects in GaMnAs - influence of annealing and growth conditions**J. Sadowski^{1,2,3)}, J. Kanski⁴⁾, L. Ilver⁴⁾, Jaroslaw Domagala³⁾, J. Bak-Misiuk³⁾, A. Mikkelsen⁵⁾, E. Lundgren⁵⁾, F. Tuomisto⁶⁾, K. Saarinen⁶⁾, M. Adell⁴⁾***1) Niels Bohr Institute, Copenhagen University, 2100 Copenhagen, Denmark, Denmark**2) MAX-Lab, Lund University, 221 00 Lund, Sweden, Sweden**3) Institute of Physics, Polish Academy of Sciences, 02- 668 Warszawa, Poland, Poland**4) Chalmers University of Technology, 412 96 Guteborg, Sweden, Sweden**5) Synchrotron Radiation Department, Lund University, 221 00 Lund, Sweden, Sweden**6) Helsinki University of Technology FIN-02015 HUT, Finland, Finland*

Increasing research activity on GaMnAs gave in recent years considerable improvements of its magnetic and electrical properties, in particular significant increase of paramagnetic-to-ferromagnetic phase transition temperature (T_c), from 110 K to 150 K (170 K in some particular case). Even though the methods of fabrication of GaMnAs with the highest T_c are more or less established and repeated by many research groups, it is not fully understood what lies behind the empirical procedures rendering GaMnAs the highest T_c. It is generally accepted that the control of structural defects present in GaMnAs in large concentrations is a key factor for its magnetic and transport properties, though the overall picture of GaMnAs defect structure is still quite fuzzy. As is well known in the case of LT GaAs, the use of low growth temperatures during Molecular Beam Epitaxial deposition of GaMnAs thin films implicates existence of variety of different structural defects such as As antisites, Ga and As vacancies, As interstitials etc.. Addition of Mn into LT GaAs makes the situation even more complicated, since Mn creates new defects states. At present the main interest has been addressed to Mn interstitials reported to be present in GaMnAs, before, mainly As antisites were concerned. Since both types of defects: Mn_I and As_{Ga} are present in GaMnAs in large concentrations (of the order of 0.5% - 2%), they both have to be taken into account to explain GaMnAs properties. In this work we demonstrate how to control independently both types of defect and what is their influence on structural, magnetic and transport properties of GaMnAs.

Alignment of SiC quantum dots on silicon substrates**V. Cimalla¹⁾, A. A. Schmidt¹⁾, K. Zekentes²⁾, Ch. Fvrster¹⁾, P. Weih¹⁾, Oliver Ambacher¹⁾, J. Pezoldt¹⁾***1) Zentrum für Micro- and Nanotechnologien, Technische Universität Ilmenau, 98693 Ilmenau, Germany, Germany**2) Microelectronics Research Group, University of Crete, Vassilika Vouton, P.O. Box 2208, Heraklion, Crete, Greece, Greece*

We demonstrate a linear alignment of self-assembled SiC quantum dots grown by molecular beam epitaxy on silicon substrates. The large lattice mismatch between Silicon and SiC of 20% stimulates a three-dimensional nucleation on the substrate. This spontaneous formation of islands is a powerful tool for the formation of quantum dots. Further more the average size of the nuclei can be easily controlled by the process conditions. However, the non-uniformities in the size and the distribution of the islands strongly limits the possible application e.g. in electronic devices. Thus, the control of the nucleation sites of SiC is a precondition for a lateral alignment of the SiC nuclei. In the technology of the formation of Ge clusters on silicon several ways to localize the nucleation sites are known, e.g., ordering on lithographically pre-patterned substrates, on dislocation networks, and along step arrays on vicinal

18:05 - 18:20

oral

18:20 - 18:35

oral

substrates. Here we use the possibilities to form well ordered monoatomic steps, biatomic steps and step bands on (100) and (111) silicon, which offer the advantage that additional processing steps to define the alignment can be avoided. These terraces promote an alignment along their step edges. The arrangement was controlled by three key parameters with the following consequences: (i) the temperature and the flux control the nucleation density, (ii) the processing time defines the size, and (iii) the terrace width is adjusted close to the average distance of the nuclei. By atomic force microscopy we demonstrate the possibility to control a lateral ordering in linear chains and in dense dot arrays.

(i) G. Jin, J.L. Liu, S.G. Thomas et al, Appl. Phys. Lett. 75, 2752 (1999).

(ii) F. Leroy, J. Eymery, P. Gentile, and F. Fournel, Appl. Phys. Lett. 80, 3078 (2002).

(iii) K. Sakamoto, H. Matsuhata, M.O. Tanner et al, Thin Solid Films 321, 55 (1998).

Wednesday, September 17th

09:00 - 10:30

Morning session

Main Building, room 213

9:00 - 9:45

Technology of high power lasers

Peter Unger¹⁾

invited oral

1) Department of Optoelectronics University of Ulm 89069 Ulm, Germany, Germany

Semiconductor lasers are gaining increasing interest because of their small size, a high electrical-to-optical power conversion efficiency, the excellent reliability of the devices, and the electronic modulation properties up to frequencies of several GHz. When properly designed, laser diodes exhibit single-mode beam properties at low output powers. At high output powers however, the beam properties are degraded due to filamentation effects. Additionally, the power is limited by device heating and Catastrophic Optical Mirror Damage (COMD). In this presentation, two approaches are discussed to achieve high optical output powers in semiconductor lasers while maintaining an excellent beam quality. The first approach uses dry-etched curved mirror facets to realize a lateral unstable resonator design. A process technology based on Chemically Assisted Ion-Beam Etching (CAIBE) is presented, which allows the etching of smooth mirror facets. The devices show good beam quality in continuous-wave operation at high output power. A scanning electron micrograph of the curved mirror of an unstable resonator laser is depicted in the figure on the left-hand side. The second approach is an optically pumped semiconductor disk laser as sketched on the right-hand side. This novel laser unifies the benefits of solid-state thin disk lasers and Vertical-Cavity Surface-Emitting Lasers (VCSELs). The undoped epitaxial structure consists of a Bragg mirror and a resonant gain region which is optically pumped by a broad-area laser diode. The second mirror is an external concave dielectric mirror resulting in a stable concentric resonator configuration enforcing single-mode operation. The whole concept is scalable allowing high optical output powers. Due to the external mirror, additional elements can be positioned inside the laser cavity like nonlinear crystals to achieve frequency doubling. Devices are presented which emit in the blue and the orange-yellow wavelength range.

9:45 - 10:30

Sensitivity of quantum dot lasers to optical feedback

Guillaume Huyet¹⁾

invited oral

1) Physic Department National College of Ireland, XXX, Cork XXXX, Ireland

Bulk and quantum well semiconductor lasers are extremely sensitive to back reflections and the optical isolators are commonly used in photonics network to prevent the appearance of instabilities that deteriorate the bandwidth. Here we will analyse, both theoretically and experimentally, the sensitivity of quantum dot semiconductor lasers to optical feedback. In particular, we will show that these devices have indeed a reduced sensitivity to optical feedback although the line-width enhancement factor is relatively large and show that this can be associated with the gain saturation characteristic of quantum dot devices.

10:30 - 11:00

Coffee break

11:00 - 12:30

Morning session - continued

Main Building, room 213

11:00 - 11:45

III-V quantum dots: from lasers to single photons

Andrea Fiore¹⁾

invited oral

1) *Institute of Quantum Electronics and Photonics Ecole Polytechnique Federale de Lausanne - CH-1015 Lausanne, Switzerland, Switzerland*

Semiconductor quantum dots (QDs) were originally predicted to provide high gain, improved temperature performance and high-frequency modulation characteristics. In the last few years, several demonstrations of QD lasers based on self-assembled growth in different material systems have pointed out the possibility of achieving very low threshold current densities and extending the wavelength range, but also the limitations due to low modal gain. Overall, it is tempting to describe QDs as an active material with a low and spectrally-broad density of states. However, the energy spectrum of each dot consists of a ladder of discrete energy states, and although this discreteness is masked by the inhomogeneous broadening in macroscopic devices, it strongly affects the device behaviour. In this presentation some of the consequences of the quantum nature of QD active regions will be addressed: On one hand, the effect of finite interlevel relaxation time on laser operation will be described. On the other hand, I will discuss the progress towards the realisation of devices incorporating single QDs in the active region, where emission can be controlled at the single photon level.

In-plane lasers incorporating 2 or 3 stacks of self-assembled InAs QDs grown on GaAs (areal density $3 \times 10^{10} \text{ cm}^{-2}$) typically show ground state (GS) lasing around 1300 nm for cavity lengths $> 1.5 \text{ mm}$ and excited state (ES) lasing (around 1200 nm) for shorter lengths, due to the higher modal gain available on the ES. We have recently found that GS and ES lasing can coexist when a device 1.5-2 mm-long is biased well above threshold. Lasing starts on the GS but a second lasing line appears on the ES as the bias is increased. The two lasing states compete for the same carriers, so that saturation of the GS power is observed at the ES threshold. The wavelength-resolved light-current characteristics are fully understood by a simple rate-equation model by assuming a intradot (GS-ES) relaxation time of 7 ps, close to the experimental 10 ps photoluminescence rise time. In fact, as bias is increased above threshold, carriers must be injected to the GS at a higher rate, which implies an increasing ES population. In short cavities, where GS lasing occurs with a nearly-saturated GS, this "relaxation bottleneck" effect is exacerbated by the very limited number of available states, so that the increase in ES population leads to ES lasing. The incomplete clamping of the ES population also affects the modulation characteristics of QD lasers, particularly in terms of the linewidth enhancement factor, which is seen to increase up to a value of $a=10$ as the bias is increased well above threshold, due to the increasingly asymmetric gain spectrum.

Carrier localisation is another intrinsic characteristic of QD active regions. We have fabricated ultrasmall (submicrometer) QD light-emitting diodes (LEDs). From the scaling behaviour of the current-voltage curves for devices with different areas, we deduce that carrier diffusion is largely suppressed in QD LEDs (diffusion length $< 100 \text{ nm}$ at room temperature), as opposed to QW LEDs (diffusion length $2.7 \text{ }\mu\text{m}$). This shows the possibility of isolating single QDs in the active region of an LEDs, in order to achieve an electrically-pumped single-photon emitter. Recent results on integrating a high-Q 3D optical microcavity with the nanoscale current injection will also be described.

Heteroepitaxial growth instabilities on Si and their modelling by kinetic Monte Carlo simulations

Friedrich Schaffler¹⁾

1) *Institut für Halbleiterphysik Universität Linz, Altenberger Str. 69, Linz A-4040, Austria*

The gate lengths in a state-of-the-art Pentium IV processor are now at 65nm and will shrink to below 20 nm in the next 10 years. At these nanoscale lengths further downscaling of critical device dimensions encounters several severe physical limitations, such as the onset of quantum effects. But also technological limitations become more and more important.

Here we report on a recently discovered phenomenon that is expected to be of significant relevance for the processing of future Si-based heterodevices with nanoscale dimensions. It is an intrinsic homoepitaxial growth instability on the Si(001) surface [PRL 83, 995 (1999)], which leads to the kinetic formation of surface undulations with amplitudes of several nm and quasi-periods of several 100 nm. Although such morphological instabilities may ultimately be exploited as self-organization phenomena, they are certainly detrimental to the properties of Si/SiGe heterointerfaces, which will become important for future high-speed devices.

We report on a systematic set of experiments to determine the dependence of the step-bunching growth instability on the deposition and wafer-miscut conditions. We found a very pronounced temperature dependence, with the maximum of the ripple formation overlapping the range of growth parameters frequently employed during standard molecular beam epitaxy of Si on Si(001). We also determined the critical exponents for the ripple amplitude and period as a function of the deposited layer thickness.

To identify the dominating mechanism, we performed kinetic Monte Carlo simulations utilizing a simplified model for the $(2 \times 1)/(1 \times 2)$ reconstructed Si(001) surface. It was found that the diffusion anisotropy of this reconstructed surface in connection with the difference of the binding energies at SA and SB steps is the main mechanism driving the kinetic growth instability. The complex adsorption/desorption kinetics at these two principal types of step edges behaves like an effective inverse Ehrlich-Schwoebel barrier, which causes the step-bunching instability of the Si(001) surface [Surf. Sci. 520, 193 (2002)].

Kinetic step bunching on Si(001) has long been associated with the strain induced by a heteroepitaxial SiGe epilayer. By performing a wide variety of experiments with Si homo- and SiGe heteroepitaxial layers and superlattices, we can rule out that strain plays any relevant role in the formation of the step bunches. On the contrary, SiGe layers tend to reduce the kinetic step bunching effect, while close to thermal equilibrium they disintegrate into Stranski-Krastanov islands on a thin wetting layer [Surf. Sci. 532 - 535, 721 - 726 (2003)]. The formation of (one-dimensional) step bunches is not an energetic favorable mechanism near thermal equilibrium and is thus of purely

11:45 - 12:30

invited oral

kinetic origin [Phys. Rev. B 64, 041301(R) (2001)]. This finding allows for a complete suppression of step bunching on Si(001) by choosing growth parameters close to thermal equilibrium. This is an important prerequisite for SiGe heterostructures with a low amount of interface roughness scattering.

12:30 - 14:30

Lunch break

14:30 - 15:30

Afternoon session

Main Building, room 213

14:30 - 14:45

Indium segregation effects in InGaAs/GaAs multiple QW heterostructures grown by MOCVD

oral **R. Akchurin³⁾, A. Y. Andreev³⁾, O. I. Govorkov¹⁾, A. A. Marmalyuk²⁾, A. V. Petrovsky²⁾, I. D. Zalevsky²⁾**

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Indium segregation effects during InGaAs/GaAs multiple QW heterostructures formation by MOCVD have been investigated. Epitaxial growth of heterostructures was carried out at 720-770C under low-pressure (~ 60 Torr) using GaAs (100) substrates. TEGa, TMin and AsH₃ were used as Ga, In and As sources, respectively. Indium concentration profiles were analyzed by high-resolution Auger-electron spectroscopy. Asymmetry of indium concentration profiles and non-identical of its content in the first grown close spaced QWs was revealed by experimental study. Two main reasons of the observed phenomenon are discussed: the influence of elastic stresses induced by lattice misfit and indium accumulation near the crystallization surface. The calculation model for simulation of In concentration profiles in single and multiple QW heterostructures and results of calculation are presented.

14:45 - 15:00

Selective epitaxial growth of Ge/Si quantum dots on patterned Si(001) substrate by ultra high vacuum chemical vapor deposition

oral **L. H. Nguyen¹⁾, V. LeThanh²⁾, D. Debarre¹⁾, V. Yam¹⁾, M. Halbwax¹⁾, D. Bouchier¹⁾**

1) Institut d'Electronique Fondamentale, UMR-CNRS 8622, 91405 Orsay Cedex, France, France

2) Centre de Recherche sur les Mecanismes de la Croissance Cristalline (CRMC2), Marseille 13009, France

In recent years, the growth of Ge/Si quantum dots (QD) has received growing interest due to the possibility for the realisation of novel QD-based electronic and optoelectronic devices. The most widely used technique to produce Ge/Si QD is based on strain-induced growth mode transition from two-dimensional (2D) to islanding growth (3D) in a highly lattice-mismatched heteroepitaxial system (Stranski-Krastanov growth). However, in view of applications of such quantum dots one of the major drawbacks is their random distribution over the substrate surface.

In this work, we study selective epitaxial growth (SEG) of Ge QD on patterned Si(001) substrates. The formation of Ge/Si QD both in single and stacking layers will be reported. For a single layer, it is shown that the growth of Si buffer layers can greatly affect the formation of Ge/Si QD. By controlling the height of the Si buffer layers, we can obtain only one dot per circular window and a high cooperative arrangement of dots on a striped window with a constant period of 80 nm. In stacking layers, we show that the vertical ordering and also the dot homogeneity can be controlled through the adjustment of the Si spacer-layer thickness.

The optical properties of these structures measured by photoluminescence spectroscopy (PL) will be also reported. In comparison with self-assembled QD, we observed, both in single and stacking layers, the absence of the wetting-layer component, confirming therefore the dot formation by selective growth. First electrical characterizations by means of current-voltage (I-V) measurements of Schottky diodes in the temperature range between 95 and 300K will be presented.

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15:00 - 15:15

Formation of Ge nanoislands on pure and oxidized Si surface by MBE

oral **A. Nikiforov¹⁾, V. V. Ulyanov¹⁾, A. G. Milekhin¹⁾, O. P. Pchelyakov¹⁾, S. A. Teys¹⁾, S. Schulze²⁾, D.R. T. Zahn²⁾**

1) Institute of Semiconductor Physics SB RAS, Lavrentjeva 13, Novosibirsk 630090, Russia, Russian Federation

2) Institut für Physik, Technische Universität Chemnitz, D-09107 Chemnitz, Germany, Germany

Phenomena of nanoislands self-organization are of interest to several areas of solid-state physics. Among these objects is the "Ge on Si" heterosystem with Ge nanoislands behaving as quantum dots. The smallest germanium

islands growing on the pure silicon surface are 15 nm in size. They can be lowered by growing germanium on the oxidized atomically pure surface prepared in situ in the MBE installation. A MBE installation "Katun-C" was used for synthesis on Si (100) substrates. An MBE growth chamber was used for oxidation at oxygen supply up to 10-4 Pa and the substrate temperature 400-500°C.

The process of Ge film growth was controlled using a RHEED patterns registrations. These observations indicate the absence of the stage of wetting layer formation. Therefore, germanium film growth on the oxidized silicon surface by the Volmer-Weber mechanism but not by the Stranski-Krastanow mechanism, which is characteristic of the growth of pure Si surface. Deformations of germanium lattice were studied by measuring in plane lattice constant a of the surface 2D unit cell. The film growth results in changes in the surface cell constant of Ge lattice against the constant of Si lattice. The difference may reach 7% as reported for the growth on pure Si(100) surface. The mode of parameter variations is similar to that observed during germanium heteroepitaxy on the pure Si(100) surface. The maximal elastic deformation occurs already in the 3D island nuclei. Depending on the thickness of the deposited Ge layer, the islands are different in size and density. At the film not thicker than 5 ML, the islands on oxidized surface are less than 10 nm in base size at the density higher than $2 \times 10^{12} \text{ cm}^{-2}$.

The work is supported by the Russian Foundation for Basic Research (Grants 03-02-16468, 03-02-16506 and 02-02-17746) and INTAS (Grant 01-0444).

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Coffee break

15:30 - 15:50

Poster session

19:00 - 21:00

Thursday, September 18th

Morning session

09:00 - 10:30

Main Building, room 213

Self-assembled semiconductor nanostructures

9:00 - 9:45

Oliver G. Schmidt¹⁾

1) Max-Planck-Institut FKF, Heisenbergstr. 1, Stuttgart D70569, Germany

invited oral

In this talk I review recent results of our group on self-assembled islands in the SiGe and InAlGaAs material system. The first part of my talk focuses on material intermixing during self-assembled island growth and on the fabrication of compact lateral quantum dot molecules as well as unstrained red-light emitting GaAs/AlGaAs quantum dots with inhomogeneous linewidths of 11 meV. The second part is devoted to the ability to laterally align self-assembled quantum dots in single and multiple layers.

We use quantum dot (QD) growth and atomically precise in-situ etching to fabricate novel quantum dot structures. The key procedure to create such structures comprises an in-situ etching process with AsBr₃ of GaAs capped InAs quantum dots. The AsBr₃ preferably etches away those regions of the GaAs, which covers the buried InAs QDs, thus causing the formation of nanometer sized holes. If these holes are overgrown with InAs again, laterally closely spaced groups of two, three, four, five, and six QDs (lateral quantum dot molecules) form around the nanoholes.

In another approach, we overgrow the nanoholes with AlGaAs/GaAs/AlGaAs. The hole morphology is preserved by the first AlGaAs growth step, whereas the GaAs preferably diffuses into the AlGaAs holes, thus forming inverted unstrained GaAs/AlGaAs quantum dots that emit in the visible wavelength regime. The GaAs/AlGaAs quantum dots show excellent optical properties including well-resolved ground and excited states with a linewidth of only 11 meV at low temperature.

Furthermore we grow In(Ga)As quantum dots on GaAs (001) substrates patterned with square arrays of nanometer sized holes. We choose the growth conditions in such a way that a perfect lateral order of InGaAs QDs occurs. We study the effect of lateral alignment as a function of stacked layers and array periodicity. In perfectly ordered arrays we observe the formation of quantum dots on second order strain energy minima.

Strain effects in heteroepitaxy of nitrides

9:45 - 10:30

Sven Einfeldt¹⁾

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

invited oral

Strain and stress are dominant features of nearly all epitaxial layers and heterostructures made out of group-III nitrides. This is mainly for the following three reasons: (1) the lattice mismatch between GaN, AlN and InN, (2) the mismatch in the coefficients of thermal expansion

between the layers and the substrate, and (3) the large and inhomogeneous density of extended defects in nitride layers. A comprehensive picture of the strains in nitride heterostructures is essential for the fabrication and optimization of most devices. In this paper, we will discuss the three main sources of strain mentioned above by means of individual examples. First, the strain variation in GaN layers grown on SiC substrates is discussed with respect to the lattice mismatch between the GaN and the Al(Ga)N buffer layer, the morphological evolution during growth and the occurrence of a phase separation in the buffer layer. Second, the inhomogeneous stress and crystallographic orientation in GaN layers grown by either pendeo-epitaxy on SiC or cantilever-epitaxy on Si substrates are analyzed in view of the thermally induced load after growth. Third, the application of SiN interlayers to reduce the dislocation density in GaN layers and the impact of these layers on the strain in GaN is presented.

10:30 - 11:00

Coffee break

11:00 - 12:30

Morning session - continued

Main Building, room 213

11:00 - 11:45

Relaxation of misfit-induced strains - from handicaps to benefits

Horst P. Strunk¹⁾

invited oral

1) Institute of Microcharacterization, University Erlangen, Cauerstr. 6, Erlangen 91058, Germany

Growth and processing of crystalline materials is in general accompanied by the creation of strain and stress, be it due to thermal gradients during cooling and heating, be it induced by intended structural processing in growth and device production. The latter is especially important to take into account because of the recent rise of large band gap semiconductor materials. For example, SiC as a material for high temperature and power devices, is not yet available as single crystals without lattice defects, although established growth procedures exist. Even worse, the group-III-nitrides cannot be grown as bulk single crystals presently, instead they have to be grown on foreign substrates that cause misfit and differential thermal stress.

This presentation gives an overview over various processes that are available to crystals to relax stress. We consider elastic, plastic and diffusive relaxation using examples from the rather well investigated epitaxial systems of the Si-Ge semiconductors group. We will then discuss current problems in III-nitride- and SiC-growth.

11:45 - 12:30

Modification of magnetic properties of ultrathin cobalt layers by an overlayer structure

invited oral

Marek Kisielewski²⁾, Zbigniew Kurant¹⁾, Andrzej Maziewski²⁾, Maria Tekielak²⁾, Andrzej Wawro¹⁾, Andrius Maneikis^{1,3)}, Lech T. Baczewski¹⁾

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3) Vilnius Gediminas Technical University, Sauletekio al. 11, Vilnius, Lithuania

New approach to modify the magnetic properties of ultrathin Co layers by introducing a special structure of coverage layer have been recently discovered and reported [1]. The double-wedge samples with the following structure: (i) monocrystalline sapphire substrate covered with Mo and subsequently with Au(111) buffer layer (ii) Co wedge with the thickness d increasing from 0 to 2 nm along "x" axis; (iii) a wedge of coverage metal A ($A=Au, Ag, Mo, Cr$) with the thickness h increasing from 0 to few nm along "y" axis; and (iv) a cap layer of metal B ($B=Au, Ag$), were grown by MBE technique. They were studied by polar Kerr effect in two experimental setups: (i) millimagnetometer with red laser beam, which locally probes the sample area of millimeter range size; and (ii) micromagnetometer based on an optical microscope. A strong influence of the overlayer designed geometry on both magnetic anisotropy and coercive field has been found. Assuming the exponential dependence $\exp(-h/h^*)$ of the spatial distribution of both parameters on silver thickness h , one can calculate h^* equal to about only 1 nm for both parameters. The size of new "chemical" domain wall constrained by the silver overlayer is theoretically analyzed, showing the possibility of wall width tuning in a wide range by changing the gradient of h . Strong modification of magnetic domain structure is observed in two regions of the sample: (i) high d , where spin reorientation transition from perpendicular to in-plane direction occurs in Co layer and (ii) low d , where magnetization remanence becomes negligible. In these two regions the fractal dimension of the domain wall, as well as the density of nucleation centers, show large increase in comparison to the in-between located region.

This work was supported by the Polish State Committee for Scientific Research (Grant No. 4 T11B 006 24) and

partly by the EU "QMDS" project, contract No. HPRN-CT-2000-00134. [1] M.Kisielewski et al. Phys. Rev. Lett. 89, 8 (2002) 87203

Lunch break

12:30 - 14:30

Afternoon session

14:30 - 15:30

Main Building, room 213

Spatial Profile of Neutral Free Radical Beam Produced by the Method of Photo-Deionization of Negative Ion Beams

14:30 - 14:45

Keiji Hayashi¹⁾, Hiroshi Konno¹⁾, Takuo Kanayama¹⁾, Takashi Oseki¹⁾

oral

1) Advanced Materials Science R&D Center, Kanazawa Institute of Technology (AMS, KIT), 3-1, Yatsukaho, Matto, Ishikawa, Kanazawa 924-0838, Japan

In order to further develop integrated quantum-functional devices it has become indispensable in recent years to ingeniously utilize selective surface reactions of neutral free radicals for the device processing. The problem encountered in the experimental study of a chemical reaction between a neutral free radical and a well-characterized material surface is how to sufficiently supply a steady-flux refined beam of neutral free radicals (RBNR), that is to say, purified beam of momentum-controlled neutral free radicals onto the surface. In order to overcome the difficulty we have proposed several experimental production approaches¹⁾, one of which is the method of photo-deionization of negative ion beams (PDINIB). When we apply RBNR to thin film growth, a steady-flux beam obtained using a CW laser is more useful than a pulsed beam. However, the power of available CW lasers is generally much weaker than the peak power of pulsed lasers. Thus, development of a multiple-pass photo-deionizer (MPDI) to enhance the photo-neutralization efficiency has been a key point to realize a practical steady-flux PDINIB apparatus. The rate of neutral free radical production by our trial PDINIB apparatus is estimated in Ref.2 based on measurement of decrease in the negative-ion beam current (DNIC).

In the present study, we could improve the S/N ratio and the spatial resolution of the DNIC measurement system 40 times and 7 times, respectively. This improved monitoring system was used for measurements of the spatial profile of the neutral-beam flux under various conditions of the neutral-beam production. At the meeting, we discuss the beam-profile controllability of the trial PDINIB apparatus based on the experimental results.

This work is supported by the Ministry of Education, Science, Sports and Culture, Japan.

1) K. Hayashi et al., J. Vac. Sci. Technol. A 20 (3), 991 (2002).

2) K. Hayashi et al., Nucl. Instrum. Methods Phys. Res. B 206C, 403 (2003).

Self-assembled Ge/Si(001) islands extending the accessible wavelength region beyond 2 μm

14:45 - 15:00

Mathieu Stoffel¹⁾, U. Denker¹⁾, G. S. Kar¹⁾, O. G. Schmidt¹⁾, H. Sigg²⁾

oral

1) Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70569 Stuttgart, Germany, Germany

2) Paul-Scherrer-Institut, CH-5232, Villingen, Switzerland, Switzerland

Low temperature capping of self-assembled Ge islands was investigated by means of photoluminescence (PL) spectroscopy. The island related PL signal systematically redshifts when the capping temperature decreases [1]. For substrate temperatures below 700°C during island growth, the emission energies are lower than the usual values reported in the literature (0.75-0.9 eV). By applying low temperature capping to Ge islands grown at different temperatures, we show that the Ge island emission wavelength can be extended up to 2.06 μm . This is related to an increased Ge content in the islands. The longest emission wavelength is obtained for hut clusters grown at 400°C which consist of pure Ge. By further decreasing the growth temperature to 360°C, the emission energy increases again. This result is explained by enhanced charge carrier confinement in extremely small Ge quantum dots.

References:[1] U. Denker, M. Stoffel, O.G. Schmidt, H. Sigg, Appl.Phys. Lett. 82, 454 (2003)

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3C-SiC:Ge alloys grown on Si (111) substrates by solid source MBE

15:00 - 15:15

P. Weih¹⁾, V. Cimalla¹⁾, Th. Stauden¹⁾, R. Kosiba¹⁾, L. Spiess¹⁾, H. Romanus¹⁾, M. Gubisch¹⁾, W. Bock²⁾, Th. Freitag³⁾, P. Fricke³⁾, Oliver Ambacher¹⁾, J. Pezoldt¹⁾

oral

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3) 3ISIMS-Labor, Institut Fresenius, Zur Wetterwarte 10, Haus 107, 01109 Dresden, Germany

One disadvantage of SiC for applications in electronic devices is the relative low carrier mobility. The creation of SiC:Ge alloys provide the opportunity to improve the electronic properties in comparison to SiC epitaxial layers. The disadvantage of this system is the relatively low thermodynamical stability and the immiscibility of Ge in SiC. As a consequence nonequilibrium material synthesis methods must be applied to achieve 3C-SiC:Ge alloys. In this work for the first time a comprehensive research of SiC:Ge thin films grown on Si (111) substrates by solid source molecular beam epitaxy (SSMBE) will be presented. The layers were grown at substrate temperatures ranging from 1173 K to 1313 K with a growth rate of 0.6 nm/min. The 120 nm thick layers were characterized by x-ray diffraction (XRD), Auger electron spectroscopy (AES), secondary ion mass spectroscopy (SIMS) and transmission electron microscopy (TEM). The structural analysis revealed that 3C-SiC:Ge alloys were grown. SIMS and AES measurements detected a Ge content of around 1 % and a significant Ge segregation at the SiC/Si interface (Fig.1). The dependence of Ge incorporation on growth temperature as well as on the strain in the SiC:Ge alloys will be discussed.

15:15 - 15:30

oral

Kinetics of heteroepitaxial growth of Ge on Si(001) at low temperature by UHV-CVD

M. Halbwax¹⁾, V. Yam¹⁾, D. Debarre¹⁾, Lam.H. Nguyen¹⁾, Y. Zheng²⁾, D. Bouchier¹⁾

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In a next future, optical interconnects will offer a solution to the limitation by metallic interconnects of working frequency of microprocessors. Micro-waveguides processed on SOI with integrated Ge photodetectors appear as an attractive option. However, the growth of a 0.4 μ m thick fully relaxed Ge film on Si with a low density of threading dislocations remains a challenge. The usually observed Stranski-Krastanov transition can be avoided by using a very low temperature deposition [i] which minimizes the Ge diffusion and favors the formation of vacancies. In situ RHEED was used to investigate the relaxation kinetics during the low temperature deposition. The Ge has been grown on Si at 330C by UHV-CVD at a total pressure of 1.8 10⁻³ Torr of hydrogen-diluted germane.

During the first 15 min. the surface was found to turn from flat to wavy, the RHEED pattern showing modulated streaks and weak 1/2 lines. For further deposition times, the surface became flat. After deposition of 45 nm of Ge (1 hour growth time), the surface exhibited a perfect 2x1 reconstruction. The variation of lattice parameter was measured as the growth proceeded and was found to increase by 2.5% after 1 hour, what is significant of a partial relaxation. In order to confirm the RHEED findings, the film roughness was measured by AFM for different deposition times. From transmission electron microscopy measurements, misfit dislocations were found to be mostly located at the interface, and emerging extended defects were identified as stacking faults. From that, it appears that the low deposition temperature process is well suited for the deposition of a thin relaxed Ge film.

[i] L.Colace et al, J.Vac.Sci.Technol.B. 17, 465 (1999).

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15:30 - 15:50

Coffee break

Friday, September 19th

09:00 - 10:30

Morning session

Main Building, room 213

9:00 - 9:45

invited oral

In situ monitoring and control optical techniques during MBE growth of III-V nanostructures

Fernando Briones¹⁾

1) Instituto de Microelectronica de Madrid (CNM-CSIC), Madrid, Spain

One of the advantages of the MBE growth process is its compatibility with a variety of in-situ monitoring and control techniques. In addition to the most extended RHEED technique, optical techniques can be easily implemented through appropriate windows of the UHV system providing relevant complementary information on the growth front.

Some of these techniques are standard such as IR pyrometry and spectral reflectivity measurements. In this talk, we will refer only to some less extended but very useful techniques that have been implemented in our laboratory in a simple and practical way: Reflectance Anisotropy (RA), Laser Light Scattering (LLS) and Laser Deflection Stress Monitoring (LDSM). Description of the principles and experimental set-ups will be followed by examples of

application to various growth processes, in particular:

- Controlling stoichiometry of low temperature InP growth using RA
- InAs/InP Quantum Wire self-assembling monitoring by LDSM
- In segregation effects during InAs/GaAs QDs formation and capping
- Surface roughness control during growth of relaxed InGaAs/GaAs layers by LLS and LDSM

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1. A new in situ III-V surface characterization technique: chemical modulation spectroscopy, P.A. Postigo, T. Utzmeier, G. Armelles and F. Briones. *Journal of Crystal Growth*, 175(1997) 298-303
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5. A growth method to obtain flat and relaxed In_{0.2}Ga_{0.8}As on GaAs (001) developed through in-situ monitoring of surface topography and stress evolution, M. U. González, Y. González, L. González, M. Calleja, J.P. Silveira, J.M. García, y F. Briones. *Journal of Crystal Growth* , 227/228, (2001) 36-40.
7. InAs/InP (001) quantum wire formation due to anisotropic stress relaxation: in situ stress measurements, J.M. García, L. González, M.U. González, J.P. Silveira, Y. González, y F. Briones, *Journal of Crystal Growth* 227/228 (2001). 975-979.
8. In situ measurements of As/P exchange during InAs/InP(001) quantum wires growth, M.U. González, J.M. García, L. González, J.P. Silveira, Y. González, L.D. Gómez, F. Briones. *Applied Surface Science* 188, 188-192 (2002).
8. Limited In incorporation during pseudomorphic InAs/GaAs growth and QD formation observed by in-situ stress measurements, J.P. Silveira, J.M. Garcia and F. Briones, *Appl. Surf. Science*, 188, 75-79 (2002)

Real-Time in situ x-ray diffraction studies on ZnSe epilayers grown on (001)GaAs

Armin S. Bader

9:45 - 10:30

invited oral

With real-time in situ x-ray diffraction (RIX) we developed a novel method to monitor strain and strain relaxation during epitaxial growth. In our setup an extremely asymmetric 113 reflection is measured using divergent X-rays. A CCD camera serves as two-dimensional detector. Thus we are able to obtain the vertical lattice constant - which is otherwise typically measured by a θ - 2θ scan - without rotating either the sample or the detector. Since the measurement time is just a few seconds, we can take time-resolved spectra which result in a relative thickness resolution in the order of 1nm.

By using a Johansson-Monochromator, we obtain convergent monochromatic X-rays focussed onto the sample. This setup provides for instance direct access to the layer thickness and hence the growth rate through the analysis of finite thickness fringes. By translating a slit through the convergent primary X-ray beam, we can separate small angular segments from the convergent beam which corresponds to a conventional ω scan. Using a stationary setup with only one moving slit, we are able to record reciprocal space maps with RIX within minutes.

We apply RIX to monitor the epitaxial growth of ZnSe on (001)GaAs and the relaxation process during growth, post-growth annealing and a cooling phase. The critical thickness of the epitaxial layer and its thermal evolution was determined and will be discussed. Inhomogeneous strain relaxation during growth and considerable relaxation during cooling down are some examples for processes that occur during heteroepitaxy. They become accessible by the novel RIX analysis tool.

Coffee break

10:30 - 11:00

Morning session - continued

11:00 - 12:30

Main Building, room 213

TEM evaluation of stress and strain in III-V structures

Andre Rocher¹⁾, Martiane Cabie¹⁾, Anne Ponchet¹⁾

11:00 - 11:45

1) CEMES, CNRS, BP 4347, F-31055 Toulouse, France, France

invited oral

The stress and strain induced by a lattice mismatch between an epilayer and its substrate are known to control most of physical properties valuable for (opto)-electronic components. In good conditions of epitaxy and when no

extended defects are seen, thin layers are considered as perfect, i.e. homogeneous and monocrystalline. This result is based on crystalline perfection of the structure: i) a sharp interface corresponding to a change of the chemical composition on one atomic plane; ii) an ideal film where all the deposited atoms take a strained position according to the elasticity theory. Within these conditions, the classical model is well established with applied strain fields directly defined by the continuity of the atomic planes through the interface. From this approach, below the critical thickness, 10 nm for 1% lattice mismatch, an epilayer is considered as perfectly strained. This representation need to be verified experimentally. Classical methods of the strain field analysis are performed by diffraction techniques and the stress field is deduced from the strain using the standard elasticity theory. The Transmission Electron Microscope (TEM) is a tool well adapted for this evaluation.

In this presentation the epitaxial strain field has been determined on X-HREM micrographs using numerical moire technique and on plan view sample using the conventional moire method. Good results are obtained for relaxed systems with misfit dislocations.

A new method of analysis of stress in epitaxial structures is described. It has been developed for determining the stress induced by a lattice mismatch in specimens such as $\text{Ga}_{0.8}\text{In}_{0.2}\text{As}/(001)\text{GaAs}$ (about 1% lattice mismatch with a critical thickness of about 10 nm). Specimens prepared for plan view TEM observations appear to be bent with a radius of several tens of μm for a substrate thickness t_s of about 0.1 to 0.3 μm , an epilayer thickness t_l of 10 nm. In these conditions the ratio t_l/t_s , which varies between 0.04 to 0.1, agrees well for determining the stress from the experimental measurement of the radius of curvature R and the Stoney formula corrected for high value of t_l/t_s . The measured misfit stress is 1 GPa, about 30% lower than the theoretical stress for the pseudomorphic structure.

The conventional representation of pseudomorphic systems based on Vegard law and the homogeneity of the materials will be discussed. In the case of ternary compounds, many physical and chemical phenomena (segregation, heterogeneous nucleation, ...) are activated during the first stages of epitaxial growth. They create a transition zone where both the crystalline structure and the chemical composition are heterogeneous. This heterogeneity is a direct manifestation of relaxation of the misfit stress and constitutes a failure as far as the concept of the critical thickness based on the crystalline perfection of the substrate, the epitaxial layer and their interface is concerned.

MOVPE growth and in situ characterization of GaN layers

Hilde Hardtdegen¹⁾

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Epitaxial growth of GaN layers is a difficult task due to the lack of lattice and thermal expansion matching substrates to GaN. Usually sapphire or SiC and lately also Si (111) wafers are employed with a lattice mismatch of 16, 3.5 and -17 %, and with a thermal mismatch of -34, 25 and 54 %, respectively. Therefore deposition strategies need to be developed which help compensate the mismatch in order to obtain high quality GaN templates for device structures. In this paper first the chemistry of the GaN growth process will be discussed. Then the growth procedure used in MOVPE will be presented. It will be shown how growth parameters influence the characteristics of the layers and how in situ characterization techniques can be used for growth monitoring and optimization. At last methods will be introduced which show how modeling and emission corrected pyrometry can be employed to increase the reproducibility of the growth process.

Closing address - J. Kossut

Main Building, room 213

11:45 - 12:30

invited oral

12:30 - 12:40

Symposium D

Colossal Magnetoresistance – New Materials and New Ideas

Organisers

Symposium D is co-organized by the Institute of Physics of the Polish Academy of Sciences as a part of activities of the Centre of Excellence CELDIS, and by Committee of Physics, Polish Academy of Sciences.

- **Henryk Szymczak**, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
- **Hanns-Ulrich Habermeier**, Max-Planck-Institut FKF, Stuttgart, Germany
- **Igor Troyanchuk**, The Institute of Solid State & Semiconductor Physics, National Academy of Science of Belarus
- **Ricardo M. Ibarra**, Universidad de Zaragoza-CSIC, Facultad de Ciencias Pedro Cerbuna, Spain
- **Eleni Gamari-Seale**, Institute of Materials Science "Demokritos", Athens

Proceedings

Symposium proceedings will be published in *Acta Physica Polonica A*.

Monday, September 15th

13:55 - 14:00

Opening address

Main Building, room 144

14:00 - 15:30

Afternoon session

Main Building, room 144

14:00 - 14:45

Strain effects in manganite thin films**Jan Aarts¹⁾, Z Q. Yang¹⁾, Y Qin¹⁾, M Y. Wu¹⁾, H W. Zandbergen¹⁾**

invited oral

1) Kamerlingh Onnes Laboratory, Leiden University, Niels Bohrweg 2, Leiden 2333 CA, Netherlands

The properties of manganite thin films strongly depend on both strain and disorder. Pure strain effects are difficult to distill, and therefore also strain engineering is difficult to perform. Moreover, the perovskite structure allows a large number of modifications, and in ultrathin films, the structure may even depend on the deposition technique. We discuss two different examples of the effects of strain. The first is in ultrathin films of $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ sputter-deposited on SrTiO_3 where, below 5 nm, we find a tetragonal rather than an orthorhombic structure, and the absence of a metal-insulator transition. The second is in thin films of $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$, also sputter-deposited on SrTiO_3 . Here we find that the charge ordering temperature T_{co} has increased significantly with respect to the bulk value. Preliminary results also indicate that T_{co} in the films is different from the orbital ordering temperature T_{oo} , contrary to the findings in bulk materials where both types of ordering are linked and occur at the same temperature.

14:45 - 15:30

Acoustical and related effects in manganite compounds.**Konstantin Dyakonov¹⁾, Alexander Goltsev¹⁾, Ludmila Kulakova¹⁾, Eduard Yakhkind¹⁾, Vladimir P. Dyakonov²⁾, Stanislaw J. Lewandowski²⁾, Henryk Szymczak²⁾, Yakov Mukovskii³⁾**

invited oral

*1) Ioffe Physico-Technical Institute (Ioffe), Polytechnicheskaya, 26, St.-Petersburg 194021, Russian Federation**2) Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland**3) Moscow State Steel & Alloys Institute, Moscow 117936, Russian Federation*

The studies of manganites have shown that strong electron-phonon interaction plays important role in the formation of CMR effect discovered in these compounds. Due to electron-phonon interaction acoustical techniques are sensitive tools for investigations of transport and structural properties of manganites.

In this report we present studies of the surface acoustic wave (SAW) attenuation and the acoustoelectric (AE) effect in the $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ (LCMO) film. We measured the SAW attenuation at 30 MHz, and at higher harmonics of this frequency. The attenuation reaches a maximum at the temperature corresponding to the peak of the film resistivity, and increases with increasing SAW frequency. The applied magnetic field reduces the attenuation. The observed behavior in the paramagnetic state of the LCMO film is in a good agreement with the relaxation type theory of the sound attenuation in conducting film. Frequency dependence of the SAW attenuation gives evidence for the linear dependence of the conductivity which probably points to a significant role of the spin disorder in the electronic transport in manganites.

The investigations of the AE effect produced by the SAW in the LCMO film have revealed an anomalous (even in the SAW direction of propagation) contribution to the acoustoelectric current, which coexists with the ordinary (odd) effect. The anomalous effect appears to depend on strong modulation of the film conductivity resulting from elastic deformations carried by the SAW. The magnetic field increases the magnitude of the anomalous AE effect while the ordinary effect is left essentially unchanged. The measurements with the SAW split into two components, propagating in the LCMO film in opposite directions, result in cancellation of the ordinary AE effect and augmentation of the anomalous one, thus confirming our understanding of the anomalous AE effect in manganites.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duża aula)

15:50 - 17:20

Afternoon session - continued

Main Building, room 144

15:50 - 16:20

Phase separation and microwave response of epitaxial and polycrystalline

oral

manganite films

Steponas Asmontas^{1,2)}, Adulfas Abrutis³⁾, Jonas Gradauskas^{1,4)}, Andzej Lucun¹⁾, Antanas Oginskis¹⁾, Valentina Plausinaitiene^{1,3)}, Algirdas Suziedelis^{1,4)}, Bonifacas Vengalis^{1,4)}

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3) *Vilnius University, Department of General and Inorganic Chemistry, Naugarduko 24, Vilnius LT-2006, Lithuania*

4) *Vilnius Gediminas Technical University, Sauletekio al. 11, Vilnius LT-2040, Lithuania*

Rare earth manganites, $\text{La}_{1-x}\text{A}_x\text{MnO}_3$ ($\text{A}=\text{Sr}, \text{Ca}, \dots$), are of great interest due to a variety of physical phenomena such as charge and orbital ordering, electronic phase separation, spin-polarized electronic transport, intrinsic and extrinsic magnetoresistance (MR), e.t.c. The highest crystalline quality manganite films demonstrate colossal magnetoresistance meanwhile polycrystalline films were found to exhibit low-field MR due to spin-polarized tunnelling between misaligned grains.

Microwave (MW) radiation has been demonstrated as a useful tool for investigation of the origin of MR in the manganites. Our recent study of resistance response of the LCMO thin films induced by MW radiation had obviously shown the dynamical phase coexistence in the material just below the semiconductor-to-metal phase transition.

Here in this work we present the succeeding experimental study of LCMO and LSMO thin films of both epitaxial and polycrystalline quality prepared either by MOCVD and magnetron sputtering on various (NdGaO_3 , LaAlO_3 and MgO) substrates. The resistance response under MW radiation ($f=10$ and 35GHz) as well as their resistance and MR were measured in the temperature range 78-300 K. The electrical resistance response measured for LCMO and LSMO films of epitaxial quality confirmed the phase separation in a narrow temperature range below the PM-FM transition temperature T_c . We point out significant increase of the resistance response with frequency for the films. Meanwhile, the resistance response of polycrystalline films has been indicated in a significantly wider temperature range (from T_c down to 78 K). It depended strongly on preparation conditions and microstructure of the material. A model based on nonuniform heating and formation of insulating regions in the strain fields of grain boundaries has been developed to explain the observed peculiarities of magnetoresistance and MW induced response for the polycrystalline manganite films.

[ABSTRAC

[ABSTRA

[ABSTRACT TRUNCATED TO 2000 LETTERS]

Mesoscopic ordering effects in single-crystalline thin films of lanthanum manganites

16:20 - 16:50

Zinaida A. Samoilenko^{1,2)}, Vladimir D. Okunev^{1,2)}, I. Pushenko²⁾, A. Abal'oshev¹⁾, M. Baran¹⁾, Piotr Gierlowski¹⁾, S. J. Lewandowski¹⁾, Henryk Szymczak¹⁾, R. Szymczak¹⁾

oral

1) *Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland*

2) *A. Galkin Donetsk Institute for Physics & Technology, National Academy of Sciences of Ukraine (DonPTI NASU), Roza Luxemburg 72, Donetsk 83114, Ukraine*

We have investigated thin films of lanthanum manganites epitaxially grown by pulsed laser deposition on single-crystalline substrates. X-ray diffraction (XRD) studies show that on the axial line of Laue pattern simultaneously occur bright well-expressed reflexes, due to coherent scattering of X-rays by planes parallel to the film surface exhibiting perfect long-range order, and weak smudged reflexes caused by coherent diffusive scattering of X-rays on fragmented planes with mesoscopic ordering. It appears that the characteristic feature of the investigated films is a superposition of coherently coupled fractions with differently scaled atomic order: long range order in the basic single-crystalline matrix, and mesoscopic order in the clusterized structure encompassing the Mn-O layers where the long-range order is disturbed by sequentially changing regions of order (clusters) and disorder (cluster/matrix interfaces). Varying the film growth parameters induces substantial changes in the matrix and in the cluster structure. The fact that cluster boundaries are smeared facilitates local changes of atomic order. Detailed analysis of diffusive scattering shows that the size of metallic clusters in the dielectric matrix varies from 40-70 Å to 140-200 Å, and their volume concentration from 1-10% to 20-50%. In the first case (small size and concentration), the metallic clusters in low temperature behave as quantum dots and their presence leads to tunneling mechanism of conductivity with characteristic $R(T)=\text{const}$ dependence at low enough temperatures. In the second case (large cluster size), the discrete energy levels become smeared and at high enough cluster concentration percolative conduction mechanism takes over. It appears also that the optical, transport and magnetic properties of the investigated films depend critically on the distribution of Mn^{2+} , Mn^{3+} and Mn^{4+} ions in the clusters.

This work was supported by Polish Government (KBN) Grant PBZ-KBN-013/T08/19.

Raman study of anharmonic and charge ordering effects on 'Pr_{0.5}Ca_{0.5}MnO₃' thin films.

16:50 - 17:20

Katerina Tatsi¹⁾, Evie Papadopoulou¹⁾, Dimitris Lampakis¹⁾, Efthimios Liarokapis¹⁾, Wilfrid

oral

Prelhier²⁾, B. Mercey²⁾

1) National Technical University of Athens (NTUA), Heroon Polytechniou 9, Athens 157 80, Greece

2) Laboratoire CRISMAT, CNRS UMR 6508, Bd Marechal Juin, Caen Cedex F-14050, France

We report novel Raman measurements made on 'Pr_{0.5}Ca_{0.5}MnO₃' thin films grown on 'SrTiO₃' and 'LaAlO₃' (PCMO/STO and PCMO/LAO respectively). The measurements were performed for a temperature range 78 K < T < 295 K and under hydrostatic pressures up to 5 GPa. In addition, different scattering polarizations (xx, xz, x'x' and x'z') have been selected for certain temperatures. In order to examine the effect of the strain from the substrate three films of different thickness have been used (50 nm, 100 nm and 180 nm). X-ray diffraction measurements have shown that the PCMO/STO film is [010]-oriented (i.e. with the [010] axis perpendicular to the substrate plane) and the PCMO/LAO film is [101]-oriented due to the lattice mismatch. The measurements have disclosed differences for the high-energy phonons related with the Jahn-Teller distortions of the octahedra. Close to the transition temperature to the charge ordered state, 'T_{CO}', and as the temperature decreases, an increase of the relative intensities of Jahn-Teller modes has been observed. From the different growth orientation of the two films the symmetry of several modes has been identified. No shift or splitting of the modes was observed due to the strains from the different substrates. Furthermore, two rotational modes of vibration of the octahedra were found to exhibit an anharmonic behavior.

Acknowledgments

This work was partially financially supported by the General Secretary of Research and Technology (GSRT) under the project 01EP13.

19:00 - 21:00

Poster session

Main Building, Main Hall (Duza aula)

19:00 - **Eu_{0.55}Sr_{0.45}MnO₃ and Sm_{0.55}Sr_{0.45}MnO₃ manganites. Comparison of properties**

poster **Anna I. Abramovich¹⁾**

1) M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation

D-1

We have studied magnetic, elastic, and transport properties of Eu_{0.55}Sr_{0.45}MnO₃ (1) and Sm_{0.55}Sr_{0.45}MnO₃ (2) ceramic. For (1) the resistivity ρ shows an insulating behaviour even at 4.2 K. Near T_C a break on the $\rho(T)$ dependence and on the temperature dependence of linear thermal expansion was observed for (1) and the sharp maximum on the $\rho(T)$ dependence and sharp volume change $\Delta V/V \sim 0.1\%$ was observed for (2). For (1) in the wide temperature region from 4.2 K to T >> T_C the sharp jump of magnetization σ , volume magnetostriction ω , and ρ takes place at H_{C1} < H < H_{C2}, i.e. under magnetic field action the phase transition from insulating AFM-like state to metallic FM-like state occurs. $\sigma(H)$ curves are not saturated at H > H_{C2} up to 80 kOe. The maximal value of σ is less than one corresponding FM ordering of Mn³⁺ and Mn⁴⁺ ions, namely 70% from latter at 4.2 K under 80 kOe. The metallic FM-like state was unchanged after field removing. Such behaviour is observed within temperature interval from 4.2 to 50 K. At temperatures from 50 to 120 K the jump of σ , ω , and ρ is maintained, but shape of $\sigma(H)$, $\omega(H)$, and $\rho(H)$ curves is changed and sample return in original state after field removing. The value of H_C decreases with increasing T within first and increases within second interval. For (2) sharp jump of σ , ω , and ρ takes place at H_{C1} < H < H_{C2} at T > T_C only, and their saturation is achieved at H > H_{C2}. For (1) MR is positive at H < H_{C1} and reaches of 6% value, at H > H_{C1} MR becomes negative and reaches of 10⁵ %. ω is negative and giant for both compounds and is observed from 4.2 K to T >> T_C for (1) and in the T_C region only for (2). We consider that different types of inhomogeneous magnetic state are realized in our compounds and reason of CMR and giant ω is the increase of FM phase volume under field action.

19:00 - **Structural and magnetic study of self-doped La_{1-x-y}Ca_xVac_yMnO₃**

poster **Vitor S. Amaral¹⁾, F. Figueiras¹⁾, J. P. Araújo²⁾, P. B. Tavares³⁾, J. B. Sousa²⁾, J. M. Vieira⁴⁾**

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D-2

3) Departamento de Química, Universidade de Trás-os-Montes e Alto Douro (UTAD), Vila Real 5001-911, Portugal

4) Departamento de Engenharia Cerâmica e do Vidro and CICECO, Universidade de Aveiro (UA), Campus de Santiago, Aveiro 3810-193, Portugal

To understand the combined effect of divalent ion and A-site vacancy (Vac) (beyond the La_{1-x}MnO₃ system) self doping polycrystalline La_{1-x-y}Ca_xVac_yMnO₃ samples were studied. Samples with excess Mn in the range x < 0.33 and y < 0.45 were prepared by standard solid-state reactions with several final conditions. Structural and phase analysis of the samples were done by X-ray diffraction with Rietveld refinement and High Resolution TEM.

Electrical and magnetic properties were also studied. The manganite structure in the studied composition range includes rhombohedral and orthorhombic phases. Most samples contain haussmanite (Mn_3O_4) phase coexisting with perovskite manganite phase. The relative contents of Mn_3O_4 were determined by calibrating the area ratio of the two main diffraction peaks of each phase. The results were applied to determine the vacancy content y_2 in the manganite phase which was found to accommodate a large fraction of vacancies, beyond $y_2=1/8$, the reported limit for $\text{La}_{1-x}\text{MnO}_3$, which is confirmed in our $x=0$ sample. A large amount of A-site vacancies ($y>0.12$) leads to a strong distortion of the manganite phase. This orthorhombic cell distortion is characterized by an increase of the cell volume (up to about 5%), keeping the shorter a lattice parameter constant, and increasing b and c. This effect is associated with the formation of a vacancy structure ordering with Mn displacement to the A-sites, leading to the perovskite related $\text{AMn}_7\text{O}_{12}$ (A=La,Ca) structure as intergrowth or interleaved planes in the main phase.

For the compositions studied, ferromagnetic metallic behavior below T_c is found, confirming the stability of the Mn sublattice. Near the optimum ($x=0.33$) stoichiometric ($y=0$) system, high vacancy content does not degrade the properties: $y_2 \sim 0.10$ (mainly La vacancies) even leads to an increase of both T_c (by 4K) and MR peak (by 14%). Further $y_2 \sim 0.20$ (Ca and La) gives properties very close to the stoichiometric sample.

Probing local symmetry changes in $\text{LaMnO}_{3.12}$ rhombohedral-orthorhombic phase transition

A.M.L. Lopes^{2,5)}, J. P. Araujo³⁾, E. Rita^{1,5)}, J. G. Correia^{1,5)}, V. S. Amaral²⁾, Y. Li²⁾, J. M. Vieira⁴⁾, R. Suryanarayanan⁶⁾, ISOLDE Collaboration⁵⁾

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5) *European Centre for Nuclear Research (CERN), Geneva CH1211, Switzerland*

6) *Laboratoire de Physico-Chimie de l'Etat Solide, bât 414, Université Paris-Sud, Orsay 91405, France*

It is well established that the $\text{LaMnO}_{3+\delta}$ system presents a rich variety of structural (rhombohedral, orthorhombic) and magnetic (antiferromagnetic or ferromagnetic) phase as a function of the oxygen content (δ) or as a function of temperature (T). To deeply understand the Rh-Orth phase transition we combined High Resolution TEM, Perturbed Angular Correlation (PAC) and X-ray powder diffraction (XRD) studies in the temperature range 10-323 K in polycrystalline sample prepared by solid state reaction method. From the PAC signal we determined the Electrical Field Gradient (EFG) parameters which are sensitive to the charge distribution around the probe vicinity and therefore to the local symmetry of the crystal. In the 10-323 K temperature range two different EFG were measured revealing two different local environments. Phase coexistence was found in large temperature range. The measured EFG were assigned to the Rh/Orth local environment. Within the same temperature range the ED-HRTEM and bright/dark field imaging results showed the existence of laths and twinning domains of different crystalline symmetry. This data and the complementary 2 line shifts observed by XRD suggest a displacive martensite like Rh-Orth phase transition.

Two-dimensional exchange interaction in perovskite-like manganites.

Maria Mikhaylova¹⁾, Natalia Bobrysheva²⁾, Do Kyung Kim¹⁾, Muhammet S. Toprak¹⁾, Therese Eriksson³⁾, Yvonne B. Andersson³⁾, Mamoun Muhammed¹⁾

1) *Materials Chemistry Division, Dept of Materials Science and Engineering, Royal Institute of Technology (KTH), Brinellvaegen 23, 2tr., Stockholm SE10044, Sweden*

2) *Chemistry Department, Saint Petersburg State University, Saint Petersburg, Russian Federation*

3) *Department of Materials Chemistry, Angstrom Laboratory, Uppsala University, Uppsala, Sweden*

Nano-dimensional effects, such as electronic phase separation and charge ordering, play an important role in GMR-manganites. These effects are associated with superexchange interaction between Mn(III) and Mn(IV) atoms in perovskite structure. The correlations between properties of $\text{La}_{1-x}\text{A}_x\text{MnO}_3$ (A - Sr, Ba) and their electronic structure are still under investigations. Problems yet to be solved include determination of local electron parameters in three-dimensional oxides. The study of low-dimensional systems such as two-dimensional perovskite-like diluted manganites based on $\text{La}_{0.75}\text{Sr}_{1.25}\text{MnO}_4$ is expected to have a solution to this problem. $\text{La}_{0.75}\text{Sr}_{1.25}\text{MnO}_4$ complex oxide contains heterovalent manganese atoms and the same type of exchange interactions. In this study the homogeneous solid solutions $y(\text{La}_{0.75}\text{Sr}_{1.25}\text{MnO}_4) - (1-y)\text{LaSrAlO}_4$ ($0.0041 < y < 0.093$) with K_2NiF_4 structure were synthesized by solid state reaction. The phase in the final product was determined by XRD. Inter-atomic distances were calculated from structure refinements performed by the Rietveld method. Magnetic susceptibility and ESR spectroscopy were used to characterize the samples. According to the results of magnetic susceptibility measurements a strong tendency to clustering in perovskite layers was observed. Thus, the local distribution of electron density was changed. The origin of such phenomenon and its influence on the properties of manganites will be discussed in details.

19:00 -

poster

D-5

19:00 -

poster

D-6

This work is supported by Russian Foundation of Basic Researches (Grant 03-03-32355).

19:00 - **Mesoscopic inhomogeneity, 55Mn NMR, CMR and magneto-transport properties of La_{0.6}Sr_{0.2}Mn(1.2-x)Fe(x)O₃ manganites**

poster **Valentin P. Pashchenko¹⁾, Aleksandr A. Shemiakov¹⁾, Aleksey V. Pashchenko¹⁾, Vasilij K. Prokopenko¹⁾, Viacheslav N. Derkachenko¹⁾, Vladimir P. Dyakonov^{1,2)}, Henryk Szymczak²⁾**

D-8 *1) Donetsk Technical-Physical Institute NASU, R. Luxemburg 72, Donetsk 83114, Ukraine*
2) Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland

Complex studies of La_{0.6}Sr_{0.2}Mn(1.2-x)Fe(x)O₃ (x = 0; 0.02; 0.05; 0.1) manganites have been performed. Interrelation between the Fe ions concentration and CMR, magneto-transport and 55Mn NMR properties is established. It is shown that the magnetoresistance is increased, temperatures of magnetic and metal-insulator transitions as well as resonance frequency of 55Mn NMR is decreased and 55Mn NMR spectrum is expanded towards lower frequencies with increasing Fe content. These particularities are shown to be due to the weakening in charge interactions between Mn ions as a result of both Fe doping and occurrence of defects of vacancy and cluster types.

19:00 - **The LaMnO₃ elastic properties in different structural phases**

poster **Serguei E. Popov¹⁾, Anatolij E. Nikiforov¹⁾**

1) Department of Physics, Ural State University, Lenin Av., 51, Ekaterinburg 620083, Russian Federation

D-9

The undoped, lightly doped and defective LaMnO₃ can be observed in four basic phases: orthorhombic O*, orthorhombic O', rhombohedral R and monoclinic P. The differences in structure of these phases are governed by the [MnO₆] rotations and Jahn-Teller distortions. That is why the elastic properties of these phases are expected to differ strongly. The experimental investigation of elastic properties of high doped manganites shows step like changes in both elastic and sound propagation properties near structural phase transitions [1].

For elucidating the role of microstructure and JT interaction influence on elastic properties of lightly doped manganites we performed a calculation of elastic constants.

The calculation and microstructure analyses were performed in interionic pair potential and shell model approximation explicitly allowing for the JT term both in crystal energy and dynamical matrix [2]. The structure of each phase was obtained by crystal energy optimization procedure. The calculation of elastic module in O' and P phase was performed within static cooperative JT effect approximation, whereas cooperative dynamical JT effect approximation in R phase was used.

The calculation revealed some interesting features like presence of a giant anisotropy of elastic modulus in orthorhombic O* phase, i.e. $(C_{11}-C_{33})/(C_{11}+C_{33}) = -49\%$. The origin of such anisotropy is anomalous interaction between apex oxygen and lanthanum shifts, which is enhanced strongly by cooperative static JT effect. The giant anisotropy appears experimentally as anisotropic compressibility of orthorhombic LaMnO₃ [3].

This work was partially supported by CRDF grant REC-005 and RFBR U02-02-96412.

[1] T.W. Darling, A. Migliori et al, Phys. Rev., B57, No. 9, p. 5093, 1998

[2] A.E. Nikiforov, S.E. Popov, Phys. Solid. State, v43, No 6, p 1132, 2001

[3] I. Loa, P. Adler et al, Phys. Rev. Lett., V87, No. 12, p 125501, 2001

19:00 - **Ellipsometric studies of NdMnO₃ single crystals**

poster **Henryk Szymczak¹⁾, Gintautas J. Babonas²⁾, Alfonsas Reza²⁾, Marek Baran¹⁾, Jan Fink-Finowicki¹⁾, Sergei V. Shiryayev³⁾, Ritta Szymczak¹⁾**

D-10

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

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The fine structure of optical spectra of manganites was the object of wide discussions. However, so far the microscopic origin of the optical transitions responsible for the optical features in NIR-VIS-UV region is still not completely understood.

In the present work the ellipsometric studies of NdMnO₃ single crystals were carried out. For the first time, all the components of the dielectric function for manganites of orthorhombic symmetry were determined. The analysis of the fine structure of the spectra and optical anisotropy was the basis for the discussion of the microscopic origin of the optical transitions responsible for the optical features. The data for NdMnO₃ were compared with those obtained for other perovskites, LaMnO₃, LaBaMnCoO₃ and LaCaCoO₃.

NdMnO₃ single crystals of orthorhombic symmetry were grown by electrodeposition technique. Ellipsometric measurements were performed by photometric ellipsometer with rotating analyzer in the spectral range 0.5-5.0 eV. The

ellipsometric data obtained on the {001}-type planes were analyzed in the model of biaxial crystal and the dielectric function components were determined. The fine structure of the dielectric function spectra was decomposed into the contributions of several Lorentzian-type lines. From the experimental data the spectra of optical conductivity and loss function were also calculated and analyzed.

Two types of electronic excitations were used for interpretation of the fine structure in the optical spectra. In the region 1-3 eV the optical features are assigned to dipole-forbidden spin-allowed intraconfigurational transitions between crystal-field split 3d levels in Mn-ions. In the region 2-3 eV the structure due to f-f transitions in Nd³⁺ ions was resolved. In the region of higher photon energies the spectra are due to charge-transfer transition 2p(O)-3d(Mn). The difference between the charge-transfer transitions in NdMnO₃ and LaCoO₃ is also noticed and discussed.

Magnetic properties of La_{0.9}Ca_{0.1}CoO₃ single crystals

Ritta Szymczak¹⁾, Marek Baran¹⁾, Jan Fink-Finowicki¹⁾, Henryk Szymczak¹⁾

1) *Institute of Physics Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw, Poland*

19:00 -
poster

Magnetic measurements were carried out on La_{0.9}Ca_{0.1}CoO₃ single crystals. In the absence of applied magnetic field, this compound is an insulator down to 4.2 K. Results of magnetization measurements suggest a spin glass ground state. An applied field of 5T drives the insulator ground state to a ferromagnetic metallic state. This field induces antiferromagnetic low spin/spin-glass-like mixed spin transition. The energy gap for ls/hs transition was estimated to be 0.12 meV.

D-11

Magnetic and structural phase transitions in the La_{0.88}MnO_x system

Igor Troyanchuk²⁾, Vladimir A. Khomchenko²⁾, Henryk Szymczak¹⁾, Klaus Barner³⁾

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3) *IY. Physikalisches Institut der Universität Göttingen, Göttingen, Germany*

19:00 -
poster

The study of crystal structure, magnetic and electrotransport properties of La_{0.88}MnO_x (2.82 ≤ x ≤ 2.96) manganites as a function of oxygen content has been carried out using X-ray, neutron diffraction, elastic modulus, magnetization and resistivity measurements. The crystal structure of investigated compounds has been found to be orthorhombic at x < 2.91 and monoclinic at x > 2.91. The gradual transition from an orbitally-ordered state to an orbitally-disordered one is observed up to the concentration range of monoclinic compounds. Strong correlation between the type of orbital state and magnetic properties has been revealed. It has been shown that orbital-ordered orthorhombic compounds (2.82 ≤ x ≤ 2.84) are antiferromagnets while orbital-disordered monoclinic ones (x > 2.92) are metallic ferromagnets which reveal the features of first-order transition to paramagnetic state. Compositions in the wide range of oxygen concentration 2.85 ≤ x ≤ 2.91 exhibit both an antiferromagnetic and a ferromagnetic component. Simultaneously, the neutron diffraction and elastic properties study suggest the coexistence of both orbitally-ordered and orbitally-disordered phases. The properties are discussed in the framework of structurally driven phase separation model.

D-12

Electroresistance of La-Ca-MnO thin films

Saulius Balevicius¹⁾, Fiodoras Anisimovas¹⁾, Piotr Cimperman¹⁾, Oleg Kiprijanovic¹⁾, Jonas Parseliunas¹⁾, Voitech Stankevich¹⁾, NERIJA ZURAUSKIENE¹⁾, Larry L. Altgilbers²⁾

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2) *US Army Space and Missile Defence Command, Huntsville, United States*

19:00 -
poster

Recently, interest in the influence of electrical fields on the resistance of manganites had increased. It has been demonstrated that dc currents induce strong resistance decrease in Pr_{0.7}Ca_{0.3}MnO₃ and La_{0.82}Ca_{0.18}MnO₃ single crystals. It was observed that pulsed electric fields also change the resistance of thin La_{0.67}Ca_{0.33}MnO₃ films. However, the reasons for the origin of this electroresistance (ER) phenomenon are still unclear. In this paper, we present experimental results demonstrating that the main reason for the appearance of this ER in thin manganite films is the existence of ferromagnetic tunnel junctions located at intergranular regions in the polycrystalline films. Epitaxial, textured and polycrystalline La_{0.7}Ca_{0.3}MnO₃ films, having 150 nm thicknesses, were deposited onto (110) NdGaO₃, MgO and lucalox substrates, respectively. The samples were designed in a co-planar shape. Two Ag tapes having width of 1 mm and spaced by a d = 20 μm gap were deposited onto the film. The samples were connected in series to 50 Ω impedance high frequency transmission line. The resistance (R) vs. electric field strength (E) dependence was investigated using 10 ns duration electrical pulses having 0.5 ns rise time and amplitude up to 500 V.

D-13

Electroresistance [R(E)-R(0)]/R(0) was investigated using electric field strengths ranging up to 80 kV/cm at temperatures ranging from 300 K to 4.2 K. Strong (up to 93%) negative ER was obtained in polycrystalline La_{0.7}Ca_{0.3}MnO₃ films prepared on MgO and lucalox substrates by laser ablation. The epitaxial films grown, using laser ablation technique on NdGaO₃ substrate demonstrated only small resistance change due to Joule heating induced by a current pulse. It was concluded that ER manifests itself in strongly inhomogeneous manganites films

exhibiting a large number of ferromagnetic tunnel junctions.

- 19:00 -
poster
D-14
- NMR study of $(\text{Sr,Ba,L a})_2\text{Fe}_{1+x}\text{Mo}_{1-x}\text{O}_6$ double perovskites**
**Dariusz A. Zajac²⁾, Czesław Kapusta²⁾, Peter C. Riedi³⁾, Marcin Sikora²⁾, Colin J. Oates²⁾,
 Damian Rybicki²⁾, Jose M. De Teresa¹⁾, Clara Marquina¹⁾, Ricardo M. Ibarra¹⁾**
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 2) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*
 3) *School of Physics and Astronomy, University of St. Andrews, St. Andrews KY16 9SS, United Kingdom*

The ^{95}Mo and ^{97}Mo nuclear magnetic resonance study of $(\text{Sr,Ba,L a})_2(\text{FeMo})\text{O}_6$ double perovskites is reported. The compounds exhibit a significant low field magnetoresistance at room temperature. Compounds with different (SrBa) - La composition, different content of cation vacancies as well as different Fe - Mo ratio have been studied. The frequency swept spin-echo spectra have been measured at 4.2K and no applied magnetic field. The parent compound, $\text{Ba}_{1.6}\text{Sr}_{0.4}\text{FeMoO}_6$, shows a main line at 59 MHz and a satellite line at 43 MHz. They are attributed to the ideal and defect (antisite neighbours and antiphase boundaries) sites of molybdenum. Both lines correspond to ^{95}Mo and ^{97}Mo resonances, which are unresolved due to very close gyromagnetic ratios. The spectrum of the 10% La doped sample, $\text{Ba}_{1.44}\text{Sr}_{0.36}\text{La}_{0.2}\text{FeMoO}_6$ shows a higher intensity of the low frequency satellite line, which indicates an increased number of defect sites. The main line is at higher frequencies compared to the parent compound and shows a satellite structure at the high frequency side. An increase of the number of vacancies at the same La doping $\text{Ba}_{1.28}\text{Sr}_{0.32}\text{La}_{0.2}\text{FeMoO}_6$ further increases the population of defect sites. In the $\text{Ba}_{1.28}\text{Sr}_{0.32}\text{La}_{0.2}\text{Fe}_{1.2}\text{Mo}_{0.8}\text{O}_6$ compound the change of the Fe-Mo stoichiometry significantly decreases the amount of antisite defects. The results are analysed in terms of the influence of doping on the Mo magnetic moments and population of defect sites. A relation to the magnetic and transport properties of the compounds is discussed.

- 19:00 -
poster
D-15
- A ^{55}Mn NMR study of $\text{La}_{0.33}\text{Nd}_{0.33}\text{Ca}_{0.34}\text{MnO}_3$ with ^{16}O and ^{18}O**
**D. Rybicki²⁾, Cz. Kapusta²⁾, P. C. Riedi³⁾, C. J. Oates²⁾, M. Sikora²⁾, D. Zajęc²⁾, J. M. De
 Teresa¹⁾, C. Marquina¹⁾, M. R. Ibarra¹⁾**
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 2) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*
 3) *School of Physics and Astronomy, University of St. Andrews, St. Andrews KY16 9SS, United Kingdom*

This report presents a ^{55}Mn NMR study of ^{16}O and ^{18}O containing $\text{La}_{0.33}\text{Nd}_{0.33}\text{Ca}_{0.34}\text{MnO}_3$. Previous bulk measurements reported in the literature reveal that at low temperatures the ^{16}O based compound shows charge ordered (CO) and ferromagnetic (FM) insulating phases, whereas the ^{18}O based compound exhibits a charge ordered insulating behavior [1]. In both compounds, application of a magnetic field can change the low temperature state to a metallic one and the field required for inducing the transitions is two times higher for ^{18}O based compounds. The ^{55}Mn spin-echo spectra were measured at 4.2K. The spectra, for both compounds, consist of a single line corresponding to the double exchange (DE) state of manganese, which is attributed to the existence of ferromagnetic metallic (FMM) regions. The intensity of the resonant line is more than an order of magnitude lower for the ^{18}O based compound compared with the ^{16}O based compound, which indicates an order of magnitude lower content of the FMM phase in the ^{18}O based compound. The Mn resonance of the CO or FM insulating phases are not observed, possibly due to a fast nuclear relaxation of the Mn^{3+} and Mn^{4+} cations. The measurements of the spectra for large pulse spacing show a pronounced minimum at the centre of the DE line. This is related to a minimum of the spin-spin relaxation time at the line centre caused by the Suhl-Nakamura interaction between nuclear spins. This interaction is effective in the large regions of uniform magnetisation, which indicates that the FMM regions are at least tens of nanometres in size for both compounds. The results are discussed in terms of phase segregation in the compounds and the influence of oxygen isotopic substitution on it.

[1] M.R.Ibarra et al, Phys. Rev. B, 57 (1998) 7446

- 19:00 -
poster
D-17
- Transport and magnetic properties of $\text{La}_{2/3}\text{Pb}_{1/3}\text{MnO}_3$ thin films**
**Mark Baran¹⁾, Marek Berkowski¹⁾, Jan Fink-Finowicki¹⁾, Stanisław J. Lewandowski¹⁾, Henryk
 Szymczak¹⁾, Bożena Krzymanska¹⁾, Sergei N. Khartsev²⁾, Yurii V. Medvedev²⁾, Ritta Szym-
 czak¹⁾**
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The magnetic and transport properties of $\text{La}_{2/3}\text{Pb}_{1/3}\text{MnO}_3$ films have been studied. The films have been epitaxially grown on LaSrGaO_4 substrate using dc magnetron sputtering technique. Magnetic properties of the films were measured using SQUID magnetometer. The Curie temperature was determined being equal 211 K for the as deposited film and 176 K for the annealed film. The small persistent photoinduced changes of magnetization have been observed, especially well seen at higher temperature region. The giant magnetoresistance effect has been observed

near the Curie temperature. It has been shown that strains strongly affect the properties of the films. Several mechanisms responsible for temperature dependence of films resistivity are analysed. The analysis shows that the Mott's variable-range-hopping (VRH) mechanism should be responsible in the investigated films for the conductivity above the Curie temperature.

Anisotropy of magnetic properties of the layered Co-based perovskite single crystals 'R₂BaCo₂O_{5+x}' (R = Dy, Tb, Eu)

Valentina I. Gatalskaya^{1,2)}, Sergej Shiryayev^{1,2)}, Georgij Bychkov²⁾, Sergej Barilo²⁾, Leonard Kurochkin²⁾, Marek Baran¹⁾, Ritta Szymczak¹⁾, Henryk Szymczak¹⁾

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2) *The Institute of Solid State & Semiconductor Physics, National Academy of Science of Belarus, 17 P. Brovka, Minsk 220072, Belarus*

We describe anisotropic magnetic behaviour of single crystals of R₂BaCo₂O_{5+x} with $x \sim 0.5$ [R = Dy (DBC), Tb (TBC), Eu (EBC)] grown first from overstoichiometric flux melt by spontaneous crystallization. The temperature and field dependences of magnetization have been obtained in region 2-350 K under magnetic fields up to 5 T at the different field configurations. With decreasing temperature the crystals revealed a similar behaviour: at HII(ab) they display the PM-WFM-AFM magnetic transitions. At 100 Oe the PM-WFM transition occurs at 295 K (DBC), 271 K (TBC) and 235 K (EBC). The AFM transition was observed at $T_N = 230$ K (DBC), 240 K (TBC) and 210 K (EBC). The calculations show that for HII(ab) $p_{eff} = 2.8 \mu_B / \text{Co}^{3+}$ (TBC) with extracting the contribution from R-ion. Note that value is close to the expected value for Co³⁺ ion full IS of $2.83 \mu_B$. For H = 1 kOe $p_{eff} = 1.8 \mu_B / \text{Co}^{3+}$ corresponding to LS/IS = 0.8 : 0.2. Ergo, magnetic field H = 5T induces the cobalt ions spin transition from LS to IS state. Similar picture is observed for DBC and EBC. The magnetic moments corresponding to WFM state are 0.07 (DBC), 0.15 (TBC) and 0.19 μ_B (EBC) at $T_{max} = 245, 265$ and 210 K, respectively. We assume that only IS Co³⁺ ions in octahedral site contribute to spontaneous moment (SM) while other Co ions in square pyramide site are PM. The AFM transition (H = 1 kOe) is a cause of SM reduction down to 0.05 (DBC), 0.002 (TBC) and 0.008 μ_B (EBC) per Co ion. For HIIc all crystals are PM and SM does not appear at lowest temperatures. But for TBC we observed the AFM ordering of Tb-sublattice at 3.44 K, which was not registered for HII(ab) up to 2 K. The M(H) dependences for HII(ab) show the FM component to survive down to the lowest temperatures. For HIIc hysteresis of M was not found in the whole temperature interval in TBC.

The work is supported by INTAS grant # 01-0278, NATO linkage grant PST CLG 979 369 and Polish KBN grant 5P03B01620.

Magnetism and superconductivity in Nd_{0.81}Sr_{0.19}MnO₃/YBa₂Cu₃O₇ multilayers

Ivan Komissarov¹⁾, Piotr Przysławski¹⁾, Maciej Sawicki¹⁾

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Magnetic oxide superlattice are attractive model system for the study coupling and interface effects. We report on the growth, structural and magnetic characterization of Nd_{0.81}Sr_{0.19}MnO₃/YBa₂Cu₃O₇ (NSMO/YBCO) multilayers. NSMO system for the doping level x=0.19 is a ferromagnetic insulator with Curie temperature of about 100 K. Multilayers with fixed NSMO thickness of 13 unit cell and varying YBCO layer thickness for 2 unit cell to 6 unit cell were sputtered on LaAlO₃ substrates. Hysteresis loops measured above and below superconductivity transition show a signature of interlayer coupling

Changes of reflection factor parameters of multilayered bigyrotropic structures

Denis G. Makarov¹⁾, Vadim V. Danilov¹⁾, Valery F. Kovalenko¹⁾

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The behavior of reflection factor for linearly polarized radiation from homogeneous multilayered structure with bigyrotropic layers in external magnetic field is investigated. Changes of width, amplitude and position of central maximum on reflection factor curve are received. It is shown, that effects of change of width and amplitude are paired on polarization (left- and right-hand circular) at change of magnetization, whereas the effect of maximum shift is various for the specified two kinds of polarization.

Atomic order and the interaction of electron and magnetic subsystems in epitaxial LaSr(Ca)MnO films

Vladimir D. Okunev^{1,2)}, Z. A. Samoilenko^{1,2)}, T. A. Dyachenko²⁾, A. Abal'oshev¹⁾, M. Baran¹⁾, P. Gierłowski¹⁾, S. J. Lewandowski¹⁾, Andrzej Szewczyk¹⁾, Henryk Szymczak¹⁾, R. Szymczak¹⁾

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2) *A. Galkin Donetsk Institute for Physics & Technology, National Academy of Sciences of Ukraine (DonPTI NASU), Roza Luxemburg 72, Donetsk 83114, Ukraine*

The effect of structure on the optical, transport and magnetic properties of single-crystalline thin LaSr(Ca)MnO films with metallic clusters coherently contained within the basic crystallographic matrix was investigated. The films were grown by pulsed laser deposition. With growth temperature T_s in the 450 C < T_s < 600 C range, the films are composed predominantly of a rhombohedral (dielectric) matrix and rhombohedral (metallic) clusters. Magnetic properties are similar to those of clusterized spin glasses and show a characteristic difference between Field Cooled (FC) and Zero Field Cooled (ZFC) magnetization M values in low (< 1kOe) magnetic fields. For growth temperature $T_s \geq 650$ C, the film structure changes to rhombic matrix and rhombic clusters. This change is accompanied by a 3-9 orders of magnitude increase of film conductivity and the appearance of a, characteristic for manganites $R(T)$ curve showing minima and maxima. magnetization at low temperatures is increased by an order of magnitude in comparison to the $T_s \leq 600^\circ\text{C}$ case.

We show that electronic, optical and magnetic properties of the films depend on the ordering of the cluster structure. There exists a threshold value of magnetic ordering, above which the magnetic subsystem starts to affect actively the film conductivity. In the case of correlated changes in the cluster structure, this leads to a percolative dielectric-metal transition. Due to the tunneling interaction mechanism between clusters, the electrical conductivity exponentially grows with the magnetization magnitude. The considerable differences in the gradients dR/dT observed for samples of nominally identical composition are caused by the changes in the interaction mechanism between atomic, electronic and magnetic subsystems.

This work was supported by Polish Government (KBN) Grant PBZ-KBN-013/T08/19.

Self-Energy Effects in Manganite Tunneling

19:00 -
poster

Vladimir M. SVISTUNOV¹⁾, Yurii F. Revenko¹⁾, Mikhail A. Belogolovskii¹⁾, Alexander Y. Gerasimenko¹⁾, Yurii G. Pashkevich¹⁾, Henryk Szymczak²⁾, Piotr Przyslupski²⁾, Ivan Komisarov²⁾

D-23

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Tunneling spectroscopy experiments on ceramic and film LCMO, LSMO, NSMO systems are reported. The electron boson interaction spectral function in a wide energy range has been reconstructed. Besides the known from Raman spectra data phonon features up to 80meV, the experimental curves clearly indicate the presence of bosonic excitations in the region above 100meV. Qualitative changes of tunneling spectra (below and above 100meV) caused by chemical modification of the oxide surface (oxygen ion displacements) after application of high voltages to the junction were revealed. Its effect is the enrichment of manganite surface layers by oxygen. After application of high biases, junction resistances were reduced and the ratio of peaks below and above 100meV was increased. These results are discussed within a scenario involving a Strong Electron-Phonon Interaction, a possible orbital ordering that has been recently predicted (observed in only LaMnO3) and Tunneling Magnetoresistance.

Irreversible Modification of Structure and Low Field Magnetoresistance of $\text{La}_{0.65}\text{Ca}_{0.35}\text{MnO}_3$ Films on Ferroelectric Substrates

19:00 -
poster

Yurii V. Medvedev¹⁾, Nikolai I. Mezin¹⁾, Yurii M. Nikolaenko¹⁾, Andriy E. Pigur¹⁾, Natalya V. Shishkova¹⁾, Vladymyr M. Ischuk²⁾, Iryna N. Chukanova²⁾

D-24

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The galvanomagnetic properties of $\text{La}_{0.65}\text{Ca}_{0.35}\text{MnO}_3$ manganite films with a thickness of 0,2 μm on the $\text{Pb}_{2.9}\text{Ba}_{0.05}\text{Sr}_{0.05}(\text{Zr}_{0.4}\text{Ti}_{0.6})\text{O}_3$ ceramics substrates have been investigated.

We have found the irreversible increase of film resistivity (ρ) in the process of number acts of inverse of direction of substrate polarization. The nature of effect is connected with a mechanical stress and local electrical currents. Wide range of monotonic increase of resistivity $\rho = 1.8 \cdot 10^{-2} - 1.8 \text{ cm}$ without destruction of macroscopic homogeneity of film conductivity is explained by appearance of a new boundaries in the structure and decreasing of size of regions without film defects. The maximal size of region (3-10 μm) is provided by the grain of ceramic substrate. The minimal size has order of 100 nm.

The films demonstrated the negative low - field magnetoresistive effect with the saturation field about $H = 1.5 \text{ kOe}$ at $T = 100 - 180 \text{ K}$ and linear decreasing type of temperature dependence.

Pressure and magnetic field effects on transport properties of ceramic and film samples $(\text{La}_{0.7}\text{Ca}_{0.3})_{1-x}\text{Mn}_{1+x}\text{O}_3$

19:00 -
poster

Vladimir I. Mikhaylov²⁾, Vladimir P. Dyakonov^{1,2)}, Valery A. Shtaba²⁾, Eduard E. Zubov²⁾, Alexey V. Pashchenko²⁾, Andrzej Szewczyk¹⁾, Kazimir Piotrowski¹⁾, Henryk Szymczak¹⁾

D-25

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Many physical characteristics including the Curie temperature T_C , the metal-insulator transition temperature T_{MI} , and resistance in doped lanthanum perovskite manganites - materials having "colossal" magnetoresistance (CMR) can be changed to a considerable extent by internal chemical as well as by external physical pressure. The pressure and magnetic field effects on transport and magnetoresistance effect (MRE) have been studied in both the epitaxial films and ceramics of manganites with excess manganese $(La_{0.7}Ca_{0.3})_{1-x}Mn_{1+x}O_3$ ($x = 0 - 0.2$). A comparison of electrical behaviour in both kinds of ceramic and film samples of similar composition under the influence of hydrostatic pressure (up to 2 GPa) and magnetic field (up to 8 kOe) has been performed. The pressure as well as magnetic field decreases the resistivity in the region of the metal-insulator transition and shifts T_{MI} to higher temperatures. T_{MI} and MRE are shown to increase with increasing manganese content.

Experimental data show that the pressure and magnetic field effects on temperatures of both metal-insulator transition (T_{MI}) and MRE peak (T_{MR}) are considerably stronger in the films than in the ceramic samples. The linear increase of T_{MI} and T_{MR} by pressure was observed up to 2 GPa, and the pressure effect is stronger in the films. The main origin of the pressure-magnetic field effects is related to increasing double exchange (DE) interaction because of both the decrease of the unit cell volume and lattice distortions. The differences in the values of pressure effect on resistivity, MRE and metal-insulator transition temperatures in ceramics and in the films of the same content are due to a difference of both the oxygen nonstoichiometry and the film strain induced by lattice mismatch between the film and the substrate.

Magnetic nonextensivity on CMR manganites

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19:00 -

poster

D-26

Although the number of works on CMR manganites amounts over three thousands, the models and theories developed to understand the physical properties of this materials fail to obtain plain agreement between experiment and theory. On other hand, another rather different area which has been growing at an analogous rate is the Tsallis generalized statistics, that satisfactorily describes nature if the microscopic interaction are long-ranged and the boundary conditions are fractals, as well as the system presents an intrinsic inhomogeneity. Such features have been invoked in recent models of manganites, as well as in the interpretation of experimental results. For instance, some authors emphasize a strong phase competition (inhomogeneity), and others have considered the formation of micro-cluster with fractal shapes, randomly distributed in the material, and the role of long-range interaction to phase segregation. In this direction, in two recent works [1,2] we proposed that manganites are better described within the Tsallis generalized statistics, where we obtained excellent agreement between experiment and theory. In the present work, we describe new evidences that manganites are non-extensive objects, with a model that foresee several types of magnetic anomalies presented in ferromagnetic manganites, namely: the strong downturn near the critical temperature, presented in many curves of the inverse of the susceptibility; the well know inflexion point, with hysteresis, presented in M vs. H curves near a first-order transition; the nature of the magnetic transition, i.e., if the magnetic transition is of first or second order; among others. Finally, we could also build a magnetic phase diagram that reproduce reliably all magnetic properties of the ferromagnetic manganites.

[1] M.S. Reis et al. Europhys. Lett. 58 (2002) 42.

[2] M.S. Reis et al. Phys. Rev. B 66 (2002) 134417.

Regularities of elastically anisotropy deformed mechanism of T-H-P influence on the phase transition and properties change in magnetic semiconductors

Piter I. Polyakov¹⁾, Stanislav S. Kucherenko¹⁾

1) Donetsk Technical-Physical Institute NASU, R. Luxemburg 72, Donetsk 83114, Ukraine

19:00 -

poster

D-27

The main aim of this paper is development of new conceptions about the general theory of structure phase transitions, phase diagrams, properties and effects in magnetic semiconductors.

An analysis of change of resistivity, magnetostriction, phase transitions of magnetic semiconductors: polycrystalline $La_{0.7}Mn_{1.3}O_3$ and monocrystalline $LaMnO_3$ under influence of temperature (T), magnetic field (H) and hydrostatic pressure (P) was carried out. The revealed magneto-, baro- and baromagneto-resistive effects have shown the same temperature T_{PP} of their peaks which coincides with the temperature T_{ms} of structural phase transition 'metal-semiconductor' and the discovered 'cooling' and 'heating' effects allow us to substantiate the linear shifting of $T_{ms}(H)$, $T_{ms}(P)$ and $H_g(T)$ [1]. A correspondence in the influence of T-H-P ($8 K \sim 2.12 kOe \sim 1 kbar$) on resistive properties, and T-H ($5.2 K \sim 2.5 kOe$) on magnetostrictive properties has been established. Sign alternation change in properties, effects and priorities of the competing influence of thermo-, baro- and magnetoelastic anisotropically deforming (EAD) strictions has been fixed. Positions of critical lines $T_{ms}(H)$, $T_{ms}(P)$, $H_g(T)$ and critical points T_X , P_X , PP_X , P_X' , $T_{PP}=T_{ms}$, T_C have been determined (fig.1). Correspondence on resistivity and magnetostriction behaviours is shown [1].

Reference

[1] P.I. Polyakov, S.S. Kucherenko, JMMM 248, ?3, pp.396-401 (2002); Pisma v Zhurn. Teh. Fiz. 28, p.8 (2002); Fiz. Nizk. Temp. 28, ?10, pp.1041-1047 (2002).

19:00 -
poster

Regularities of elastically anisotropy deformed mechanism of T-H-P influence on the phase transition and properties change in magnetic dielectrics

Piter I. Polyakov¹⁾, Stanislav S. Kucherenko¹⁾

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D-28

From the resonance properties of monocrystalline low-temperature magnetic dielectric $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ a correspondence of T-H-P influence ($1 \text{ K}^{-4} \text{ kOe}^{-3} \text{ kbar}$) on structural phase transition has been determined [2]. The thermomagnetic and thermobaromagnetic effects are revealed [3]. Their peaks have the same temperature $T_{PP}=0 \text{ K}$, which coincides with the temperature T_{ST} of structural phase transition, by analogue with magnetic semiconductors [1]. An sign alternation 'cooling' and 'heating' effects in $T_P(\text{H})$, $T_P(\text{H},\text{P})$ shifting are also found. The critical points P_X , PP_X , T_X and P have the temperature parameter $T_P=9.2 \text{ K}$ which is highly different from the known $T_N=4.3 \text{ K}$ in these materials.

From the various established critical lines $T_P(\text{H})$, $H_g(\text{T})$ and points P_X , PP_X , T_X the value of correspondence and sign alternation of T-H-P influence through the EAD mechanism is defined. The identical of this mechanism in magnetic semiconductors and magnetic dielectrics is proposed.

Presented results allow to predict the participation of EAD mechanisms in realization of well-known colossal magnetoresistive effect and of superconductivity effect on HTSC structures.

Using of these mechanisms allow to rename the law of correspondence states as the law of elastically deformed correspondence, and revealed regularities of T-H-P influence as the law of elastically anisotropy deformed stresses and sign alternation.

References

[1] P.I. Polyakov, S.S. Kucherenko, JMMM 248, ?3, pp.396-401 (2002); Pisma v Zhurn. Teh. Fiz. 28, p.8 (2002); Fiz. Nizk. Temp. 28, ?10, pp.1041-1047 (2002).

[2] A.A. Galkin, V.A. Popov, P.I. Polyakov et.al. Fiz. Nizk. Temp. 2, ?1, pp.49-60 (1976); Fiz. Tverd. Tel. 17, ?10, pp. 2123-2126 (1975);

[3] P.I. Polyakov, S.S. Kucherenko Proceeding of NMMM-18, Mosc. State Univer., Mosc., p.714, 2002.

[4] J.Van Den Handel, H.M. Gijsman and N.J. Poulis, Physica, 1952, v.18, ?11, p.8

19:00 -
poster

Non-equilibrium Kondo effect in a single-channel quantum dot asymmetrically coupled to two reservoirs

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D-29

Non-equilibrium Kondo effect in a quantum dot coupled to two metallic leads is studied theoretically. The leads can be either ferromagnetic or nonmagnetic. However, of particular interest is the case when the leads are made of a system with large spin asymmetry of transport properties, like giant and/or colossal magnetoresistance materials. Owing to the spin asymmetry, the effective coupling between the dot and reservoirs can be strongly spin dependent, too. Electron interaction on the dot is taken into account via the Hubbard term for a single-channel model with an arbitrary value of the correlation parameter U .

The non-equilibrium Green function technique is used to calculate electric current and density of states. The key point of the approach is a consistency of the approximations used to calculate the lesser and retarded (advanced) Green functions within the equation of motion method. These functions are calculated on equal footing, ie, within the same approximation scheme. The description generalizes in several ways some of the earlier descriptions based on the equation of motion method. (i) It applies to nonmagnetic and magnetic systems. (ii) The description is gauge invariant (the electrostatic potential due to the charge on the dot is included). (iii) It applies to equilibrium as well non-equilibrium situations.

The key difference between our approach and some of the earlier descriptions based on the equation of motion technique originates from a difference in the predictions on the occupation numbers. This difference gives rise to a difference in the density of states at the Fermi level as well as in the corresponding Kondo anomaly in the differential conductance at small bias. The difference is particularly large in the case of asymmetric junctions, even in equilibrium situations. The model predicts spin splitting of the Kondo peak in the density of states in the magnetic case, as well as some anomalous behavior in external magnetic field.

19:00 -
poster

2D freezing of magnetic nanoparticles in a perpendicular field

Jean-Claude Levy¹⁾, Abdelwahab Ghazali²⁾

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2) Groupe de Physique des Solides Universit s Paris 6 et 7 (GPS), 2 place Jussieu, Paris 75005, France

D-30

2D magnetic nanoparticles are known to lead to numerous structures, experimentally ^{'1'} and theoretically ^{'2'}. Here we want to focus on the case of magnetic nanoparticles in a strong perpendicular magnetic field which aligns all magnetic dipoles. Then the dipole-dipole interaction is repulsive as well as the hard core interaction, and a confinement is required to stabilize the structure. These interactions are simulated by means of a Monte-Carlo computation. The energetic difference between a triangular lattice of perpendicular dipoles and a square lattice with the same density is quite weak. Thus the solidification temperature of confined perpendicular dipoles is expected to be very low. This is confirmed by numerical simulations which show that the solid phase contains triangular lattice parts as well as many defects as known in a Wigner glass. As a matter of fact this liquid-solid transition reveals to be a freezing one because of the continuous variation of energy per particle through the transition. In the solid phase, heating is linked with localized particle displacements towards a more irregular structure.

^{'1'} F. Kun et al., Phys. Rev. E 64, 061503, (2001).

^{'2'} A. Ghazali and J.-C.S. Levy, Phys. Rev. B 67, 064409, (2003).

Magnetoresistance Observation In Ferromagnet-Electroactive Polymer-Metal Junction

19:00 -

Alexei N. Lachinov¹⁾, Natalia V. Vorobjeva¹⁾, Alexandr R. Doroshenko¹⁾

poster

1) Institute of Molecules and Crystal Physics, Ufa Scientific Centre of Russian Academy of Sciences, 151 Prospekt Oktyabrya, UFA 450075, Russian Federation

D-31

It was reported previously that in multilayer metal-polymer-metal structures magnetic properties change of any electrode may result in considerable changes in the charge transport through the polymer film. Magnetic properties changes were realized by variation of the sample temperature near the Curie point.

The purpose of this work was to find conditions for charge transport control in a ferromagnet-polymer-metal system with the aid of the external magnetic field. In organization of the experiment we proposed that electroactive polymer transport properties were defined by the surplus charge (electron) and macromolecule functional groups interaction. The probability of the interaction depends on the spin orientation of the injected electron. One can control the spin orientation by the external magnetic field influence over the ferromagnetic electrode domain structure.

Polycrystalline nickel was used as the ferromagnet; poly(phtalidilidenbyphenililene) (PPB) as the electroactive polymer. For less magnetostrictive effects (tensions) in the magnetic sample it was chosen as a plate placed normally to the magnetic field force lines. The conducting state of the system was chosen as the initial one. With magnetic field increase up to ~150 mT we see great current fluctuations. At 160 mT the current decreases down to a minimal value and the sample transfers to the low-conducting state. The resistance changes by four orders of magnitude. With the magnetic field decrease we see the return of the conductance to the initial state under less magnetic field values ~ 148 mT. It was noticed that the field values under which one can see changes of the system conductivity depended on the sample history. The minimal field value under which we could see the changes was ~ 20 mT. For the results analysis magnetization curves of the samples were obtained and various charge transport parameters. The polymer thickness and initial conductivity value influences were also investigated.

Magnetoresistance of Ge-Si whiskers in the vicinity to metal - insulator transition

19:00 -

Anatoly A. Druzhinin¹⁾, Igor P. Ostrovskii¹⁾, Natalya S. Liakh¹⁾

poster

1) Lviv Polytechnic National University, 12 Bandera, Lviv 79013, Ukraine

D-32

Magnetoresistance of Ge-Si whiskers with germanium compositions up to 11 at. % was studied in the range of magnetic field up to 14 T. The whisker concentration was close to critical one for metal-insulator transition. Character of dependencies of the whisker magnetoresistance on magnetic field is determined by level of the whisker doping. For insulator samples quadratic field dependence of magnetoresistance is found, while for metallic ones the dependence is exponential. In the whiskers with large germanium content (4-11 at. %) anomalous positive magnetoresistance as well as negative magnetoresistance is observed. Negative magnetoresistance is shown to rise with increase of germanium content. The dependencies found are discussed.

(Cd,Hg)Te Defect Structure and its Magnetoresistive Properties, Modified by Pulse Laser Irradiation

19:00 -

Bohdan Kotlyarchuk²⁾, Apollinariy O. Zaginey²⁾, Yuriy E. Syvenkyy²⁾, Maciej Oszwaldowski¹⁾

poster

1) Politechnika Poznanska, Instytut Fizyki (PUT), Nieszawska 13a, Poznan 60-965, Poland

2) Institute of Applied Problems of Mechanics and Mathematics (IAPMM), 3B Naukova Str., Lviv 79060, Ukraine

D-33

The work is aimed of the theoretical and experimental investigations on the management possibilities of the laser beam processing of semiconductor crystals (Cd,Hg)Te.

The nonlinear mathematical model of the laser beam heating process was developed, when millisecond pulses of ruby laser were used. The constructed nonlinear model allowed to determine the dynamics of heating and melting

process in material, depending on energetic and space-time characteristics of pulse laser radiation. Distribution of the extended structural defects in (Cd,Hg)Te crystal near-surface layers and micro-sections was determined.

The increased density of structural defects being formed by laser treatment in the near-surface region of the (Cd,Hg)Te samples leads to the essential reduce of the survival time of the current carriers in the very region. The considerable reduce of the survival time of the unstable current carriers in the defective zone creates the conditions for the origin of the magneto-concentrated effect in the intersecting electric and magnetic fields. The Lorentz force deflects the current carriers towards the surface with the increased rate of recombination. This leads to the growth of electrical resistance in the samples.

The following structures have been examined: the n-(Cd,Hg)Te base - the p-(Cd,Hg)Te modified with laser irradiation; the p-(Cd,Hg)Te base with different concentration of the holes - the modified layer p-(Cd,Hg)Te. These samples had one surface treated by ms-width laser pulse with level of its energy close to the threshold. In magnetic fields 1.8 Tl we have determined an essential discrepancy in the Volt-Ampere characteristics of the samples. This discrepancy depends on the direction of the magnetic field. The influence of the thickness of the base and the ratio of the concentration of the majority carriers in the layers over the magneto-concentrated effect has been investigated.

19:00 - **Magnetoresistance of semimagnetic semiconductors**
 Bahram M. Askerov¹, Tariyel H. Ismailov¹, Sofiya R. Figarova¹, Mehdi M. Mahmudov¹
 poster 1) *Baku State University, Department of Physics (BSU), Z.Khalilov str. 23, Baku, Azerbaijan*

D-34 The influence of the exchange interaction between charge carrier spins and d - electron spins of Mn^{2+} on the magnetoresistance of $Hg_{1-x}Mn_xTe$ semimagnetic semiconductor is theoretically investigated. Transverse and longitudinal magnetoresistances are studied. Analytical expressions for the transverse magnetoresistance depending on the magnetic field magnitude, temperature and band structure parameters, namely exchange interaction constant and band gap, are obtained. It is supposed that the relaxation time is proportional to the density of states, which is calculated taking into account quasilocal acceptor levels. The cases of degenerate and non-degenerate electron gas are considered. It is shown that the exchange interaction essentially influence on the magnetoresistance magnitude. It is obtained that for certain compositions (X) and magnetic field magnitudes transverse magnetoresistance reaches the giant value. It is noted that the magnetoresistance dependence on temperature is determined both the exchange interaction and availability quasilocal acceptor levels.

The longitudinal magnetoresistance of the non-degenerate electron gas weakly depends on exchange interaction. While in the case of degenerate electron gas longitudinal magnetoresistance strongly depends on exchange interaction.

Comparison of the received theoretical dependences with experimental data available in the literature is carried out. The qualitative agreement is received.

19:00 - **Magnetic properties of low-doped Nd_{1-x}Ca_xMnO₃ manganites**
 Vladimir A. Khomchenko², Igor Troyanchuk², Henryk Szymczak¹, Ritta Szymczak¹
 poster 1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*
 2) *The Institute of Solid State & Semiconductor Physics, National Academy of Science of Belarus, 17 P.Brovka, Minsk 220072, Belarus*

D-35 The study of crystal structure, elastic and magnetic properties of $Nd_{1-x}Ca_xMnO_3$ ($x \leq 0.15$) perovskites has been carried out. The ferromagnetic component is shown to increase under hole doping and, simultaneously, the temperature of orbital order-disorder phase transition decreases. The mechanism of concentrational transition from weak ferro-

magnetic state ($x=0$) to ferromagnetic one ($x>0.15$) is discussed using two-phase model according to which the samples consist of weak

ferromagnetic and ferromagnetic phases exchange coupled at their boundary. It is found that interaction between different

magnetic phases leads to spin reorientation which takes place for $0.06 \leq x \leq 0.1$ compounds around $T_{eff} \sim 9$ K. For the

$Nd_{0.92}Ca_{0.08}MnO_{2.98}$ sample, the metamagnetic behaviour is revealed in the temperature range from 5 to 20 K. H vs.

T as well as T vs. x magnetic phase diagrams are proposed. The appearance of orientational transitions is explained on the basis of magnetic !

analogue of Jahn-Teller effect taking into account that the magnetic moments of Nd ions are ordered parallel to the moments

of Mn ions in ferromagnetic phase, and opposite to the direction of weak ferromagnetics vector at $T > T_{eff}$ in weak ferromagnetic phase.

Tuesday, September 16th

Morning session

 Main Building, room 144

11:00 - 12:30

Spectacular Effect of Doping upon Magnetism and Conductivity in Manganites
Bernard RAVEAU

11:00 - 11:45

invited oral

Manganites are very sensitive to the presence of impurities on the Mn sites of the perovskite structure. In this respect, the doping of orbital-charge ordered manganites, such as $\text{Ln}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ or $\text{Ln}_{0.4}\text{Ca}_{0.6}\text{MnO}_3$ by various elements is spectacular. Starting from the antiferromagnetic CE-type insulator, two classes of materials can be generated, depending on the nature of the dopant : for non magnetic elements (Al, Ga, Ti, Sn, In, Sc, Mg) and iron, spin glass insulators are obtained whereas for magnetic cations (Cr, Co, Ni, Cu, Rh, Ir, Ru), metallic ferromagnets are generated. As a consequence, the doping at Mn sites modifies dramatically the magnetic phase diagrams of the manganites, even for the LaMnO_3 phase alone which by doping with various cations (Zn, Li, Mg, Co, Ni) becomes ferromagnetic, due to valency effects.

At very low temperature, ($T < 5\text{K}$) anomalies in the magnetic and transport properties are induced in the charge ordered manganites, either by doping at the manganese sites with various cations, or by doping at the calcium sites with larger cations like barium and strontium. Plotting the magnetization versus the applied magnetic field, a step like behaviour is observed which is strongly influenced by the nature of the dopant, its content, thermal cycling etc. Importantly, a correlated effect is observed on the resistivity which can vary by several orders of magnitude in the form of steps. This effect is also detected on the variation of the specific heat versus the applied magnetic field. These properties are closely related to the phase separation phenomena that appear in the manganites. This peculiar behaviour also detected from neutron diffraction data, can be interpreted by a martensitic like mechanism.

Design Rules for Manganites with Novel Magnetic and Electronic Properties
Bogdan Dabrowski¹⁾

 1) Northern Illinois University (NIU), Faraday Hall W216, DeKalb 60115, United States

11:45 - 12:30

invited oral

We have been systematically developing strategies for making new perovskite manganites with novel magnetic and electronic properties. This effort requires two-fold approach: development of understanding the dependence of properties on chemical and structural factors and development of ability for synthesis of desired compounds. We show that similar to other single valent 3d systems the magnetic superexchange interactions in RMnO_3 manganites (R = Rare or Alkaline Earth's) are dependent on the Mn-O-Mn bond angle that is a function of interatomic distances R-O and Mn-O. The local structural disorder on the R-site suppresses magnetic interactions. Recently, by comparing disordered (randomly mixed R/Ba) and ordered (forming Ba/R/Ba layers along the c-axes) perovskites we have demonstrated much more conspicuous effects of structural and charge disorder for mixed-valent manganites. We show that by stabilizing ordered structure; i.e., by suppressing local structural and charge disorder, for R = La and Pr a substantial increase of T_c (from 340 and 200 to 365 and 335 K, respectively) can be achieved.

To achieve these compounds, we have been systematically developing special synthesis techniques for extending chemical composition ranges far beyond those previously achieved, developing rules for predicting what compositions it should be possible to make by these special techniques, and establishing methods for selectively ordering or disordering mixtures of metal atoms on the R or B sites. Our design rules for the synthesis of metal-oxide compounds enable the chemical compositions and crystal structures of these materials to be controlled in order to achieve the desired magnetic and electronic properties for applications.

Work at NIU was supported by the NSF-DMR-0105398 and by the State of Illinois under HECA. At ANL work was supported by the U.S. Department of Energy, Division of Basic Energy Science ? Materials Sciences, under contract No. W-31-109-ENG-38.

Lunch break

12:30 - 14:00

Afternoon session

 Main Building, room 144

14:00 - 15:30

Orbital correlations and magnetic phase transitions in lightly doped manganites and cobaltites
Igor Troyanchuk²⁾, **V.A. Khomchenko**²⁾, **Henryk Szymczak**¹⁾, **K. Barner**³⁾

14:00 - 14:45

invited oral

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

2) The Institute of Solid State & Semiconductor Physics, National Academy of Science of Belarus, 17

P. Brovka, Minsk 220072, Belarus

3) IY. Physikalisches Institut der Universitat Gottingen, Gottingen, Germany

The results of X-ray and neutron diffraction study as well as the elastic, magnetic and magnetotransport properties of $\text{La}_{0.88}\text{MnO}_x$, $\text{La}_{1-x}\text{Bi}_x\text{MnO}_3$, $\text{Nd}_{1-x}\text{Ca}_x\text{MnO}_3$, $\text{Eu}_{1-x}\text{Ca}_x\text{MnO}_3$ manganites and $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$, $\text{Nd}_{1-x}\text{Ba}_x\text{CoO}_3$ cobaltites have been presented. The transition to ferromagnetic state is observed for these systems under oxygen or Bi, Ca, Sr, Ba doping. In manganites, the transition to ferromagnetic state correlates with the change of the type of Jahn-Teller distortions from static to quasistatic and then of dynamic ones. The quasistatic and dynamic orbital correlations favour ferromagnetic state, while A-type antiferromagnetic state is typical for the static Jahn-Teller distortions. The possible types of orbital structures and their relationship with the magnetic structures are discussed. It is argued that the canting magnetic structure in manganites is not realized. The transition to ferromagnetic state occurs via the formation of inhomogeneous state with different type of orbital correlations and magnetic structure. The key role of intrinsic chemical inhomogeneity in the formation of inhomogeneous magnetic states in solid solutions of manganites and cobaltites is emphasized. The influence of orbital correlations on the formation of ferromagnetic state and metal-insulator transition in cobaltites is discussed.

14:45 - 15:30

Probing the phase separation in the doped manganites by the magnetic resonance methods

invited oral

Nikita V. Volkov¹⁾, Guerman A. Petrakovskii¹⁾, Klara A. Sablina¹⁾

1) L.V.Kirensky Institute of Physics Siberian Branch of the Russian Academy of Science (IPH SB RAS), Akademgorodok, 50, Krasnoyarsk 660036, Russian Federation

Here we present the magnetic resonance studies which confirm the existence of the phase separation picture in the $\text{Eu}_{0.7}\text{Pb}_{0.3}\text{MnO}_3$ and $\text{La}_{0.7}\text{Pb}_{0.3}\text{MnO}_3$ single crystals. These studies demonstrate that the magnetic resonance method is an effective tool for probing inhomogeneous states in the manganites. The measurements were performed with a conventional technique and with using unconventional schemes of the magnetic resonance method: a spectrometer operating in a wide frequency range with a pulsed external magnetic field; a spectrometer with combined influence of the microwave irradiation and dc current. The magnetic resonance studies display the coexistence of two absorption lines in spectra, which are related to different regions in the samples. The inhomogeneous state takes place in $0.65T_C - 1.15T_C$ temperature range, where the CMR effect is observed. Studying the microwave frequency dependence of the spectra is the main feature of our investigation. This method is very powerful for the determination of the magnetic state of the coexisting phases. The frequency-field dependencies of the spectra allow to conclude that these phases are paramagnetic (PM) and ferromagnetic (FM) ones. Moreover studying of the frequency dependences is found to be an effective tool for probing the sensitivity of the mixed-phase state to the magnetic field. The behavior of the spectra suggests that there takes place the scenario of the phase separation and the mixed state is not related to the simple chemical inhomogeneity. The changes of the conductivity induced by the microwave resonance absorption are observed in a vicinity of T_C , where inhomogeneous PM-FM state is the ground state for the sample. The changes are not related to a trivial heating of the sample by the microwave radiation. We propose the mechanism connected with the change of equilibrium concentrations of the coexisting phases, this change takes place when the system is under magnetic resonance conditions.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duža aula)

15:50 - 17:10

Afternoon session - continued

Main Building, room 144

15:50 - 16:35

Long length scale interaction between magnetism and superconductivity in $\text{La}_{0.3}\text{Ca}_{0.7}\text{MnO}_3/\text{YBa}_2\text{Cu}_3\text{O}_7$ superlattices

invited oral

Santamaria Jacobo¹⁾, V Pena¹⁾, Z. Sefrioui¹⁾, D. Arias¹⁾, C. Leon¹⁾, M. Varela³⁾, S. J. Pennycook³⁾, J. L. Martinez²⁾

1) Universidad Complutense de Madrid, Avda. de la Complutense s/n, Madrid 28040, Spain

2) Instituto de Ciencia de Materiales de Madrid, Cantoblanco, MADRID 28049, Spain

3) Oak Ridge National Laboratory, Solid State Division, Tennessee, United States

The competition between magnetism and superconductivity has been the focus of considerable research effort in recent years. The interplay between these two antagonistic long range orderings gives rise to quite exotic phenomena like spatial modulation of the order parameter, pi-junctions, etc, whose study, apart of its fundamental interest, may also open the way to important applications in the field of spintronics. The new oxides (high T_c (HTS) and colossal magnetoresistance (CMR) materials) offer a new scenario to explore this interplay at the nanometer scale. The spin polarization of the conduction band of the manganites is expected to suppress the superconductivity over very short length scales (0.1 nm) into the ferromagnet, and the short coherence length of the superconductor will make superconductivity to survive over very short length scales. In this talk, I will explore the issue of the ferromagnetic / superconducting (F/S) proximity effect in $\text{La}_{0.3}\text{Ca}_{0.7}\text{MnO}_3(\text{LCMO})/\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) superlattices. We

have grown LCMO/YBCO superlattices by high oxygen pressure sputtering technique epitaxially on (100) SrTiO₃ changing the thickness of the individual layers between 1 and 15 nm. A structural study using x-ray refinement and transmission electron microscopy shows sharp interfaces with a high degree of structural perfection. Magnetization (SQUID) and transport measurements show the "coexistence" of magnetism and superconductivity. While the thinnest LCMO layers (3 unit cells) leaved the superconducting critical temperature almost unchanged, thicker LCMO layers result in a systematic reduction of the critical temperature over a wide thickness interval of the manganite layer. These results suggest a long nanometer scale length for superconductivity depression into the ferromagnet. We discuss this result in terms of the F/S proximity effect.

+ Work supported by MCYT MAT 2000- 1468, Fundacion Ramon Areces, CAM.

[ABSTRACT TRUNCATED TO 2000 LETTERS]

Magnetoelastic properties of (La_{0.8}Ba_{0.2})_{0.93}MnO₃ single crystal.

Yuriy Bukhantsev¹⁾, Bohdan Kundys¹⁾, S. Vasiliev¹⁾, Ya. M. Mukovskii²⁾, A. Nabialek¹⁾, R. Szymczak¹⁾, Henryk Szymczak¹⁾

16:35 - 17:05

oral

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

2) *Moscow State Steel & Alloys Institute, Moscow 117936, Russian Federation*

We have investigated magnetic and magnetoelastic properties of (La_{0.8}Ba_{0.2})_{0.93}MnO₃ single crystal over temperature range of 4.2-150 K in magnetic fields up to 12T. The basic magnetostriction constants of the (La_{0.8}Ba_{0.2})_{0.93}MnO₃ single crystal have been estimated using longitudinal and transversal magnetostriction measurements data, which were carried out along different crystallographic directions of single crystalline sample. The variation of magnetic domain structure in applied external magnetic field has also been analyzed using magnetization hysteresis loop measurements.

Acknowledgement

This work was supported by KBN research grant 2P03B07824

NMR study of (Sr,Ba,La)₂Fe_{1+x}Mo_{1-x}O₆ double perovskites

Dariusz A. Zajac²⁾, Czesław Kapusta²⁾, Peter C. Riedi³⁾, Marcin Sikora²⁾, Colin J. Oates²⁾, Damian Rybicki²⁾, Jose M. De Teresa¹⁾, Clara Marquina¹⁾, Ricardo M. Ibarra¹⁾

17:05 - 17:35

oral

1) *Universidad de Zaragoza-CSIC, Facultad de Ciencias, Pedro Cerbuna 12, Zaragoza 50009, Spain*

2) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland*

3) *School of Physics and Astronomy, University of St. Andrews, St. Andrews KY16 9SS, United Kingdom*

The ⁹⁵Mo and ⁹⁷Mo nuclear magnetic resonance study of (Sr,Ba,La)₂(FeMo)O₆ double perovskites is reported. The compounds exhibit a significant low field magnetoresistance at room temperature. Compounds with different (SrBa) - La composition, different content of cation vacancies as well as different Fe - Mo ratio have been studied. The frequency swept spin-echo spectra have been measured at 4.2K and no applied magnetic field. The parent compound, Ba_{1.6}Sr_{0.4}FeMoO₆, shows a main line at 59 MHz and a satellite line at 43 MHz. They are attributed to the ideal and defect (antisite neighbours and antiphase boundaries) sites of molybdenum. Both lines correspond to ⁹⁵Mo and ⁹⁷Mo resonances, which are unresolved due to very close gyromagnetic ratios. The spectrum of the 10% La doped sample, Ba_{1.44}Sr_{0.36}La_{0.2}FeMoO₆ shows a higher intensity of the low frequency satellite line, which indicates an increased number of defect sites. The main line is at higher frequencies compared to the parent compound and shows a satellite structure at the high frequency side. An increase of the number of vacancies at the same La doping Ba_{1.28}Sr_{0.32}La_{0.2}FeMoO₆ further increases the population of defect sites. In the Ba_{1.28}Sr_{0.32}La_{0.2}Fe_{1.2}Mo_{0.8}O₆ compound the change of the Fe-Mo stoichiometry significantly decreases the amount of antisite defects. The results are analysed in terms of the influence of doping on the Mo magnetic moments and population of defect sites. A relation to the magnetic and transport properties of the compounds is discussed.

Thursday, September 18th

Morning session

09:00 - 10:30

Main Building, room 144

Magnetoressistance and magnetic properties of the double perovskites

Rudolf Gross¹⁾, J.B. Philipp¹⁾, P. Majewski¹⁾, L. Alff¹⁾, A. Erb¹⁾, T. Graf²⁾, J. Simon³⁾, D.D. Sarma⁴⁾

9:00 - 9:45

invited oral

1) *Walther-Meissner-Institute (WMI), Walther-Meissner Str. 8, Garching D-85748, Germany*

2) *Walter-Schottky-Institute (WSI), Garching D-85748, Germany*

3) *Institute for Anorganic Chemistry, Bonn D-53117, Germany*

4) *Indian Institute of Science, Bangalore 560 012, India*

The Curie-temperature and the saturation magnetization of double perovskites is known to depend strongly on structural and electronic degrees of freedom. We have performed a detailed experimental study of the system A_2CrWO_6 with $A = Sr, Ba$ and Ca . The use of different alkaline earth ions with different ionic radii on the A site results in a significant variation of the tolerance factor. The Curie temperature of polycrystalline A_2CrWO_6 samples was found to be strongly reduced for Ca_2CrWO_6 and Ba_2CrWO_6 as compared to Sr_2CrWO_6 due to a tolerance factor significantly deviating from unity resulting in deviations from the ideal perovskite structure. Electron doping by La^{3+} substitution on the A site also reduces the Curie temperature and the saturation magnetization both due to disorder and band structure effects. We also have fabricated high quality epitaxial thin films of Sr_2CrWO_6 and studied their magnetotransport properties [1,2].

[1] J.B. Philipp et al., Appl. Phys. Lett. 79, 3654 (2001).

[2] J.B. Philipp et al., J. Appl. Phys. 93, 6853 (2003).

9:45 - 10:30

Growth of manganite single crystals and their properties

Yakov M. Mukovskii¹⁾

invited oral

1) Moscow State Steel & Alloys Institute, Moscow 117936, Russian Federation

Features of manganite single crystal growth by the non-crucible floating zone melting method with radiation heating are discussed. Compounds $(La_{1-x}A_x)MnO_3$ ($A = Sr, Ca, Ba$) with $x < 0.2$ can be fabricated using feed rods of $(La_{1-x}A_x)_yMnO_3$ composition with $y = 0.93-0.97$ because Mn is evaporated from the melt larger than other elements. Due to the fact that the distribution coefficient ($K = C_{Solid}/C_{Liquid}$) for Ca and Ba is considerably less than unity growing of such crystals with $x > 0.2$ is rather unstable, and element distribution along an ingot is inhomogeneous. To fabricate quality crystals travelling solvent floating zone method was used.

Results of real structure effect on various physical properties are demonstrated. Some results obtained at perfect single crystals such as $R(T)$ dependence, structural transitions, neutron dynamics, polaron formation, etc.

10:30 - 11:00

Coffee break

Main Building, Main Hall (Duža aula)

11:00 - 12:30

Morning session - continued

Main Building, room 144

11:00 - 11:45

The Effect of Rich Cr Doping of Perovskite Manganites on the Mn-site. A neutron diffraction study

invited oral

Eleni Gamari-Seale

Abstract. Neutron powder diffraction study of the rich doped manganite with Cr on the Mn-site, $Nd_{0.6}Ca_{0.4}(Mn_{1-x}Cr_x)O_3$ in powder form, revealed the magnetic unit cell retains the CE type structure as the parent compound $Nd_{0.6}Ca_{0.4}MnO_3$ but the charge ordered (CO) and orbital ordered (OO) observed in the parent compound is destroyed. The Mn and Cr form antiferromagnetic zig-zag chains along the a-axis. These planes are stacked along the b axis but with opposite spins. At low temperature there is a sign of canted structure. The Nd sublattice orders at low temperature in a ferromagnetic way. The Nd magnetic moment obtains the small value of $0.5\mu_B/10\nu$ at low temperatures.

11:45 - 12:30

Magnetic reversal and relaxation in La-Ca-Mn-O based exchange-biased multilayers

invited oral

Ioannis Panagiotopoulos³⁾, Nikolaos Moutis¹⁾, Christos Christides²⁾

1) Institute of Materials Science "Demokritos", 15 310 Ag.Paraskevi, Athens 153 10, Greece

2) Department of Engineering Sciences, School of Engineering, University of Patras, Patras 26 110, Greece

3) Department of Materials Science and technology, University of Ioannina, Ioannina 45110, Greece

In mixed valence manganese perovskites the competition between the various types of interactions as double exchange, Jahn-Teller coupling, Coulomb, and antiferromagnetic superexchange which are sensitive to the structural parameters and doping creates a rich phase diagram with various types of magnetic, orbital and charge ordering. Thus they present an ideal system for building exchange-biased antiferromagnetic (AF), ferromagnetic FM superlattices since the structural compatibility of the AF and FM layers permits coherent growth of the superlattice that satisfy the conditions for magnetic coupling at the interfaces.

Magnetic and magnetotransport measurements in La-Ca-Mn-O based FM/AF multilayers prepared by pulsed laser magnetic measurements have revealed the presence of an exchange-biasing mechanism at low temperatures.

The blocking temperature (T_B) distribution has been estimated by the dependence of H_{EB} on a field cooling process that includes field reversal at a intermediate temperatures. The derived T_B distribution as well as the

exponential thermal decay of H_{EB} can be attributed to the competing interactions and phase separation at the AF/FM interfaces that give rise to a spin-glass-like magnetic disorder. The asymmetry of the magnetization reversal has been probed by (i) magnetoresistance measurements (ii) temperature dependence of the left and right coercive fields (iii) and magnetic viscosity measurements. The magnetic relaxation follows a $\ln(t)$ dependence at 5 K, which is a universal feature of a slow relaxation process, whereas above the blocking temperature of $T_B=70\text{K}$ the time decay requires an additional exponential term. Below T_B , the observed loop asymmetries in the irreversible susceptibility and the magnetic viscosity S reveal two inequivalent reversal mechanisms between the field increasing and field decreasing branches of the hysteresis loop.

Lunch break

12:30 - 14:00

Afternoon session

Main Building, room 144

14:00 - 15:30

NMR in ferromagnetic manganites

Pavel Novak¹⁾, **Michail M. Savosta**²⁾

14:00 - 14:45

1) *Institute of Physics, Academy of Sciences of the CR, Cukrovarnicka 10, Praha 6 162 53, Czech Republic*
 2) *A. Galkin Donetsk Institute for Physics & Technology, National Academy of Sciences of Ukraine (DonPTI NASU), Roza Luxemburg 72, Donetsk 83114, Ukraine*

invited oral

Nuclear magnetic resonance is a suitable tool to provide insight in the nature of the complex physics of the ferromagnetic manganites as it probes locally the magnetic states and their dynamics. The combination of NMR and dc magnetization studies proved to be of particular importance when determining the nature of the magnetic transition.

In this work we present selected results of an extensive study of the temperature dependence of ^{55}Mn nuclear magnetic resonance and relaxation in the mixed valence ferromagnetic manganites, completed in several cases by the ^{139}La NMR and dc magnetization measurements. In particular the crossover between the second- and first-order magnetic transition in $\text{La}_{0.7}\text{M}_{0.3}\text{MnO}_3$ ($M=\text{Ca, Sr, Ba}$) manganites is analysed and the temperature dependence of the spin-lattice and spin-spin nuclear relaxation in $\text{La}_{1-x}\text{Na}_x\text{MnO}_3$ system is used to determine the polaronic nature of the charge carriers. NMR evidence of the magnetic phase separation in ferromagnetic manganites is also given and the charge density wave in $\text{La}_{0.84}\text{Sr}_{0.16}\text{MnO}_3$ is revealed.

Spontaneous Ferromagnetic Domains in CMR Manganites

Grzegorz Jung^{1,2)}

14:45 - 15:30

1) *Ben-Gurion University of the Negev, P.O.Box 653, Beer-Sheva 84105, Israel*
 2) *Institute of Physics Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw, Poland*

invited oral

Rare-earth manganites of the composition $\text{R}_{1-x}\text{A}_x\text{MnO}_3$, where R stands for a trivalent rare earth ion such as La, Nd, Pr or Gd and A for a divalent alkaline ion, exhibit the colossal magnetoresistance effect (CMR) resulting from a subtle balance between spin, orbital, charge and lattice degrees of freedom. CMR manganites have a complex phase diagram in which metal-insulator transition accompanies the magnetic ordering transition. CMR is found on a magnetic field scale of several Tesla, which is not very appealing for applications. This stimulated investigations of extrinsic magnetoresistance effects such as; grain-boundary magnetoresistance, spin-polarized tunnelling and domain-wall magnetoresistance, promising a large magnetoresistance ratio in low magnetic fields.

The extrinsic effects may dominates transport properties of classical and CMR ferromagnets. Resistivity increase at low temperatures in lightly doped $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ (LCMO) manganites has been associated with spin-dependent tunnelling between adjacent ferromagnetic domains. A serial arrangement of intrinsic tunnel junctions leads to pronounced resistance oscillations. It has been speculated that intrinsic tunnel junctions are located within the domain walls. Surprisingly, little is known about the magnetic domain structures in CMR perovskite single crystals. In this presentation we concentrate on spontaneous magnetic domains visualized and investigated by means of the magneto-optical technique and their relation to the low temperature transport properties of LCMO manganites

Coffee break

Main Building, Main Hall (Duża aula)

15:30 - 15:50

- 15:30 - 16:00
oral
- An NMR study of $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{Mn}_{0.97}\text{Ga}_{0.03}\text{O}_3$**
**Colin J. Oates¹⁾, Czesław Kapusta¹⁾, Peter C. Riedi²⁾, Marcin Sikora¹⁾, Dariusz A. Zajac¹⁾,
 Damian Rybicki¹⁾, Christine Martin³⁾, C. Yaicle³⁾, Antoine Maignan³⁾**
 1) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland
 2) School of Physics and Astronomy, University of St. Andrews, St. Andrews KY16 9SS, United Kingdom
 3) Laboratoire CRISMAT - UMR 6508, ISMRA et Université de Caen, 6 Boulevard de Marechal JUIN,
 14050 Caen Cedex, France

An NMR study of $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{Mn}_{0.97}\text{Ga}_{0.03}\text{O}_3$ is reported. The parent $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ compound is a charge ordered insulator. Gallium doping destroys the charge order and brings about metallicity and a step-like behavior of the electrical resistivity and magnetization in the applied magnetic field. Spin-echo spectra were measured on polycrystalline samples at zero external field and 4.2 K. They consist of an unresolved $^{69,71}\text{Ga}$ peak centered at 74 MHz, a ^{55}Mn double exchange (DE) line at 375 MHz and ^{141}Pr quadrupole-split spectrum centered at 605 MHz. The Ga, Mn and Pr hyperfine fields amount to 6.6 T, 35.7 T and 48.4 T, respectively. The Mn DE line corresponds to metallic ferromagnetic regions. A variation of spin-spin relaxation time T_2 across the line shows that the size of the DE regions is at least 10 nm in size. The Pr hyperfine field is significantly lower than the free ion value, which is 338 T. The quadrupole splitting, 78 MHz, is one order of magnitude larger than the free ion value, which indicates a large lattice contribution to the electric field gradient at the Pr site. From the analysis, information on the spectroscopic state of Pr ion is derived and a relation to the magnetic properties of the compound is discussed.

- 15:50 - 17:50
- Afternoon session - continued**
 Main Building, room 144

- 16:00 - 16:30
oral
- Giant volume magnetostriction in manganites and its connection with colossal magnetoresistance**
Liudmila I. Koroleva¹⁾, Roman V. Demin¹⁾, Alexey V. Michurin¹⁾, Anna I. Abramovich¹⁾
 1) M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation

Giant volume magnetostriction $\sim 10^{-4}$ - 10^{-3} near Curie point TC is found in $\text{A}1\text{-xSrxMnO}_3$ (A = Sm, Nd and La). This discovery make possible the magneto-mechanical devices on a basis of manganites. In this work the elastic, magneto-elastic, magnetic and electrical properties of $\text{La}1\text{-xSrxMnO}_3$ (x = 0.1, 0.125, 0.15, 0.3) single crystals, and $\text{A}1\text{-xSrxMnO}_3$ (A = Nd, Sm; x = 0.33, 0.4, 0.45) ceramics are studied. For all the samples a volume magnetostriction w is negative and the $|w|(T)$ curves go through maximum near of TC. At $T \uparrow TC$ thermal expansion Dl/l exceeds linear on T thermal expansion. The behavior of CMR and w near TC are similar in $\text{La}1\text{-xSrxMnO}_3$: isotherms are not saturated up to the maximum field of measurement ~ 120 kOe; both of them are negative; the maximum magnitudes, taken place near TC, decrease with the rise of x. Similar behavior of CMR and w was observed for $\text{A}1\text{-xSrxMnO}_3$ (A = Nd, Sm, x = 0.33). However the behavior of w and CMR was different in compounds with x = 0.4 and 0.45 of $\text{Sm}1\text{-xSrxMnO}_3$ system, in which ferromagnetic (F), anti-ferromagnetic (AF) of A-type and of CE-type charge ordering (CO) clusters were observed from neutron diffraction. Both F and A-type AF clusters break down in the TC - region and CO AF clusters remain at $T > TC$. Here the transition of CO clusters to F state, induced by magnetic field H, is accompanied by the sharp jumps on isotherms of magnetization, magnetoresistance and volume magnetostriction at $H \sim HC1$ and their saturation is achieved at $H \sim HC2$. This transition leads to CMR and giant magnetostriction ($\sim 10^{-3}$); to say, CMR and giant magnetostriction take place in that case when F ordering regions increase by the magnetic field at $T \uparrow TC$. Peculiarities of w, Dl/l and CMR near TC are explained magnetic two-phase F-AF state due to strong s-d exchange [1].

[1] A. Yanase and T. Kasuya. J. Phys. Soc. Japan 25 (1968) 1025

Friday, September 19th

- 09:00 - 10:30
- Morning session**
 Main Building, room 144

- 9:00 - 9:45
invited oral
- Tailoring magnetic anisotropy in doped LaMnO_3 thin films**
Hanns-Ulrich Haberman¹⁾, Zhihong Wang¹⁾, Georg Cristiani¹⁾
 1) Max-Planck-Institut FKF, Heisenbergstr. 1, Stuttgart D70569, Germany

Magnetic anisotropy in ultrathin films of doped rare earth manganites with on their high degree of spinpolarization attracts increasing interest, both, from fundamental physics aspects as well as aspects due to its potential application in novel spintronics devices.

We approach the problem by two different experimental routes. One is based on the application of SrTiO₃ single-crystal (100) oriented substrates with a vicinal cut along the [010] direction to tailor the in-plane magnetic anisotropy along the [100] direction, the other makes use of the compressive epitaxial strain between a CaMnO₃ layer and the ferromagnetic La-Sr-Mn-O film to generate a perpendicular magnetic anisotropy. The films are produced by the pulsed laser deposition technique and characterized by structural [x-ray, TEM] as well as transport and magnetic measurements.

Giant magnetoresistivity in single crystals of rare earth layered cobaltites
Sergei N. Barilo¹⁾, Sergei V. Shiryayev¹⁾, Georgii L. Bychkov¹⁾, Denis V. Sheptyakov⁴⁾, Zhixian Zhou³⁾, Scott Alexander³⁾, Jack E. Crow³⁾, Andrew Podlesnyak²⁾, Peter Allenspach²⁾, Albert Furrer²⁾

9:45 - 10:30

invited oral

1) *The Institute of Solid State & Semiconductor Physics, Belarus NAS (ISSSP), P. Brovka, Minsk 220072, Belarus*

2) *Laboratory for Neutron Scattering, ETHZ & PSI Villigen (LNS), Switzerland*

3) *The National High Magnetic Field Laboratory (NHMFL), Tallahassee, United States*

4) *Laboratory for Neutron Scattering ETHZ & PSI Villigen (LNS), Villigen, Switzerland*

It was shown recently LnBaCo₂O_{5+delta} (Ln-112, Ln being rare earth ion, 0 < = delta < = 1) layered cobaltites present giant magnetoresistivity (GMR) phenomena accompanied by fascinating features with structural, metal-insulator, and magnetic field induced transitions, as well as charge and orbital ordering phenomena on the verge of Co³⁺ spin-state transitions. Early work focused on studies sintered polycrystalline and powder samples. High quality single crystals are essential to understand nature of GMR phenomena in the layered cobaltites, which is differing cardinal from the nature of colossal magnetoresistance in the wellknown rare earth manganites. The original approach to flux growth was developed to provide large and high quality single crystals of the oxygen deficient layered cobaltates. Fortunately, we found co-crystallization of the Ln-112 phase and new cobaltite phases in wide ranges of flux melt composition and temperature for the most of Ln₂O₃ - BaO - CoO systems. The data on primary crystallization fields of the phases, which appear in parts of corresponding Gibbs triangles at temperature in the range 1373 - 1523K allow us to presume a tentative (T-x) quasi binary cut of the system phase diagram. Here we also report on first growth of the Ln-112 (Ln = Pr, Eu, Gd, Tb, Dy) single crystals as large as 6 x 6 x 1.5 mm³ and confirm GMR property of the compounds by measurements of the crystals in external fields up to 30T. Highly anisotropic negative GMR effect (up to 80% at 4T, 40K) was registered in Gd-112 single crystal. Data of neutron powder diffraction of Tb-112 and magnetic susceptibility of weakly twinned Gd-112 single crystal unambiguously shown the layered cobaltites are easy axis magnets with spontaneous magnetization alignment along a or b-axis of the structure. Metamagnetic behavior of cobalt sublattice in the basal plane of Eu-112 single crystal was observed to serve as a base for constructing of generic H-T magnetic phase diagram.

Coffee break

10:30 - 11:00

Main Building, Main Hall (Duža aula)

Morning session - continued

11:00 - 12:30

Main Building, room 144

Oxides for spintronics

Manuel Bibes¹⁾, Vincent Garcia¹⁾, Martin Bowen¹⁾, Pierre Seneor¹⁾, Manuel Munoz¹⁾, Agnes Barthélémy¹⁾, Karim Bouzehouane¹⁾, Stéphane Fusil²⁾, Vincent Cros¹⁾, Julian Carrey¹⁾, Michael Besse¹⁾, Annie Vaures¹⁾, Jean-Pierre Contour¹⁾, Albert Fert¹⁾

11:00 - 11:45

invited oral

1) *Unité Mixte de Physique CNRS-Thales, Domaine de Corbeville, Orsay 91404, France*

2) *Université d'Evry, Rue du Pere Jarlan, Evry 91025, France*

In spintronics, to obtain large effects it is of paramount importance to master sources of highly spin-polarized charge carriers. The simplest way to achieve this is to use half-metallic materials, like mixed-valence manganites (A_{1-x}A'_xMnO₃). When integrated as electrodes in magnetic tunnel junctions (MTJs), manganites have proved to yield large tunnelling magnetoresistance (TMR) values, which underscores their usefulness for spintronics applications. In this talk, we will review recent results obtained at Orsay in manganite MTJs. At low temperature, a TMR of 1800 % is obtained in a La₂/3Sr₁/3MnO₃ / SrTiO₃ / La₂/3Sr₁/3MnO₃ junction. This corresponds to a spin-polarization of 95% for La₂/3Sr₁/3MnO₃ (LSMO) at the interface with SrTiO₃ (STO). The bias voltage dependence of the TMR shows a very peculiar behaviour, from which quantitative information on the spin-dependent DOS above EF can be extracted. We have also studied the temperature dependence of the TMR in MTJs with STO and TiO₂ barriers. In both systems, the TMR vanishes in the 280K range. Even if, at low temperature, the spin-polarization of the LSMO / TiO₂ interface is only 70 % as opposed to 95 % in the case of LSMO / STO, the decay upon rising T of both PLSMO/STO and PLSMO/TiO₂ is very similar, and differs from that of the spin-polarization of a free LSMO surface. We will discuss the possible reasons for this behaviour.

A possible candidate to obtain TMR at room temperature is Sr₂FeMoO₆ (SFMO), which has a TC above 420K. The growth of SFMO films is problematic and the films usually show a morphology incompatible with a classical lithography process. We have circumvented this difficulty by defining junctions with areas in the 10-100 nm² range, thanks to a new technology of nanolithography, based on nanoindentation. At low temperature, a positive TMR of 45% is obtained in a SFMO / STO / Co nanojunction. This confirms the large negative spin-polarization of SFMO predicted by band-structure calculations.

11:45 - 12:30

Specific heat studies of magnetic and structural phase transitions in La_{1-x}Sr_xMnO₃ compositions

invited oral

Andrzej Szewczyk¹⁾

1) *Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland*

Manganites show a variety of magnetic and structural phase transitions, which have a diverse influence on their thermal properties. With respect to this, thorough studies of temperature dependence of specific heat for several compositions of the series La_{1-x}Sr_xMnO₃ for x < 0.19 were performed using a relaxation method over the temperature range from 2 K to 385 K, in zero magnetic field, B, and in the field B=7 T.

As expected, the transition from the ferromagnetic to paramagnetic phase was visible on the temperature dependence of specific heat as a well-pronounced maximum, strongly influenced by magnetic field. Basing on these studies, the adiabatic change of temperature induced by a change in B, i.e., the main parameter characterizing the magnitude of the magnetocaloric effect, was determined. It was equal to ~3.5 K at a change of B by 7 T, which demonstrates unequivocally that the magnetocaloric effect in the studied compounds is small, despite of the fact that large B-induced isothermal entropy changes were found in numerous works, by analyzing the magnetization measurements.

An unusual behaviour was found for the structural transition from the rhombohedral (R) to the orthorhombic (O*) phase. For compositions for which the transition took place above the Curie temperature, e.g., for x=0.185, it was visible as a well-pronounced maximum, not affected by B, and not showing a temperature hysteresis. When the transition occurred below the Curie temperature, e.g., for x= 0.155, no anomaly at the transition temperature was observed. The only indication of the presence of the transition was a steep change of the temperature relaxation time, which indirectly demonstrated the change of the thermal conductivity of the sample.

A qualitatively different anomaly accompanied the other structural transition between two orthorhombic phases, O'-O*. At this transition, a bend on the temperature dependence of specific heat was observed, e.g., for x=0.155.

12:30 - 14:00

Lunch break

14:00 - 15:00

Afternoon session

Main Building, room 144

14:00 - 14:30

Local Probe Studies of high-Tc Superconductors and CMR Oxides Using Radioactive Isotopes

oral

V. S. Amaral²⁾, **J. P. Araujo**³⁾, **A.M.L. Lopes**^{2,5)}, **E. Rita**^{1,5)}, **J. G. Correia**^{1,5)}, **U. Wahl**¹⁾, **P. B. Tavares**⁴⁾, **ISOLDE Collaboration**⁵⁾

1) *Instituto Tecnológico Nuclear (ITN), Sacavém 2686, Portugal*

2) *Departamento de Física and CICECO, Universidade de Aveiro (UA), Campus de Santiago, Aveiro 3810-193, Portugal*

3) *Departamento de Física and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal*

4) *Departamento de Química, Universidade de Trás-os-Montes e Alto Douro (UTAD), Vila Real 5001-911, Portugal*

5) *European Centre for Nuclear Research (CERN), Geneva CH1211, Switzerland*

The route to induce high-Tc superconductivity and CMR relies on the doping of the parent antiferromagnetic insulator compound which is commonly performed by partial or total replacement of elements, incorporation of vacancies and/or oxygen. In such intrinsically disordered systems, microscopic phenomena like point or extended defects or the lattice site of dopants are known to play a crucial role on their electric and magnetic properties, pointing to the existence of key microscopic phenomena for which atomic-scale studies are highly desirable. Nuclear hyperfine techniques are extremely useful in providing atomic scale information on the probe-element interactions with the lattice host. In particular, the measurement of the electric field gradient (EFG) and/or the magnetic hyperfine fields (MHF) at specific probe-element nuclei provides direct information on the local charge distribution and/or local spin density transfer, thus providing a fingerprint of the lattice site and the specific point defects located at the probe's neighbourhood. In this way, nuclear techniques using radioactive isotopes like γ - γ , e^- - γ Perturbed Angular Correlations (PAC) and the Electron Emission Channelling (EC) techniques were used to provide

relevant information on the electronic environment and the lattice location of specific elements or probes on high-Tc superconductors and CMR oxides. We present studies on: a) Unusual properties of cadmium doped manganites b) Mercury doping of $\text{YBa}_2\text{Cu}_3\text{O}_7$ high-Tc superconductor c) Oxygen doping of Hg-based high-Tc superconductors and structure of oxygen defects.

Charge-ordering and magnetoelastic coupling effects on the magnetocaloric properties of manganites

14:30 - 15:00

Vitor S. Amaral²⁾, Mario S. Reis^{1,2)}, A. M. Gomes¹⁾, J. P. Araújo³⁾, P. B. Tavares⁴⁾, J. S. Amaral²⁾, I. S. Oliveira¹⁾

oral

1) *Centro Brasileiro de Pesquisas Físicas (CBPF), R. Dr. Xavier Sigaud 150 Urca, Rio de Janeiro-RJ 2229-180, Brazil*

2) *Departamento de Física and CICECO, Universidade de Aveiro (UA), Campus de Santiago, Aveiro 3810-193, Portugal*

3) *Departamento de Física and IFIMUP, Universidade do Porto (UP), Rua do Campo Alegre, 687, Porto 4169-007, Portugal*

4) *Departamento de Química, Universidade de Trás-os-Montes e Alto Douro (UTAD), Vila Real 5001-911, Portugal*

A study of the magnetocaloric properties of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$, $\text{La}_{1-x-y}\text{Y}_y\text{Ca}_x\text{MnO}_3$ and $\text{Pr}_{1-x}\text{Ca}_x\text{MnO}_3$ manganites with ferromagnetic and charge-ordered states is presented. The ferromagnetic Lanthanum manganites show a negative entropy change peak ΔS under the application of a magnetic field at the Curie temperature. However, the magneto-elastic coupling effects lead to a first-order paramagnetic-ferromagnetic transition and an increased ΔS compared to simple ferromagnets. This effect is analysed in the framework of the Landau theory of phase transitions which shows a comparable influence of the magnetoelastic couplings and the ordinary magnetic ordering effect.

In contrast, the $\text{Pr}_{1-x}\text{Ca}_x\text{MnO}_3$ system shows a richer electric and magnetic phase diagram. For $0.15 < x < 0.30$ a ferromagnetic insulator phase is established. A more complex electric-magnetic phase diagram is found for $0.30 < x < 0.85$, where the charge-ordering effect coexists with an antiferromagnetic insulator order. The results for $x=0.25$ and 0.30 show a simple ferromagnetic phase transition effect. For the samples above the onset concentration for the charge ordering (~ 0.30) an anomalous magnetic entropy change is observed below the charge ordering temperature (TCO). This effect is associated with a positive contribution from the magnetic entropy change due to charge-ordering, superimposed to the negative contribution due to spin-ordering. Such positive entropy contribution can be understood as the increase of accessible states due to the increase of electron mobility, under an applied magnetic field. Moreover, at low temperatures, when the magnetic field induced metal insulator transition becomes irreversible, we find extremely large values of the magnetic entropy change. For $x=0.32$, ΔS reaches -20.8 J/kg-K , under a 4T magnetic field.

Symposium E

Materials for Medical Applications

Symposium organisers

- **Malgorzata Lewandowska-Szumiel**, Medical University of Warsaw, Department of Biophysics and Human Physiology; Department of Transplantology&CTB
- **Alma U. Daniels**, University of Basel, Switzerland
- **Joost D. de Bruijn**, Bilthoven, Netherlands
- **Ralf-Peter Franke**, Ulm University, Germany
- **Malgorzata Lewandowska**, Warsaw University of Technology, Faculty of Materials Science and Engineering, Poland
- **Luigi Ambrosio**, Institute of Composite & Biomedical Materials (IMCB-CNR), Italy

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Monday, September 15th

Opening address

13:55 - 14:00

Afternoon session - Joost D. de Bruijn, Malgorzata Lewandowska Szumiel

14:00 - 15:30

Biomimetic hydrogels for tissue reconstruction

Luigi Ambrosio¹⁾, Paolo A. Netti²⁾

14:00 - 14:30

1) Institute of Composite & Biomedical Materials (IMCB-CNR), Piazzale Tecchio, 80, Naples 80125, Italy

invited oral

2) Department of Materials & Production Engineering, University of Naples "Federico II" (DIMP-CRIB), Piazzale Tecchio, 80, Naples 80125, Italy

One of the principal goals of the biomedical engineering is to design of biomaterials capable to replace, substitute or repair natural tissues. Over the past twenty years the research in this field has led to the formulation of novel highly compatible materials and to new methods to obtain materials of natural origin able to better interact with biological tissues. However, a major problem with these materials is that they do not reproduce the functional and biological properties of the natural tissues. An ideal biomaterial must possess a complete set of biophysical, structural and biological properties. Our approach to the design of novel biomaterials is inspired to nature guidelines to mimic the behavior of natural tissue. Following this approach we have been able to design prosthesis for dental implant, bone substitutes, intervertebral disc, ligaments, and scaffolds for tissue engineering.

Replication of the structure and the functions of the extracellular matrix in vitro is performed by forming a semi-Interpenetrated Polymer Network (s-IPN) between Collagen type I and Hyaluronic acid (Hyal)

The rheological behaviour of the semi-IPN collagen/HA have been analysed to evaluate the effect of Hyaluronic Acid and collagen in the composite gel. The presence of Hyaluronic Acid induces a reinforcement of the collagen network due to the Hyal interaction with collagen during fibrillogenesis. This is also confirmed by the electron micrographs which show that collagen fibres and HA are strongly interconnected and the collagen fibers network is fully developed despite the presence of the HA.

The effect of collagen scaffold properties such as stiffness and morphology on the cellular processes of growth, migration and remodelling that occur within 3D polymer matrices have been analysed. The results indicate that is possible in principle to control cell growth and migration by modulating the physical properties of the material.

Physical & Mechanical Properties of Tissue Engineering Scaffolds & Constructs

14:35 - 15:05

Alma U. Daniels²⁾, Dieter Wirz²⁾, Martin Stolz³⁾, Matteo Moretti¹⁾, Ivan Martin¹⁾, Ueli Aebi³⁾

invited oral

1) Tissue Engineering Res. Group, University Hospital, Basel, Switzerland

2) Lab. for Orthopedic Biomechanics, Univ. Basel, Basel, Switzerland

3) Inst. for Structural Biology, Univ. of Basel, Basel, Switzerland

INTRODUCTION: To date, engineering of load-bearing tissues has focused mainly on two difficult tasks: persuading harvested and cultured cells to proliferate while achieving and maintaining phenotype (e.g., chondrocytes), and coaxing them to begin making tissue (e.g., cartilage). Both tasks are undertaken in scaffold materials, which become constructs implanted to replace damaged tissue. Many materials have been tried empirically, with some success. Attention is turning increasingly to two questions: (1) Why do some scaffold materials better encourage desired cell behavior? (2) Can scaffolds and constructs really bear loads? This paper focuses attention on two sets of scaffold and construct properties which must be measured to help answer these questions, discusses methods available, and considers methods needing development.

PHYSICAL PROPERTIES: Answers to (1) lie partly in scaffold surface chemistry and nano-texture. Scaffolds are also porous 3D materials whose internal architecture must provide sufficient surface area for cell attachment and the right kind and amount of empty space (pore structure) for movement of cell nutrients and waste, and formation of tissues. Frequently, % porosity is the only pore parameter reported. Of equal importance are pore surface area, size range, cross-sectional shape, connectivity and tortuosity. Besides SEM image analysis, applicable techniques include BET surface area, mercury porosimetry, and gas pycnometry. Experience using these tools on a bio-derived scaffold will be described.

MECHANICAL PROPERTIES: Answering (2) requires recognizing that implanted, load-bearing constructs should quickly become identical to normal tissue in stiffness, strength, fatigue resistance, coefficient of friction, and strength of attachment. If not, a construct may subject surrounding tissue to abnormal loads, abrade surfaces against which it articulates, lose structural integrity before it becomes normal tissue, and/or become detached. Simulated-use tests must be devised and employed to help predict what may occur. Novel micro- and nano-scale mechanical and

thermal methods will be discussed.

15:10 - 15:25

Tissue engineering for reconstructive orthopedic surgery - challenges and perspectives

oral **Grzegorz T. Benke¹⁾, Andrzej Górecki¹⁾**

1) Medical University of Warsaw, Orthopaedics Dept, Lindleya 4, Warszawa 02-005, Poland

The progress in orthopedic surgery is mutually connected with advances in material engineering, from biologically inert materials of early sixties, to the present-day biomaterials with preprogrammed bioactivity and predictable tissue response.

In contemporary bone surgery, however, the main trend is to replace, not restore damaged tissues. The greatest promise from emerging technologies of tissue engineering is to facilitate restoration of destroyed bone, cartilage, tendons and ligaments with implants containing living cells. To fulfill these promises different materials for cell growth, with different mechanical and biological properties are needed. Additionally there is need for controlled biodegradation of implanted materials, from weeks in soft tissue to months in bone reconstructive surgery.

For clinical applications in humans close collaboration of clinicians, engineers and specialists in cell biology is crucial.

Acknowledgments

This work was supported by the State Committee of Scientific Research (grant No. 05/PBZ-KBN-082/2002/06).

15:30 - 15:50

Coffee break

15:50 - 17:25

Afternoon session - continued - Luigi Ambrosio, Mikhail I. Shtilman

15:50 - 16:20

Biodegradable polyester nano- and microspheres by dispersion polymerization and macromolecular self-assembly

invited oral

Stanislaw Slomkowski¹⁾, Stanislaw Sosnowski, Mariusz Gadzinowski, Izabela Radomska-Galant²⁾

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Standard procedures used for fabrication of polymeric objects consist of polymerization, isolation, purification of synthesized polymer followed by shaping polymeric material into a desired form. However, there is growing an interest in processes with all these steps proceeding parallel. Apparently, parallel synthesis of macromolecules and their self-assembly into desired objects is less time consuming and more energy efficient. Recent studies proved that this way could be used for fabrication of nano- and microobjects with accuracy not attainable in any other way.

We developed a method (based on dispersion polymerization of lactides and epsilon-caprolactone) suitable for direct synthesis of microspheres. In this paper there will presented results of studies on the mechanism of formation of polyester microspheres and on the post-synthesis treatment allowing formation of particles with controlled degree of crystallinity.

According to developed method the microspheres with diameters from 0.5 to 7 mm and with narrow diameter polydispersity were obtained. Molecular weight of polymers in synthesized microspheres also could be controlled. The second method leading to particles is based on synthesis of block copolymers composed of biodegradable polyester block and hydrophilic blocks of polyglycidol and poly(ethylene oxide), followed by assembling the copolymers into nanoparticles. Diameters of formed nanoparticles could be varied, depending on molecular weight of hydrophobic (polyester) and hydrophilic blocks, from 22 to 60 nm. Their diameter polydispersity was narrow ($D_w/D_n < 1.2$). Procedures suitable for loading of the mentioned above microspheres and nanoparticles with bioactive compounds will be discussed.

Strategies suitable for using microspheres and nanoparticles as building blocks of materials porous on nano- and micrometer level will be discussed.

This work was supported by the State Committee of Scientific Research, grant No. 05/PBZ-KBN-082/2002/06.

16:25 - 16:40

Poly(styrene/alpha-tert-butoxy-omega-vinylbenzylpolyglycidol) microspheres for immunodiagnosics. Principle of a novel latex testbased on combined electrophoretic mobility and particle aggregation measurements

oral

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Principle of a novel latex agglutination test based on changes in electrophoretic mobility of particles with immobilized antigen in the presence of antibody is described. Poly(styrene/alpha-tert-butoxy-omega-vinylbenzylpolyglycidol) microspheres (P(S/PGL)) were synthesized by a one step soap-free emulsion polymerization of styrene and alpha-tert-butoxy-omega-vinylbenzylpolyglycidol macromonomer with number average molecular weight $M_n=2700$ and polydispersity $M_w/M_n=1.10$. The monodisperse latex particles with number average diameter $D_n=220$ nm and the surface fraction of polyglycidol equal $f=0.42$ mol% were obtained. Adsorption and covalent immobilization of human serum albumin (HSA) onto these particles were investigated. The hydroxyl groups of polyglycidol chains required activation with 1,3,5-trichlorotriazine, prior to protein attachment. It has been found that human serum albumin (HSA) was efficiently covalently bound with the surface of latex particles. The maximal surface concentration of covalently immobilized HSA was equal 1.2 mg/m².

A new type of antibody-antigen detection system based on combined measurement of changes in zeta potential and size of aggregates formed caused by immunoreactions of HSA immobilized on latex particles with anti-HSA (as antibody) was designed. In the model immunodiagnostic assay for anti-HSA involving P(S/PGL) particles with covalently bound HSA (P(S/PGL)-HSA), electrophoretic mobility and aggregation of microspheres were simultaneously measured. This approach allowed detection of anti-HSA in the serum in the range of anti-HSA concentrations from 0.1 to 200 µg/ml. The highest changes in electrophoretic mobility values were registered for P(S/PGL)-HSA-anti-HSA aggregates when microspheres with surface concentration of covalently immobilized HSA equal $\Gamma_{HSA_{cov}}=0.92$ mg/m² have been used.

In-vitro test and mathematical modelling on the controlled-release behavior of indomethacin from PLA-PEO nanospheres

16:45 - 17:00

Yun-Suk Jo¹⁾, Min-Cheol Kim²⁾, Do-Kyung Kim¹⁾, Charn-Jung Kim²⁾, Young-Keun Jeong³⁾, Kyung-Ja Kim³⁾, Mamoun Muhammed¹⁾

oral

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Drug delivery systems (DDS) have been focused as the most effective way to administer highly insoluble drug under physiologic conditions to the targeted site in a controlled manner. We report the synthesis of poly(lactic acid-co-ethylene oxide) (PLA-PEO), fabrication of PLA-PEO amphiphilic DDS and investigations of releasing behaviors of indomethacin as a model drug. The ring-opening polymerization (ROP) is commonly used technique for the synthesis of well-defined homo/copolymers from lactones or lactides. Instead of Sn(II) 2-ethylhexanoate which is well-known initiator for the ROP, a bioresorbable substance Fe(II) lactate was used to polymerize poly(D,L-lactide) (PDLLA) and poly(L-lactide) (PLLA) with poly(ethylene glycol) (PEG) to yield a series of PLA-PEO amphiphilic copolymers in the different compositions. DSC and XRD analysis results indicated that all copolymers were successfully synthesized and especially Fe(II) lactate was revealed as an effective initiator to yield highly crystalline PLLA-PEO copolymers. Emulsion/evaporation technique was used to fabricate amphiphilic nanospheres and resulted in a homogeneous size distribution with a low polydispersity index. Under physiologically similar condition, in-vitro test was conducted via dialysis method in phosphate saline buffer solution (PBS). The releasing behaviors were compared to two different types of mathematical models including diffusion and dissolution models. It was confirmed that key parameters such as volumetric ratio (V_r) between organic and aqueous phase, interaction parameter (K_p), and encapsulation efficiency (EE), affected to the overall characteristic regarding controlled-release of the entrapped drug. As the system had higher V_r value, the releasing profile tended to move toward dissolution model so that overall efficiency (ζ) were evaluated to optimize DDS for a specific type of drug or materials under consideration of various parameters to be manipulated.

Novel Biodegradable and Biocompatible Polymers for Anti-cancer Drug Delivery Systems

17:05 - 17:20

Zbigniew Jedliński¹⁾

oral

1) *Centre of Polymer Chemistry, Polish Academy of Sciences, Curie - Skłodowskiej, Zabrze, Poland*

Polymeric materials are playing nowadays an important role in medical applications as implants in reconstructive surgery and in drug delivery systems. However many natural polymers like cellulose and its derivatives, starch, hyaluronic acid and many others polymers produced in the nature being biocompatible, show some drawbacks as very high molecular weight, broad molecular weight distribution and not reproducible biological properties.

Therefore the natural copolymer of R,3-hydroxybutyric acid marketed by American Company Zeneca under the

trade name Biopol, which is the biodegradable analog of natural polymer could be used only for production of packing materials.

In my lecture biodegradable and biocompatible polymeric materials are presented, which have been recently used in novel drug delivery systems. Drug delivery systems with suitable polymers as drug carriers show new vistas in chemotherapy. Using discovered in our laboratory catalyst system we are able to synthesize supramolecular polymeric anticancer drug conjugates and drug nano-dispersion.

Using this novel approach to drug delivery, the drug activity is increased and some side effects e.g. cardiotoxicity can be eliminated.

The useful discussions and cooperation with prof. P. Grieb (Institute of Experimental and Clinical Medicine PAS, Warsaw), prof. J. Kawiak (Department of Clinical Cytology, Medical Center of Postgraduate Education, Warsaw) and prof. Z. Krawczyk (Institute of Oncology, Gliwice) as well as financial support by KBN 7 T08 E 059 are acknowledged.

References:

1. Z. Jedlinski, J. Polym. Sci. Part A: Polym. Chem., 40, 2158 (2002)
2. Z. Jedlinski, Accounts Chem. Res., 31, 55 (1998)

19:00 - 21:00

Poster session

19:00 -

poster

E-1

Hard Tissue Compatibility of Titanium Phosphide Layers Obtained by Diffusion in Ti-HA Interface

Cheol Sang Kim^{1,3)}, Ki Eun Hwang²⁾, Sung Joo Heo⁴⁾

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Previous studies on phosphide and oxide films formed by heat treatment of HA dip coated-Ti showed various properties with regard to both composition and morphology in the interfaces according to heating temperature. HA dip coated Ti screws were heated for 1 hour in a graphite furnace with protective argon atmosphere at 800(TiP1), 850(TiP2) and 900°C(TiP3) after producing a vacuum (10^2 Torr). The adhered HA coating layers on the metal surfaces were removed with strong jet spray of distilled water and subsequently sterilized according to standard procedures for metallic implant devices. Nine adult New Zealand white rabbits, aged 9 to 10 months, were used in the experiments. The three screw-shape specimens including non-treated Ti (c.pTi) screw were implanted in each animal, two in each medial proximal tibia according to the surgical protocol of the Branemark implant system. After 4 weeks, the rabbits were sacrificed with a lethal dose of pentobarbital injection and then the osseointegration was determined by an immediate measurement of the removal torque of the specimens. The result obtained demonstrated that the phosphide and rough Ti surface layers yielded higher the removal torque values than non-treated Ti surface. The mean values (standard deviation in parentheses) were 15.3(9.0), 33.8(17.9), 27.7(11.5) and 37.5(23.7) N-cm for c.pTi, TiP1, TiP2 and TiP3, respectively. Oxide thickness and mean pore size of the heated Ti surfaces increased relative to elevated heating temperature, the maximum phosphorus concentration (about 7 at.%) diffused from the HA layer was obtained via heating at 800°C. From results of this experiment, a conclusion can be reached that the chemical composition of Ti phosphide layer with the surface morphology of Ti materials play an important role in bone bonding process on Ti implants.

19:00 -

poster

E-2

In vitro reaction of human fibroblasts with gold - and L-aspartic acid - functionalized superparamagnetic iron oxide nanoparticles.

Maria Mikhaylova¹⁾, Yun-Suk Jo¹⁾, Do Kyung Kim¹⁾, Catherine Berry²⁾, Adam Curtis²⁾, Mamoun Muhammed¹⁾

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2) Centre for Cell Engineering, University of Glasgow, Glasgow G12 8QQ, United Kingdom

Colloidal magnetic nanoparticles have found effective applications in drug and gene delivery, magnetic resonance imaging (MRI), tissue engineering, and hyperthermic therapies for past decade. In these respect, 3D suspensions of magnetic nanoparticles with various functional surface layers were produced and tested in-vitro and in-vivo. Unusually strong magnetic properties and biocompatibility are considered to be one of the important issues for

engineering of new materials for biomedical applications. In this research the surface of superparamagnetic iron oxide nanoparticles (SPION) was functionalized with two biocompatible materials such as gold and L-aspartic acid to study their reaction with human fibroblasts, thus providing information about cytotoxicity, endocytosis and particles adhesion to cells. Au-coated SPION sample was prepared via process based on the reduction of corresponding metal ion on the surface of particles. The chemisorption process was used to modify surface of SPION with L-aspartic acid. Surface functionalized SPION samples were incubated with human fibroblasts for 6 and 24 hr. Cell-particles interaction were observed by light, fluorescence and scanning electron microscopy. Cells incubated with L-aspartic acid-coated SPION have similar gross cell morphologies to control cells, indicating no adverse cell reaction to the presence of the particles. With regard to the cell cytoskeleton, prominent stress fibre formation and tubulin clearly radiating out from the tubulin organising centre by the nucleus was observed, again exactly as the control cells. L-aspartic acid-coated SPION sample demonstrated an increase in clathrin production, particularly at the cell filopodia, suggesting that the particles are partially being up-taken into the cell body via endocytosis; however this is not causing any cell damage after 24 hours culture. Au-coated SPION sample do affect cell size and appear to induce smaller filopodia with cell/particle debris.

Model of Regeneration Processes Prognosis at Application of Natural Bioimplants

19:00 -

Lyudmila I. Donchenko¹⁾

poster

1) Scientific Research Institute for Traumatology & Orthopedics of Donetsk State Medicine University (DonIT&Or), 106, Artema St, Donetsk 83048, Ukraine

E-3

Bioimplantation is an actual direction of advanced medicine. Development of various kind of implantable biomaterials with the given strength characteristics, preservation of vital biomechanical properties, with the low immunogenicity and high level inductivity of regenerative processes causes the appropriateness of studies of biochemical reactions after implantation of biomaterials. This will allow one to elucidate the peculiarities of regeneration processes at application of different kinds of bioimplantable materials.

In this work we have studied the electrolyte exchange and enzyme activity after transplantation with the application of natural materials. The content of macro-elements and enzyme activity at auto- and allo-transplantation of Achilles' tendon were investigated for thirty rabbits of Chinchilla breed in the comparative aspect.

We have investigated blood sera for both groups of animals after transplantations of tendons after 7, 14, 21, 45 and 90 days.

It was revealed that the contents in the blood serum of sodium, amylase acid phosphatase activity which were determined at earlier terms after transplantation are the informative data of prognosis of regeneration processes evolution. The obtained results were the basis for development of algorithm of regeneration processes prognosis. Approbation of developed algorithm at clinic conditions was revealed the high accuracy (84%) of true prognosis.

An in vitro study of polymer based composites

19:00 -

Bozena D. Konieczna²⁾, Marta Blazewicz²⁾, Barbara Czajkowska¹⁾

poster

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2) University of Science and Technology, Department of Biomaterials, Krakow (AGH-UST), Krakow, Poland

E-4

Most of the tissues existing in human body have structures, which can be simulated with fibrous composite materials. The main problem associated with designing biocompatible composites for reconstruction of damaged or missing tissues is the ability of mimicking such structures. Physical, chemical and mechanical properties of composite materials should be similar to those of the native tissues. Another very important property of polymer-based fibrous composite materials, which can relatively easily be modified, is their surface microstructure. This surface microstructure depends on the way of preparation, type of polymer matrix and kind of reinforcement used.

This work aimed at determination of biological properties of composites obtained from carbon fibres and polymer matrix, which could possibly be utilised as implant materials in reconstruction of cartilage tissue. Two types of samples based on short carbon fibres, differ with polymers were tested. The samples were prepared by casting technique. MTT tests were carried out in the presence of hFOB-1.19-line human osteoblasts and HS-5-line human fibroblasts. The results show differences in viability of living cells.

These results suggest that if biocompatibility of polymer-based fibrous composites could be achieved then their possible use as implant material could be considered feasible and shall consequently be investigated.

Influence of Bioimplants on Immune Response

19:00 -

Natalya V. Shishkova²⁾, Lyudmila I. Donchenko¹⁾, Sergey A. Tkachenko³⁾

poster

1) Donetsk Institute of Traumatology & Orthopedics, Donetsk State Medicine University, 72, R.Luxemburg St., Donetsk 340114, Ukraine

2) Donetsk Phys.&Tech. Institute of the NAS of Ukraine (DonPhTI), R.Luxemburg, Donetsk 83114, Ukraine

E-5

3) Kiev State Medicine University (KSMU), Ukraine

The development and processing of some kind new implantable biological materials is impossible without understanding of processes taking place in organism after implantation and methods of their estimation.

In this work we have studied the peculiarities of immune response at application of auto- and allo-implants as the natural biological implantable materials for Achilles' tendon for rabbits of Chinchilla breed. The comparative estimation was realized in dynamics of experiment.

It was established that peculiarity of immune response on biological implant of allotransplant is characterized by more expressed inhibition of lymphoid chain of immune system for the early post-surgery period than on auto-plastics and by normalization of immune system indexes for the later terms. This allows us to recommend the given allo-transplant for reconstructive surgery at the tendon injuries.

19:00 -
poster
E-6

The influence of heat treatment temperature on bioactivity of TiO₂ sol-gel coatings

Bożena Pietrzyk¹⁾, Leszek Klimek¹⁾

1) *Technical University of Łódź, Institute of Materials Engineering, Stefanowskiego 1/15, Łódź 90-924, Poland*

One of the most important feature of material, which can replace or join bone, is bioactivity which consist in the formation of the bonelike apatite layer on its surface in body environment, or also in simulated body fluid (SBF). It is known that some sol-gel coatings like Si-, Nb-, Zr-, Ti- oxides show this property, but the nature of this effect is not quite clear yet.

TiO₂ sol-gel coatings were obtained by a dip-coating process using titanium(IV)butoxide as a sol precursor. The influence of heat treatment temperature of sol-gel TiO₂ coatings on their bioactivity was investigated.

The bioactivity of TiO₂ coatings was determinated as ability to form a layer of hydroxyapatite on their surfaces by soaking in SBF. The surface morphology and chemical composition of the surfaces of TiO₂ coatings were studied by scanning electron microscopy (SEM) and energy disperse X-ray microanalysis (EDX), respectively. The bioactivity of TiO₂ coatings was correlated with their bond configuration which were characterised by FTIR.

19:00 -
poster
E-7

Corrosion resistance investigations of prosthetic dental alloys coated by TiN

Dorota Ryłska¹⁾, Jerzy Sokołowski²⁾, Leszek Klimek¹⁾

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2) *Medical University of Łódź, Łódź, Poland*

Ni- and Co-based alloys, widely used in prosthetic dentistry, are submitted to corrosion processes and some allergic reaction may occur. The purification of metal intraosseous implants by the modification of alloy surface is employed to avoid their noxious influence. One of these methods is deposition of TiN layers characterized by high hardness, strength, wear resistance and good biotolerance. The high passivity of coatings should ensure their good corrosion resistance.

This paper presents the results of investigations of the corrosion resistance of PVD deposited TiN layers onto typical dental alloys. Two types of coatings-TiN and TiN+Ti₂N were deposited on the Ni-Cr-Mo type alloy (Hearenium NA typically used for casts of immovable elements facing by ceramics) and on the Co-Cr-Mo type alloy (Wironit used for movable framing casts). These coatings were examined by means of diffraction techniques and X-ray microanalysis due to determine the chemical composition and structure of the layers. The approximate thickness of layers was stated by X-ray method. Then the corrosion behaviour was examined. The number of ions from the metallic base penetrating through the coating into the corrosive solution (artificial saliva) was determined. Also, the potentiodynamic polarization technique was used to determine the corrosion resistance of coated samples. Tests were performed in a solution of 0.9% NaCl with the potential change rate of 1 mV/s in the potential range from -200 mV to 1000mV. The results of electrochemical corrosion tests of coated samples were compared with results for based alloys. The surfaces of samples before and after corrosion tests were observed by means of SEM.

The main statement is that the coatings of titanium nitrides improve the corrosion resistance.

19:00 -
poster
E-8

Nanocrystalline diamond (NCD) - a new and promising material for medical applications

Bogdan Walkowiak^{1,2,3)}, Wiesława Okroj^{1,3)}, Iwona Przybyszewska^{1,3)}, Marta Pirek^{1,3)}, Witold Szymański^{1,3)}, Paweł Koseda^{1,3)}, Witold Jakubowski^{1,3)}

1) *The Centre of Excellence NANODIAM - Institute of Materials Science and Engineering, Poland*

2) *Technical University of Lodz, Poland, Poland*

3) *Department of Molecular and Medical Biophysics, Medical University of Lodz, Poland*

Our recent effort was focused on examination of nanocrystalline diamond layers, synthesized on a metal surfaces, allowing for controllable biocompatibility and bioactivity. The NCD layer was formed in a vacuum reactor chamber

by a radio frequency plasma activated chemical vapor decomposition of methane (RF PCVD). This method was pioneered by Mitura and Niedzielski, and currently is subjected to several modifications. Structural and chemical analysis of carbon coating, produced by this method, revealed the presence of nanocrystalline diamond layer (96% pure diamond), apart from other allotropic forms of carbon. About 300-500 nm in thickness the NCD layer smoothly changes into a diffusion interlayer of carbides and the base. The NCD coating exhibits very promising mechanical features and meets very well our expectations in respect to the above described phenomena. Protein deposition and blood platelet adhesion to NCD surface is very significantly reduced, when compared to medical steel, and this coating is also resistant to a microbial colonization. The NCD surface manifests also positive bioactivity accelerating of free radicals decay in blood plasma.

Nanocrystalline Diamond Coatings - antyallergic barrier onto medical implants

19:00 -

Katarzyna Bąkiewicz¹⁾, Beata Kręcisz²⁾, Marta Kieć-Świerczyńska²⁾, Piotr Niedzielski¹⁾

poster

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E-9

Allergy, the main human disorder development of the world civilization.

Many types of allergy can be observed in different pathological situations.

Nowadays, more and more directions of medicine are interested in allergic reactions as a response to changes of homeostasis of a living organism.

Biomedical Engineering as an interdisciplinary science which is mainly connected with the application of medical implants in human organism should to start the examinations which will answer the basic question: Do medical implants protect against allergy in human environment?

There are examples of very strong, dangerous allergic processes after implantation of medical implants without protecting barrier in human body.

The new, pioneer examinations with Nanocrystalline Diamond as a biocompatible and bioactive biomaterial in human body proved these assumptions on the allergic reaction level.

The results of Irritation Reaction (IR) and Allergic Reaction type IV (hypersensitivity delayed reaction; contact eczema) with Diamond Powder Particles on the human skin in lobular tests show the complete biocompatibility with human body; we have not observed any contact allergic reaction to the suspension of Diamond Powder Particles.

The results of examinations with Nanocrystalline Diamond Coatings (NCD) onto metallic samples as a simulation of metallic implants in living organism proved the antiallergic bioactivity of diamond in the contact reaction as a barrier for metals.

The safe future of medical implants in human organism is directly connected with the application of Nanocrystalline Diamond Coatings as an antiallergic barrier to different types of metal implants.

Effects of Chemical Composition of Titanium Material Surface on SAMs Formation by Functional Groups of Alkanethiol and Alkanesilanes

19:00 -

Cheol Sang Kim^{1,2)}, Kyung Shin³⁾, Bock Choon Pak⁴⁾

poster

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E-10

Self-assembled monolayers(SAMs) have become an essential technique for biochemical modification using specific peptides on a biomaterial surface. Previous studies have reported effects of their terminal functionalities including the organic molecules on apatite formation in a simulated body fluid. Also biological responses are influenced by the surface chemical characteristics of SAMs. In this study, the adsorption behavior of the alkanethiol and two alkanesilanes on the modified titanium surfaces were examined using an electrochemical quartz crystal nanobalance(EQCN). Furthermore, the formation of SAMs on three working electrode surfaces: Au coated Ti, commercialized pure Ti(c.pTi) and TiO₂ were examined. Resolution limit obtained for the constructed EQCN was about 0.1 ng. The mass change ratio on the surfaces versus immersion time was precisely investigated using the EQCN. The quantity of Thiol-I SAM adsorbed on c.p Ti is about 67% compared with the quantity formed on Au coated titanium substrate. However, there was no SAM formation on the TiO₂ surface with thick titanium oxide layer obtained by electrochemical oxidation method. The SAMs amount (for organosilanes) adsorbed on TiO₂ surfaces of Silane-I[(CH₃O)₃Si(SH₂)₃NHCH₂CH₂NH₂] and Silane-II[H₂N(CH₂)₃Si(OCH₃)₃] were approximately 19% and 14% more than that for c.pTi. Results obtained from this study show that by altering titanium surface chemistry

and introducing functional groups, titanium surface may be modified into a functional bioactive surface and can be used in the same way as Au substrate.

19:00 -
poster
E-11

CPC Precipitation Behavior of Titanium Phosphide Surfaces Obtained from Heat Treated Ti6Al4V buried in Hydroxyapatite Paste

Cheol Sang Kim^{1,4)}, Kyung Yi Shin²⁾, Ki Eun Hwang³⁾, Min Young Jung³⁾

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The calcium phosphate compounds (CPC) precipitation behavior of titanium phosphide surfaces obtained from heated Ti6Al4V buried in hydroxyapatite (HA) paste was investigated using a combination of XRD, ICP and AAS. The morphological and compositional studies in the interfaces were performed by SEM and scanning Auger electron spectroscopy. The cylindrically shaped specimens were heated at 800, 850 and 900°C for 1 hour in a protective argon atmosphere. The precipitated amounts of Ca and P on the specimens were measured after immersion for 2, 4, 8 weeks in 50 ml SBF using ICP and AAS. The heating process led to formation of porous surfaces and stable Ti phosphide layers in HA-Ti material interfaces. Quantities of precipitated Ca and P on the heat-treated surfaces were significantly higher than those on non treated Ti6Al4V surface, and the quantity increased with increment in pore spaces due to elevated temperature. The approximate Ca/P ratio of the CPC precipitated on non-treated Ti6Al4V is 2.2 signifying a high non-crystallinity, and the ratio of CPC formed on titanium phosphide surfaces ranged from 1.62 to 1.83. Hence, the results show that the composition and morphology in the interfaces of phosphide and oxide films show a variety of in-vitro CPC precipitation behavior. This method could be suitable for the surface modification of titanium materials which in-turn will be able to improve the biological performance of titanium implants.

19:00 -
poster
E-12

Calcium phosphate structure controlled by organic matrix (multilayer film)

B. Jachimaska¹⁾, C. Gergely, J.C. Voegel, F.J.G. Cuisinier²⁾

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Biomineralization is general process by which inorganic salt crystals are formed in biological environments. In order to study the mechanisms of the biomineralization, polyelectrolyte multilayers film were used to induced nucleation and growth of inorganic crystals. Nucleation and growth are controlled by specific interactions between mineral crystals and organic or inorganic molecules. Still there are many unanswered questions especially concerning the essential components involved in the initiation of mineralization and in the regulation of crystal growth. Alternate layer by layer adsorption is a powerful and convenient technique for preparation of organized molecular films. This surface may precisely be an appropriate substrat for cristal nucleation.

The aim of this study was to investigate the influence of polyelectrolyte films to calcium phosphate nucleation. Two situation where studied: when the polyelectrolyte surface is formed by polyallyamine (PAH) positive surface and polystyrenesulfonate (PSS) as negative surface. The results were also compared with bare surface. The proces of nucleation of calcium phosphate was followed in situ using Optical Waveguide Lightmode Spectroscopy (OWLS) and AFM liquid cell. This technique allows focusing on the process occurring directly at the surface. Crystal morphology was determine by scanning electron microscopy (SEM). The SEM images shows that three dimensional crystallites are formed on the PSS terminated film and two dimensional on the PAH terminated film.

Acknowledgement:

The research was carried out under 5th European Framework Programme:: Surface Improvement On Metal Implants - SIMI, Project G5RD- 2000-00423.

19:00 -
poster
E-13

Extra cellular matrix used in-vitro model system for hydroxyapatite formation

Emilia V. Pecheva¹⁾, Liliana Pramatarova¹⁾, Manfred F. Maitz²⁾, Mihn T. Pham²⁾, Alexey Kondyurin³⁾

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3) *Institute of Polymer Research, Dresden 01069, Germany*

Many are the hypotheses related to the mechanisms of biomineralization process, particularly hydroxyapatite (HA) nucleation and growth. Surface topography is regarded as one of them, as well as supersaturated solutions, the presence of hydroxyl groups, chemistry of the nucleation points, organic components and etc.

The aim of this work is to investigate the influence of the organic components deposited on different solid surfaces upon HA formation. For this purpose the surfaces of stainless steel, silicon and silica glass substrates are coated with extra cellular matrix (ECM) to promote interactions with bone-forming cells. The ECM proteins are known as ligands in reactions with cell surface receptors involved in bone physiology. The osteoblast-like cell line SAOS-2 is allowed to synthesize and assemble its own ECM on the substrates under standard cell culture conditions. Cells are then selectively removed resulting in substrates coated with a thin film of ECM.

The so-modified samples are immersed in a solution, supersaturated with respect to Ca and P ions (so called simulated body fluid, which resembles the composition of human blood plasma) and a layer is grown. X-ray diffractometric techniques (XRD and EDX), spectroscopic (FTIR and Raman), microscopic techniques (SEM) are used to highlight the likeness of the grown artificial biomimetic HA/ECM protein structure with natural bone tissue.

A homogeneous HA layer is found on the so-modified samples, which consists of particles with regular sphere shape with mean diameter size of 5 micrometers. Well resolved IR and Raman peaks reveal the formation of crystalline HA layer, self organized inside the ECM matrix.

Growth of hydroxyapatite layers on modified by ion beam patterned solid surfaces for studying the biomineralization

19:00 -

Emilia V. Pecheva¹⁾, Liliana Pramatarova¹⁾, Manfred F. Maitz²⁾, Mihn T. Pham²⁾, Alexey Kondyurin³⁾

poster

1) Institute of Solid State Physics, Bulgarian Academy of Sciences (ISSP-BAS), 72, Tzarigradsko Chaussee Blvd., Sofia 1784, Bulgaria

E-14

2) Forschungszentrum Rossendorf (FZR), Dresden 01314, Germany

3) Institute of Polymer Research, Dresden 01069, Germany

In this work we continue with the study of the hypotheses related to the process of biological mineralization, i.e. surface induced nuclei and hydroxyl (OH) groups, surface morphology, use of supersaturated solution and organic components, and etc. We study particularly the formation of hydroxyapatite (HA, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) in aqueous systems, which is often met in the nature in bones, teeth, shells, kidney stones, and etc.

This study has been taken to investigate the behavior of surface modified stainless steel, silicon and silica glass materials, used as in-vitro model systems, upon inducing HA formation in a simulated body fluid (SBF). Patterning by ion implantation through molybdenum mask with a definite design is the method chosen for a selective surface modification of the three classes of materials. Na^+ ion implantation and subsequent thermal treatment at 873 K in air is employed for inducing OH- groups in the fluid through ion-exchanging processes in SBF.

Investigation of the surface chemistry, morphology and elemental composition of the grown layers is done by using Fourier transform Infrared (FTIR) and Raman Spectroscopy, and Scanning Electron Microscopy (SEM), coupled with Energy Dispersive X-rays (EDX). FTIR and Raman Spectroscopies show the formation of hydroxyapatite layers on the surfaces of stainless steel, silicon and silica glass. Morphology of the layers, observed by SEM, shows that they consist of sphere-like particles homogeneously distributed on the whole surface of the samples.

Immobilization of analgetic AV-101 into calcium alginate gels

19:00 -

Rinat M. Iskakov¹⁾, Valentina K. Yu¹⁾, Dmitry Y. Boldyrev¹⁾, Y Y. Fomicheva¹⁾, Erkeshe O. Batyrbekov¹⁾, Kaldybai J. Praliev¹⁾, Bulat A. Zhubanov¹⁾, D K. Berlin²⁾

poster

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2) Oklahoma State University, 107 Physical Science, Stillwater 74078, United States

E-15

A new analgetic drug AV-101 was immobilized to calcium-alginate gel beads with average diameter of 1 mm. A series of alginate gel contains with various mannuronic/guluronic (M/G) ratios was selected to control the diffusion of the drug. Release of the drug from the alginate beads in vitro into physiological sodium-containing solutions was studied. As found, a discontinuous time of the Fickian diffusion of the drug depending on the M/G ratio was followed by a burst release of the remaining drug. The burst release was due to a swift disintegration of calcium alginate with the exchange on sodium ions. The preceding discontinuous lag time promotes a free dissociate exchange of sodium-calcium in M units, while the burst disintegration leads to fast dissociation of G units. The lag time can be controlled by M/G ratio of calcium alginate gels. The lag time increases if a content of M units decreases. The increase of M blocks was led to more extensive swelling of the alginate gel beads. Such way could be promising for a controlled drug delivery or the use in implants with controlled drug effect.

Modification of Acetylsalicylic Acid by Oligomers of 3-Hydroxybutyric Acid

19:00 -

Maria Juzwa¹⁾

poster

1) Centre of Polymer Chemistry, Polish Academy of Sciences, Curie - Skłodowskiej, Zabrze, Poland

E-16

Many drug-polymer delivery systems were proposed offering improved efficacy, reduced toxicity and better patient compliance as compared with conventional forms of drugs. Various synthetic and natural polymers were used in these systems [1]. However many polymers proposed as carriers were not biomimetic and not biodegradable.

Several methods of drug modifications were studied in our laboratory, and new drug-polymer conjugates containing synthetic oligo(3-hydroxybutyrate) as polymeric carriers were synthesized [2,3] attention has been paid to modifications of nonsteroidal anti-inflammatory drugs (NSAIDs). The results of the present study have revealed that in the reaction of *b*-butyrolactone with acetylsalicylic acid the regioselective ring opening and oligomerization of *b*-butyrolactone takes place and drug-polymer conjugates are prepared [4,5].

The structure of synthesized compounds was controlled by GPC and confirmed by elemental analysis, ¹H NMR, ESI-MS, FTIR spectra.

The obtained modified acetylsalicylic acid has shown prolonged activity and lower toxicity than the drug (aspirin) itself. Further studies on the usefulness of modified aspirin for medical applications are underway.

Acknowledgements

Financial support by KBN 7 T08 E 059 and POLPHARMA Company are acknowledged.

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19:00 -

poster

E-17

Uniform aminosiloxane films for immobilization of biomolecules: a sol-gel approach

Kristjan Saal^{1,2)}, Tanel Tätte^{1,2)}, Indrek Tulp²⁾, Ilmar Kink¹⁾, Rünno Lohmus¹⁾, Uno Mäeorg²⁾, Ago Rinke²⁾, Ants Lohmus¹⁾

1) *Institute of Physics, University of Tartu, Riia str. 142, Tartu 51014, Estonia*

2) *Institute of Organic and Bioorganic Chemistry, University of Tartu, Jakobi 2, Tartu 51014, Estonia*

Aminosilanes are widely used for covalent linkage of biomolecules to glass in biosensor and DNA chip fabrication. First introduced by Lyubchenko et al [1], silanization of mica and other substrates with trialkoxyaminoalkylsilanes has become a common technique in AFM studies of immobilised biological species. As a drawback of this relatively simple method, it has been pointed to the poor reproducibility in formation of uniform siloxane layer [2]. The formation of homogeneous siloxane (mono)layer of trialkoxysilanes is extremely sensitive to the presence of surface silanols on the substrate as well as to the quantity of pre-adsorbed water on the substrate and in silanizing environment [3]. In this work we present further developments of the approach reported previously [4] on the preparation of uniform aminofunctionalized siloxane films using sol-gel technique. We show that these films can be used in SPM studies of covalently immobilized biological species as well as supports in DNA chip fabrication.

In our experiments 3-aminopropyltrimethoxysilane and tetramethoxysilane were co-polymerised linearly, followed by deposition onto glass substrate from methanol solution and subsequent gelation to three-dimensional siloxane layer. FTIR and ¹³C NMR spectroscopy and MALDI-TOF mass spectrometry were applied to unveil chemical characteristics of linearly polymerised siloxane chains. AFM was used to probe the topographic features of the films. High-resolution phase contrast imaging revealed molecular sub-structure of the film, showing clearly densely packed linear siloxane chains. Spectroscopic analysis of the samples confirmed the presence of aminopropyl groups, pointing to their ability for covalent coupling of biomolecules.

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19:00 -

poster

E-18

Polymeric complexes of sorbic acid.

Olga V. Suprun¹, Mikhail M. Lotter¹, Mikhail I. Shtilman¹, Aristidis M. Tsatsakis²

The work is devoted to investigation of polymeric complexes formed by sorbic (trans, trans 2, 4-hexadiene-1-carboxylic) acid (SA) fungicide and poly-N-vinylpyrrolidone (PVP). Formation of PVP and SA complexes in mediums of several organic solvents with different parameter of solubility was considered.

For estimation of immobilizing ability of polymers with different molecular weight method of competitive complexing with use of fluorescent probe of 1-anilinonaphthyl-8-sulfoacid, which is in the free state in the form of no

fluorescent dimmer, was used. Obtained results showed that samples with higher molecular weight had greater complexing ability (from fluorescent analysis excitation wave length is 363 nm and peak emission wave length is 480 nm).

For complexes stability analysis the method of equilibrium dialysis was also used. In this case it was also demonstrated that stability of complexes increases with increase of polymeric carrier molecular weight.

Level of polymeric complexes fungicidal activity was evaluated by the standard biotest, the inoculation (*Fusarium oxysporum* f.sp. *radicis-cucumerinum*) was carried out in Petri dish on agar plates, prepared with the use of medium, containing polymer derivatives of SA.

As it was shown, polymeric complexes of SA possess higher level of activity than low-molecular acid. At the same time level of activity increased with the decreasing of polymeric carrier molecular weight.

Polymeric esters of SA, prepared by interaction of acid and water-soluble polymeric epoxy-containing carriers with different molecular weight show lower fungicidal activity than polymeric complexes of acid.

Lower activity of systems in which acid was bind by covalent (ester) bond is based on lower speed of polymers hydrolysis. Speed of hydrolysis of these polymeric ethers was analyzed by HPLC method.

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Shtilman M.I., Tzatzarakis E., Tsatsakis A.M. et al. // Food Additives and Contamination, 2000, V.17, T 12, P.965-971

Porous epoxycontaining hydrogels.

Alexandr A. Artyukhov, **Vasily S. Kozlov**, **Mikhail V. Khazanchuk**, **Ilya A. Fadushin**, **Aristidis M. Tsatsakis**, **Mikhail I. Shtilman**

19:00 -
poster

The influence of synthesis conditions on properties of porous epoxycontaining hydrogels obtained by radical copolymerization of 2-hydroxyethylamine and epoxycontaining monomers of allylglycidilic ether and glycidilmethacrylate in the presence of cross-linking agent in water frozen systems was studied.

N, N'-methylenebisacrylamide (MBAA) was used as cross-linking agent. By varying of its abundance in the base mixture we can obtain gels with the different ability for swelling and penetrability.

Realization of the gel synthesis in water solution at low temperature (-15°C - -20°C) allows attempting of carrier high porosity. Gels obtained at lower temperature (-30°C) have lower penetrability and better swelling.

Increased concentration of initiator regarding to the monomer mixture leads to the higher product yield.

Due to the porous structure features gels are stable in the solutions with different ionic force. Microscopic study of gels shows that they contain transparent pores with the size of tens and hundreds of microns according to synthesis conditions.

Studying of synthesized polymer systems in water saturated condition by structure-dynamic method shows that with the application of load we can observe two deformation areas, first for extrusion from macropores and second for deformation of cross-linked areas.

Using this data the distances between polymer phase lacing knots were calculated. Their values lay between 20000-60000 D.

Presence of highly reactive epoxy groups in polymers enables the immobilization of different proteins in mild conditions without extra activation of the carrier.

Studying of the immobilization conditions influence on the amount of immobilized albumin allows discovering of optimal process parameters and shows that amount of immobilized protein reaches 150 mg/g of the carrier.

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Shtilman M.I. Immobilization on polymers.-VSP: Utreht-Tokyo-1994.-479p.

Water-soluble metal complexes based on derivatives of poly-N-vinylpyrrolidone

Maria V. Klyagina, **Denis A. Lovyagin**, **Dmitriy A. Lovyagin**

19:00 -
poster

New polymeric carrier, able to bind different biologically active ligands, epoxy-containing poly-N-vinylpyrrolidone was synthesized.

The polymer was prepared in conditions of Darzan reaction. It was shown that by this method it is able to obtain with high yield water-soluble polymers which contain up to 20 molar % of epoxycontaining rings. Prepared polymer did not dissolve in the reaction spirit medium due to the presence of amide groups and precipitated.

The effect of reaction conditions - temperature, reaction time, concentration, reagents ratio and molecular weight of initial poly-N-vinylpyrrolidone on yield and containing of epoxy-groups was studied.

This method pays attention as beforehand prepared for medical purpose polymer with known molecular-weight distribution, suitable for creation of preparations, assigned for introduction into the organism. Process was controlled with determination of polymers yield and abundance of epoxy groups. Polymers structure was also confirmed by infrared spectral method.

It was determined that these polymers are able to interact with amino acids. Analysis with AMR method showed that addition of amino group passes on the atom of carbon that is a part nitrogen-containing cycle.

E-20

It was shown that synthesized polymers and their complexes with transitional metals have different types of biological activity determined by experiments with several tissue cultures.

Interconnection between metal and ligand was studied by photometry, chelatometry, potentiometry, viscometry. For determination of donor atoms, interacted with metal ions, the analysis of obtained polymers with IR and PMR spectrography methods was carried out. The estimates were made about the structure of synthesized polymeric complexes of different metals and ligand groups, placed in side chains.

References

Shtilman M.I., Tashmuhamedov R.I. et al. // 27th Intern. Symp. on Contr. Release of Bioact. Mater. [Paris,2000].- CRS.- 2000.- P.7009.

19:00 -
poster
E-21

Oxidation and degradation of polyethylene in hip implants

Janusz S. Otfinowski²⁾, Joanna Kowal¹⁾, Barbara Czajkowska⁴⁾, Anna Więcek³⁾

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3) *Faculty of Chemistry, Jagiellonian University, Kraków, Poland*

4) *Collegium Medicum, Jagiellonian University, Kraków, Poland*

Polyethylene is generally used as a component in the majority of hip joint prostheses. A very important downside of this material is excessive wearing of some of the polyethylene cups leading to premature loosening of the hip prostheses.

Based on numerous observations, it has been determined that polyethylene wear is not always identical. Differences observed in the polyethylene wear are not easily explained, as they neither depend on the period of use nor on the factors that may affect the value of load on hip prosthesis such as weight and physical mobility of patients

A question arises whether in cases of premature and excessive wear of polyethylene bearings which cannot be explained by exclusively mechanical factors, the chemical structure of implanted material has changed making the material more susceptible to mechanical tear and wear.

To answer that question the authors made IR spectrophotometric studies of 13 samples collected from used ultra-high molecular weight - UHMWPE (DIN 58834) - polyethylene cups removed from patients after several years of use. The results of spectrophotometric studies on used polyethylene samples have been compared with 8 samples of new, not yet used polyethylene cups.

The analysis of infrared spectra demonstrates a conspicuous difference in the chemical structure of both types of polyethylene cups. In the spectrum of the exploited material the presence of additional, complex carbonyl band ranging from 1800 to 1600 has been observed, corresponding to the stretching vibrations of the groups >C=O, which does not occur in the spectrum of an unused bearing. It may be concluded that the cause of premature wear-off of polyethylene cups is oxidation and accompanying polymer degradation. Chemical degradation and oxidation of the material lower its mechanical resistance and may accelerate the process of polyethylene cup wear-off.

19:00 -
poster
E-22

Life time of polymeric composite implants on the basis creep and fatigue tests

Jan Chłopek¹⁾, Grzegorz Kmita¹⁾, Patrycja Rosół¹⁾

1) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland*

In living organism longterm working implants are subjected to mechanical stresses as well as aggressive body fluids. While aging, they can change their mechanical and biological properties. Investigations presented in this study show the influence of conditions simulating human body on the mechanical properties of biostable (polysulfone) and bioresorbable (poly(lactide-co-glicolide)) polymers and their composites reinforced with medium-modulus carbon fibres. Stability of investigated materials was estimated on the basis creep tests (static load) and under cyclic load. As a result of creep tests the data enabling to estimate longterm samples behaviour under mechanical tension biological environment were obtained. On the basis of completed experimental data critical force, under which the material can work for long time without failure was determined and the life time for remote load and acceptable deformation of investigated samples was estimated.

As a result of cyclic stress we can observe forming and development of cracks leading to failure. During the analysis of fatigue behavior of composite elements, one fact need to be taken into account, which is their quite different fatigue behavior than that of their components. Hence, carrying out the experiments on the medical device in conditions close to its work environment is very important. Within this study an analysis of fatigue behavior of composite polymer screw was realized. On the basis of chosen micromechanical models a trial of lifetime prediction of joining screws was undertaken (in presumed conditions of simulated body fluid and under cyclic load).

19:00 -
poster
E-23

Surface Characterization of the Radiation Processed Polymer Materials for Medical Uses

Vasile Cornelia¹⁾, Mihaela C. Pascu²⁾, Bogdan S. Munteanu¹⁾, Loredana E. Nita¹⁾, Anca Cojocariu²⁾

1) *Petru Poni Institute of Macromolecular Chemistry of the Romanian Academy, Aleea Grigore Ghica Voda 41A, Iasi, Romania*

2) *"Grigore T. Popa" Medicine and Pharmacy University, 16 University, IASI Ro 6600, Romania*

The surface properties of several complex polymer materials compatibilized by adding block copolymers coupled with electron beam irradiation have been determined. The investigation methods were sessile drop method, electron microscopy and protein absorption. The protein absorption leads both to enhance and to test the biocompatibility. It has been established the optimum radiation dose to achieve a good biocompatibility for the polyethylene/polystyrene and polypropylene/EPDM blends used in medical purposes.

Type of radicals formed in select siloxanurethane polymers under irradiation method of sterilization.

19:00 -

Izabella Legocka¹⁾, Monika Celuch¹⁾, Jarosław Sadło³⁾, Janusz Kozakiewicz²⁾

poster

1) *Institute of Nuclear Chemistry and Technology, Dorodna 16, Warsaw 03-195, Poland*

2) *Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warsaw 01-793, Poland*

E-24

3) *Institute of Nuclear Chemistry and Technology (IChTJ), Dorodna 16, Warsaw, Poland*

In this preliminary study, experimental segmented poly(siloxaneurethanes) of varying length segment, designed for medical scaffolds for tissue engineering were exposed to electron beam and gamma radiation.

The films of samples packed in double sterilization pouches were irradiated with standard dose of 28 kGy used for sterilization.

The samples were irradiated using accelerator type LAE 13/9 with nominal energy of 10 MeV and 60Co gamma ray source named Minejola 1000.

None inert gas was applied. The effect of radiation on radicals formation was investigated. ESR (Electron Spin Resonance) method was used for detection present of radicals in irradiated materials. There was no signals for samples irradiated in ambient temperature, so it was necessary to carry out analogical experiment in lower temperature. Some signals with different intensity from paramagnetic species were observed for samples irradiated in temperature of liquid nitrogen. In order to examine kinetics of formation and decay of radicals there were carried out same experiments in various temperatures from liquid nitrogen to ambient temperature.

In the same time there were observed no differences in IR and DRS (Diffuse Reflected Spectroscopy) spectrum of radiated and irradiated samples. It can be result of the very fast recombination process of new-created radicals. After modification selected poly(siloxaneurethanes) with scavengers of radicals there was observed decrease intensity of signals in ESR spectrum.

Elaboration and characterization of biodegradable scaffolds from poly(L-lactide-co-glycolide) copolymer synthesized with low-toxic zirconium acetylacetonate.

19:00 -

Elzbieta Pamula¹⁾, Marta Blazewicz¹⁾, Barbara Czajkowska²⁾, Piotr Dobrzynski³⁾, Maciej Bero³⁾, Janusz Kasperczyk³⁾

poster

1) *University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland*

2) *Collegium Medicum, Jagiellonian University (CM UJ), ul.Św.Anny 12, Kraków, Poland*

E-25

3) *Centre of Polymer Chemistry, Polish Academy of Sciences, Curie - Skłodowskiej, Zabrze, Poland*

Designing and manufacturing of appropriate scaffold materials for a particular application are of a key importance in tissue engineering. The fundamental are such properties of scaffolds as biocompatibility, adequate degradation time, appropriate biodegradation rate (not too fast in a first stage of contact with cells and tissues) and proper porous microstructure.

The polymer material used in this study was synthesized with the use of low-toxic zirconium compound, instead of tin compounds, which are commercially used in manufacturing of implant materials from aliphatic polyesters. The material was studied in vitro in order to check its biocompatibility. The results of tests of viability of human osteoblast line (Saos2) clearly show that the polymer obtained with the use of zirconium is much more biocompatible than that synthesized with the tin compound. Several types of porous polymer materials were obtained via solvent casting / particulate leaching technique, using various porogens. A very high open porosity (more than 85%) and different size and distribution of pores were achieved. The degradation of scaffolds as a function of incubation time in PBS at 37°C was analyzed by FTIR, SEM and XPS. Viscosity and measurements of relative weight were also made. The results of biological, textural, physico-chemical properties of obtained materials, regarding their behaviour in conditions simulating biological environment show that they could be used as scaffolds for tissue engineering of cartilage and bone.

Comparison of properties of porous alumina as a potential cell carrier formed by foaming and polymeric sponge methods

19:00 -

Zbigniew Jaegermann¹⁾, Joanna Karaś¹⁾, Sławomir Michałowski¹⁾

poster

E-26

1) *Institute of Glass and Ceramics (ISiC), 9, Postepu Str., Warsaw 02-676, Poland*

Technology of manufacturing of porous ceramic biomaterials is an important element of elaboration of multifunctional specialised implants used by medicine. One of the roles of implants consists in bone or cartilage stimulation through the implantation of the porous grafts that contain tissue cultured in vitro. The biomaterial is a carrier of cell culture and after the implantation it helps in the tissue restoration. Porosity of the ceramic material used, as well as the shape and size of pores have to be controlled in order to obtain optimal conditions for cell culture. Porous materials properties depend to a considerable extend on the forming method applied. Therefore the present poster is a comparison of properties of porous materials formed by chemical foaming and by structural sponge methods.

Two batches of alumina porous implants were prepared: one formed by chemical foaming and the other using polyurethane sponge. Both methods were described. A comparison concerning apparent density, total porosity and compressive strength, as well as texture properties of both materials have been conducted.

The analysis of the results demonstrated that it is possible to obtain standard quality of alumina foams with a determined pore size, porosity value and adequate mechanical properties when forming the material with the use of structural sponges. All the parameters mentioned above can be controlled and steered, therefore alumina foams could be a proper carrier of implanted cells used as a stimulant agent for bone and cartilage restoration.

Acknowledgement

This work was supported by the State Committee of Scientific Research (grant No. 05/PBZ-KBN-082/2002/06)

19:00 - **Structural-strength aspects of glass/metallic composite based on Co-Cr-Mo implantation alloy**

poster **Zbigniew Oksiuta²⁾, Krzysztof Kurzydłowski¹⁾, Jan R. Dąbrowski²⁾**

E-27

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

2) *Białystok Technical University, Faculty of Mechanical Engineering, Wiejska 45C, Białystok 15-351, Poland*

This paper presents the analysis of research to determine influence of bioglass (15%CaO, 9%P₂O₅, 76%SiO₂) addition 0%, 5%, 10% wt. on a structural and mechanical properties of sintered samples based on Co-Cr-Mo alloy. Cylindrical samples 20mm diameter and 10mm length were prepared according to the following scheme:

- 1) blending of Co-Cr-Mo powder alloy with bioglass addition of 5%, 10%wt. in a ball mill, 2) uniaxial cold compaction at pressure of 600MPa,
- 3) sintering in the horizontal tube furnace at the temperature 1230°C in a dry argon atmosphere, for 1 hour,
- 4) rotary cold repressing on a PXW 100A press at pressure of 600MPa, number of rotations i=12, with spinning upper tool movement,
- 5) heat treatment at the temperature of 1230°C for 1 hour in argon atmosphere, cooling in the cold area of the furnace.

The measurement of hardness, tensile and compressing strength and ultrasonic tests were performed. The microstructure of cross-sections specimens after sintering, repressing and heat treatment were analysed.

The microstructure of samples after heat treatment exhibited homogeneous distribution of bioglass particles (average size 120 microns) in the Co-Cr-Mo matrix with an average grain size 40 microns.

The compressing tests has shown that an addition to the Co-Cr-Mo matrix 5%, 10%wt. of bioglass caused an increase of the yield strength (from 495MPa to 610MPa for 10% wt. of bioglass), but decrease of UCS and plastic strain of specimens. The tensile strength data (as we assumed) have shown that samples without any bioglass addition received higher mechanical properties in comparing with the glass/metal composites. Non-destructive ultrasonic investigation revealed that the composite samples have lower Young and shears modulus in comparison with samples without any addition. Thus, from the mechanical point of view, the composites with 10% of bioglass addition may be suitable material for further investigation.

19:00 - **Glass-ionomeric materials for otologic surgery**

poster **Joanna Karas¹⁾, Tadeusz Floriańczyk²⁾, Lidia Ciołek¹⁾, Zbigniew Jaegermann¹⁾**

E-28

1) *Institute of Glass and Ceramics (ISiC), 9, Postepu Str., Warsaw 02-676, Poland*

2) *Warsaw University of Technology (PW), Warsaw, Poland*

The aims of the investigation conducted in the Institute of Glass and Ceramics was to prepare glass-ionomeric materials for otologic surgery. The synthesis of the materials developed on the basis of glass powders belonging to the system Na₂O-CaO-SiO₂-Al₂O₃-P₂O₅-F and setting liquids which were copolymers of acrylic and itaconic acids.

1. Glass-ionomeric cement. Once the components are mixed, a plastic pulp is obtained. It sets in a determined period of time and the process does not provoke temperature increase. The cement shows high formability, good adhesion to wet surfaces so the interface between the cochlear implant and the bone is very resistant. The cement allows the wide range of anatomical reconstructions of bone defects that are result of disease processes or surgical removal. The anatomic reconstruction allows to restore primary and optimal function of the treated tissues.

2. Glass-ionomeric implants made in two sizes provide possibility of reconstruction of ossicular chain. The implants shape corresponds to that of a plate with a stem tapering towards the end. In both sizes the angle between the stem and the plate is of 75°. Implants have smooth surface and rounded edges. The method of manufacturing allows perfect mapping of complicated shapes. Quick and effective, total and partial reconstruction of the ossicular chain is possible. This allows to restore the right function of the middle ear. In many cases application of these implants allows significant reduction in operation time.

3. Granulated glass-ionomeric material is used in two granular ranges: 1,0 - 1,6 mm and 1,6 - 3,2 mm. The method of manufacturing allows to obtain porous material for filling cavities and channels of the temporal bone. The biological in vitro as well as in vivo tests of these materials were conducted; clinical examination are in course.

Microstructure of the ceramic - polymer composites for dental applications

Joanna Siejka¹⁾, Malgorzata Lewandowska¹⁾, Krzysztof Kurzydowski¹⁾

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

19:00 -
poster

Composites are perspective materials for permanent fillings in dentistry. The microstructure of these composite materials is composed of polymer matrix and ceramic reinforcing particles. In the present work the quantitative studies of the microstructure of commercial fillers have been carried out. To this end scanning electron microscopy (SEM) and computer image analysis have been used. The equivalent diameter of ceramic particles, their shape and volume fraction were measured and the data have been used for a finite element model explaining their properties.

E-29

The influence of quality of dental alloys used for cast prosthetic completions on their corrosion resistance.

Sokołowski Jerzy²⁾, Rylska Dorota¹⁾, Klimek Leszek¹⁾

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2) Medical University of Łódź, Łódź, Poland

19:00 -
poster

E-30

Very often to the brand-new centrifugal casted NiCr-based alloys for prosthetic completions there are added some amounts of recasted alloys up to the 25% in weight concentration. The aim of presented paper is to compare the corrosion resistance of brand-new Ni-Cr alloys with such prepared castings. Corrosion resistance investigations were made by using the potentiodynamic polarisation method. The number of ions from the alloys passing to the artificial saliva was determined by means of X-Ray fluorescence analysis. Also the X-ray micro analysis of alloys and SEM observations were used to determine the chemical composition and structure of samples. The results show the decrease of corrosion resistance of alloys with recasted additions in comparison with brand-new Ni-Cr

Characterization of nanostructured hydroxyapatite ceramics densified at high-pressure and temperature

Jianmin Shi¹⁾, Bogdan F. Palosz²⁾, Stanisław Gierlotka²⁾, Arndt Klocke³⁾, Ulrich Bismayer¹⁾

1) Mineralogisch-Petrographisches Institut, University of Hamburg, Hamburg, Germany

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19:00 -
poster

E-31

Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, HA) is the main inorganic component of bone and dental tissues. Synthetic HA ceramics are bioactive and biocompatible materials due to the similarity in composition and crystal structure to biomineral HA. The low mechanical strength and fracture toughness of HA ceramics limit their applications to powders, coatings, porous bodies and nonload-bearing implants. It has been found that nanostructured materials have mechanical properties substantially different from their coarse-grained counterparts. We compacted nanosized HA powders under high-pressure and-temperature. The resulting materials were studied using X-ray diffractometry, scanning electron microscopy, and infrared spectroscopy. The microhardness and the fracture toughness of compacted HA ceramics were evaluated using a Vickers indentation method. It is found that all compacted samples under high pressure are composed of nanosized clusters which contain 10-20 nm HA grains. Elongated clusters were observed in the sample densified at higher temperatures. XRD and FTIR results indicate an increase in crystallinity and crystal perfection with increasing temperature. No obvious difference in microhardness was observed between samples, however, the fracture toughness increases with increasing temperature while the translucency of the densified materials is reduced. The difference in mechanical and physical properties of the nanostructured HA ceramics could be explained in terms of grain growth and water evaporation during high-pressure and high-temperature sintering.

Bioglasses as fillers of bone defects

Lucyna Zamorska¹⁾, Maria Laczka²⁾, Katarzyna Cholewa-Kowalska²⁾

19:00 -
poster

E-32

1) Collegium Medicum, Jagiellonian University (CM UJ), ul. Św. Anny 12, Kraków, Poland

2) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland

Bioactive glasses and glass-ceramics can be applied as fillers of bone defects due to their stimulating effect on the regeneration of the bone tissue. Four kinds of such materials of CaO-P2O5-SiO2 system with the symbols: RM, RMC, S2 and A2 have been obtained in laboratory conditions. The RM and RMC materials were produced by the melting method, and the S2 and A2 materials by the chemical sol-gel method. The final products had the form of granules 100-800 µm. The materials were characterised with respect to surface development and behaviour in simulated body fluid SBF. It has been found that in SBF the materials become covered with a surface hydroxyapatite layer, which is evidence of their bioactive properties. In order to estimate the biocompatibility of the obtained materials they have been subjected to selected tests in vivo. The granules were implanted in the skeleton muscle of rats in order to determine the progress of regeneration and the degree of tissue biocompatibility. Intensive phagocytosis of the material particles in the case of gel-derived A2 and S2 implants was observed. The melted materials RM and RMC showed low level of phagocytosis by macrophages, however these materials were surrounded by the so-called cells around the alien body. Certain reduction of metabolism was observed in the muscle fibres situated near the RM and RMC biomaterials, whereas near the A2 implants-good, progressive regeneration of the muscle fibres was observed. Attempts have been made to explain the observed, differentiated biological phenomena stimulated by biomaterials, using the supplementary immuno-histochemical tests.

Analysis of the mechanical properties in polymeric composite materials with short leather fibers

19:00 -
poster
E-33

Józef Wójcik¹⁾, Miriam Sánchez Pozos¹⁾, Flavio S. Vázquez Moreno¹⁾

1) UNIVERSIDAD AUTONOMA DEL ESTADO DE MEXICO (UAEM), Cerro de Coatepec s/n Fac. Ingeniería, Toluca 50110, Mexico

Leather is one of the most utilized materials through out the history of human kind. However leather waste is discarded in the environment (municipal landfills) with no care whatsoever, turning these landfills through time into huge sources of chrome pollution. The objective of this work is to demonstrate that the composites formulated with treated leather fibers and used as filling in a polymeric matrix may be utilized in some applications as substitutes of leather. These materials are suitable for making products that usually are made of leather (shoe heels, bags, insoles, etc). However, they can be used as materials for hospital equipments that may be advantageous from the economical point of view. On the other hand, application of this type of materials may reduce the environmental pollution, which is beneficial for the human health.

Two basic types of matrixes were considered due to their commercial and scientific importance: PMMA and PVC. These matrixes were reinforced by the following fibers: non modified leather short fibers, silanos leather short fibers and PMMA grafted leather short fibers. The mechanical properties of these different materials were determined. These properties are of fundamental importance for their potential applications.

Tuesday, September 16th

11:00 - 12:30

Morning session - Luigi Ambrosio, Alma U. Daniels

11:00 - 11:30

Hybrid technology as a basis for bone- and cartilage tissue engineering

invited oral

Clemens van Blitterswijk¹⁾

1) IsoTis NV, P.O. Box 98, Bilthoven 3720 AB, Netherlands

In the repair of bone- and cartilage tissue both the biological activity and the mechanical properties of the implant are relevant. Optimal and sufficiently complex biological properties can be best provided by using cultured cells while the mechanical properties of constructs are still largely dependent on scaffold properties. As a consequence, tissue engineering is a subtle interplay between biological- and material sciences. In this presentation we will demonstrate some of the strategies that were developed in our group in order to develop appropriate hybrid technology for tissue engineering.

Although bone and cartilage are quite different tissues, as clearly exemplified by the absence of vascularization in mature cartilage whereas the presence of vascularization is pivotal for bone metabolism, they require rather similar approaches in tissue engineering.

First: scaffolds need to be manufactured that can be truly designed in order to provide a fully functional three-dimensional system with appropriate porosity and sufficient mechanical properties. In our hands conventional production methods do not have adequate capabilities and much better results are obtained by employing 3-D printing technology. By these methods we have made promising scaffolds for cartilage replacement with a family of degradable PEO/PBT copolymers. For bone substitution rapid prototyping technology resulted in highly reproducible calcium phosphate scaffolds.

Second: another critical factor in tissue engineering is supplying sufficient nutrients to the cells in the scaffolds,

even in the center. This involves the use of bioreactor technology. We have successfully cultured up to 10 cc of human osteoprogenitor cells in bioreactor systems. Cells were multiplied to useful numbers and retained osteogenic properties after implantation.

Finally: one of the major challenges in multiplying cells further lies in retaining differentiation capacity. Recent findings by our group indicate that the use of tissue-inductive materials may play an important role in achieving this goal, thereby possibly reducing or substituting the use of growth factors.

The main challenge in tissue engineering will be in combining all developments as described in the above into one practical technology.

Synthetic osteoinductive biphasic calcium phosphates as scaffolds for bone tissue engineering

11:30 - 12:00

Joost D. de Bruijn¹⁾, Mirella van den Doel¹⁾, Clemens van Blitterswijk¹⁾, Klaas de Groot¹⁾, Huipin Yuan¹⁾

invited oral

1) IsoTis NV, P.O. Box 98, Bilthoven 3720 AB, Netherlands

Porous calcium phosphates have been proposed as bone tissue engineering scaffolds due to their osteoconductive properties and widespread clinical use as synthetic bone graft. Recently, microporous calcium phosphates have been described that are capable of inducing osteogenic differentiation of stem cells and bone formation in ectopic sites. In this study, we investigated such an osteoinductive biphasic calcium phosphate (BCP1200) as a potential bone tissue engineering scaffold and compared this to two non-osteoinductive calcium phosphates (BCP1300 and HA60).

Bone marrow derived mesenchymal progenitor cells were isolated from the Os Ilium of goats, expanded and seeded onto all three types of porous calcium phosphate in a concentration of 8.16 million cells per cc scaffold. After implanting the tissue engineered implants and control samples in the dorsal muscle of goats for twelve weeks, histology and histomorphometry was performed to evaluate the amount of newly formed bone in the available pore area.

Control samples of the osteoinductive biphasic calcium phosphate showed a limited amount of newly formed bone tissue in the inner pores (0.9 +/- 1.1%), confirming its osteoinductive properties. None of the control BCP1300 or HA60 samples showed any bone formation, while the tissue engineered samples of these materials only occasionally revealed some bone formation. On the other hand, the tissue engineered and osteoinductive BCP1200 samples showed many areas of bone formation. At twelve weeks, bone was still actively being formed as indicated by the presence of seams of cuboidal osteoblasts and osteoid, and filled 15.9 +/- 5% of the available pore area. These results indicate that the osteoinductive biphasic calcium phosphate BCP1200 is a superior bone tissue engineering scaffold as compared to the non-osteoinductive calcium phosphates HA60 and BCP1300, by retaining or inducing an osteogenic phenotype of seeded mesenchymal progenitor cells.

Differentiation of cells from adipose tissue into osteoblasts in the presence of biomaterials in vitro (preliminary report)

12:05 - 12:15

Radosław Olkowski²⁾, Edyta Sienkiewicz-Łatka²⁾, Malgorzata Lewandowska-Szumiel²⁾, Wojciech Lisik¹⁾, Maciej Kosieradzki¹⁾, Zbigniew Wierzbicki¹⁾

oral

1) Department of General and Transplantation Surgery, Medical University of Warsaw, Warszawa, Poland

2) Department of Biophysics and Human Physiology, Department of Transplantation & CTB, Medical University of Warsaw, Warszawa, Poland

Mesenchymal stem cells (MSC) are multipotent cells which can be induced to differentiate into various types of cells. The most popular source of MSCs is bone marrow. However, some cells obtained from adipose tissue may have potential to grow into bone and cartilage [Zuk P.A. et al. Tissue Engineering 7: 2001, 211-225], and thus, this type of tissue can be a promising source of cells for tissue engineering, especially that collecting adipose tissue is less invasive and less painful for patients when compared to bone marrow.

The aim of the experiment was to determine if omentum-isolated cells can reach the osteogenic potential in vitro in the presence of different biomaterials used for bone reconstruction. Human cells obtained from omentum major were used. Human bone harvested cells served as a control. Cells were cultured on various biomaterials, i.e.: steel, titanium, aluminium oxide, hydroxyapatite and polystyrene, in a culture medium with ascorbic acid and in the same medium supplemented with dexamethasone. Cells were cultured for 7 days under standard conditions. Alkaline phosphatase (AP) activity was measured as an indicator of osteogenic potential of cells.

AP activity was enhanced significantly in response to dexamethasone in all analyzed groups. This effect was dependent on biomaterial used as a support.

The results support the expectation that omentum-isolated cells can differentiate into osteoblast-like cells, although further experiments are needed for confirmation. The cells dependence on support during differentiation should be taken into account when cells are to be used in biomaterial scaffolds as tissue engineered implants.

Acknowledgment: This work was supported by the State Committee of Scientific Research (grant No. 05/PBZ-KBN-082/2002/06).

12:15 - 12:25

oral

Omentum major as potential source of osteogenic cells for tissue engineering (preliminary report)

Pawel Kowalczyk¹, Malgorzata Lewandowska-Szumiel¹, Wojciech Lisik², Maciej Kosieradzki², Maciej Siński³, Zbigniew Wierzbicki², Jacek Przybylski⁴

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2) Department of General and Transplantation Surgery, Medical University of Warsaw, Warsaw, Poland

3) Department of Internal Medicine and Hypertension; Medical University of Warsaw, Warsaw, Poland

4) Department of Biophysics and Human Physiology, Medical University of Warsaw, Warsaw, Poland

For skeletal tissue engineering, autologous stem cells are obtained from bone marrow (BM). However, BM procurement procedures may be painful and may yield a low number of stem cells upon processing. There is a need for other convenient stem cells sources.

The omentum major is potential source of mesenchymal tissues. Thus the aim of this study was to determine whether stem cells potentially present in the human omentum major could differentiate into osteogenic tissue. Cells harvested from tissue obtained during surgery, which would be otherwise discarded, were used in the experiment. The collected cells were incubated in a 96-well plate (15000 cells per well) per week in control media (i.e. DMEM + 10% FBS) without vitamin C (C-) and with vitamin C (C+), and media enriched with: vitamin D3 (C+ D3+), dexamethasone (C+ DEXA+) or demineralized bone matrix (C+ DBM+). Human osteoblasts were used as a control. Osteocalcin level and alkaline phosphatase activity (AP) were compared between groups.

The addition of C+ DBM into medium elevated the level of osteocalcin both in omentum-derived cells and osteoblasts. Whereas C+ DEXA+ and C+ D3+ increased AP activity in all cell populations.

It is suggested that the omentum major contains multipotent cells with osteogenic potential. Thus omentum may be taken into account as potential source of stem cells for skeletal reconstruction.

Acknowledgments

This work was supported by the State Committee of Scientific Research (grant No. 05/PBZ-KBN-082/2002/06).

12:30 - 14:00

Lunch break

14:00 - 15:30

Afternoon session - Joost D. de Bruijn, Stanislaw Slomkowski

14:00 - 14:25

invited oral

Amphiphilic polymers of N-vinylpyrrolidone. Behavior in water solutions. Liposome modification.

Mikhail I. Shtilman¹, Andrei N. Kuskov, Vitaliy V. Nutskov, Aristidis M. Tsatsakis, Vladimir P. Torchilin

1) D.I. Mendeleev University of Chemical Technology of Russia (MUCTR), Miusskaya Sq., Moscow 125047, Russian Federation

Amphiphilic polymers of N-vinylpyrrolidone, which contain fragments of water-soluble polymer and hydrophobic end groups and are able, to build into liposome coats were synthesized.

These polymers were obtained by two-phase method. First the radical polymerization of monomers (N-vinylpyrrolidone, acrylamide) in presence of initiator (dinitrile of azobisisobutyric acid) and functional chain transmitters (mercaptanes) was carried out.

Changing the mercaptane amount, yield and molecular weight of polymers can be controlled. Polymers with different molecular weight (Mn=1500-14000) were synthesized. Coincidence of molecular weights obtained by titration and by ebullioscopy corroborates that these polymers have only one functional end group.

For introduction of a long chain aliphatic group the reaction between polymer with carboxylic end group and stearylamine in the presence of dicyclohexylcarbodiimide or reaction between polymer with amine end group and chloranhydride of stearic acid was carried out.

Obtained polymers were soluble in water and as it was shown form associates with the compounds, which contains hydrophobic fragments. Also polymers form micelles in water solutions. The influence of polymer molecular weight on critical concentration of aggregation was studied. Obtained modified liposomes were of 165-190 nm sizes.

Synthesized polymers protect liposomes from destroying effect of polycation. These studies were carried out by means of fluorescent marker and method of studying of modified-by-polymers liposomes fraught by pyren.

Behavior of modified by synthesized polymers of N-vinylpyrrolidone liposomes was studied on white linear mice. It was determined that introduction of polymers increases time of liposome circulation in blood and decreases liposome capture by liver.

References

V.P. Torchilin, M.I. Shtilman et al. // *Biochim. Biophys. Acta.* 1994. V.1195. P.181-184.

Do PMMA Bone Cements Differ in Long-Term Stability?

Alma U. Daniels¹⁾, Gladius Lewis²⁾, Dieter Wirz¹⁾, E. W. Morscher¹⁾

1) *Lab. for Orthopedic Biomechanics, Univ. Basel, Basel, Switzerland*

2) *Mech. Engineering Dept., Univ. of Memphis, Memphis, United States*

14:30 - 14:45

oral

INTRODUCTION: Correlation studies performed on registries of clinical total hip replacement (THR) suggest that the type of commercial, PMMA-based bone cement used is a major determinant of the procedure's clinical longevity (Havelin LI, et al. *J. Bone & Joint Surg.* 77:10, 1995). However, laboratory studies of cement initial mechanical properties have not offered clear-cut explanations for clinical performance differences. This pilot study was performed to investigate the possibility that clinical differences may be due to differences in physico-chemical stability of the principal cement component—powdered PMMA—because this could result over time in development of property differences in PMMA-based cements. Isothermal microcalorimetry (IMC) provides a means for comparing and predicting long-term stability of materials, even when they degrade extremely slowly. IMC has shown that gamma radiation sterilization (gR) increases the degradation rate of UHMWPE used in THR components, while ethylene oxide (EtO) does not. (Charlebois s, Daniels AU, Lewis G, *Biomaterials* 24:291-296, 2003).

MATERIALS & METHODS: In this study, 3 commercial bone cement starting powders with differences in sterilization (EtO vs. gR), and molecular weight (MW > or < 350 kD) were compared: P (EtO, MW > 350), SU (EtO, MW < 350), SP (gR, MW < 350). Exothermic heat flow rate (proportional to degradation rate) was determined in air at 25°C by IMC.

RESULTS: Heat flow rates were all very low, due to the good inherent stability of PMMA. However, compared to P (expected to be most stable), heat flow rates were about 2X higher for SU and about 3.7X higher for SP.

DISCUSSION & CONCLUSIONS: Results suggest cements made from higher MW, EtO-sterilized PMMA powders will be more stable over time. Use of cement P (produced from EtO-sterilized, MW > 350 kD powder) correlates with higher longevity in THR clinical registries. Further IMC studies are underway, and studies of polymerized cements are planned.

Dynamic mechanical behaviour of plain and gentamicin loaded bone cements

Roberto De Santis²⁾, Francesco Mollica, Dante Ronca, Luigi Ambrosio¹⁾, Luigi Nicolais

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2) *Institute of Composite & Biomedical Materials (IMCB-CNR), Piazzale Tecchio, 80, Naples 80125, Italy*

14:50 - 15:05

oral

Cements based on poly(methyl-methacrylate) (PMMA) are used for anchoring the prosthetic components to contiguous bones. The aim of this work is to characterize experimentally two different bone cement formulations through DMA in a water chamber.

CMW-1 and CMW1-G (from DePuy) bone cements were tested. In order to investigate the dependence of material properties on water uptake and monomer release, each cement sample was randomly divided in two groups conditioned in a water chamber at 37°C for 2 hours (sample 1) and 1 week (sample 2) respectively. Mechanical tests were carried out using the 3 point bending method (ASTM D790) in a water chamber at 37°C.

The radical polymerization of PMMA does not proceed to completion. Thus, beside water absorption, conditioned specimens (samples 2) are characterized also by the release of unreacted monomer.

Our results indicate that at low frequency sample 1 dissipate more energy than sample 2, while at high frequency no difference was observed between the samples. The higher damping factor values of sample 1 at 0.01Hz clearly indicate that the unreacted monomer plays an important role on the main chain mobility. As the unreacted monomer is released through a prolonged conditioning process (sample 2), the dissipating capability at low frequency drastically drops, while dissipation values similar to sample 1 are measured at high frequency. This effect at high frequencies may be related to the small role played by the solvent thermodynamic quality on the time-scale and activation energy for local conformational transitions. Thus the residual unreacted monomer, in vivo applications of bone cements, mainly affect the relaxation or creep in a transient manner, while water absorption mainly increase the damping capability at high frequency, thus affecting the side chain mobility.

Effect of polysiloxaneurethaneurea elastomer structure on free radical formation in sterilisation by E-beam/gamma irradiation

Janusz Kozakiewicz³⁾, Izabella Legocka²⁾, Jarosław Sadło²⁾, Magda Brzozowska³⁾, Monika Celuch¹⁾, Jarosław Przybylski³⁾

1) *Institute of Nuclear Chemistry and Technology, Dorodna 16, Warsaw 03-195, Poland*

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3) *Industrial Chemistry Research Institute (ICRI), Rydygiera 8, Warsaw 01-793, Poland*

15:10 - 15:25

oral

Samples of polysiloxaneurethaneurea elastomers were prepared by moisture-curing of the siloxaneurethane prepolymers terminated with NCO groups at 25°C and 50% R.H. Prepolymers were synthesized from polysiloxanediols of different chain length and diisocyanates using different NCO/OH molar ratio. Sterilisation of samples was made using 28 MeV E-beam or gamma rays to reach 28kGy dose.

In order to investigate free radical formation in sterilisation process, samples were studied using EPR (Electron Spin Resonance) technique 15 min after irradiation had been completed. For samples irradiated at R.T. no free radicals were observed while the clear signals corresponding to paramagnetic fragments of different structures of polymer chain were recorded by EPR for samples exposed to gamma rays at liquid nitrogen temperature. This result indicated that though free radicals were formed in sterilisation process, either recombination or transformation proceeded very quickly at R.T., so no evidence of free radicals was found at that temperature.

FTIR studies did not reveal any distinct differences between samples investigated before and after sterilisation what suggested that mostly recombination leading to crosslinking, not transformation leading to formation of new functional groups was responsible for total decay of free radicals at R.T. Mechanical properties of sterilised and not sterilised samples of the same chemical composition will be compared in order to confirm that assumption.

15:30 - 15:50

Coffee break

15:50 - 18:00

Afternoon session - continued - Clemens van Blitterswijk, Ralf-Peter Franke

15:50 - 16:15

Alternative methods bring a new input in biocompatibility testing

Malgorzata Lewandowska-Szumiel¹⁾, Krzysztof Sikorski²⁾, Andrzej Szummer²⁾, Janusz Komender³⁾

invited oral

1) *Medical University of Warsaw, Department of Biophysics and Human Physiology; Department of Transplantology & CTB, Chałubińskiego 5, Warszawa 02-004, Poland*

2) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

3) *Medical University of Warsaw, Department of Transplantology & CTB, Centre of Biostructure, Chałubińskiego, Warsaw 02-004, Poland*

Due to the growing offer of candidate biomaterials there is a need of reliable, effective and quick biocompatibility tests. In this respect alternative techniques seem to be an attractive tool which significantly limits the number of experimental implantations to animal tissues. Moreover, it seems that testing procedures in vitro make possible observations of such aspects of implant-tissue interaction, which are difficult to observe by routine methods.

Three examples of original procedures for examination of biomaterials for bone tissue reconstruction are shown, each of them concentrated on a particular aspect of implant-bone interaction, i.e.:

-micromotion which exist between load-bearing implants and host bone -

experimental simulation of the mutual implant-tissue displacement in cell culture has been proposed, followed by a brief discussion of its influence on human bone derived cells (HBDCs) cultured on the surface of titanium, hydroxyapatite and alumina.

-elastic strain of metallic stem of endoprosthesis induced by a loading - influence of elastic strain applied to the Ti6Al4V samples on HBDs cultured on their surface is examined in order to confirm that osteoblasts in vitro are effected by elastic strain of metallic support. This may be one of the factors responsible for the loosening of endoprosthesis.

-presence of wear particles in tissue adjacent to implant - the method for observation of biomaterial particles phagocytosis by cells in culture by means of electron microprobe has been proposed as an instrument for detecting the risk of undesirable elements appearance near the implants.

Finally, using alternative techniques as an invaluable assistance in tissue engineering seems a promising perspective for both the development of biomaterials and alternative methods.

16:20 - 16:35

Biocompatibility study of nitrided titanium alloy for bone implantation by laser flow cytometry, laser scanning cytometry and confocal microscopy

Elzbieta Czarnowska²⁾, Agnieszka Sowinska²⁾, Bożena Cukrowska²⁾, Michał Godlewski³⁾, Tadeusz Wierzchoń¹⁾

oral

1) *Warsaw University of Technology, Koszykowa 75, Warsaw 00-662, Poland*

2) *The Children Memorial Health Institute, Department of Pathology (CMHI), al. Dzieci Polskich 20, Warsaw 04-736, Poland*

3) *Warsaw Agricultural University, Department of Animal Physiology, Warsaw, Poland*

One of the parameters that allow assessment of the biocompatibility of innovative materials for implant and prosthesis is cellular adhesion. Therefore the aim of our study was to evaluate the ability of fibroblasts and osteoblasts to adhere to samples with TiN + Ti₂N + AlphaTi(N) surface layers produced on titanium alloy by glow discharge nitriding processes in comparison to titanium alloy using laser flow cytometry (FACS), laser scanning cytometry (LSC), and confocal microscopy. Cells were cultured on samples for 48 hours. Data obtained from FACS showed the presence of fibronectin receptors on cells that adhered to both titanium alloy and nitrided surface layers. However, live cells did not express fibronectin, in contrast to dead cells that positively stained for this protein what could suggest intracellular storage of fibronectin. Fibronectin production by adhered cells was confirmed by investigations in LSC and confocal microscopy. Both intra- and extracellular presence of fibronectin in cells adhering to both

type of samples was shown. The amount of dead cells harvested from samples was about 30% for both types of biomaterials, as measured by FACS. LSC analysis showed that 5-12% of adhered cells died through mechanisms of apoptosis. These results suggest that strong adhesion to material blocks the mechanisms involved in cell spreading, which can induce cell apoptosis.

In conclusion, results confirms the good biocompatibility of TiN + Ti₂N + AlphaTi(N) surface layers and its potential in bone surgery applications. Employment of LSC together with confocal microscopy for analysis of cell behaviour in contact with biomaterials seems to be very useful, but FACS analysis of chemically harvested cells should be carefully taken into account in the study of cell adhesion processes.

Cytokine secretion during in vitro cellular interaction with titanium alloy and nitrided surface layer produced under glow discharge conditions

16:40 - 16:55

Bożena M. Cukrowska²⁾, Agnieszka Sowinska²⁾, Elzbieta Czarnowska²⁾, Tadeusz Wierzchoń¹⁾

oral

1) *Warsaw University of Technology, Koszykowa 75, Warsaw 00-662, Poland*

2) *The Children Memorial Health Institute, Department of Pathology (CMHI), al. Dzieci Polskich 20, Warsaw 04-736, Poland*

Implantation of a foreign material into human tissue leads to inflammatory and repair reactions, that involve releasing pro-inflammatory mediators such as TNF-Alpha, IL-6 and IL-1Beta. Investigations on cytokine production by cells cultured in contact with new biomaterials can be useful to identify materials better suited for prosthesis and bone implants. The aim of our study was biocompatibility of nitrided surface layers produced under glow discharge conditions on titanium alloy in comparison to titanium alloy in terms of activation of cytokine production and release from human fibroblasts and osteoblasts, i.e. cells responsible for proper biomaterial integration with tissues surrounding the implant. Cells were cultured on disc-shaped specimens prepared from titanium alloy and with surface layer TiN+Ti₂N+AlphaTi(N) type for 24 and 48 hours and cytokine contents in culture medium were measured using enzyme linked immunoassay (ELISA) systems. TNF-Alpha induction was not observed in either 24- or 48-h cultures exposed to both types of samples. IL-1Beta was found only after 48h cultivation. Titanium alloy induced higher amounts of IL-1Beta than the nitrided surface layer. IL-6 production was high already after 24h cultivation regardless of the type of titanium sample and increased after 48h. In summary, it was shown that these biomaterials did not activate the typical pro-inflammatory mediator. However, both types of materials activated production of IL-1Beta and IL-6, i.e. cytokines, which despite their pro-inflammatory action, can also play a positive role in bone remodeling. It should be stressed that the biocompatibility of biomaterials is dependent on the balance between a variety of cytokines inducing biological mechanisms connected with bone integration of prosthesis and implants. Our preliminary study shows that IL-6 and IL-1Beta could play an important role in these processes.

Influence of surface roughness of flat TiAl6V4 discs on cell differentiation in vitro and on tissue integration in vivo

17:00 - 17:15

Ralf-Peter Franke¹⁾

oral

1) *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany*

Round Ti Al6 V4 alloy samples with the same surface texture were assayed in human bone marrow culture and in NMRI wild type mice. In the mice the samples were implanted both in the interscapular region and onto the temporal bone of the head after preparation of a rectangular periosteal flap which was then used to fix the sample subperiostally.

The new samples were examined by Scanning Electron Microscopy (SEM) and by Atomic Force Microscopy (AFM). New samples were enzymatically digested and again examined by SEM and AFM. After the exposition in cell cultures or the implantation in mice a fraction of the samples was prepared for immune histology and assayed in Confocal Laser Scanning Microscopy (CLSM) for the development/advent of cells of the leucocytic line or of the mesenchymal line respectively.

Another fraction was enzymatically digested, as described before, and examined by SEM and AFM. A third fraction of the animal implanted samples was examined in a mechanical testing device for the shear strength of the implant-bone-bonding.

Results:

There was practically no influence of the enzymatic digestion of new Ti Al6 V4 on the surface roughness of the samples.

Surface roughness has an influence on the development of cells of the leucocytic line on the samples in human bone marrow culture.

The surface roughness of Ti Al6 V4 samples decreased after implantation in the interscapular region as well as in the temporal headbone. This was especially true for roughness structures of the size of 20-40µm. The reduction surface roughness of samples implanted in the interscapular region was more pronounced in the submicron range. Surface roughness in the submicron range seems to be important for tissue-integration and coincides with the amount of gap bridging and mineralised tissue constructs between implant and bone.

17:20 - 17:35

oral

Bioactivity of Carbon Particles**Stanisław Mitura¹, Katarzyna Bąkowicz¹***1) Technical University of Łódź, Institute of Materials Engineering, Stefanowskiego 1/15, Łódź 90-924, Poland*

Biocompatibility of materials with living organism onto medical implants was the first step to develop the next examinations. The results of these researches have not proved exactly this assumption. Biocompatibility and biotolerance of diamond and graphite have been observed on macroscopic level (in vivo) in the pioneer researches with carbon coatings.

Carbon Particles - Diamond Powder Particles (DPP) and Graphite Particles have been examined on molecular, macroscopic and clinical level.

On the molecular level Diamond Powder Particles (DPP) have very high bioactivity. This activity involves toxic processes of cell membrane and damage of nucleus. DPP inhibits these toxic reactions and prevents the different human disorders, for example: Alzheimer Disease, Parkinson Disease, Diabetes Mellitus, Myocardial Infarctus.

The comparison between Graphite Particles and Diamond Particles shows that mainly graphite has very high biocompatibility with a living organism on the molecular level but its bioactivity is not significant.

In vivo examinations with Carbon Particles proved the biocompatibility with living organism (the contact reaction with peritoneum) and these results indicate that only DPP is bioactive.

In vivo DPP inhibits inflammatory process in peritoneum stimulated by LPS (lipopolysaccharide).

Graphite Particles are neutral for this reaction.

The clinical examination (patch tests) proved the very high biotolerance of both Carbon Particles. The results show the absence of Irritation Reaction (IR) and Allergic Reaction on the skin in patients with allergy after exposure to Carbon Particles in the patch tests.

Graphite Particles is neutral for this reaction.

The clinical examination (patch tests) proved the very high biotolerance of both Carbon Particles. The results show the absence of Irritation Reaction (IR) and Allergic Reaction on the skin patients with allergy after exposition of Carbon Particles in the patch tests.

17:40 - 17:55

oral

Microacoustical NDE of pyrocarbon materials for cardiovascular prosthetic application**Yulia S. Petronyuk¹, Vladimir N. Zygan²***1) Institute of Biochemical Physics RAS (IBCP), Kosygin 4, Moscow 119991, Russian Federation**2) Kirovo-Chepetsk Chemical Plant, Kirovo-Chepetsk 613 020, Russian Federation*

Primary atomic structure of pyrocarbons is close to atomic arrangement of natural graphite (stacks of hexagonal atomic planes). Further microstructure formation critically depends on pyrolysis conditions. Low-temperature (LT) pyrolysis gives different types of microstructure but only isotropic pyrocarbon is of special interest. The exceptional mechanical properties in combination with chemical and biological inertness have determined the main field of isotropic pyrocarbon application: the material is employed for manufacturing cordial prosthetic devices. Cardiovascular applications impose hard demands on pyrocarbon materials. Reliability of prosthetic devices depends on microstructure of pyrocarbons and presence of microdefects, on local mechanical properties and their distribution over the specimen body. We have developed high frequency focus ultrasound methods for evaluating local mechanical properties and microstructure, for visualizing defect distribution inside the material body and non-destructive inspection of material to be employed for prosthesis production.

Microstructure characterization has been performed by acoustic imaging of structural elements inside the material body. The layered structure resulted from a large-scale microscopic ordering has been observed. Acoustic microscopy technique has been employed for revealing inclusions, cracks and grain structure. Layer-by-layer imaging provides 3D reconstruction of matter structure.

The elastic characteristics of pyrocarbons have been measured by microacoustical technique. Despite the substances being considered as isotropic materials, the ultrasonic measurements revealed elastic anisotropy. It seems the anisotropy is correlated with occurrence of large-scale ordering in pyrocarbons. A part of specimens reveals an elastic orthotropy (transversal isotropy), while others demonstrate a more complicated anisotropy. A complete set of elastic moduli has been measured for the orthotropic material.

Wednesday, September 17th

11:00 - 12:30

Morning session

12:30 - 14:00

Lunch break

14:00 - 15:30

Afternoon session - Alma U. Daniels, Malgorzata Lewandowska-Szumiel

Biocompatibility of metallic implants for reconstructive surgery; choice of material, design, processing, clinical application

14:00 - 14:25

Ralf-Peter Franke¹⁾

invited oral

1) *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany*

Until the year 2010 the most important medical implants are assumed to be the total hip prosthesis (THP), the total knee prosthesis (TKP) and the vascular stent according to the WHO. Metals are the major constituents of the hip and knee implants and so far of vascular stents. Metallic implants also play an important role in the reconstructive surgery in the fields of tumour and trauma therapy.

The high strength and fatigue resistance as well as availability, easy processing and moderate prices explain the important roles gained by metals in implantology. A particular importance is given to titanium and its alloys, because these metals may be integrated into surrounding tissues, and especially hard tissues, and exhibit chemical and mechanical bonding, indicative of good biocompatibility/histocompatibility and biofunctionality.

A well engineered coating of implants with very thin calcium-(phosphate)layers seems to be further advantageous.

The use of metallic implants is not without problems. Different and high rates of corrosion influence the choice of an implant material, since fretting corrosion among others may contribute strongly to the production of wear particles which might lead to a particle disease and to implant loosening consecutively. The corrosive behaviour is influenced by the industrial processing and the surface structure of the implant.

Important, too, is the affinity of metallic surfaces to microbial contaminants and biofilms. The rate of implant infection, necessitating the riskful exchange of the implant, even in good clinics, can reach 2 %-3 %, as taken from literature.

The careful design for first time implants and for exchange implants has to face the problems of stress shielding and loss of bone stock.

The major influence of surgical training is demonstrated by the Scandinavian implant register study.

Study on the biocompatibility of c.p. titanium surgical clips

14:30 - 14:45

CATALIN POPA¹⁾, DINDELEGAN GEORGE²⁾, CANDEA VIOREL¹⁾

oral

1) *Technical University of Cluj-Napoca, Department of Materials Science and Materials Technology (UTCN), Bd. Muncii, Cluj-Napoca 3400, Romania*

2) *University of Medicine and Pharmacy "Iuliu Hatieganu" of Cluj-Napoca, Cluj-Napoca, Romania*

ABSTRACT

C.p. titanium surgical clips are widely used in medicine, for endoscopic or laparoscopic surgery, gynecology, etc. Some of the clips may be removed at the end of the intervention but most of them remain in the body as long-term implants. Analysis of soft tissue adjacent to explants reveals often the effects of migration of metal ions, ranging from minor pigmentation to incipient stages of metallosis. These effects are chiefly determined by the injuries of the oxide layer in the bending area but electro-chemical causes are acting as well. Commercially available clips are generally surface treated in order to increase the thickness and stability of the oxide layer responsible for the passivity of c.p. titanium.

This work approaches the causes that may determine a faulty clinical behaviour of c.p. titanium surgical clips. Biocompatibility was regarded as general property that characterises the behaviour of the biomaterial in a given application.

Clip-shaped samples (with a proportionally reduced size), as well as flat samples, subjected to heat or electro-chemical treatments were tested in vivo (Wistar rats) and in 3% NaCl solution. The effect of narrow scratches or cracks on the oxide surface was analysed in the context of the elastic distortion cells resulted on the outer / inner sides of the bending area. The effect of the microstructure was considered as well. To conclude on the biocompatibility of surgical clips, the bending force was also considered.

The study shows that by an appropriate constructive design of the clips, as well as by an differential heat treatment leading to a graded structure in the active bending area, the biocompatibility of clips can be improved.

Thin layers of TiN fabricated on metallic titanium and polyurethane by pulsed laser deposition

14:50 - 15:05

Bogusław Major¹⁾, Reinhold Ebner²⁾, Tadeusz Wierzchon⁴⁾, Waldemar Mroz³⁾, Wolfgang Waldhauser²⁾, Roman Major¹⁾, Michal Wozniak⁴⁾

oral

1) *Institute of Metallurgy and Materials Sciences, Polish Academy of Sciences, Reymonta 25 St, Krakow, Poland*

2) *Joanneum Research Forschungsgesellschaft mbH, Laser Center Leoben, Steyrergasse, Graz A8010, Austria*

3) *Institute of Optoelectronic Military University of Technology (MUT), Wotowska, Warsaw 02-507, Poland*

4) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

Successful use of titanium and its alloys in medicine arises from its promising effects in trauma treatments due to biocompatibility and corrosion resistance. Titanium nitride (TiN) is regarded as a potential biomaterial for blood-contact applications. Titanium nitride thin layers were fabricated by pulsed laser deposition (PLD) using a Nd:YAG laser on two types of biomaterials like: metallic titanium and polyurethane. Formation of the uniform smooth surface was stated in both cases. Application of scanning electron microscopy (SEM) for examination of the cross-section of the materials revealed the form of diffusion layers with continuous transfer from the deposited film to the substrate. Transmission electron microscopy (TEM) examinations performed on the thin foils prepared from the cross-section of the metallic titanium covered with the TiN confirmed the diffusion character of the deposited layer, moreover presenting a fine grained microstructure of the deposited TiN phase. Texture examinations were carried out for the deposited TiN as well as for the titanium substrate. Application of the pseudo-position sensitive detector in texture examination allow to draw the pole figures of residual stresses, while using the X-ray diffraction method for residual stress measurement ($\sin^2\Psi$) made possible to measure their values which were for the TiN phase in the range -8 to -10 GPa for films fabricated on the metallic titanium substrate and of order of -4 to -5 GPa for the polyurethane substrate. Morphology of the surface of the deposited layers was examined by application of atomic force microscopy (AFM). The results revealed contribution of the deposition parameters as well as the thickness of the layer to the crystallite sizes and vertical diameter.

TiNi-based shape memory alloys for medical application

V. G. Pushin²⁾, Ruslan Valiev¹⁾

15:10 - 15:25

oral

1) *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation*

2) *Institute of Metal Physics, Ural Division of Russian Academy of Sciences, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation*

Among the various types of materials with thermoelastic martensitic transformations and shape memory TiNi-based alloys have the highest strength and plastic properties. They also exhibit unique shape-memory effects (SME) and reproducibility (single-way and reversible, or two-way, SME, hyperelasticity, damping, etc), as well as high reliability and good mechanothermal, mechanocyclic, thermocyclic lifetimes. In addition, they exhibit weldability, corrosion resistance, and biocompatibility; have a relatively simple chemical composition and are characterized by a good workability of the metallurgical process and subsequent industrial procedures. As alloys with a SME, they are irreplaceable in critical articles and state-of-the-art devices in medicine. In this paper the main classes of medical TiNi -based alloys and, accordingly, types of medical application of given materials as instruments and implants are considered. The novel modern methods and new approaches for improvement of structure and properties of TiNi shape- memory alloys have been discussed.

Coffee break

15:30 - 15:50

Afternoon session - continued - Ralf-Peter Franke, Maria Laczka

15:50 - 17:45

New generation of bioactive, ceramic materials

Maria Laczka¹⁾

15:50 - 16:20

invited oral

1) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland*

Bioglasses and bioactive glass-ceramics used in medicine and dentistry as bone substitutes have unique properties; they are able to joint with living tissues by hydroxyapatite surface layers as well as to stimulate tissues to regeneration. Lately a special attention was given to the new generation of these biomaterials (glasses and glass-ceramics) produced by the sol-gel method. Gel-derived biomaterials are characterised by higher bioactivity, especially concerning their effect on the faster reconstruction of living tissues. Gel-derived materials of various chemical and phase compositions were obtained in laboratory conditions in the form of powders, compact and porous sinters as well as in the form of coatings on inert base. The simulated body fluid (SBF) test and biological "in vitro" tests were carried out for gel-derived biomaterials using as a control bioglasses produced by traditional melting way. Moreover, "in vivo" examinations were conducted. In these experiments gel-derived bioglasses in the form of 100-800 μm granules were implanted to the defect in rabbit shank bone. Heterograft made of alien bone tissue was used as a control material. The progression of bone reconstruction was estimated on the basis of radiological tests, scanning mirtoscopy (SEM) and EDAX analysis, structural examinations of fillers (FTIR, XRD) and histopatological tests. It has been found that the process of reconstruction of bone tissue proceeds faster in the presence of gel-derived bioglass in comparison with its when heterograft was used. Results of our experiments indicate the high bioactivite of gel-derived biomaterials.

Morphological and structural study of silica based implants in bone

P N. De Aza²⁾, Zofia B. Luklinska¹⁾

16:25 - 16:40

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oral

2) Instituto de Bioingenieria, Universidad Miguel Hernandez, Edificio Torrevalio, Avda. Ferrocarril s/n, Elche 03202, Spain

The paper reports on the structural morphology of the interface between a porous glass ceramic (A3) based on the $\text{SiO}_2\text{-CaO-MgO-K}_2\text{O-Na}_2\text{O-P}_2\text{O}_5$ system and the surrounding bone in rat tibias. After devitrification the glass was composed of diopside and calcium phosphate phases.

In physiological environment, porous implants are highly favourable, as their open structure secures large surface areas for biochemical processes.

The interfaces formed after implantation for 6, 8 and 12 weeks were examined by analytical scanning and high-resolution transmission electron microscopy.

The new bone was found growing in direct contact with the implants. Gradual degradation processes of the A3 glass ceramic encouraged the bone ingrowths into the pores of the implanted material. At 12 weeks, most of the pores were filled with osseous tissue and vascularization process of the implant was well established.

Significant morphological remodelling at the interface formed a disorganised bone tissue structure, followed by lamellar bone arrangement. Advanced cellular activity and collagen fibrils deposition were also observed.

Selected area diffraction pattern of the interface displayed distinctive spots from the crystalline implant in addition to characteristic arcs associated with the mature bone.

In the area of mature bone, Ca/P ratio reached value for pure HA, while inside of the material, the ratio was consistently higher at 2.00. The reason for this increased ratio could be continuous release of Ca ions from the implant. Secondly, the bone could enrich itself with carbonate ions, producing carbonate enriched HA of variable CO_3^{2-} content

In conclusion, the porous A3 material demonstrated bioactive-degradable properties right at the interface and inside the whole volume of the implant. The material is expected to be satisfactory in applications related to repair or replacement of living bone, where a progressive substitution of the implant is required.

Non-elastic behaviours of high-porosity ceramics and ceramic-polymer composites

16:45 - 17:00

Yuriy M. Podrezov¹⁾, S O. Firstov¹⁾, M Szafran³⁾, Krzysztof Kurzydowski²⁾

oral

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2) Warsaw University of Technology, Faculty of Materials Science and Engineering, Woloska 141, Warsaw 02-507, Poland

3) Warsaw University of Technology, Woloska 141, Warsaw 02-507, Poland

High porosity ceramics and ceramic-polymer composites are now widely applied in a number of fields and in particular in medicine. The present paper is focused on mechanical properties of these materials and possible ways for their improvement. One of them is via increasing relative stiffness. The other is by rising the fracture toughness and deformation energy, which can be achieved by increasing the volume fraction of pores due to non-elastic behaviours of high porosity ceramics under bending. The third method is to increase absorbing capacity of strain energy. In the paper the sensitivity of these three effects to the microstructure of pores and solid phase is analysed and methods for microstructure optimisation are proposed.

Fracture mechanisms of porous ceramics transform with increasing porosity from a brittle cleavage typical of monolithic materials to cracking of separated defects in high-porosity ones. According to the model proposed, four stages of process can be identified: (a) loading without micro-cracking, (b) stable, non-localised micro-cracking before reaching the stress maximum, (c) stable localised microcracking after stress maximum point and (d) unstable (catastrophic) fracture. These stages results in non-linear behaviour of high porosity ceramics under bending and compression making these materials attractive for in terms of damping capacity.

The proposed model of fracture can be used to determine the dependence of the number of cracks per unit area on the level of applied stresses. The important conclusion, that can be derived from this model is that properties of the high porosity ceramics can be improved by infiltrating them with polymers due to relaxation of stress concentrations.

New hydroxyapatite based nanomaterials for potential use in medical fields

Roxana M. Piticescu²⁾, Gabrielle C. Chitanu³⁾, Madalina L. Popescu²⁾, Witold Lojkowski¹⁾, Agnieszka Opalińska¹⁾, Tomasz Strachowski¹⁾

17:05 - 17:20

oral

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

2) Institute for Non-ferrous and Rare Metals, 102 Biruintei Blvd., Pantelimon 73957, Romania

3) Petru Poni Institute of Macromolecular Chemistry of the Romanian Academy, Aleea Grigore Ghica Voda 41A, Iasi, Romania

Hydroxyapatite (HAP) is a biocompatible material with good osteoconductive properties. This ceramic material was intensively studied due to its similitude to structure and composition with natural bone. Its utilisation it is however limited due to its low mechanical strength. The aim of this paper is to find the most suitable reinforcing agent or additive for HAP in order to obtain biological active nanomaterials with improved mechanical properties. Different powders of pure HAP, zirconia-reinforced HAP and HAP-polymer composites have been prepared by the hydrothermal method. Yttria-doped zirconia helped the crystallization of HAP and inhibited the grain growth as showed by XRD patterns. In the case of polymer-HAP composites results obtained from XRD, SEM, BET and picnometric densities measurements made us to presume that some physical bonds were formed between organic and inorganic phase and the same inhibition effect of grain growth was observed for a concentration threshold. Expected applications of these new materials are in the field of dental implants or as filling material for implants.

17:25 - 17:40

oral

Porous hydroxyapatite, tricalcium phosphate and polylactide as reconstructive material in treatment of maxillar's hemangioma-case report**Lukasz Wysienki^{2,4}, Andrzej Kukwa², Anna Slosarczyk¹, Krzysztof Wlodarski³***1) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland**2) Medical University of Warsaw, Division of Dentistry, Department of Otolaryngology (ENT), Sepinska 19/25, Warszawa 00-925, Poland**3) Department of Histology and Embryology, Medical University of Warsaw, Poland**4) Department of General and Experimental Pathology, Medical University of Warsaw, Warszawa, Poland*

Large hemangioma treatment is great challenge for reconstructive technics in head and neck surgery . The tumor infiltrates and damages surrounding tissue and can cause dangerous for live bleeding. Ceramic and polymeric scaffolds can be helpful material for reconstruction during surgery procedure of its tumor. Authors show case report of maxillars's hemangioma treatment where hydroxyapatite, tricalcium phosphate and polylactide were implanted as scaffolds. The patient underwent 2 years follow up with no episodes of bleeding, with good cosmetic and physiologic effect.

19:00 - 21:00

Poster session

Symposium F

Functional Nanostructured Materials for Optoelectronics and other Applications

Symposium organisers

- **Donats Millers**, Institute of Solid State Physics, University of Latvia, Riga, Latvia
- **Ulrich Herr**, Ulm University, Germany
- **Witold Lojkowski**, High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Warsaw
- **Wieslaw Strek**, Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Wrocław
- **Hans-Jörg Fecht**, Ulm University, Germany

Proceedings

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High Pressure Research Center "UNIPRESS", Polish Academy of Sciences:

- SPUB MSN and other

Monday, September 15th

13:55 - 14:00

Opening address

Main Building, Small Hall (Mała aula)

14:00 - 15:30

Afternoon session

Main Building, Small Hall (Mała aula)

14:00 - 14:30

2D and 3D-photonic crystals fabricated in silver-doped glasses

Yuri S. Kaganovskii¹⁾

invited oral

1) *Bar-Ilan University, Ramat-Gan 52900, Israel*

Glasses containing diffusion layers (of 20 -30 microns thick) doped by Ag⁺ ions were used for direct laser recording of 2D and 3D photonic crystals. After reduction of Ag⁺ ions in a hydrogen atmosphere at elevated temperatures, neutral Ag atoms formed nanoclusters of 20-nm radius. 2D and 3D photonic crystals were fabricated by pulsed laser irradiation using four or five coherent intersecting beams. Under irradiation the clusters absorbing light energy are heated to high temperatures and become mobile due to formation of liquid shells around the clusters. Adjacent clusters moved to each other and towards the irradiated surface in local temperature gradients. We present theoretical analysis of temperature distributions around nanoclusters of various sizes and calculate kinetics of cluster motion both for nanosecond and sub-picosecond pulsed irradiation.

14:30 - 15:00

Photonic Crystals

Daniel C. Meisel¹⁾, **Markus Deubel**²⁾, **Martin Hermatschweiler**¹⁾, **Kurt Busch**³⁾, **Wolfpeter Koch**²⁾, **Georg von Freymann**¹⁾, **Alvaro Blanco**¹⁾, **Christian Enkrich**²⁾, **Martin Wegener**^{1,2)}

invited oral

1) *Forschungszentrum Karlsruhe GmbH, Institut für Nanotechnologie (FZK), Postfach 3640, Karlsruhe 76021, Germany*

2) *Universität Karlsruhe (TH), Institut für Angewandte Physik, Wolfgang-Gaede-Str. 1, Karlsruhe 76131, Germany*

3) *Universität Karlsruhe (TH), Institut für Theorie der Kondensierten Materie, Wolfgang-Gaede-Str. 1, Karlsruhe 76131, Germany*

Materials have special optical properties if the refractive index is modulated periodically in space on a wavelength scale. They are of great interest with respect to future photonic devices. The propagation properties of light in such materials are strongly depending on the details of the structure. This corresponds to the complex band structure of the respective dispersion relation.

Present investigations aim at a realization of materials with a so called complete photonic band gap. The fabrication of such structures is still a challenge, therefore different technological approaches are being studied. Corresponding model calculations are expected to predict and understand the optical properties.

Several ways to fabricate and to characterize Photonic Crystals are discussed. Holographic lithography and its crystallographic background is described in more detail. This includes the dependence of the lattice type and the basis element inside the Wigner-Seitz-cell on the configuration of the interfering laser beams. Design freedom and restrictions are outlined. Direct laser writing is discussed, too. The combination of both methods is expected to be particularly useful.

For best results a high refractive index contrast is needed calling for infiltration of the photoresist structures which serve as templates.

15:00 - 15:30

Nanomagnetism: Surfaces, Interfaces and Finite Size

Branko Stahl¹⁾

invited oral

1) *Technische Universität Darmstadt, Darmstadt, Germany*

At surfaces and interfaces the magnetic order and magnetization dynamics is modified due to the lack or change of nearest-neighbor coordination. As an example it will be shown, how in thin film or nanoparticle systems the surface or interfaces influence the superparamagnetic behavior and the electronic structure. For instance, in FePt nanoparticles surface anisotropy is dominating and related to the chemical state of the surface atoms. Furthermore, the electronic structure of the metallic core is strongly influenced by surface states. This leads to size dependent features. Besides the scientific interest in the properties of such technical nanostructures and materials there is a link to magnetoreception in biology, i.e. to highly sensitive magnetic nanostructures in homing pigeons.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duža aula)

Afternoon session - continued

Main Building, Small Hall (Mała aula)

15:50 - 17:20

Nano-scale surface interactions Studied by TEM-SPM**Rynno Lohmus¹⁾, Donats Erts²⁾, Ants Lohmus¹⁾, Hakan Olin³⁾, Ilmar Kink¹⁾, Justin D. Holmes⁴⁾**

15:50 - 16:20

1) *Institute of Physics, University of Tartu, Riia str. 142, Tartu 51014, Estonia*2) *Institute of Chemical Physics, Riga LV-1586, Latvia*3) *Physics and Engineering Physics, Chalmers University of Technology (CTH), Göteborg 41296, Sweden*4) *Department of Chemistry, Cork University Col, Ireland, Ireland*

invited oral

Scanning probe microscope (SPM) in combination with transmission electron microscope (TEM), so called TEM-SPM, was used for adhesion and force interaction studies of metallic and semiconducting nanowires and nanocontacts. TEM-SPM is an attractive combined tool for direct investigation of the structure, electronic properties, and interactions in atomic scale. In TEM-SPM the size and shape of the tip and sample, as well as the size of contact area and the type of interaction (elastic/plastic), are observed directly.

In short, our TEM-SPM consists of a piezo tube which is used for fine motion, and of a geared stepping motor for rough z-motion. For force measurements we used an AFM cantilever. The force was measured by direct measurement of the displacement of the AFM tip. The TEM-SPM was inserted into a Philips CM200 field emission gun TEM. TEM-SPM was used to investigate force interactions between two gold samples and mechanical properties and force interactions of Si and Ge nanowires.

When approaching gold coated AFM tip to sharp Au tip an anomalous high value of the jump-to-contact distance was found. We ascribe this to an enhanced surface diffusion of gold atoms towards the tip-sample gap due to the van der Waals forces, leading to an avalanche situation where the gap is quickly filled until the ordinary jump-to-contact distance. Neck formation was observed in the gap between two nanocontacts during the jump to contact. It showed up when the gap between two gold nanoasperities was decreased below 1 nm.

The contact radius at zero applied loads were measured and compared with adhesion theories. The results were in the Maugis transition region, between the limiting cases of the Derjaguin-Müller-Toporov (DMT) and the Johnson-Kendall-Roberts (JKR) models.

Nanowire of diameter less than 1 nm was created in the gap between two Au nanocontacts by lateral AFM tip movement. The rupture force of Au nanowire was 9 nm, which corresponds to about 1 nN per atom.

Semiconducting nanowires: properties and architectures**Donats Erts¹⁾, Boris Polyakov¹⁾, Edgars Saks¹⁾, Hakan Olin²⁾, Justin D. Holmes³⁾**

16:20 - 16:50

1) *Institute of Chemical Physics, Riga LV-1586, Latvia*2) *Chalmers University of Technology, Goeteborg S-412 96, Sweden*3) *Department of Chemistry, Cork University College, Cork, Ireland*

invited oral

One-dimensional quantum wires are expected to play important role in future electronic and optical devices. We have investigated semiconducting nanowires made by a novel supercritical fluid solution-phase technique [1]. Conductivity and force interactions in individual semiconductor nanowires were investigated by using an in-situ probing technique using a TEM-SPM [2,3], which is a combination of the scanning probe microscope (SPM) and the transmission electron microscope (TEM). Free standing Si nanowires were high resistive with exhibited metallic behaviour. Nonconductive gap was observed in Ge nanowires. The electromechanical properties of these wires were characterised and a prototype nanorelay was demonstrated.

Uniform mesoporous thin films, with pores orientated almost perpendicular to the substrate surface are used as templates for unidirectional semiconducting nanowire growth. Structure of films and polycrystalline material containing Si and Ge nanowire arrays was investigated [4]. Luminescence of mesoporous nanowires was determined, as function of nanowire size. The optical properties can be controlled by the effective change of pore size inside mesoporous silica.

Quantum-chemical simulation of the silicon nanowires is carried out by semi-empirical CLUSTER code.

1. J.D. Holmes, K.P. Johnston, R.C. Doty, B.A. Korgel, *Science*, 287, 1471 (2000).2. D. Erts, H. Olin, L. Ryen, E. Olsson, A. Thölen, *Phys. Rev. B*, 61, 12725 (2000).3. D. Erts, A. Lohmus, R. Lohmus, H. Olin, A.V. Pokropivny, L. Ryen, K. Svensson, *Appl. Surf. Sci.* 188, 460 (2002).4. K. M. Ryan, D. Erts, H. Olin, M. A. Morris, J. D. Holmes. *J. Amer. Chem. Soc.*, 125, 6284 (2003).**Science of Functionalized Nanoparticles****Horst Hahn^{1,2)}**

16:50 - 17:20

1) *Technische Universität Darmstadt, Institute of Materials Science, Petersenstr. 23, Darmstad 64287, Germany*

invited oral

2) *SusTech GmbH & Co. KG Darmstadt, Petersenstr. 20, Darmstadt 64287, Germany*

The behavior of materials exhibiting structural components in the nanometer regime has been found to differ from that of conventional materials with structures in the micrometer range or larger. In many cases, such as the plasticity of metals and the electronic properties of materials, new mechanisms have been identified which do not occur in larger dimensions. The functionalization of nanostructures results in improved dispersibility in liquid and solid media. As a consequence of organic functional layers, dispersions of nanoparticles in aqueous and organic solvents can be stabilized for extended periods of time. This is only possible as the agglomeration is prevented almost completely due to the surface modification. Similarly, the organic shells around magnetic nanoparticles can be used to deposit ordered two- and three-dimensional arrays on surfaces. Additionally, three-dimensional packing of magnetic particles with excellent separation can be achieved and superparamagnetic properties can be established. Functionalization of inorganic nanomaterials occurs in natural systems as well. An example is found in the magnetic orientation system of the beak of the homing pigeon. The bio-mimicking of these functional bio-nanomaterials offers new opportunities for the basic understanding of the orientation system and for technological applications.

The large potential given by the opportunities of designing materials with desired properties and property combinations by using nanotechnological approaches has resulted in a worldwide industrial interest.

19:00 - 21:00

Poster session

Main Building, Main Hall (Duża aula)

19:00 - poster F-1 Luminescence properties of a multi-component glass co-implanted with Si and Er

Francesco Enrichi¹⁾, Giovanni Mattei¹⁾, Cinzia Sada¹⁾, Elisabetta Borsella^{1,5)}, Domenico Pacifici²⁾, Giorgia Franzo²⁾, Francesco Priolo²⁾, Fabio Iacona³⁾, Michel Prassas⁴⁾

1) *INFM - Università di Padova, via Marzolo, 8, Padova 35131, Italy*

2) *INFM - Università di Catania, via S. Sofia, 64, Catania 95123, Italy*

3) *CNR - IMM, Stradale Primosole, 50, Catania 95121, Italy*

4) *Corning SA, 7 bis Avenue de Vilvins, Avon 77211, France*

5) *ENEA, via E. Fermi, 45, Frascati - ROMA 00044, Italy*

Incorporation of Si-nc in Er doped silica is known to strongly enhance the infrared luminescence of Er³⁺ at 1.54μm. The enhancement is believed to be due to an energy transfer process from Si-nc to Er. In this work we investigate the formation of Si nano-aggregates and their role in the energy transfer process to Er³⁺ ions for a multi-component glass host. These materials can offer better performances than silica in terms of Er solubility and band broadness for integrated Er-doped optical amplifiers and their study is therefore very interesting for optoelectronic applications. The precipitation of Si and the enhancement of the 1.54μm Er emission were studied for different post-implantation annealing temperatures. In particular the optical properties of the glass were investigated by means of photoluminescence (PL) spectroscopy and PL decay dynamics and the results were compared with those of a reference silica substrate treated at the best performing annealing conditions for that material in terms of enhancement of the Er emission. These data are presented and related to the structural properties of the material. Moreover the implications of these results on the future development of an Er doped optical amplifier will be discussed.

19:00 - poster F-2 Structure and electric properties of nanocrystalline CdS-CdSe films sputtered on substrates with silver sublayer

Alexander K. Fedotov²⁾, Anis Saad³⁾, Alexander V. Mazanik²⁾, Oleg Stukalov¹⁾, Alexander V. Petrov⁴⁾, Ditmar Fink⁴⁾, Tadeush Figielski⁵⁾

1) *Institute of Solid State and Semiconductor Physics of, 17, P. Brovka Street, Minsk 220 072, Belarus*

2) *Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus*

3) *2Al-Balqa Applied University, Salt, Jordan*

4) *Hahn-Metner-Institut, Berlin D-14109, Germany*

5) *Institute of Physics PAN, al. Lotnikow 32/46, Warsaw 02-668, Poland*

The main goal of this work was to investigate the possibility for manufacturing of nanocrystalline Cd_xSe_{1-x} films to improve their homogeneity and the properties reproducibility. In these experiments the n-type 'CdS_{0.2}Se_{0.8}' films of 0.50-0.65 μm

19:00 - poster Nanocrystals of CdTe and CdMnTe: Growth, Optical and Magneto-optical Properties

Andriy Savchuk¹⁾, Oleg Panchuk¹⁾, Petro Feichuk¹⁾, Maksym Kovalenko¹⁾, Igor Stolyarchuk¹⁾, Volodymyr Fediv¹⁾

F-3

1) Chernivtsi National University, Chernivtsi, Ukraine

The II-IV based semimagnetic semiconductors and related nanostructures belong to an attractive materials owing to their unique properties and because of new possibilities of application in spintronic devices. We report on growth, optical and magnetooptical characterization of CdTe and CdMnTe nanocrystals embedded in different dielectric matrices. For their growth different physical and chemical techniques have been used: i) growth by melting of the semiconductor-doped fine powder borosilicate glass with the post heat treatment; ii) embedding of semiconductor nanocrystals into polymer matrix; iii) growth of nanocrystals embedded in glass matrix by pulsed laser deposition technique; iv) growth of nanocrystals by rf magnetron sputtering; v) fabrication of nanoparticles by the colloidal chemistry technique. Comparative structural and optical study of the grown CdTe and CdMnTe nanocrystals demonstrates advantages and drawbacks for the used methods.

Results of magneto-optical experiments on CdMnTe nanocrystals suggest of coexistence both the quantum size effect and the exchange interaction of the band electrons with the magnetic ions.

Possible application of semimagnetic semiconductor nanocrystals in development of spin filter devices is discussed. This work was supported in part by INTAS grant No.01-0354.

Formation and optical properties of CuInSe₂ and CuInTe₂ nanocrystals in silicate glass

19:00 -

Valerij S. GURIN²⁾, Ivan V. Bodnar¹⁾, Alexandra P. Molochko¹⁾, Nina P. Solovei¹⁾

poster

*1) Belarusian State University of Informatics and Radioelectronics (BSUIR), P. Brovka str. 6, Minsk 220000, Belarus**2) Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus*

F-4

A creation of nanoscale semiconductors with the proper control of properties is of great significance in nanomaterials technology. The nature of semiconductors determines basically optical features, and the next factors are: size and crystalline structure of nanoparticles, matrix composition, interaction of the particles with environment, etc. The synthesis of silicate glasses with ternary semiconductor nanoparticles CuInSe₂ and CuInTe₂ has been developed by us recently, and in the present work, we consider studies of post-treatment of these glasses that reveals noticeable effect upon optical properties.

The bulk crystalline precursors CuInSe₂ and CuInTe₂ were produced by means of the single-temperature method with composition control by chemical analysis and XRD and doped to the glass system SiO₂-CaO-R₂O (R = Na, K, Li) under synthesis by the high-temperature melting in reducing conditions. The post-treatment was performed by heating under 500-600°C during various time intervals. TEM evidenced spherical nanoparticles those were initially in the glass remain concentration with slight growth. Optical absorption (studied in the visible and near IR ranges) of the glasses varies significantly only at 600°C treatment (that temperature is close to the beginning of matrix softening). Transmittance of the glasses becomes lower, and the excitonic maximum (appearing in the range 550-600 nm) grows in the case of CuInSe₂, while in CuInTe₂ nanoparticles it only red shifted (0.05 eV).

The studies showed that in our preparation procedure nucleation and growth of CuInSe₂ and CuInTe₂ nanocrystals in silicate glass occur at the step of primary melting. The method is useful for development of new optical materials for optoelectronic and non-linear optical applications.

Formation of Luminescent Rare Earth Fluoride Nanoparticles in Silicate Matrices

19:00 -

Heike Kaetker¹⁾, Ulrich H. Kynast¹⁾, Marina M. Lezhnina^{1,2)}

poster

*1) University of Applied Sciences Muenster, FB1, Applied Materials Sciences (FHMS), Stegerwaldstrasse 39, Steinfurt 48565, Germany**2) Mari State Technical University, Institute of Physics (MSTUYO), Lenin-pl. 3, Yoshkar-Ola 424 000, Russian Federation*

F-5

Fluoride coordination provides a beneficial, sometimes crucial chemical environment to optical phenomena generated from rare earth ions. Examples may be found in several luminescence effects. Due to high ionicity and correspondingly low phonon frequencies, access to efficiency can be gained in the NIR emission range and eventually NIR to VIS upconversion. Furthermore, especially in materials based on Eu³⁺, low lying charge transfer states can be suppressed, as required for e.g. two photon emission (downconversion, quantum cutters).

Hosted by nanoporous silicate matrices, initially, parent fluoride complexes were prepared in the voids of the matrix and subsequently converted into encapsulated nanoscaled fluoride and oxifluoride particles by thermal decomposition. In the resulting host-guest hybrid materials the porous structure of the matrices can be maintained, such that surface coordination effects of the nanoparticles to the matrix and subsequently penetrating water or other volatile chemicals can in suitable rare earth systems be monitored using luminescence spectroscopy, despite encapsulation. TEM measurements, absorption spectra ranging from VUV to NIR, accompanying luminescent properties and stoichiometric parameters of both, zeolite based materials and sol-gel derived silicas as hosts are elucidated.

19:00 - **Circular polarization photoluminescence of the defects in SiO₂**
 poster **Natalia N. Vandysheva¹⁾, Evgeny N. Vandyshev¹⁾, Konstantin S. Zhuravlev¹⁾, Vladimir S. Aliev¹⁾**

1) Institute of semiconductor physics (ISP), Lavrentiev, Novosibirsk, Russian Federation

F-6

Recently, the interest to investigations of the systems that are capable to change a spin condition of charge carriers has been increased. These systems have been used as media for quantum computers. In the present paper a circular polarization photoluminescence of the defects in SiO₂ has been investigated. A circular polarization degree is a sensitive indicator of a spin state of the charge carriers. Grown at the room temperature 0.5 mm SiO₂ was irradiated with electrons. The dose of irradiation was 0.3 K/cm². An Ar⁺ laser ($\lambda = 488$ nm) with a power of 14 mW was used for photoluminescence excitation. The circular polarization photoluminescence spectra were recorded using quarter-wave plate and Glan prism. The measurements were carried out at the room temperature. In the photoluminescence spectrum of the investigated samples a wide band with a maximum at ~ 650 nm was observed. The origin of this band was associated with a non-bridging oxygen atom (-Si-O \cdot). A circular polarization degree was calculated as $P=(I_+ - I_-)/(I_+ + I_-)$, where I₊ and I₋ are the intensities of a clockwise polarized and a counterclockwise polarized photoluminescence, correspondingly. The circular polarization degree equals to 3.8%. We also have investigated the circular polarization photoluminescence of the silicon nanocrystals in SiO₂. But these measurements have not given a beneficial effect. The spin relaxation time was calculated and equaled to 2.24 microseconds. The mechanism of this effect is discussed.

The work was supported in part by the Russian Foundation for Basic Research (grant no.02-02-17719).

19:00 - **Zinc oxide nanopowders obtained by the microwave-hydrothermal route**
 poster **Tomasz STRACHOWSKI²⁾, Ewa Grzanka^{1,2)}, Adam Presz²⁾, Edward Reszke⁴⁾, Ludomir SLUSARSKI³⁾, Witold Lojkowski²⁾**

1) University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland

2) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

3) Technical University of Łódź, Institute of Polymers, Stefanowskiego 12/16, Łódź 90-924, Poland

4) Ertec Poland Edward Reszke (Ertec), Rogowska, Wrocław 54440, Poland

F-7

Zinc oxide nanopowders have been successfully prepared via microwave hydrothermal process. The reaction were carried out in hydrothermal conditions under a pressure of up to 4 MPa using a microwave reactor. The application of microwave energy for the processing of ceramics has become an attractive area of research and innovation recently. Zinc oxide was prepared by the decomposition of different zinc saltz in an aqueous solution under alkaline conditions or by the addition of TEA (triethanolamine) and urea. The effects of reaction temperature, synthesis time and synthesis conditions on the particulate properties such as the particles morphology and grain size are discussed. Zinc oxide particles of various shapes and sizes were produced.

Keywords : Nanopowders, Zinc Oxide, Hydrothermal method, Chemical synthesis.

19:00 - **Microwave driven hydrothermal synthesis of Pr-doped zirconia nanopowders**
 poster **Agnieszka Opalińska³⁾, Ewa Grzanka^{1,3)}, Adam Presz³⁾, Cristina Leonelli²⁾, Witold Lojkowski³⁾**

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3) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

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Zirconia is an important ceramic material with useful mechanical, thermal, optical and electrical properties.

Nanocrystalline zirconia and Pr-doped zirconia powders were synthesized via a hydrothermal method using a microwave reactor under pressure up to 5,5 MPa and temperatures up to 250 C. Oxides nanopowders with particle size in access of 5 to 100nm and Pr content (0 to 20mol%) were obtained under different experimental conditions such as pH solution, temperature, pressure and reaction time. Power properties including crystalline phase, crystalline size, particle size distribution, degree of agglomeration and luminescence properties were investigated.

Aim of this work is to obtain nanocrystalline powders doped with rare earth element for optoelectronic applications. Microwave hydrothermal synthesis is versatile technology to obtain doped zirconia nanopowders of high purity.

19:00 - **Microwave driven hydrothermal synthesis of iron oxide - the effect of process parameter on the properties of the nanopowders**

poster **Robert Fedyk¹⁾, Martin Arnault, Witold Lojkowski¹⁾**

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

F-9

In recent years a lot of attention has been given to the synthesis of magnetic iron oxide nanoparticles due to their numerous applications. For example they are of great interest for information storage, jet printing, pigments and in medicine and biology. Each application requires particular properties from iron oxide particles which depend on their shape and grain size. Therefore it is important to develop various synthesis routes of magnetic nanocrystalline particles.

In our work we used the microwave driven hydrothermal synthesis MDHS because both microwaves and pressure allow synthesis of iron oxide nanoparticles along kinetic pathways different from conventional methods. The most common methods of synthesis of iron oxide particles are solid state transformation and precipitation from iron salts. Aim of this work is to check influence of several parameters namely the power, pH, concentration of iron salts (ferric chloride and ferrous sulfate), citrate ions and addition of cobalt on the size distribution and magnetic properties of particles obtained by MDHS.

Powder precursors for nanoceramics: cleaning and compaction

Grzegorz Kalisz^{2,3)}, Anna Swiderska-Sroda²⁾, Stanisław Gierlotka²⁾, Ewa Grzanka^{1,2)}, Bogdan F. Palosz²⁾, Svetlana Stelmakh²⁾

1) *University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland*

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3) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

19:00 -
poster

F-10

Purity and pre-compaction of powder precursors are the key factors determining the quality of nanoceramics. We investigated desorption and re-sorption of gaseous impurities on nanocrystalline powders of diamond and silicon carbide using thermo-gravimetry and mass spectroscopy techniques. Compaction was investigated by means of SAXS and gas porosimetry under pressures up to 7.7GPa. The amounts of gases adsorbed by nanocrystalline powders stored in air correspond to one monolayer of molecules. In a flow of purging gas (Ar, He) weakly adsorbed molecules (mainly H₂O) desorb at temperatures below 200°C. Desorption of relatively strongly bound molecules (O₂, CO, CO₂, N₂) requires temperatures up to 500°C. Above 500°C oxidation of the surface by residual oxygen from the purging gas takes place. Freshly desorbed samples exposed to air for 0.5 h not show any considerable contamination. Those results provide a guidance for conditions of cleaning the nanopowders before sintering. Effective densification of nanopowders requires application of extreme pressures. At 2.5GPa the relative density of both diamond and SiC compacts is only about 0.5. At 7.7GPa the densities reach 0.66 for SiC and 0.75 for diamond. Extreme pressures close pores much more efficiently in diamond than in SiC. This difference is due to a difference in the morphology of the two materials. Diamond nanopowders, produced by detonation technique, undergo an intense chemical treatment what causes a considerable agglomeration. Silicon carbide powders (synthesized by gaseous methods) exhibit a pronounced fractal structure. SAXS data show that during compaction this structure is being preserved and the density of the compact remains low until the pressure reaches approx. 1GPa. Above that pressure the voids in the compact form 2-dimensional aggregates. As the pressure increases those aggregates gradually dissociate into one-dimensional chains of voids and, finally (at approx. 6GPa) form isolated voids.

Studies on preparation and characterisation of the carbon nanoforms

Ryszard J. Kalenczuk¹⁾, Ewa Borowiak - Paleń¹⁾

1) *Szczecin University of Technology, Centre of Knowledge Based Nanomaterials and Technologies (Know-MatTech), Pulaskiego 10, Szczecin 70-322, Poland*

Since the discovery of carbon nanotubes by Iijima carbon nanomaterials have been pronounced as the most perspective material family for nanoelectronic applications. For future using the methods of production as well as for characterisation of such materials should be prepared. The catalytic methods used by us for preparation of different carbon nanoforms have been presented. The nanomaterial composed of the graphene structure and it's changes to the other structures has been studied.

During this study the new boron nitride nanocarbon multitubes for various application has been performed. Such prepared material has been found as a very different comparing with hexagonal carbon boron nitride known in macroscale.

In the next step the procedure for preparation of the carbon boron nanotubes has been performed. The fairly high concentration of born has been measured. Introducing of boron guest atoms to the structure of single wall nanotubes leads to the formation of the new metallic like nanostructure with the possible designing of electronic properties.

Starting from the carbon source the possibility of the production of the carbon nanomaterials with the extremely high structural stability with semiconducting as well as with metallic properties has been shown. Set of characterization techniques were proposed.

19:00 -
poster

F-11

Fabrication of Lu-based nanocrystalline phosphors

Eugeniusz Zych¹⁾, Joanna Trojan-Piegza¹⁾

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19:00 -
poster

F-12

A few synthesis techniques of Lu-based nanocrystalline phosphors will be presented and the structural variations between the products will be discussed. Preparation of lutetium oxide activated with various lanthanides using combustion synthesis will be presented and the influence of the organic fuel on the products characteristics will be shown. Homogeneous precipitation of RE-doped Lu hydroxide and its subsequent decomposition to oxide will be discussed together with some properties of the final products. Influence of surfactants addition on the route of the precipitation will be shown. Some basic information on the sintering properties of the various powders will be shown.

Depending on the synthesis technique the materials consists of particles whose sizes vary between 5-200 nm. The spectroscopic properties vary to some extent with the particles sizes. The most profound influence on the products characteristics is observed when surfactants are introduced to the solution where precipitation occurs. The surfactant may even alter the final product composition.

19:00 -
poster
F-13

The influence of colloidal silica structure on the ceramic moulds properties

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2) *Foundry Research Institute (FRI), 73 Zakopianska Street, Cracow 30-418, Poland*

Viscosity of nanosized colloidal silica suspensions, used as binders in the investment casting, was determined as a function of their weight fraction reaching 50%. A new capillary viscometer was used whose construction eliminated the suspension sedimentation effects. The influence of suspension ionic strength at fixed pH equal 9,5 was systematically studied in order to assess the magnitude of electroviscous effects. It was found that for the low silica concentration range suspension viscosity increased much more rapidly than the prediction of Einstein's or Betchelor's theories. This discrepancy was accounted for by postulating a loose, gel-like structure of colloid silicas used in experiments. Hence, the apparent hydrodynamic radius of silica clusters was considerably larger than the primary particle size in accordance with direct microscope observations of the structure of silica sols. Based on this postulate an apparent density of the silica sols was found to be 1,2 g/cm³ instead of 2,3 g/cm³ as determined from the suspension dilution method. Also for higher concentration range experimentally determined viscosity increased more rapidly with increased sol concentration than predicted by the Dougherty-Krieger model derived for hard particles. The deviation was attributed to the secondary electroviscous effect stemming from the electrostatic interactions among silica particles in sheared suspensions. This effect has quantitatively been interpreted in terms of the Russel's theory. On the other hand, for the very high concentration reaching 50% the experimental results were accounted for by using the effective hard particle concept. Exploiting our experimental findings a sensitive method of determining the structure and apparent density of silica sols in aqueous media was proposed. Simultaneously with the investigations mentioned above, the mechanical properties of thin layers of ceramic containing colloidal silica suspension were investigated.

19:00 -
poster
F-14

Influence of the physicochemical factors on the nanostructure of colloidal silicate arrangement

Andrzej B. Baliński

The methods of measurement of the size of colloidal nanostructure elements in hydrated sodium silicate and of the decay of these sizes were discussed in the aspect of the used method of Photon Correlation Spectroscopy. Hydrated sodium silicate possessing different physical and chemical proprieties, obtained by different physicochemical methods, was investigated. The bases of the estimation of nanostructure in hydrated sodium silicate by application of the counted coefficients were described.

The obtained results of investigations enabled qualification of the influence of physicochemical factors on the nanostructure in the examined materials, defined by the size of small colloidal particles and characterization of the decay of this size in nanoparticle clusters.

The performed investigations of the cohesion-adhesion values of bonds in the system of "sand grain - hydrated sodium silicate - ester" show that it is advisable to modify the nanostructure in hydrated sodium silicate in the aspect of improvement of the binding proprieties of this material.

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19:00 -
poster
F-15

Growth and investigation of oxide heterostructures containing half-metallic Fe₃O₄

Bonifacas Vengalis¹⁾, Lisauskas Vaclovas¹⁾, Kristina Sliuziene¹⁾, Vytautas Petrauskas¹⁾, Se-

bastiaan Van Dijken²⁾, Mazhar Bari²⁾, M Venkatesan²⁾, Michael Coey²⁾1) *Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania*2) *Dept. Physics, Trinity College, College Green, Dublin 2, Ireland*

Magnetite, 'Fe₃O₄', with an inverse cubic spinel structure (a=0.8396 nm) is known as a ferrimagnetic oxide exhibiting electrical conductivity due to hopping of spin-polarized electrons between ferrimagnetically ordered Fe²⁺ and Fe³⁺ states. It is promising for novel applications based on spin-polarized transport due mainly to high Curie temperature ('T_c' = 858 K) which is an important advantage compared to other half-metallic oxides such as 'La_{1-x}Sr_xMnO₃' (360 K), 'Sr₂FeMoO₆' (450 K), 'CrO₂' (395 K). For most of the applications, there is increasing need of fundamental and technological research of various hybrid device structures composed of high quality 'Fe₃O₄' layers, conducting underlayers and isolating barriers. In this work, thin films of 'Fe₃O₄' were grown in-situ at T=300-500°C on both lattice-matched MgO and epitaxial conductive ITO/YSZ(100), 'LaNiO₃'/'NdGaO₃'(100), 'La_{0.66}(Ca,Sr)_{0.34}O₃'/'NdGaO₃' underlayers by sputtering of metallic Fe target under a fixed Ar:'O₂' (30:1) gas mixture pressure of about 5 Pa. Reflected high energy electron diffraction (RHEED) and XRD investigations revealed epitaxial quality of the magnetite films grown on MgO and ITO/YSZ meanwhile the films prepared on perovskite underlayers were highly textured. We were focussing on in- and out-of-plane resistivity, resistance anomaly at the Verwey transition point ('T_V' = 120 K), magnetoresistance, magnetization and current versus voltage to elucidate the effect of substrate and deposition conditions on major parameters of the grown 'Fe₃O₄' films and to investigate interface properties of the 'Fe₃O₄'/'In₂O₃<Sn>', 'Fe₃O₄'/'LaNiO₃' and 'Fe₃O₄'/'La_{0.66}(Ca,Sr)_{0.33}O₃' heterostructures. Current flow in the bilayer structures was modeled to investigate stability of the interfaces and to evaluate diffusion of oxygen in different layers.

Ceramic composites of the Al₂O₃-ZrO₂ type obtained by the Direct Coagulation Casting Method**Anita Tomaszewska-Grzęda¹⁾, Mikolaj Szafran¹⁾**1) *Faculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, Warsaw 00-664, Poland*

The Direct Coagulation Casting (DCC) is a new and very interesting method of molding samples from ceramic casting slips of high concentration of the solid phase. It consists in concentrating the casting slip via internal slow change of pH round each ceramic powder particle, which causes changes in the electrostatic repulsion forces of double electric layers in favor of the Van der Waals attraction forces.

The role of enzymes in the DCC process consists in the decomposition of an appropriately selected substance which results in slow liberation over the whole volume of molecules changing the pH or also in the synthesis of salts modifying the double electric layer.

The 15 nm grain size nanopowder of zirconia used in the study was purchased from "CEREL" Boguchwała as was the "gel", the hydrated mixture of zirconia salts, which produces nZrO₂ upon calcination. The paper discusses the differences between the properties of Al₂O₃ grains modified with the additions of: a) zirconia nanopowder, b) the "gel", and c) zirconia powder of grain size comparable to that of Al₂O₃. The addition of zirconia is intended to increase the fracture toughness of alumina. It is assumed that the addition of the nanopowder of zirconia, due to its small particle size, may form nanolayers around the grains of Al₂O₃ or fill the spaces between them.

The results of using the urease-urea system and the properties of ceramic casting slips, green samples and after sintering with aluminium oxide, Al₂O₃-ZrO₂, and Al₂O₃-nZrO₂ composites are presented in the paper.

The obtained results of studies show a considerable probability of obtaining on the future Al₂O₃-ZrO₂ and Al₂O₃-nZrO₂ composites.

19:00 -

poster

F-16

Correlation between high-pressure ZrO₂ electrical properties and crystallite size**Anna N. Trefilova¹⁾, Ilia V. Korionov¹⁾, Alexey N. Babushkin¹⁾, Witold Lojowski²⁾, Agnieszka Opalińska²⁾**1) *Department of Physics, Ural State University, Lenin Av., 51, Ekaterinburg 620083, Russian Federation*2) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

We studied correlation between the sizes of crystallite and resistance ZrO₂ 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 Y₂O₃ 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.

1. At a pressures of about 35 GPa the ZrO₂ resistance decreases by 3-4 orders of magnitude. The temperature dependence of the resistance of the nanocrystalline samples at all pressure has activation nature ($R=R_0 \exp(E_a/kT)$).

2. The temperature dependence of the bulk material resistance (crystallite size near 500 nm) has metallike type (with positive temperature coefficient) at pressure 47 GPa and temperature 320 K.

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poster

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3. For all samples at pressures 40-43 GPa take place maximums of activation energy and local maximums of resistance. It is possible to suspect existence of structure-phase transition in ZrO₂ at pressure about 42 GPa.

4. The activation energy E_a and parameter R_0 (bound with parameters of charge carriers) depends from crystallite size. Activation energy is between 0.1-1 eV (size 10 nm) and 0.001-0.01 eV (size 56 nm). Whereas the value of activation energy for bulk ZrO₂ is approximately 0.01 eV. It is possible to suspect, that the surface effects essentially change a ZrO₂ conductivity mechanism at high pressures.

This work was supported by CRDF grant REC-005 for Ural Center of Research and Education "Advanced materials" and RFBR grant 01-03-96494.

19:00 -

poster

F-18

Phase transitions in multicomponent chalcogenides of silver at high pressure by a method of impedance spectroscopy

Olga L. Kobeleva¹⁾, Alexey N. Babushkin¹⁾, Sergey N. Shkerin²⁾, Natalia R. Tatarinovich¹⁾, Yulia A. Lis'ih¹⁾

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2) Institute of High Electrochemistry, S.Kovalevskaya, 20, Ekaterinburg 620219, Russian Federation

Last years in Laboratory of Physics of extreme influences on substance of USU the various multicomponents chalcogenides of silver and copper were synthesised and the researches. The large interest the represents are research of properties of these compounds at high and super-high pressure. In the present work were investigated chalcogenides of silver AgPbAsS₃, AgSnAsS₃ and AgSnSbS₃ at pressure 15GPa-50GPa by a method of impedance spectroscopy. These compounds are mixed electronic-ionic conductors, with low temperatures of a appearance the ionic current. Their research at high pressure represents the large interest and it is necessary for specification of areas of their possible application. For generation of pressure up to 50GPa used the carbonado-diamond anvil cell.

Were received the godographs of an impedance of compounds at pressure 15GPa-50GPa and the dependences of conductivity on frequency are investigated. The hysteresis of conductivity is investigated at gradual the unloading a sample. The influence of borders an electrode / sample was analysed at various pressure. From the data of conductivity and tangent of a corner the dielectric losses the region of existence of phase transitions in samples are received. The influence of structure of a sample on its electrical properties at high pressure is analysed. At the analysis of results the contribution of an impedance of the anvil in a com-mon impedance was taken into account. For the description of results the circuit was offered, into which the elements describing various processes occurring in a sample (of resistance, capacity, element of a constant phase) were entered.

The researches are executed at partial financial support CRDF (grant No REC-005) and RFBR-URAL (grant No 01-03-96494).

19:00 -

poster

F-19

Relaxation processes in ZrO₂ at high pressures

Ilia V. Korionov¹⁾, Anna N. Trefilova¹⁾, Alexey N. Babushkin¹⁾, Witold Lojkowski²⁾, Agnieszka Opalińska²⁾

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2) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

It is well known that materials electrical properties changed in time after pressure treatment.

We studied relaxation processes in ZrO₂ at the pressures in range 22 - 50 GPa. 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 Y₂O₃ 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. We found times dependence of resistance at different pressure for all samples.

The analysis of experimental data has shown that the time function of electric resistance most precisely described by exponential function for the sample with crystallite size 10 nm and for polycrystal sample up to pressure 44-45.5 GPa. After pressure treatment higher than 45.5 GPa the character of the relaxation changed and resistance increases with increasing time. Relaxation processes in ZrO₂ with 56 nm crystallite size does not take place.

It can be seen, that relaxation times depend on pressure and crystallite size.

19:00 -

poster

F-20

Transport phenomena in ammonium halides under high pressures

Galina V. Tikhomirova¹⁾, Alexey N. Babushkin¹⁾

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The conductivity of polycrystalline ammonium chloride (NH₄Cl), fluoride (NH₄F), and bromide (NH₄Br) under high uniaxial pressures from 15 to 50 GPa in the temperature range 77 to 400 K was studied.

For all three materials a sharp decrease in resistivity by orders of magnitude from the value more than 10⁸ Ohm to kOhms was observed at some threshold pressure P_c being about 42, 27 and 15 GPa for NH₄F, NH₄Cl and NH₄Br, respectively. Similar sharp transition was observed near P_c in the temperature dependence of resistivity. P_c values correspond to a stationary state, which was reached after sufficiently long exposure of samples under stress. The time of the pressure treatment necessary to stabilize this jump in resistance is quite different for these three materials. Initial loading of the samples by the pressure of 50 GPa causes the change in the resistance only after the exposure under stress during a month for NH₄F, about 10 days for NH₄Cl and seconds for NH₄Br. There exist the correlation between the times of such a treatment, as well as values of P_c, and the density of the materials (the atomic weight of halogens F, Cl and Br). Relaxation times of conductivity are different at increasing and decreasing pressure. The relaxation time is essentially larger near P_c.

At the pressures and temperatures above this transition points, a hysteresis in the resistance of all three materials in dependence on temperature was observed, indicating the existence of intermediate states. The magnitude of the hysteresis decreases with the number of cycles of applying and removing pressure. All ammonium halides show metal-like behaviour under high pressures similar to that of alkali halides.

This work was supported by CRDF grant REC-005 for Ural Center of Research and Education "Advanced materials" and RFBR grant 01-03-96494.

Effect of the DAC treatment on the nanomaterials of type Si-O

Natalya B. Efros²⁾, Boris M. Efros²⁾, Natalya V. Shishkova²⁾, Andrzej Misiuk¹⁾

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poster
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Extensive experiments studies of the - IV elements have been made in recent years. Motivations have included the rich variety of phase and structural transitions.

Different SiO_{2-x} defects can be created in Czochralski grown silicon, Cz - Si, by appropriate pre-annealing at atmospheric pressure (10⁵ Pa). Some data concerning the effect of enhanced hydrostatic pressure on creation of defects in the nanomaterial Si - SiO_{2-x} system have been reported for defects - containing Cz - Si subjected to the cyclic hydrostatic pressure treatment.

The increase of defect concentration in the hydrostatic pressure - treated Cz- Si nanomaterial samples with initially present SiO_{2-x} precipitates can be considered as a proof of hydrostatic pressure - induced massive creation of defects on before - created oxygen - related defects. However, in the case of some DAC - treated samples, a misfit dislocation network was not directly proved to be created because of too small sample dimension in comparison to the resolution of the spectroscopy and X-ray methods.

The study of these nanomaterial samples was carried out by spectroscopical methods under pressure too. In particular, it was revealed that the sample with high defect density indicated the transparency ~ 55-70% in the wavelength range 800-1100 nm with some maximum at 930 nm at the initial state the effect possibly connected with the sample non-homogeneity. At application of the high pressure (~5 GPa) the value of transparency decreases up to ~10% in the wavelength range of 800-925 nm and later on transparency value increases sharply to ~100% at 1100 nm wavelength. Further pressure rise (up to ~10 GPa) resulted in an increase of the transparency value up to 20% at 800-900 nm range and later on it increases up to 55% at 1100 nm wavelength. Thus with the pressure rise, the absorption edge of forbidden zone moved to higher wavelengths.

Electrical properties in C₃N₄ under high pressure

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19:00 -
poster
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During a recent years a great attention was given to the problem of an investigation of phase transitions in C₃N₄ as by M.L.Cohen and co-authors were theoretical predicted the diamond-like properties of beta-C₃N₄. We investigated the phase transitions in the two samples of this material, which were prepared by different methods (chemical (1) and shock (2)), in the carbonado-diamond anvil cell with using the methods of electrical measurement on d.c. and impedance spectroscopy. The first sample were investigated at pressure 15GPa-43GPa, a second sample at pressure less 33GPa. A dependences of the electrical resistances R of the samples at the T=300K, in the 10³Hz<f<8*10⁵ Hz were made. A hodographs of impedance and a tangency of an angle of dielectric loss dependencies were got. For the sample No.1. impedance measurement give a little hysteresis and a weak indication on the existence of phase transitions at 15GPa-25GPa and 33GPa-35GPa. After unloading the sample on 2-4-cycles input-output, the

R is sharply increased more than 10^4 times and large hysteresis is appeared (d.c.). After holding under pressure 43GPa during 6 days hysteresis has vanished. After fast increasing of pressure till 43GPa (2 minutes) some reaction has taken place and after unloading was got a graphite dust and transparent small crystallite of unknown nature. The preliminary results obtained shows the new phase appearing from C3N4 under pressure till 43GPa at room temperature and phase transitions under 15-25GPa and 33-35GPa possible with changing composition. For the sample No.2. the results are differ from results for sample No.1. The resistance is decreasing with increase the pressure.

So the method of preparing samples and their purity is essential for behavior their electrical conductivity.

This work in part has been supported by RFBR (grant No.01-03-96494,02-03-32699), CRDF (grant No.REC-005).

19:00 -
poster
F-23

Automated setup for ultrahigh pressure treatment of materials

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Vladimir I. Osotov¹⁾**

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High pressure treatment by various type of apparatuses is known to change the microstructure of substances in different ways. Thus, for example, the hydrostatic contraction above phase transition point tends to shredding of some binary compounds into nano-particles. Thermal, mechanical, etc. properties of such inhomogeneous materials depend both on the intrinsic parameters of substances and also on the shape and concentration of particles contained. In present paper the original high pressure apparatuses, recording automated systems, and methods of materials treatment are discussed. The combined technique is able to provide both the various types of pressure treatment and allow to distinguish the physical and geometrical parameters of substances under pressure.

The apparatuses include: the two-layered chambers of piston-cylinder type for hydrostatic pressure up to 2.3 GPa, the solid-media pressure chambers of Bridgman anvils type made from steel, tungsten carbide, tungsten-free titanium hard alloys and synthetic diamond (boron nitride) up to 5, 10 and 30 GPa, and nature diamond (sapphire) anvils. The parts of the apparatuses are made from non-magnetic corrosion-resistant titanium alloys. Stationary apparatuses allow to perform the contraction and to observe the variations of electrical, thermal and volumetric properties during phase transformations, while autonomous ones are used for thermal, electrical magnetic and also for optical, synchrotron and neutron measurements up to 10 GPa. The examples of application of the above technique for treatment and investigation of different classes of semiconductor micro-samples and the molecular crystals are given in the work.

The work was supported by the Russian Foundation for Basic Research, Gr. No. 01-02-17203 and US Civil Research and Development Foundation (US CRDF), TGP-599, TGP-656.

19:00 -
poster
F-24

The Sock-wave Model of the Effect of Superdeep Penetration of Powder Particles into Metallic Materials

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At the special conditions of loading a small part of the torrent of explosion accelerated powder (dispersed) particles can penetrate into metal barriers on depth, in 100 - 1000 time superior the size of particles. The phenomenon is known as effect of superdeep penetration (SDP). Following conditions are necessary for SDP: the speed of particles is not less than 300 m/s; the sizes of particles is less than 500 microns; the time of loading is more than 100 ms; the average density of torrent is more than 1 g/sm³.

New physical model of the effect of superdeep penetration (SDP) has been created. The model allows to explain the mechanism of SDP and to get mentioned above experimentally stated conditions of the phenomenon. The model is based on description of shock-wave interaction between the penetrating particles and target material.

Under the action of fluctuating field of stresses the mobile cavities in target material are appear. Such mobile cavities (transport capsules) carry the particles inside of target. The carried particle is passively moving in the capsule and does not spend energy for the deformation of a material.

The following criterion of a possibility of SDP is obtained:

$$(4\pi/3)(h/c\Delta t)^{1/3} > 1$$

where h is the height of a layer of powder particles in an explosive booster, Δt is the duration of loading, c is the velocity of sound.

According to this criterion, the penetration of particles can occur if $h/\Delta t > 5 \cdot 10^{-2}$ mm/ μ s, that corresponds to the torrent density $\rho > 1$ g/sm³.

It is established, that the value of characteristic time of process is proportional to diameter of particles. For the diameter of particles more than 500 microns this value exceeds the time value of a plastic relaxation of metals. In this case the target is deformed plastically; the transport capsules does not arise.

This work has been supported by RFBR (grant No. 03-03-33028).

Propagation and Interaction of Dodecahedral Converging Shock Waves in Steel Balls

19:00 -

Alexey E. Kheifets²⁾, Vitaliy I. Zel'dovich²⁾, Irina V. Khomskaya²⁾, Nataliya Y. Frolova²⁾, Boris V. Litvinov¹⁾, Nikolay P. Purygin¹⁾, Vladimir I. Buzanov¹⁾

poster

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F-25

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Physical metallurgy and computer simulation methods were used to study the propagation and interaction of shock waves in steel balls subjected to convergent dodecahedrally symmetric shock waves with the pressure 50-200 GPa. Conditions for the energy cumulation and realization of the regular and the irregular type of interaction of shock waves were studied. Based on the results of microstructural investigations of intact samples, parameters of the shock-wave loading were calculated such as the pressure, the residual temperature, the time of isentropic flow of the substance under load, and the pressure profile behind the shock-wave front.

The Mach (irregular) regime of the waves interaction was realized in the steel ball of diameter 40 mm [1]. Based on the obtained experimental data, the thermodynamic parameters of the shock-wave loading for this ball ($R_{sp}/R_{expl} = 1/2$, R_{sp} - the ball's radius, R_{expl} - the thickness of explosive layer on the ball's surface) were calculated. When simulating the shock-wave motion, we used the real geometry of the boundaries of contact discontinuity (Altschuller patterns).

The regular regime was realized in 60-mm steel balls ($R_{sp}/R_{expl} = 3/4$). A computer simulation of the shock-wave motion based on the results of a metallographic investigation of microstructural changes in the loaded material permitted us to reconstruct the picture of the shock-wave motion and describe the spatial distribution of pressures in the ball and to restore the pressure profile of the interacting shock waves [1].

This investigation was carried out with bounds of Presidium of the RAS program "Thermal physics and mechanics of intensive energy influence"

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Hugoniot of C₆₀ fullerite and sound velocity in shock-compressed fullerite

19:00 -

Vladimir V. Milyavskiy¹⁾, Andrey Z. Zhuk¹⁾, Aleksander V. Utkin¹⁾, Eugene B. Zaretsky²⁾

poster

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Hugoniot of C₆₀ fullerite and sound velocity in shock-compressed fullerite were experimentally studied at the pressures range from 0.3 to 48 GPa.

In our experiments we used polycrystalline C₆₀ samples with a density 1.64 g/cc. The samples were loaded by impact of metal plates accelerated by gas gun (projectile velocities up to 0.53 km/s) or by preliminary calibrated explosive projectile systems (projectile velocities up to 5.3 km/s). The continuous monitoring of rear surface velocity of the samples was performed using laser interferometer VISAR through PMMA (low-pressure region) or water (high-pressure region) windows. To determine velocities of impactors and (or) shock velocities, contact gages or piezoelectric gages were used.

The Hugoniot of fullerite has a set of peculiarities, which are connected with a series of polymorphic phase transitions. For C₆₀ fullerite a transformation to a dense carbon phase is observed with a transition onset pressure 15 GPa, the stability limit of the fullerite structure under single-step shock compression. The two-wave structure of the transition is overdriven to a single wave above 33 GPa.

Previous to this work, behavior of fullerite under shock-wave compression was experimentally studied only with the use of recovery assemblies of various configurations (see, for example, review [1] for details). Our experimental results give new information about shock-induced phase transitions of C₆₀ fullerite and may be used as a basis for construction of the wide-range equation of state.

We thank L.G. Khvostantsev and I.V. Ustinov (IHPP RAS) for preparation of the samples. The work was supported by Russian Academy of Sciences and RFBR.

- [1] V.V. Milyavskiy, A.Z. Zhuk, K.V. Khishchenko, Defect and Diffusion Forum 208-209 (2002) 161.

19:00 - **Molecular adsorption on GaAs studied by HREELS**
 poster **Ana Maria Botelho do Rego²⁾, Ana Maria Ferraria²⁾, Manuel Rei Vilar¹⁾, Jamila Elbeghdadi¹⁾, Ron Naaman³⁾**

F-27 *1) ITODYS (CNRS - Université Denis Diderot) (ITODYS), Place Jussieu, F-75005 Paris, France*
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GaAs is a promising material for building devices for chemical sensing. For this purpose, a molecular monolayer coating is used both to stabilize the surface towards the oxidation and to selectively recognize the analyte molecules or ions (the analyte). Therefore, the molecules for the coating need to be bifunctional organic molecules: one of the functionalities binds covalently to the surface of the device and the other one to the analyte. The study of the quality and the extension of the molecular adsorption to the surface is then crucial. In fact, the stability of the sensing surface depends on the quality of the molecular coverage. The number of adsorbed molecules is also important to assure a good sensitivity of the device to the analyte.

High-resolution electron energy loss (HREELS) is an analysis technique highly sensitive to the extreme surface (~ 1 nm) and to the molecular orientation. HREELS is here used to study the influence of solvent (acetonitrile) water content on the adsorption of phenylphosphonic acid on gallium arsenide. Both using the vibrational and the electronic energy loss range, it is shown that there is a poor molecular adsorption for water contents ranging from 0 to 4% in volume: HREELS spectrum is always a combination of the substrate and the adsorbed molecule spectra. For a water content of 5% in volume there is an abrupt jump in the HREELS spectra shape: they become typical of phenyl group in the electronic region. In the vibrational region, the typical C-H frequencies of aliphatic chains disappear showing that the extreme surface is exclusively covered by phenylgroups. Simultaneously, the arsenic oxide disappears and the gallium oxide grows suggesting that the adsorption sites are constituted by gallium oxide surface hydroxyl groups. Also for the samples where a large adsorption occurred, surfaces become negatively charged under electron irradiation attesting the existence of a large number of traps for the incident electrons.

19:00 - **Growth of InAs quantum dots on a low lattice-mismatched AlGaSb layer**
 poster **Naokatsu Yamamoto¹⁾, Kouichi Akahane¹⁾, Shinichiro Gozu¹⁾, Naoki Ohtani¹⁾**

F-28 *1) Communications Research Laboratory, Basic and Advanced Research Division (CRL), 4-2-1, Nukui-Kitamachi, Koganei, Tokyo 184-8795, Japan*

Quantum dots (QDs) have been investigated to realize high-performance opto-electronic devices. A use of a high lattice-mismatch is generally reported to fabricate self-assembling QDs, such as growth of InAs QDs on GaAs with its 7.2% lattice-mismatch. However, an emission wavelength from these InAs QDs appears around 1000nm, because an energy-band in these dots is strongly affected by a compressive strain from GaAs layer. This stress also causes a difficulty of stacked-QDs structure fabrications. In this paper, we report a growth technique of InAs QDs on AlGaSb buffer layer in the low lattice-mismatched (1.3%) InAs/AlGaSb system. We also discuss about a long wavelength emission from these QDs.

A GaAs buffer layer was formed on a GaAs (001) substrate at 580 deg.C by using molecular beam epitaxy (MBE). An AlGaSb layer on a 200 nm-thick AlSb buffer layer was formed on the GaAs sub. at 500 deg.C. InAs QDs of 1.7 or 4 ML (growth rate: 0.1 ML/s) were grown on the AlGaSb/GaAs with an As-flux (5.5×10^{-7} Torr) irradiation. A size and density of QDs were estimated by using atomic force microscopy (AFM). Photoluminescence (PL) of Ar ion laser (514 nm) excitation was also measured from InAs QDs covered with AlGaSb layer and multi-stacked QDs embedded in AlGaSb layers.

From AFM measurements, an average size and density of the 1.7 ML InAs QDs are found to be 45nm-diameter, 7nm-height, and 5×10^{10} /cm². This result exhibits a structural selectivity between the QD layer and a flat hetero-interface under growth conditions change in the InAs/AlGaSb system. A broad PL spectrum (FWHM:0.25eV) at a peak of approx. 1200nm is also observed from 1.7 ML InAs QDs covered with AlSb at 300 K. Therefore, it is expected that InAs QDs grown on AlGaSb are useful to realize opto-electrical QD devices, because these QDs emit the high intensity luminescent content around 1300nm as the long wavelength, and a growth of multi-stacked QDs layers is possible due to the low lattice-mismatch.

19:00 - **Thermoelectric power of Czochralski silicon containing electrically active oxygen nanoclusters**

poster **Andrzej Misiuk¹⁾, Vladimir V. Shchennikov²⁾, S. V. Popova²⁾, S. N. Shamin²⁾, A. V. Galakhov²⁾, V. P. Galakhov²⁾, Sergey V. Ovsyannikov²⁾**

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Czochralski grown single crystalline silicon, Cz-Si, contains oxygen admixture, in a concentration up to about 0.004 %, typically in the form of interstitials, Oi. At annealing Oi's are gathering creating oxygen and silicon containing

clusters with increasing (with annealing temperature) dimensions. In particular, annealing of Cz-Si at about 720 K results in producing of the oxygen-silicon nanoclusters exhibiting electrical activity (thermal donors, TDs [1]). Thermoelectric properties of Cz-Si with different concentrations of TDs were investigated in this work at quasi-hydrostatic pressures (HP) up to 16 GPa. The Cz-Si samples were cut from the (001) oriented Cz-Si wafers subjected to prolonged pre-annealing (up to 40 h) under atmospheric pressure to create TDs, as determined by infrared (FTIR) and electrical measurements.

Thermopower of TDs-containing Cz-Si was measured in the pressure apparatus with anvils from synthetic diamond and hard alloy [2]. The X-Ray emission spectra were determined using the ultrasoft spectrometer; some microhardness characteristics of TDs-containing Cz-Si were also determined.

Calculation of the electronic structure of investigated samples was performed in the Local Spin Density approximation. Among others it has been stated that:

- the absolute value of thermoelectric power is dependent on the TDs concentration; it decreases with HP up to HP = 7-10 GPa, at which pressures the transition to the metallic phase takes place; - the transition pressure as well as other HP - related characteristics of the Cz-Si samples are dependent on the TDs concentration. It has been proven that the thermoelectric power measured under HP is highly sensitive to the changed concentration of oxygen nanoclusters (TDs) and of other microdefects.

The work was supported by RFBR 01-02-17203.

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2. V.V.Shchennikov, Fiz.Met.Metal., 67 (1989)93.

Decoherence and Relaxation of the Exciton in Quantum Dots

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19:00 -
poster
F-30

Possible mechanisms of dephasing and relaxation of an exciton confined in a quantum dot (QD) are considered in order to establish the substantial time limitation for the coherent control of the exciton with relevance to its application in quantum information processing. It is shown that in spite of the discretisation of the spectrum of carriers confined in QD, which is responsible for the increase of their phonon relaxation time compared to the life time of the band carriers in bulk, the coherence is limited to the time of the order of picoseconds (according to the experiment [1]). This is the time of formation of the exciton-polaron [2],[3],[4] accompanied by the energy transmission into the acoustical phonon subsystem. The role of the acoustical and optical phonons in the decoherence and relaxation is discussed in detail. The estimations of the characteristic times for the InAs/GaAs QD are performed.

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"Ultralong Dephasing Time in InGaAs Quantum Dots", Phys. Rev. Lett. 87, 157401(2001), "Coherent Light-Matter Interaction in InGaAs Quantum Dots: Dephasing Time and Optical Rabi oscillations", Phys. Stat. Sol. B 233, 391 (2002), [2]L.Jacak, J.Krasnyj, D.Jacak, P.Machnikowski, "Anharmonicity-Induced Polaron Relaxation in GaAs/InAs Quantum Dots", Phys. Rev. B 65, 113305 (2002), [3]L.Jacak, J.Krasnyj, D.Jacak, P.Machnikowski, "Magneto-polaron in weakly elliptical InAs/GaAs quantum Dot", Phys. Rev. B 67, 035303 (2003), [4]L.Jacak, P.Machnikowski, J.Krasnyj, P.Zoller, "Coherent and incoherent phonon processes in artificial atoms", Eur. Phys. J. D 22, 319 (2003)

Thermally enhanced lateral carrier transfer characteristics in InAs/GaAs quantum dot heterosystem

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19:00 -
poster
F-31

Carrier dynamics for quantum dot (QD) heterosystem are the subject of intense material and device research. Photoexcitation dependence of inter-dot lateral carrier transport has been studied by temperature-dependent photoluminescence (PL). For comparison, two InAs/GaAs QD samples with different dot-size as well as size uniformity were carried out by metal-organic vapor phase epitaxy (MOVPE). Considering the thermal-sensitive PL spectra, the so-called V-shape behaviors were characterized to the corresponding carrier dynamic processes. At low temperatures, the full width at half maximum (FWHM) of 88 and 79 meV for the two QD samples, respectively, reveals that the optimal growth conditions improve the size uniformity. Meanwhile, the dot-size distribution is the

dominant contribution to the inhomogeneous broadening of PL spectra. With increasing the temperatures as well as the excitation power, the so-called negative temperature phenomena are by far observed. The narrowing of the FWHM for all confined states is associated with the lateral thermally activated carrier transfer between inter-dots at the temperature range of 110 and 175 K. The photoexcited carriers have enough thermal energy not only to overcome the matrix barriers but also to redistribute to the local minimum states. Significantly, the thermalization of carriers in the less uniformity heterodot system is profound, while the better uniformity one exhibits more thermal-stability. As the temperature further increases, the FWHM of excited state extends once again, since both the electron-phonon scattering and thermal redistribution mechanisms dominate the carrier relaxation processes. All experimental results are consistent with the rate equation calculations as well. The observation of transmission electron microscopy images will be also shown.

- 19:00 -
poster
F-32
- New type of paramagnetic silver clusters in sodalite: Ag_8^{n+}**
Jaroslav Sadlo¹⁾, Jacek Michalik¹⁾, Hirohisa Yamada²⁾, Yuichi Michiue²⁾
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Metal clusters exhibit novel electronic, optical and chemical properties owing to quantum size effect. Small cationic silver clusters have been efficiently stabilized in zeolites after hydrogen or gamma irradiation reduction. Depending on their framework structure, cation capacity and water presence zeolites offer possibility to stabilize silver clusters with different size and charge. They can be formed in two different agglomeration pathways. In the first one the agglomeration is radiation-induced process initiated by Ag^0 atoms and following by the reactions of Ag^+ cations with Ag^0 and later with Ag_2^+ and Ag_3^{2+} clusters during gradual thermal annealing. Finally Ag_4^{3+} tetramers are formed and it was postulated that they are located in octagonal prisms of zeolite rho structure. The second agglomeration pathway was observed in Ag-A zeolite. In the course of dehydration at 150°C Ag^+ become mobile and they agglomerate filling some cages with six Ag^+ cations. The assembly of six Ag^+ by trapping radiation-produced electron forms Ag_6^{5+} cluster which is paramagnetic and can be observed by EPR. In the present work we investigated $\sim 100\mu\text{m}$ crystallites of silver sodalite with hydroxyl guest anion. The XRD analysis showed sodalite structure with unit cell dimension of 0.8784 nm. Chemical composition was checked using electron microprobe analyzer and ICP technique; in both cases silver loading was higher than 7.8 per unit cell. EPR spectra of sodalite, γ -irradiated at 77K and recorded just after irradiation, show 9-line, almost isotropic signal with $g_{iso}=1.987$ and hyperfine splitting of 8.3 mT due to Ag_8^{n+} cluster. Its intensity decreases during thermal annealing and signal decays completely above 200K. We tentatively postulate that octamer consists of four Ag atoms located inside sodalite cages which interact with four Ag located in hexagonal windows of adjacent sodalite cages.

- 19:00 -
poster
F-33
- Studies of multiwall carbon nanotubes using Raman spectroscopy and AFM**
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The rapid progress of interest in carbon nanotubes research started with a development of sensitive tools for their observation and characterization. Raman spectroscopy and atomic force microscopy (AFM) has been successfully applied for studying carbon nanotubes length and diameter, electronic type (metallic or semiconducting), and whether nanotubes are separated or in bundle. Due to the specific combination of strong van Hove singularities of the phonon density of states and Raman resonance effects it is possible to measure Raman scattering spectra of small bundle of carbon nanotubes or even of single nanotube. Recent progress in understanding of the Raman spectra of relatively simple single wall carbon nanotubes (SWNT) stimulate the further Raman studies of more complicated multiwall carbon nanotubes (MWNT). We present the preliminary results of Raman scattering measurements of multiwall carbon nanotubes. The studied nanotubes have been grown by electric arc method and chemical vapor deposition (CVD) method. The nanotubes have been carefully dissolved, separated and then characterized by AFM. The micro-Raman spectra of the objects are taken with different wavelength excitations and in the temperature range 4K - 300K. Basically the spectra are quite similar to the well known single wall carbon nanotube spectra. The major Raman bands of single wall nanotubes are found in the spectra. In particular the disorder effects are visible due to the pronounced D band at $\sim 1350\text{ cm}^{-1}$. Metallic and semiconducting type of conductivity is distinguished through an analysis of the G (LO) mode at $\sim 1600\text{ cm}^{-1}$. All the results will be discussed and compared with the literature results.

- 19:00 -
poster
F-34
- Arc plasma synthesis of carbon encapsulates containing Fe-Nd-B nanocrystallites**
Michał Bystrzejewski¹⁾, Andrzej Huczko¹⁾, Hubert Lange¹⁾, Jan Kozubowski²⁾, Michał Woźniak²⁾, Marcin Leonowicz²⁾, Waldemar Kaszuwara²⁾
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02-507, Poland

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.

In this paper, the formation of novel nanostructures was achieved by DC arcing of carbon anodes filled with $\text{Nd}_2\text{Fe}_{14}\text{B}$ material. The influence of operational parameters (e.g. pressure, sublimation rate, anode composition) on the product characterization was studied. The optical emission spectroscopy was carried out on-line to determine the temperature and C_2 radial concentration fields. The solid products were analyzed by using different techniques. Fullerenes content was evaluated spectrophotometrically. The morphology of the nanostructures was studied by electron microscopy (SEM, HRTEM). The formation of carbon encapsulates was confirmed. Starting material and discharge products were subjected to magnetic measurements. The hysteresis loops proved that the processing affected the physical properties.

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Microstructural characterisation of RF sputtered ZnO thin films on SiC

Adrian Trinchi¹⁾, Wojtek Wlodarski¹⁾, Sandro Santucci²⁾, Maurizio Passacantando²⁾, Carlo Cantalini³⁾, Giorgio Sberveglieri⁴⁾

19:00 -
poster

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In the recent years there has been intense effort in the preparation of nano-sized thin film metal-oxides, largely due to their markedly different physical and chemical properties with respect to bulk materials. Nanocrystalline ZnO has attracted much interest due to its potential applications in solar cells, chemical sensors, pigments and rubber additives, electrical, acoustic and luminescent devices.

The microstructural characterization of r.f. magnetron sputtered ZnO thin films deposited on 6H-SiC is presented, together with a comprehensive investigation of the films properties as a function on annealing temperature and film thickness. These structures, with some modifications, are utilised as Schottky diode hydrogen gas sensors.

The films after annealing in static air at temperatures of 300, 500, 700 and 900°C for 1 hour have been characterized by Scanning Electron Microscopy (SEM), Rutherford Backscattering Spectrometry (RBS), X-ray Photoelectron Spectroscopy (XPS) and X-ray Diffraction (XRD) techniques. The morphology was studied by SEM which reveal the ZnO grows in cylindrical columnar grains, normal to the SiC surface, which have mean diameter of around 100-120 nm and lengths dependent on the film thickness.

XRD experiments revealed the formation of hexagonal ZnO (JCPDS card. 36-1461) was obtained by annealing the films at temperatures higher than 700°C. The surface chemical composition of these films was investigated by XPS. Selected-area XPS depth profiling of the samples was carried out by using cyclic Ar+ sputtering at low ion energy. The separation of different chemical species of the same element and quantitative analysis of experimental depth profiles enabled us to completely reveal the chemical composition of the films investigated. In such a way, the samples of mixed oxides, annealed at different temperatures, were characterized and compared. The influence of the films chemical composition on the gas-sensitive properties is also discussed.

Structural and catalytic properties thin films $\text{CuO}_x\text{-CeO}_2\text{-x}$ deposited by means of laser ablation

19:00 -
poster

Agnieszka Kopia¹⁾, Magdalena Chmielowska¹⁾, Christine Leroux²⁾, Sebastian Saizek²⁾, Jan Kusiński¹⁾, Jean Raymond Gavarri²⁾

1) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland

2) Universite de Toulon et du Var, Laboratoire Materiaux et Microelectronique de Provence, Av. de l'Universite Bat. R., La Garde B.P. 132, France

F-36

The structural analysis of thin cerium dioxides films doped with Cu, elaborated by the laser ablation techniques for catalytic and gas sensor application, was the general aim of this study. The thin films deposited on a (100) silicon

substrate were of nanocrystalline structure with well-developed texture. The morphology as well as the privileged films orientation are changed with the volume fraction of Cu. The observed changes affect on the catalytic properties of the obtained materials. It was confirmed by the performed catalytic tests with CH₄.

19:00 - **Investigation of the silica sol-gel thin films containing Mn, Pd and Pt.**

poster **Alexey E. Muravyev¹⁾, Sofia S. Mikhailova¹⁾**

1) Physical-Technical Institute UrBr RAS (PTI UrBr RAS), 132 Kirova str., Izhevsk 426001, Russian Federation

F-37

Mn, Pt and Pd were incorporated in a silica gel during the preparation of the sol. Doped silica thin films were obtained by heat treatment at 450C in a nitrogen atmosphere and studied by XPS and AFM.

From the XPS and AFM data it was found that Mn was uniformly distributed through the film. As we moved away from the outer surface to the bulk we could see that the fraction of unoxidized manganese increased. Judging by the above data, the fraction of unoxidized manganese increased with depth. Pt and Pd mainly concentrate in the subsurface layers both as oxides and pure metals. The differences in the O1s- and Si2p-spectra can be attributed to the structural variations in the Si-O bond length and the Si-O-Si bond angle that occur in the films.

According to the AFM investigations of the Pt- and Pd- doped films inclusions 600x600 nm form there. In Mn-doped films a pronounced polymeric structure with "coils" of different sizes form. Changes in the film surfaces structure in the process of the AFM scanning imply larger strength of the manganese doped films in comparison with the platinum- and palladium-doped films.

Based on the XPS and AFM results a conclusion was drawn on the relationship between the physical-chemical structure of the investigated films and the peculiarities of their surface morphology and properties.

19:00 - **Peculiarities of formation and growth of thin polymer plasma-deposited films**

poster **Alevtina M. Lyakhovitch¹⁾, Anna M. Dorfman¹⁾, Maxim A. Shirobokov¹⁾**

1) Physical-Technical Institute UrBr RAS (PTI UrBr RAS), 132 Kirova str., Izhevsk 426001, Russian Federation

F-38

Plasma polymerization is the process of generating a new type of materials with properties essentially different from traditional polymers. Low temperature plasma enables the formation of thin protective plasma-polymerized films, deposited from organic compound vapor on metal surfaces. However, the properties of such coatings can change owing to the formation of polymers with non-uniform chemical composition and structure in case the mechanism of their growth is changed. The purpose of the work presented is to study the peculiarities of the formation and growth of the polymer films deposited in the plasma of saturated hydrocarbons using the atomic force microscopy and other techniques.

In the present paper it has been stated that several film growth mechanisms take place in the plasma of saturated hydrocarbons. Along with the film growth at active sites formed after the C-C and C-H bonds rupture, the film also grows through the active sites formed at the expense of the relaxation energy of internal stresses. It is these active sites that stimulate the film growth through "cones", the shape, number, sizes and location of which reflect the degree of the polymer potential stress and the distribution of its surface energy. The change in the polymer film growth mechanism is governed by the level of internal stresses in the film. The polymer film growth mechanism influences the physicochemical properties of the film, namely, its thickness, strength, permeability, protective ability, as well as roughness and hydrophobicity of the surface. During the film deposition onto an uncooled substrate there is a critical film thickness, which depends on ultimate internal stresses. The increase of the monomer chain length makes changes in the film surface morphology; the macromolecular species formed in gas phase become larger, the cross-linking degree of plasma-polymerized films increases.

19:00 - **Silicon nitride on a thin amorphous silicon as a mask for chemical and thermal treatments**

poster **Valentina A. Yakovtseva¹⁾, Vitaly P. Bondarenko¹⁾, Marko Balucani²⁾, Aldo Ferrari²⁾**

1) Belarusian State University of Informatics and Radioelectronics (BSUIR), P.Brovka str. 6, Minsk 220000, Belarus

F-39

2) Universita 'La Sapienza' di Roma, Via Eudossiana 18, Roma 00184, Italy

A mask to protect selective regions of a silicon substrate against adverse effects of environment during chemical and/or electrochemical and thermal treatments comprises a silicon nitride layer on a thin buffer layer of amorphous silicon. The thin amorphous silicon layer is deposited on the surface of the silicon substrate. The silicon nitride layer is then deposited over the amorphous silicon layer. The composite structure is patterned and etched to form openings in the coating. The following chemical and/or electrochemical treatments in solutions of hydrofluoric acid or the like and thermal treatments in oxidizing ambient or the like effect only the surface of the exposed regions, while the silicon nitride layer secures masked silicon regions against adverse effect of environment. The thin buffer amorphous silicon layer between the silicon substrate and the silicon nitride layer minimizes stress and dislocations that can be created in the underlying silicon region by the silicon nitride layer during a thermal treatment and prevents the silicon nitride from undercutting problem during chemical (electrochemical) treatments in the solutions

of type of hydrofluoric acid that occurs in widely used protective coating consisted of a silicon nitride layer on the thin layer of silicon oxide.

Properties of nanostructured carbonaceous films containing Ni nanocrystals

Elzbieta Czerwosz^{2,4)}, Piotr Dłuzewski¹⁾, Tarun Sharda⁵⁾, Halina Wronka⁴⁾, Mirosław Kozłowski^{3,4,6)} 19:00 -

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3) *Institute of Physics PAN, al. Lotników 32/46, Warsaw 02-668, Poland*

4) *Industrial Institute of Electronics (PIE), Długa, Warsaw 00-241, Poland*

5) *Seki Technotron (SEKI), 5-6-30 Kiba, Koto-ku, Tokyo 135-0042, Japan*

6) *Institute of Physics PAS (IF PAN), Warsaw, Poland*

poster

F-40

Over the past few years, there has been an increasing interest in using carbonaceous films as the cold electron emitting cathodes in field emission displays (FED) and other field emission devices such as sensors and detectors. Major advantage of using carbonaceous films (e.g. diamond-like, nano-cluster carbon and propose by us carbonaceous films containing metallic nanocrystals) as cold cathodes are: wide temperature operating range; reliable sensing performance; simplicity in fabrication process; flexibility in the choice of substrate; compatibility with silicon microfabrication process and cost efficiency. The use of a catalyst such as Ni enhances the sensor performance (e.g. stability, selectivity and sensitivity over a wide range of temperature).

The authors present a new carbonaceous films (containing Ni nanocrystals) obtained by physical vapor deposition (PVD). The films were prepared by evaporation of 'C₆₀/C₇₀' mixture and nickel acetate from two separated sources. The Ni content was changing from 3 to 40 wt % in individual film volume. Such Ni content changes caused the structural, morphological and topographical differences in the films with different Ni content. These properties were studied with transmission electron microscopy (TEM), selected area electron diffraction (SEED) and Raman spectroscopy. With these methods it was found that Ni nanocrystal size diminishes and fullerenes are decomposed to graphite or amorphous carbon with increasing Ni content.

Electron emissive properties were studied in vacuum diode system where film was placed as a cathode and an anode was a metal tip or metal polished plate. I-U characteristics for these films exhibited that electric field threshold decreases with increasing Ni content.

Ion implanted nanolayers in AlN for direct bonding with copper

Jerzy Piekoszewski^{1,2)}, Wiesława Olesińska³⁾, Jacek Jagielski³⁾, Dariusz Kaliński³⁾, Marcin Chmielewski³⁾, Zbigniew Werner²⁾, Marek Barlak²⁾ 19:00 -

1) *Institute of Nuclear Chemistry and Technology, Dorodna 16, Warsaw 03-195, Poland*

2) *The Andrzej Soltan Institute for Nuclear Studies (IPJ), Świerk, Otwock 05-400, Poland*

3) *Institute of Electronic Materials Technology (ITME), 133 Wólczyńska, Warsaw 01-919, Poland*

poster

F-41

Aluminum nitride (AlN) is gaining increasing interest as an attractive substrate material for electronic applications in high power density packing owing to such features as: high thermal conductivity, good electrical insulation, thermal expansion similar to silicon and non-toxicity. The requirement of heat dissipation imposes the need of forming low thermal resistance (thin) joint with a high thermal conductivity metal, preferably copper. According to recent literature the direct bonding (DB) of the substrate to the conductor is considered as the most promising technique.

Recently satisfactory result of AlN-Cu bonding was demonstrated by addition of 1-1.5 at % of oxygen as an active element to AlN-Cu system without intentional modifying of the substrate surface.

In our approach it is expected that formation of nano-size interface layer with enhanced content of favorable additives introduced by ion implantation into AlN and pre-oxidation of Cu should result in even better wettability between the bonded elements.

For ion implantation into commercially available AlN substrates Ti, Fe and O ions have been chosen. The choice of Ti was dictated by a commonly known beneficial effect of this element on metal-ceramic joints quality. There is also some evidence that similar behavior can be expected of iron. Oxygen implantation is considered as a low temperature alternative to thermal oxidation of AlN surface - known to improve adhesion of copper layer.

The implantation was carried out with the doses of $5 \times 10^{15} \text{ cm}^{-2}$, $2.5 \times 10^{16} \text{ cm}^{-2}$, $1 \times 10^{17} \text{ cm}^{-2}$ at ion energy of about 140 keV. Cu foils were oxidized in air at 380°C for 10 min. DB process was performed at 1085°C in flowing nitrogen. The best results of shear strength, sufficient for electronic applications, were obtained for Ti implanted and additionally post-implantation oxidized substrates. The results are discussed in terms of possible compositional phases formed during the technological process.

Nanostructured layers in high temperature-pressure treated silicon implanted with helium

Barbara Surma^{1,3)}, Andrzej Misiuk¹⁾, Vito Raineri²⁾, Artur Wnuk³⁾, M. Prujarczyk¹⁾, Andrzej Bukowski^{3,4)} 19:00 -

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poster

F-42

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4) *CEMAT Silicon, Wólczyńska 133, Warsaw 01-919, Poland*

Helium- and hydrogen- implanted single crystalline silicon have become of remarkable technological interest because of importance of He- and / or H₂- filled microcavities created at annealing in Si:He and Si:H for producing the gettering active areas and for Si wafer splitting (Smart Cut process). The estimated pressure in the He/H₂ filled bubbles is in the Giga Pascal (GPa) range [1] and defects of different kind and concentrations are created in annealed Si:He and Si:H. The shear stress at the H/He bubble / Si matrix boundary can be tuned by annealing the Si:H, Si:He and Si:H,He samples under enhanced hydrostatic pressure of inert gas ambient (HT-HP treatment [2]). The HT-HP treatment results, between others, in the decreased strain between the Si matrix and the He- or H -containing nanostructured layers and in creation of more numerous defects in Si:He [2,3]. The effect of HT-HP treatment on Si:He (prepared by Si implantation with He⁺ at energy, E, up to 300 keV, doses, D, up to $1 \times 10^{17} \text{ cm}^{-2}$) at up to 1270 K under hydrostatic Ar pressure up to 1.2 GPa was investigated in the present work by photoluminescence (PL), X-Ray, TEM and related methods. Among others it has been stated that enhanced HP at annealing of Fz-Si:He (E = 150 keV, D = $2 \times 10^{16} \text{ cm}^{-2}$) at 1070-1270 K results in modified structure of the buried nanostructured layer promoting creation of dislocations (enhanced intensity of dislocation-related D1 PL line) and point defects clusters. Also the recently obtained results concerning the HT-HP treatment effect on Si:H,He structures will be presented and discussed.

Our investigations are currently supported (at 2002 - 2004) by the Polish Committee for Scientific Research (grant no. 4T08A 03423).

1. Xiang Lu, et al., Appl. Phys. Lett. 71 (1997) 1804.
2. A.Misiuk, et. al., Mater. Phys. Mech. 5 (2002) 31.
3. A.Misiuk, et al., Comput. Mater. Sci. 21 (2001) 515.

19:00 - **Modeling of photoresistors based on semiconductor multilayer variable-GAP structures**

poster **Bogdan S. Sokolovskii¹⁾**

1) *Ivan Franko National University, 50 Dragomanov Str., Lviv, Ukraine*

F-43

In this report the results of theoretical study of peculiarities of photocarrier transport in periodic semiconductor multilayer variable-gap structures are presented. It is investigated in detail the case of sawtooth-like photosensitive structure with linear coordinate dependence of energy gap within each layer. The structure is supposed to be illuminated with polychromatic light which uniformly generates photocarriers. The problem is solved in quasi-neutral approximation on the basis of diffusion-drift equation complemented by appropriate boundary conditions being followed from the continuity of carrier concentrations and current densities at multilayer variable-gap structures interfaces.

Band-gap grading due to spatial variation of semiconductor composition gives rise to appearance of quasielectric built-in fields which significantly affect the photocarrier transport. The coordinate dependence of photocarrier concentration manifests the nonmonotonic shape with its maximum being reached in the regions of smallest band gap. The presence of quasielectric fields gives rise to reduction of the sweep-out effect and to increase of the photo-sensitivity at large values of bias voltage. The photoelectric amplification coefficient of such photoresistors at high voltages is shown to be much greater than that based on an homogeneous material.

19:00 - **Ab initio calculations of copper nanostructures on oxide substrates**

poster **Oleg Sychev²⁾, Yuri F. Zhukovskii^{1,2)}, Eugene A. Kotomin¹⁾, Gunnar Borstel²⁾**

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2) *Universität Osnabrück, Barbarastr. 7, Osnabrück D49066, Germany*

F-44

Recent achievements in microelectronics and other high technologies result in the necessity in both improving and further development of copper applications. One of the most actual issues is increasing demand for epitaxially grown Cu films on nonconducting substrates used in integrated circuits, for instance, fabrication of smooth copper films to be served as a growth template for device structures, such as tunneling magnetoresistance devices. MgO substrates in a combination with a Fe/Pt seed layer provide the superior film quality needed. Cu/MgO interface is also of great importance in other technological applications, including catalysis, metal-matrix composites, etc.

To clarify the nature of interfacial bonding in the Cu/MgO(001) interface, we performed DFT calculations for copper adhesion on the perfect magnesia surface with metal coverage varied from 1/4 monolayer (ML), 1/2 ML (with regular net and striped adatom distributions), up to 1 ML substrate coverage. The adhesion associated with polarization and charge redistribution turns out to be the dominant contribution to the bonding on the regular Cu/MgO(001) interface. The most favorable positions for the adsorption of quasi-isolated (1/4 ML) copper atoms are found to be above the surface O²⁻ ions on the (001) substrate. Due to mismatch between the lattice constants of Cu and MgO crystals the absolute values of bonding energy per adatom for 1 ML and 1/2 (striped) ML (0.33 and 0.37 eV, respectively) are markedly smaller than for 1/4 and 1/2 (net) Cu ML where adatoms may be considered as quasi-isolated (0.62 and 0.65 eV). Because of high mobility of adatoms along a surface (with a low energy barrier), we can predict high probability for the aggregation of Cu atoms into nanoclusters as compared to the mechanism of

layer-by-layer film growth on MgO(001) substrate. Qualitatively, our present results are in agreement with earlier experimental and theoretical studies on the Me/MgO interfaces.

Sputter-deposited tungsten carbide

Pavo Dubcek¹⁾, Nikola Radic¹⁾, Sigrid Bernstorff²⁾, Kreso Salamon³⁾, Ognjen Milat³⁾

1) RUDJER BOSKOVIĆ INSTITUTE, BIJENIČKA C. 54, ZAGREB 10 000, Croatia (local name: Hrvatska)

2) Sincrotrone Trieste, Basovizza 34012, Italy

3) Institute for Physics, Zagreb 10 000, Croatia (local name: Hrvatska)

19:00 -

poster

F-45

Tungsten-carbide thin films were produced by reactive magnetron sputtering (argon + benzene) onto monocrystalline silicon substrates. Substrates were in a fixed position relative to the two adjacent cylindrical magnetrons. A series of samples were prepared, with benzene partial pressure varied from 1% to 10% of the total working gas pressure and the substrate temperature held at RT, 200°C, and 400°C, while the substrate potential was floating potential or biased (-70 V) with respect to discharge plasma.

Grains in the films, few nm in size, were investigated by GISAXS using a two dimensional CCD detector in order to obtain the information about the bulk of the films from the off-specular plane scattering. For the higher values of the benzene partial pressure, the generated films consist of densely packed tungsten carbide grains with an amorphous, carbon rich matrix in between. The lower benzene pressure resulted, in some cases, in isolated carbon rich particles buried in tungsten carbide. While the bias influence was minor, the influence of the substrate temperature on the grain size is evident.

Fabrication of 3D Hierarchical Structures

Muhammet S. Toprak¹⁾, Maria Mikhaylova¹⁾, Yun-Suk Jo¹⁾, Do Kyung Kim, Mamoun Muhammed¹⁾

1) Materials Chemistry Division, Dept of Materials Science and Engineering, Royal Institute of Technology (KTH), Brinellvaegen 23, 2tr., Stockholm SE10044, Sweden

19:00 -

poster

F-46

Conventional methods of preparing macroporous metal oxides involve mixing the particles with some type of binder. Because of nonuniformity in particle size, it is often difficult to control the shape of the pores, and consequently a low porosity with a broad size distribution is obtained. Templating methods provide an alternative approach to fabricate macroporous solids. Recently developed colloidal crystal templating techniques have permitted chemical preparations of macroporous materials having three-dimensionally ordered arrays of pores with diameters from tens to hundreds of nanometers. Macroporous inverse opal structures, templated from opals, have different optical and magnetic properties from the bulk material they are formed. Properties of these materials can be tuned through the control of the opal periodicity. An inverted opal can be envisaged as the negative replica of an opal with a periodic distribution of interconnected spherical cavities.

The main goal is the synthesis of inverse opals made of functional materials. In this work, fabrication of inverse opals made of different functional particles is reported. Wetting behaviors of opals are considered in choosing the appropriate solvents. Two different methods have been employed, namely chemical batch deposition (CBD) and electrochemical deposition. In CBD method, thermodynamic modeling was employed to obtain the optimum conditions for the formation of the particles with the desired phase within the channels of the opal-like structures. When loading is finished, the precursor is decomposed and reduced when appropriate. The polystyrene template is then removed by chemical etching or pyrolysis, to obtain the macroporous structures. Further modification of the system is also performed to produce functionalized materials. Structural and physical properties of the prepared inverse opal structures were investigated.

Fabrication of GaSb microlenses by photo and e-beam lithography and dry etching

Ewa Papis¹⁾, Anna Piotrowska¹⁾, Eliana Kaminska¹⁾, Tadeusz T. Piotrowski¹⁾, Krystyna Golaszewska¹⁾, Jacek Ratajczak¹⁾, Jerzy Katcki¹⁾, Jerzy Wrobel²⁾

1) Institute of Electron Technology (ITE), al. Lotników 32/46, Warsaw 02-668, Poland

2) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

19:00 -

poster

F-47

GaSb and related semiconductors are well recognised for their potential application in mid-infrared optoelectronics and thermophotovoltaics. We have recently demonstrated fabrication of DH InGaAsSb/AlGaAsSb/GaSb mid-IR photodetectors with detectivity ranged from 5×10^{10} to 2×10^{11} cmHz^{1/2}/W, depending on the active area and cutoff wavelength.

One of the possible way of improving photodetector performance is a monolithical integration of device structure with hemispherical/hyperhemispherical microlenses enabling effective concentration of IR radiation and increasing device detectivity.

In this work we present the results of fabrication lenses arrays in GaSb substrate using both resist reflow/dry etching and binary optics techniques. Photo and electron beam lithography techniques were used to define lenses pattern. Surface morphology, diameters and shape of the lenses after dry etching were determined by optical microscopy with Nomarski contrast, SEM, and Tencor Alpha-step profilometer.

In the case of the resist reflow technique photoresist cylinders were melt into spherical lenses by heating on a hot plate at temperature over 200°C, depending on the lens diameter and thickness. In the pattern transfer process the control of the relative etch rates of the photoresist and substrate was critical to achieve the desired microlens properties. The sputter etching in Ar⁺ was chosen to etching refractive microlenses. Binary optics technology requires anisotropy etching with high selectivity between mask and substrate. Reactive ion etching in BCl₃ plasma with RF power of 10W was used to transfer preshaped AZ PF514 electronresist into GaSb substrate. We have obtained high quality spherical microlens arrays with diameters up to 10μm and circular gratings with 400nm linewidth and 1μm period.

This work is partly supported by the State Committee for Scientific Research under the grant No 4 T11B03922

Presentation typ

[ABSTRACT TRUNCATED TO 200

[ABSTRACT TRUNCATED TO 2000 LETTERS]

19:00 - **Nanolevel Structuring - Design of Novel Functional Highly-Organized Solids and Materials**

poster **Vladimir Smirnov¹⁾, Olga Osmolowskaia¹⁾**

1) Chemistry Department, Saint Petersburg State University, Saint Petersburg, Russian Federation

F-48

The perspectives of the high precision solid-state chemical synthesis of highly-organized nanostructured solids and materials of various levels of microscopic organization are discussed. The accuracy of the synthesis is ~0.1 nm. The structural organization of substances, i.e., space ordering of substance structural units, can be performed by the self-organization processes or the processes of forced structural organization.

We prove that the chemical processes which can be controlled at the atomic-molecular level are of great importance to synthesize artificial structures of various topologies. The control is performed by sequential surface chemical reactions of functional groups (active centers) with low-molecular substances. This synthesis allows the space-time organization of chemical interaction between molecules and solids to be produced. In this context this process is similar to the organization of biosynthesis processes.

Using the formation of a two-dimensional surface oxide nanolayers with a thickness of 5-50Å as an example we consider the possibility to create a variety of space distributions of atoms of an artificially synthesized substance and represent the examples of oxide nanostructures, containing d -and f-elements.

Some peculiarities of the highly-organized structures of a "frame-within-a frame" type of material (Fe-based composites as an example) will be discussed. It is important that the temperature of such process is significantly below than a melting point of these metal .

This work was financially supported by the Russian Foundation for Basic Research (Grant No. 03-03-06161) and by the Scientific School Program (grant No. NSh-2236.2003)

19:00 - **Size effects in dynamics of dipolar planar nanosystems**

poster **Henryk Puzkarski¹⁾, Jean-Claude S. Levy²⁾, Maciej Krawczyk¹⁾**

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2) Universite Paris 7 Denis Diderot (LPTMC), 2 Place Jussieu, Paris 75251, France

F-49

The dipolar field static and dynamic components are calculated in thin magnetic stripes in saturating field applied parallel or perpendicularly to the sample surface. Lattice sums, a generalization of the sums used in theoretical treatment of infinite samples with short-range interactions, are calculated numerically, and found to be in good agreement with our analytical approximations proposed for a confined system. The equations of motion are derived for a system with pure dipolar interactions, and magnetostatic modes propagating perpendicularly to the sample surfaces are calculated. The corresponding frequency spectra and mode profiles are computed numerically with emphasis laid on size effects. The lowest-frequency modes, exhibiting typical antiferromagnetic profile, are identified as surface-localized modes. The profiles of the highest-frequency modes are found to be close to the uniform mode profile. These results will be compared with available experimental ferromagnetic measurements reporting the observation of resonance spectra with well-resolved magnetostatic modes, and following consequences for the magnetic reversal dynamics will be discussed.

19:00 - **Temperature dependence of microwave resonance absorption studies of α-iron and iron carbide nanoparticle agglomerates in a diamagnetic matrix**

poster **Grzegorz Gąsiorek²⁾, Nikos Guskos³⁾, Janusz Typek²⁾, Tomasz Bodziony²⁾, Urszula Narkiewicz¹⁾, Walerian Arabczyk¹⁾, W Konicki¹⁾, A E. Anagnostakis³⁾**

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2) Technical University of Szczecin, Institute of Physics (TUS), al. Piastów 48, Szczecin 70-311, Poland

F-50

3) *National Technical University of Athens (NTUA), Heroon Polytechniou 9, Athens 157 80, Greece*

Samples of α -iron and iron carbide (Fe₃C) nanoparticle agglomerates (with a typical size of α -iron nanoparticles in the 14-15 nm range) dispersed at a concentration of 0.1 % in nonmagnetic matrix of wax have been prepared. The samples have been characterized by XRD and SEM spectroscopy. Magnetic resonance measurements of the samples, using X-band electron paramagnetic resonance spectrometer, have been carried out in 300-8 K temperature range. A very intense and very broad magnetic resonance line has been recorded in all samples with various weight ratios of α -irons and iron carbides. For samples at room temperature the resonance signal could be fitted by one (for low iron concentration) or two (for high iron concentration) Lorentzian-shape lines: one centered near zero magnetic field (around 20-100 mT), and the other at higher magnetic field (about 520-550 mT). Both lines displayed a strong decrease of their integral intensity with decreasing concentration of iron. With increasing concentration of the Fe₃C nanoparticles the intensity and the linewidth of the resonance absorption signal shows an unusual behavior as a function of temperature. The shape of resonance line is changing with temperature; it becomes more flat at lower temperatures. Amplitude of the resonance absorption signal is decreasing with temperature down to 40-60 K and then increasing again with further temperature decrease down to 8 K. The resonance line is shifting with temperature increase towards higher magnetic fields and the integral intensity is increasing.

The effect of milling mode on the hydriding properties of nanocrystalline Mg₂Ni intermetallic

JERZY BYSTRZYCKI²⁾, TOMASZ CZUJKO²⁾, Robert Varin¹⁾, JAROSŁAW MIZERA³⁾

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3) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

19:00 -

poster

F-51

Mg₂Ni intermetallic is attractive hydrogen storage materials because of their lightweight and high absorption capacity. However, the improvement of its hydriding/dehydriding kinetics is especially important. The most typical mechanical ball milling processes applied to produce nanostructured intermetallic powders from pre-alloyed cast ingots are carried out in either planetary (Fritsch) or shaker (mixer, Spex) ball mills. The movement of balls in these mills is completely uncontrolled and chaotic. This may be one reason of different results reported in that matter so far.

In the present work, the hydrogen sorption properties of nanocrystalline Mg₂Ni intermetallic prepared by mechanical (ball) milling under controlled shearing/impact mode were studied. The pre-alloyed intermetallic powders were subjected to ball milling under various controlled milling conditions such as shearing, high-energy shearing and impact in a magnetic Uni-Ball-Mill 5. The structures of milled and hydrided powders were investigated by XRD and SEM with EDS detector. The hydriding properties of the treated samples were evaluated by monitoring the absorption P-C isotherms by the conventional constant-volume method using Sieverts semi-automatic apparatus. Changes of structure during processing and hydriding properties of nanostructured Mg₂Ni intermetallic powders will be shown and discussed.

Polymeric nanocomposites of complex ferrite

Dokyung Kim¹⁾, Muhammet S. Toprak¹⁾, Maria Mikhaylova¹⁾, Yun-Suk Jo¹⁾, Thomas Tsakalagos²⁾, Mamoun Muhammed¹⁾

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2) *Department of Ceramic and Materials Engineering, Rutgers University, Piscataway, New Jersey 08854, United States*

19:00 -

poster

F-52

The main objective of this study is to prepare the polymer-ferrite nanocomposites consisting of ferrite nanoparticles embedded in a polymeric matrix that can be used for radar absorption. Thermodynamic modeling of the complex ferrite system has been performed for the controlled coprecipitation by selecting the working pH ranges where all the ions involved in the composite could be precipitated simultaneously. The method employed is very convenient to synthesize the multicomponent systems with a homogenous distribution of compositions.

The ferrite nanoparticles were characterized using several different techniques such as XRD, DSC, TGA, TEM, and SEM. Prepared nanocrystalline complex ferrite powders have been annealed at different temperatures to investigate the effect of annealing temperature on the particle size and crystallinity. Also, an investigation of a colloidal suspension of the complex ferrite was performed by monitoring the distribution of the particles size and the zeta potentials as a function the pH. Dynamic light scattering and ESA were respectively used to measure the isoelectric point (IEP) of the ferrite particles to decide further modifications on the surface of the nanoparticles.

Afterward, the polymer nanocomposites were prepared by dispersing the complex ferrite nanoparticles in a polymeric matrix by sonication. The dispersion experiments were classified as two categories; with or without modifications of the surface of nananoparticles. The effect of surface modifications with silane (APTMS) as a coupling agent and the coating of attachment/conjugation of the ferrite nanoparticles to the polymeric matrix were investigated by

FTIR and DSC techniques. The other detailed procedures and properties will be presented during the presentation.

19:00 - poster
F-53

New Composite: Cyanine Dye/Polymer For Stable Holographic Recording
Yaroslav I. Vertsimakha¹⁾, Anatoly B. Verbitsky¹⁾, Alexander A. Ishchenko²⁾, Sergey L. Studzinski³⁾, Oksana P. Budnyk⁴⁾, Ruslan A. Lymarenko⁴⁾, Alla V. Savchuk⁴⁾

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4) *International Centre "Institute of Applied Optics" NASU, Kudryavskaya Str. 10G, Kyiv 04053, Ukraine*

The stable holographic gratings recording is found in cyanine dye/polymer composites under illumination of continuous He-Ne laser (632 nm) at low intensity.

We study cyanine dyes with different molecular structure. As polymer matrix we use various vinyl polymers. The composite samples were prepared by spin-coating method from dichloroethane solution.

The effect of cyanine dyes concentration on the diffraction efficiency of amplitude grating is studied. It is shown that in the researched range the diffraction efficiency rises with the dye concentration decrease.

The recording was performed in the spectral region of dye molecules aggregates displaying. The possible mechanism of the holographic gratings formation in studied composites based on cyanine dyes is discussed.

These composite materials can be used as flexible low-cost media for the holographic recording.

19:00 - poster
F-54

Studies of Polymer-dye Structures and Polymer-dye / V₂O₅ xerogel Heterostructures Photosensitive in Wide Spectral Region
Yaroslav I. Vertsimakha¹⁾, Anatoly B. Verbitsky¹⁾, Alexander S. Kutsenko²⁾, Alexander A. Ishchenko³⁾, Natalia A. Berezina⁴⁾, Vladimir G. Syromyatnikov⁴⁾

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4) *Kyiv National Taras Shevchenko University, Department of Chemistry, Volodymirskaya Str. 64, Kyiv 01033, Ukraine*

Optical and photovoltaic properties of polymer composites with high (up to 40 %) concentration of Cl-containing polymethine dye (meso-Cl) and their heterostructures with V₂O₅ xerogel films consisting of nanofibres have been studied. As polymer matrix different carbazole polymers: polyvinylcarbazole (PVK), polyepoxypropylcarbazole (PEPK), octylmetacrylate-vinylcarbazole (VK-OMA), 3-Iodine-octylmetacrylate-vinylcarbazole (3I-VK-OMA) were used. Meso-Cl / polymer films were prepared by spin-coating method from dichloroethane solution. V₂O₅ xerogel films were made by sol-gel method.

It is shown that few types of aggregates are formed in meso-Cl / polymer films. This leads to the significant widening of absorbance spectral region in short wavelength side (to 500 nm) comparing of molecular absorbance in the solutions. The efficiency of charge carriers photogeneration for aggregates is greater than for single dye molecules. Because of this meso-Cl - polymer composites are photosensitive at 500-950 nm range.

It is found that the increase of photosensitivity is observed for double-layer heterostructures with V₂O₅ especially in the region of xerogel absorption due to charge carriers photogeneration in V₂O₅ layer.

The structures of 3I-VK-OMA have maximal photosensitivity among studied polymer matrixes.

The effect of molecular structure of polymers, dye aggregation and V₂O₅ xerogel films quenching on photovoltaic properties of components and structures is discussed.

Photosensitivity observed for above structures is sufficiently greater than that of NBDB/C60 heterostructures, and the spectral region of their sensitivity is wider than of HITC dye based polymer structures.

Therefore studied double-layer structure can be used for the development of plastic photoconverters sensitive in near-IR region, including organic solar cells.

This work is supported by INTAS grant 00-00506.

19:00 - poster
F-55

Nanocrystalline iron-carbon fillers for polymers
Urszula Narkiewicz¹⁾, Walerian Arabczyk¹⁾, Tomasz Bodziony³⁾, Jan Subocz²⁾, Jerzy Majszczyk³⁾

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3) *Technical University of Szczecin, Institute of Physics, Szczecin, Poland*

The nanocrystalline iron samples were carburised with CO:CO₂ (98:2) mixture at the temperature of 450 °C. The obtained product contained 29 % of Fe₃C, 35 % of Fe₃O₄ and 36 % of C in form of carbon fibers. This composite

was used as a filler for epoxy resin.

The electric and magnetic properties of the obtained polymer composites were studied.

The obtained polymer product is semi-conductive. The conductivity increases with increasing content of the iron-carbon filler in the polymer composite. The filler has no effect on the dielectric properties of the polymer composite. The results of the EPR studies show a superparamagnetism of the polymer composite. A dramatic change of the EPR signal intensity is observed at the temperature of 80 to 81 K. This effect is connected with the II order phase transition at the temperature between 80 and 81 K.

Growth of CdS - PbS nanostructured double films on silicon substrate by SILAR techniques

Judita Puiso¹⁾, Seppo Lindroos²⁾, Sigita Tamulevicius¹⁾, Markku Leskela²⁾, Ginutis Balcaitis³⁾, Asta Guobiene¹⁾

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The successive ionic layer adsorption and reaction (SILAR) technique was used to deposit CdS and PbS nanostructured films. PbS thin films were grown from diluted solutions 0,4 M $\text{Pb}(\text{CH}_3\text{COO})_2$, 0,2 M $\text{Pb}(\text{CH}_3\text{COO})_2 + 0,2 \text{ M C}_6\text{H}_{11}\text{NO}_3$ (triethanolamine), 0,4 M $\text{Pb}(\text{NO}_3)_2$ as lead and 0.4 M CH_3CSNH_2 (thioacetamide) as sulfur precursors. CdS nanostructured films were grown from 0,1 M CdCl_2 as cadmium and 0,05 M Na_2S as sulfur precursors. Ionic layer by ionic layer process took place at room temperature and normal pressure. XRD, AFM and SEM and XPS characterized the growth of CdS - PbS multilayers on Si. Especially the effect of the different PbS buffer layer to the growth of CdS was studied. The PbS films were polycrystalline and cubic and CdS films were polycrystalline and hexagonal. The best crystallinity of the double layer films was achieved when lead nitrate was used as precursor. According to SEM and AFM the films were smooth.

19:00 -
poster

F-56

Preparation and electrical properties of modified polyimide films with fullerene contents

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2) Technical University of Szczecin, Institute of Electricity, Sikorskiego 37, Szczecin 70-313, Poland

At present, numerous researchers are focused on polyimide (PI) modifications that should lead to extension of potential PI application areas. This paper presents the effects of certain modifications of PI on dielectric relaxations process in the PI films. Modification of PI were carried by addition of some amount of fullerene nanoparticles (0.1-2.0 % wt) in polyamidoacids of different structures with subsequent thermal polycyclization of obtained compositions.

The essential differences in relaxation were observed in low frequency range and high temperature range. So far, as in PI composites, the relatively steady dipole structure of activation energy at 0,1 eV can be observed, the addition of fullerenes induces charge relaxation. Its the time constants and activation energies depend on the amount of C₆₀. In that case a wide extent of changes in activation energy values ranging from 0,45 eV up to 1,6 eV is particularly noticeable.

Another effect of C₆₀ presence is an evident decrease in dielectric permittivity ϵ_r for the entire range of temperatures applied and decrease in dielectric loss factor $\tan \delta$ for the temperatures lower than 300 K. A constant temperature of extrema $\tan \delta$ and a decrease in their values along with higher fullerene contents can indicate that the additions of C₆₀ do not generate qualitative changes in main dipole relaxation of time constant τ_1 , however they cause the main dipole relaxation volume reduction. It can indicate that first, the dipole relaxations fill free spaces in the nanoparticles-PI composites whereas the space charge is likely to be formed at the phase boundary between the main PI chain and the fullerene.

19:00 -
poster

F-57

The immiscible polymer blends thin layer formation by sorption from organic solutions

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A number of technologically important properties of polymeric materials are controlled by their surface chemistry and morphology. For polymer blends, the equilibrium composition of the surfaces is different from the bulk composition.

The present work is aimed to form PS(polystyrene) /PMMA (polymethylmethacrylate) blends polymeric layers (25-75 weight % concentration) based on toluene solutions of 0.5 weight % concentration and characterize these

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poster

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layers on various substrates (SiO₂ glass, Si crystalline substrate) exposed to plasma and chemical processing.

The dependence of the processes of formation and morphology of PS/ PMMA blend films on solution concentration, type and conditions of substrate processing was studied by such methods as ellipsometry, laser light scattering, scanning electron microscopy (SEM), electronic spectroscopy for chemical analysis (ESCA) and atomic force microscopy (AFM).

It is shown, that as a result of sorption processes of interaction between polymer macromolecules and substrate surface, thin polymeric layers with a structure determined by the substrate properties, solution concentration and temperature are formed. It is demonstrated that due to interaction of growing films with the substrate, morphology of thin film have a tendency to nanostructurisation, and periodic nanostructures can be formed.

- 19:00 -
poster
F-59
- Catalytic properties of metal cluster-containing polymers**
Anatolii D. Pomogailo²⁾, Svetlana I. Pomogailo¹⁾, Gulzhian I. Dzhardimalieva¹⁾
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 2) *Institute of Problems of Chemical Physics Russian Academy of Sciences (IPCP RAS), Institutskii pr., 18, Chernogolovka 142432, Russian Federation*

Immobilized metal cluster complexes are of interest as potential catalysts for varied processes. We have elaborated the methods of synthesis of Rh₆- and Os₃-containing monomers and their polymers [1,2]. The cluster-containing polymers were obtained by two ways: i) copolymerization of cluster-containing monomers with traditional monomers such as styrene, acrylonitrile, 4-vinylpyridine, etc.; ii) by polymer-analogous reaction between macroligands and metal carbonyl clusters. The cluster-containing polymers obtained can be considered as precursors of metal nano-sized polymer systems with defined dimensions and compositions of the cluster fragments. Heterogenization of the homogeneous catalysts allows one to enhance their activity and to isolate and study the intermediates which are catalytically active. We found that the immobilized metal clusters studied were effective catalysts for hydrogenation and oxidation reactions. Thus, polymer Rh- and Os carbonyl clusters show a high activity in the hydrogenation of cyclohexene or styrene in the repeated cycles. Os₃-containing copolymers were active in the oxidation of cyclohexene by molecular oxygen and oxidative dehydrogenation of 2,3,6-trimethyl hydroquinone. The possible mechanism of these reactions in the presence of polymer-immobilized clusters and the intermediate active species are also discussed.

[1] S.I. Pomogailo, G.I. Dzhardimalieva, A.D. Pomogailo, *Macrom. Symp.*, 2002, 186, 155.

[2] S.I. Pomogailo, G. I. Dzhardimalieva, A.D. Pomogailo. In book "Interfacial Effects and Novel Properties of Nanomaterials", Series "Solid State Phenomena", Trans Tech. Publs., Zürich, 2003, Vol. 94.

- 19:00 -
poster
F-60
- Multiple scattering use for varying the luminescence spectra of the luminophors based on fine-dyspersated matrixes**
Vasil P. Yashchuk¹⁾, Eugen O. Tikhonov²⁾, Olga A. Prygodjuk¹⁾, V I. Bezrodny²⁾
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 2) *Institute of Physics NASU (IP), Prospect Nauki 46, Kyiv 03028, Ukraine*

Usually one changes the luminophore to change the luminescence spectrum, but such tuning is coarse. Nevertheless in some cases slide tuning of the luminescence spectrum is required. It can be realized using multiple scattering on fine-dispersed particles embedded into the luminophor, if the luminophor luminescence and absorption spectra are overlapped. Such situation takes place when laser dyes are used. We have shown that we can realize a slide turn the luminescence spectrum of the dye within $\Delta\lambda=20\text{nm}$ by means of variation of the particle concentration, sample thickness, or dye concentration. The efficiency of luminophors does not significantly change. The results of our experiments can be used for information protection and for development of scintillators with the spectrum that as much as possible coordinated with the maximum sensitivity of receiver.

- 19:00 -
poster
F-61
- A novel thermosensitive composite fabricated with Au nanoparticles, poly lactide, and poly(N-isopropylacrylamide)**
Yun-Suk Jo¹⁾, Do-Kyung Kim¹⁾, Muhammet S. Toprak¹⁾, Maria Mikhaylova¹⁾, Mamoun Muhammed¹⁾
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Using a thermally responsive polymer with the intrinsic chemical and physical nature of stimuli-sensitive smart materials, it is possible to fabricate environmental switches for the controlled-release of pharmaceutical drug. Particularly, much attention has been paid to the biomedical applications of poly(N-isopropylacrylamide) (PNIPAAm)

because of its unique reversible transition at a specific lower critical solution temperature (LCST). A Poly(N-isopropylacrylamide-b-poly(D,D-lactide)) (PNIPAAm-PDLA) diblock copolymer template was prepared as a micelle of sub-micrometer diameter, entrapped by an outer shell composed of a poly(L,L-lactide-b-poly(ethylene glycol)) (PLLA-PEG) copolymer. The outer surface of the structures was modified by the self-assembly of Au nanoparticles to enhance the heat transfer rate from external irradiation to the polymeric nanospheres. It is associated with our objective to force the inner sphere to collapse by thermal change and then efficiently fill the cavity of the structures with a hydrophilic drug such as bovine serum albumin (BSA). From UV/VIS, DSC spectroscopy, TEM micrographs, and a quasi-elastic light scattering (QELS) spectrometer at 25 °C, the results demonstrate the successful formation of structures of PLLA-PEG@PNIPAAm-PDLA prepared by a modified-double-emulsion method with two different types of diblock copolymers composed of an inner-shell for thermal response loaded with BSA and a biodegradable outer-shell. Since the rate of drug release can be solely controlled by the outer-shell composition, it is possible to manipulate the variation in composition and enantiomeric substitutes for a specific purpose to administer the drug in a controlled fashion. The unique transition of PLLA-PEG@PNIPAAm-PDLA nanocapsules physiologically permits various kinds of feasible applications for controlled drug release systems.

Properties of TiO₂ films prepared by spray pyrolysis method

Ilona Oja¹, Arvo Mere¹, Malle Krunks¹, Claus-Henning Solterbeck², Mohammed Es-Souni²

19:00 -

1) Tallinn Technical University (TTU), Ehitajate tee 5, Tallinn 19086, Estonia

poster

2) University of Applied Sciences of Kiel, Kiel, Germany

TiO₂ thin films were prepared by cost-effective spray pyrolysis method. The solution containing titanium(IV)isopropoxide as a titanium source, acetoacetone and ethanol was deposited onto glass or quartz slides and n-type Si(100) wafers at substrate temperatures of 275-500 °C using pulsed spray solution feed.

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SEM, XRD, FTIR, UV-VIS, ellipsometry and impedance spectroscopy methods were applied to characterize as-deposited and annealed films.

Homogeneous films are formed at growth temperatures of 315-500 °C. Film thickness of 80-500 nm could be controlled by the substrate temperature and number of spray pulses. The formation of TiO₂ anatase films at temperatures above 300 °C is proved by FTIR. As-deposited films prepared at temperatures below 450 °C were amorphous, whereas crystalline films could be grown above 500 °C according to XRD. TiO₂ films prepared at low temperatures contain residues of incompletely decomposed precursors as minority secondary phases. The annealing of as-deposited films at 700 °C in oxygen atmosphere results in well-crystallized chemically pure anatase phase if films were deposited at substrate temperatures of 315-435 °C. The films deposited at 500 °C and annealed under the same conditions contain additionally rutile phase according to FTIR. The formation of interfacial SiO₂ layer is confirmed by FTIR if TiO₂ film on Si substrate is annealed at 700 °C in oxygen. Annealed TiO₂ films show refractive index close 2.30 and exhibit relative dielectric constant value of 8.5 at 10 kHz.

Tuesday, September 16th

Morning session

11:00 - 12:30

Main Building, Small Hall (Mała aula)

Semiconductor nanostructures for infrared applications

NERIJA ZURAUSKIENE¹, Steponas Asmontas¹, Adolfas Dargys¹, Jurgis Kundrotas¹, Griet Janssen², Etienne Goovaerts², Saulius Marcinkevicius⁵, Paul M. Koenraad³, Joachim H. Wolter³, Rosa Leon⁴

11:00 - 11:30

invited oral

1) Semiconductor Physics Institute, A.Gostauto 11, Vilnius LT-2600, Lithuania

2) Department of Physics, University of Antwerp, Antwerp, Belgium

3) Department of Physics, Eindhoven University of Technology, Eindhoven, Netherlands

4) Jet Propulsion Laboratory, California Institute of Technology, Pasadena, United States

5) Department of Physics-Optics, Royal Institute of Technology, Stockholm, Sweden

Intensive research in the field of self-assembled quantum dots (QDs) has resulted in rapid improvement in the understanding of the principal properties of QDs for developing new quantum devices such as quantum dot infrared photodetectors (QDIP). QDs provide two main advantages for this application: 1) the ability to absorb the normally incident infrared photoexcitation, and 2) long (up to ns) photoexcited carrier lifetimes. Normal incidence photoexcitation is desirable for the fabrication of a two-dimensional focal plane array for passive location imaging applications. The long lifetimes ensure higher responsivity of the detector allowing increased temperature operation. Due to discrete energy levels QDs could be used for tunable narrow-band detection or "fingerprint" of various objects observed from the space. In addition, for QDIP operation at space conditions it is important to investigate also influence of high energy particles irradiation on optical properties of QDs systems. Small size (~10 nm at the base) InAs/GaAs QDs were investigated using Optically Detected Magnetic Resonance

(ODMR) and time-resolved photoluminescence (TRPL) techniques. The ODMR spectra were detected via modulation of the total intensity of the QDs emission induced by 95 GHz microwave excitation and exciton fine structure was studied. Very long life times (up to 10 ns) of photoexcited carriers were observed in this system at low temperatures and excitation intensities promising higher responsivity of such QDs for QDIP development. The effects of proton and alpha particles irradiation on carrier dynamics were investigated on different InGaAs/GaAs and GaAs/AlGaAs QD and QW systems. The obtained results demonstrated that carrier lifetimes in the QDs are much less affected by proton irradiation than that in QWs. A strong influence of irradiation on the PL intensity was observed in multiple QW after high-energy particles irradiation.

11:30 - 11:45

oral

Intersublevel Relaxation Dependence of Carrier Hopping in self-organized InAs Quantum Dot Heterostructures

Hui-Tang Shen³⁾, Ya-Fen Wu¹⁾, Yung-Hsiang Lin³⁾, Chao-Ching Cheng³⁾, Ray-Ming Lin³⁾, Tzer-En Nee³⁾, Nien-Tze Yeh²⁾

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3) Department of Electronic Engineering, Chang Gung University, 259 Wen-Hwa 1st Road, Kwei-Shan, Tao-Yuan, Taiwan, Province of China

A reduction of dimensionality of the electron motions in nano-structures brings new phenomena in semiconductor physics. The correlations between intersublevel relaxation and interdot carrier hopping are an important issue for quantum dot (QD) heterosystems. All InAs QD samples grown on (100) 2 ω -titled toward (111)A Si-doped GaAs substrates with different size uniformity and dot density were carried out by metal-organic chemical vapor epitaxy (MOVPE). The observed photoluminescence redshifts for all samples investigated here revealed that the QD formation was controlled by the growth conditions. The full width at half maximum (FWHM) depended on dot size uniformity at low temperatures. With increasing temperature, the hot carriers on the excited states overcame the potential barrier to redistribute in heterodot system. Moreover, not only phonon bottleneck effect, but also size variation led to the observed abnormal shrinkage of FWHM. Considering carrier-photon rate equations, interestingly, the longer relaxation time to the ground state of ~ 200 ps was obtained in the QD samples with more size-inhomogeneity, while the shorter time of ~ 30 ps in those with more size-homogeneity. Up to temperature of 325 K, the FWHM for all the confined states increased with temperatures was dominantly due to both the electron-phonon scattering as well as thermal distribution. The images taken from the transmission electron microscopy were also shown.

11:45 - 12:00

oral

Microstructure and Electrical Properties of Doped ZnO Varistor Nanomaterials

Xueya Kang¹⁾, Tu MinJing¹⁾, Zhang Ming²⁾

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A sol-gel method of preparation doped ZnO varistor nanomaterials is described, The influences of doped ZnO nanomaterials for varistor microstructure and electrical properties (nonlinear coefficient α , breakdown voltage V_{1mA} , dielectric constant ϵ , and dielectric loss $\tan \delta$) are investigated. Compared with the conventional mixed oxide technique, varistor ceramic of prepared by nanometer materials showed more homogeneous microstructure, smaller grain sizes, higher densities and excellent electrical properties.

12:00 - 12:30

invited oral

Luminescence mechanisms: from single crystals to nanocrystals

Larisa Grigorjeva¹⁾, Donats Millers¹⁾

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According to considerable progress in the synthesis of nanocrystals the investigation of luminescence properties of these systems became actual. The overview of luminescence models (excitonic, activator and defect associated) in bulk materials and nanocrystals will be presented.

The excitonic luminescence is very sensitive to grain size and the quantum confinement effect of exciton luminescence (as well as absorption) was observed in a numerous semiconductor nano systems. The decay time of the excitonic luminescence is short (in subnanosecond region at RT) and together with the luminescence intensity enhancement due to quantum effects the commercial interest (fast scintillators or/and displays) will be satisfy.

The luminescence of defect states (donor-acceptor recombination or free electron/hole recombination on charged traps) in nanocrystals as a rule are associated with surface states. The role of nonradiative recombinations due to surface defects will be observed in luminescence decay times and luminescence yield decreasing with grain size decreasing. The surface effects of nanocrystals embedded in matrix can strongly differ from those for free standing nanocrystals since interface layer between nanocrystal and matrix play an important role. These surface and interface states changes during the thermal annealing of system. The luminescent properties of material are an efficient

indicator of these changes due to the luminescence high sensitive to any modification of defect states. The results of investigations of doped nanocrystals show that impurity ions are incorporated at surface and near surface states mainly. The luminescence quenching due the efficient association of impurity (like surface aggregates) and the growths of probability of nonradiative transitions in local impurity centers will be observed.

Lunch break	12:30 - 14:00
Afternoon session Main Building, Small Hall (Mała aula)	14:00 - 15:30
Si nanocrystals and erbium co-doped glasses for optical amplifiers Elisabetta Borsella ^{1,2)} 1) <i>INFN - Università di Padova, via Marzolo, 8, Padova 35131, Italy</i> 2) <i>ENEA, via E. Fermi, 45, Frascati - ROMA 00044, Italy</i>	14:00 - 14:30 invited oral
<p>The rapid development of optical telecommunications leads to a strong demand in low cost basic components to facilitate the deployment of the networks. A considerable effort is focused on Er-doped planar amplifiers (EDWAs) with the aim to solve the manufacturing and cost/performance issues. The SINERGIA (Si nanocrystals and erbium co-doped glasses for optical amplifiers) Project addresses the material and technological issues related to increase the optical gain per unit length of Er-doped silica (SO) and specific glasses (SG) for the realization of highly performing EDWAs, together with the exploration of extension to EDFAs (Er-doped fiber amplifiers). The technological approach concerns the co-doping of SO and SG with Er and Si nanocrystals (Si-nc), and is based on the observation of more than two orders of magnitude enhancement in the intensity of the 1.54 μm emission of Er³⁺ when this element is co-doped in dielectrics with Si-nc. An enhancement of more than three orders of magnitude of the effective Er absorption cross section (σ_{eff}) is also observed in these systems. The σ_{eff} value is comparable to that of the excitation cross section of the Si-nc at the same wavelength (0.46 μm), suggesting a strong interaction between Si-nc and Er ions, leading to an efficient energy transfer from the Si-nc to the Er ions. The enrichment of silica host with Si-nc increases its refractive index and consequently the index contrast with the cladding pure silica layer. This could result in small optical-mode dimensions and bending radius. A determining advantage is lying in the indirect excitation of Er ions, i.e. through the interaction with nc Si, by using a broad-band pumping source of low cost, instead of expensive lasers with pumping wavelengths adequate for the narrow absorption bands of Er ions at 0.98 and 1.48 μm. First consolidated results will be presented both on the modelling and on the implementation of synthesis routes and performance of co-doped glasses.</p>	
Synthesis and optical properties of nanocrystalline yttrium aluminium garnet doped with RE-ions Wieslaw Strek ¹⁾ , Dariusz Hreniak ¹⁾ , Piotr Mazur ³⁾ , Witold Lojkowski ²⁾ , Stanisław Gierlotka ²⁾ 1) <i>Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Okolna 2, Wrocław 50-422, Poland</i> 2) <i>High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland</i> 3) <i>Institute of Chemistry, Opole University (OU), Oleska 48, Opole 45-052, Poland</i>	14:30 - 14:45 oral
<p>Synthesis of nanocrystalline yttrium-aluminium garnet doped with RE³⁺-ions (RE= Eu³⁺, Tb³⁺, Nd³⁺, Yb³⁺) via the sol-gel derived method has been presented. The luminescence spectra and decay profiles of luminescence have been measured. The influence of grains size on structure and luminescence properties of the obtained materials has been investigated. The influence of incorporating of nanocrystals into sol-gel silica glasses on their physicochemical properties has been shown and discussed.</p>	
Luminescence behaviour BaTiO₃ nanocrystallites doped with RE-ions prepared by the sol-gel method Dariusz Hreniak ¹⁾ , Wieslaw Strek ¹⁾ , Robert Pązik ²⁾ , Georges Boulon ³⁾ 1) <i>Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Okolna 2, Wrocław 50-422, Poland</i> 2) <i>Institute of Chemistry, Opole University (OU), Oleska 48, Opole 45-052, Poland</i> 3) <i>Laboratoire de Physico-Chimie des Matériaux Luminescents, UMR 5620 du CNRS (UCBL), 10 rue A.M. Ampere, Villeurbanne Cedex 69622, France</i>	14:45 - 15:00 oral

The preparation of barium titanate nanocrystallites doped with RE³⁺-ions (Eu³⁺, Nd³⁺, Yb³⁺) by sol-gel method has been presented. The effect of sintering temperature on optical properties of obtained nanocrystallites has been investigated. The emission and absorption spectra and luminescence decays measurements were performed. The attention was paid into the size-effects and influence of concentration of active ions on emission properties of RE³⁺:BaTiO₃ nanocrystallites. The effect of BaTiO₃ grain sizes on photoluminescence properties of nanocrystallites has been noticed. To explain this differences a detail analysis of luminescence spectra has been performed.

15:00 - 15:15

oral

Grains of porous silicon embedded in SiO₂: Studies of optical gain and electroluminescence

Katerina Luterova¹⁾, Jan Valenta²⁾, Josef Bursik³⁾, Vojtech Kresalek⁴⁾, Bernd Honerlage⁵⁾, Ivan Pelant¹⁾, Katerina Dohnalova¹⁾

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Recent reports on experimental observation of optical gain in silicon nanostructures in the visible region, performed at several laboratories all over the world, have triggered an extraordinary surge of interest in silicon lasing. However, attempts aimed at reproducing the red stimulated emission from "standard" silicon nanocrystals (sized 3-5 nm) at some other laboratories either failed or did not come to definite conclusions. Therefore, more detailed measurements of optical gain in a wider variety of samples containing Si nanocrystals are required in order to unravel whether or not the observation of optical gain is an intrinsic property of Si nanocrystals, etc.

We have performed a detailed study of optical gain in layers of densely packed Si nanocrystals using variable stripe length (VSL) method in combination with shifting excitation spot (SES) method. In selected samples we have observed a distinct difference in behaviour between VSL and SES curves, indicating the occurrence of positive optical gain of $\sim 24 \text{ cm}^{-1}$. Preliminary reports on transport and electroluminescence measurements in thin films of SiO₂ doped with porous silicon grains, prepared by spin-coating technique, will also be discussed.

15:15 - 15:30

oral

Luminescence of ZrO₂ and ZnO nanocrystals

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The luminescence of ZrO₂ undoped and Pr doped different size nanocrystals has been studied within temperature range 300-800K. Luminescence of ZnO nanocrystals was studied in the range 85-500K.

Two main luminescence bands at $\sim 1.9 \text{ eV}$ and $\sim 2.75 \text{ eV}$ were observed in all undoped ZrO₂ nanocrystals at RT. The intensity of luminescence at 2.75 eV strongly depends on temperature due to two reasons - temperature quenching of luminescence and reduction of the number of luminescence centers. Annealing of nanocrystals at 800K in the vacuum changes the luminescence spectrum. Band at $\sim 2.75 \text{ eV}$ diminish completely whereas band at $\sim 1.9 \text{ eV}$ survived.

It is suggested that the Zr-oxygen complex is responsible for luminescence band at $\sim 2.75 \text{ eV}$ and the high stability defect can account for the luminescence band at $\sim 1.9 \text{ eV}$. The weak luminescence is observed at 1.9 eV even at 800K. The decay of luminescence is fast for both bands at RT. Close to 90% from the luminescence total intensity decays within 60ns, however a weak slow decaying component at 2.75 eV is observed also.

The luminescence band at $\sim 2.75 \text{ eV}$ is strongly suppressed in ZrO₂:Pr due to effective energy transfer from intrinsic excitations to Pr and the luminescence band at $\sim 1.9 \text{ eV}$ became dominant in doped nanocrystals. The decay of this luminescence band after excitation pulse reveals a fast stage (70% from initial intensity decays during 50ns) followed by slower one extending to the microsecond range.

ZnO nanocrystals at 85K shows the known luminescence bands peaking at $\sim 3.4 \text{ eV}$ (excitons, bound excitons) and at $\sim 2.0 \text{ eV}$ (recombination at defect states). The luminescence intensity dependence on temperature is different for 3.4 eV and 2.0 eV band, however both bands were observable at RT. It is pointed out that the ZnO nanocrystals produced by different methods shows differences in luminescent properties.

15:30 - 15:50

Coffee break

Main Building, Main Hall (Duža aula)

15:50 - 17:20

Afternoon session - continued

Main Building, Small Hall (Mała aula)

SiC nanocrystals embedded in polymer matrix : a new electro-optical nanocomposite 15:50 - 16:05**Johann Bouclé^{1,3)}, Abdelhadi Kassiba¹⁾, Nathalie Herlin-Boime²⁾, Ivan V. Kityk⁴⁾** oral*1) Université du Maine, LPEC CNRS-UMR 6087, Av. O. Messiaen, Le Mans 72085, France**2) Service des Photons, Atomes et Molécules (CEA/SPAM), bat 522, Gif sur Yvette cedex 91191, France**3) Service des Photons, Atomes et Molécules (SPAM/LFP), CEA de Saclay, bat 522, Gif sur Yvette cedex 91191, France**4) Physics institute (WSP), al. Ar. Krajowej 13/15, Czestochowa PL-42201, Poland*

Semi-conducting silicon carbide nanocrystals (ncSiC) are promising materials due to their tunable electronic and optical properties. The grain sizes, crystalline polytypes, surface states and defect content play a key role in the physical response of the particles. To take advantage of the nanosized crystal properties, functional hybrid materials based on ncSiC incorporated in an organic host are developed. In this context, it was recently shown that the incorporation of SiC nanocrystals (10-30 nm) in polymer matrix leads to an enhancement of the second- and third-order nonlinear optical susceptibilities of the nanocomposite.

This work is devoted to the electro-optical effect of such kind of nanocomposites (e.g. application as Pockels modulators). The different steps of the elaboration process were developed and improved. This includes the ncSiC synthesis by laser pyrolysis, the particle dispersion in the initial solvent/polymer blend and the deposition of the films by spin-coating to achieve high optical quality composites.

Electro-optical characterization of the composites shows an enhancement of the non-linear optical response of a polyvinyl carbazol matrix with the incorporation of the ncSiC. The corresponding Pockels coefficient may be improved through the nanoparticle properties (surface states, C/Si ratios...) and the homogeneity of their distribution in the organic matrix (concentration, global alignment of the SiC nanoparticles in the matrix under static electric field...). Discussion of these aspects gives a better understanding of the effect in this new kind of hybrid systems.

Luminescent nano-sized ZnS and ZnO particles 16:05 - 16:20**Michael Bredol¹⁾, Holger Althues** oral*1) Fachhochschule Münster, Stegerwaldstraße 39, Steinfurt 48565, Germany*

The conjugation of luminescent nano-sized doped ZnS or ZnO particles with various polymeric and biochemical substrates requires particle surfaces with well defined chemical characteristics and covalently bonded anchor groups. Huge progress has been made in this respect recently, but still the interplay between luminescent properties and surface chemistry remains a delicate theoretical and preparational task. In this work, the ratio of room temperature luminescence from (bulk) defect sites and Mn²⁺ sites (bulk and surface) has been used in a series of particles with varying particle size to study the influence of ligands present on the surface. Coupling of the ligands to the particle surface has been achieved by thio groups. Although most of the preparation of the nano-particles has been performed in aqueous solution, redispersability and transfer into non-aqueous solvents has been investigated as well.

Recent developments in the synthesis of iron-based nanostructures by laser pyrolysis: integrating structural analysis with experimental method 16:20 - 16:50**Rodica Alexandrescu¹⁾, Ion Morjan¹⁾, Florian Dumitrache¹⁾, Ion Voicu¹⁾, Iulia Soare¹⁾, Ion Sandu¹⁾** invited oral*1) National Institute for Lasers, Plasma and Radiation Physics (NILPRP), Atomistilor Str.111, Bucharest R-76900, Romania*

The CO₂ laser pyrolysis of gas phase reactants provides a powerful and versatile tool for producing nanostructures of various chemical compositions. Typically, the laser pyrolysis technique is based on a cross-flow-configuration reactor, in which a CO₂ laser orthogonally irradiates a narrow and uniform reaction zone, achieved by the confinement (by means of a coaxial buffer gas flow) of the reactant gas stream emerging from a nozzle. In this way, very pure nano-scale materials (the reaction takes place in the gas phase, far from polluting walls) may be prepared. Their properties and structures are controlled by the main process parameters (nature of the gas (vapor) precursor, gas flow rate, pressure, laser power). Being based on the resonance between the laser wavelength and the absorption bands of the gas precursors, the technique cannot avoid some drawbacks like to be addressed to materials possessing gaseous precursors. The reaction yield may be small, depending mostly on the precursors of the specific synthesized nanostructures.

Iron-based materials have a huge number of applications: magnetic storage media, sensors, catalysts etc. New approaches to their functionality are opened if particles in the nanometer size range are used. We present here comprehensive investigations of different iron-based nano-phase compounds prepared by laser pyrolysis. Sensitized iron pentacarbonyl-based mixtures were used. Various structural characteristics of the reaction products were

evidenced: filamentary shaped iron nanostructures, iron carbides, gamma iron oxide and composites. Mean size diameters ranging from 1.5 to 9 nm and sharp particle distributions were often obtained. The combined results of several analytical methods (TEM, HREM, EELS, XRD, IR and Raman spectroscopy, thermal analysis) were used to characterize the different morphologies and crystallographic features.

16:50 - 17:05

oral

Practical Synthesis of Disubstituted 2,7- Fluorenes, Conjugated Nanomaterials for Electronic and Optoelectronic

Joanna Cabaj²⁾, Jadwiga, Maria Sołoducho¹⁾

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A compounds containing fluorene units, which have substituted alkyl chains and because of that some amphiphilic character, were found to form stable monolayers (Langmuir-Blodgett layers) [1]. Because of the rigidity, planar rings and third-order nonlinear optical properties they can also form a novel pi-conjugated, processible, transparent and thermally stable copolymers. Materials obtaining from above compounds can be used in many electronic and optoelectronic devices [2].

2,7-bis(1,4-dioxane[2,3-c]thiophene)9,9-dialkylfluorene and 2,7-bis(thiophene)-9,9-dialkylfluorene have been synthesized via a Stille-type coupling reactions [3] in which dibromodialkylfluorene was reacted with 2-(trimethylthio)thiophene or 2-methylated (1,4-dioxane[2,3-c]thiophene) to give 9,9-dialkyl-2,7-bis(thiophene)fluorene or 2,7-bis(1,4-dioxane[2,3-c]thiophene)9,9-dialkylfluorene, using Pd(PPh₃)₂Cl₂ or Pd(PPh₃)₄ as catalyst (Scheme 1). A series of bis(thiophenes)- and bis(dioxanethiophenes) based on fluorene rings were prepared for conductive material and characterized by ¹H NMR and ¹³C NMR.

Further work is in progress in order to understand the role of disubstituted fluorenes in these reactions and also in polymer results.

The quantum-chemical calculations were performed to assess the usefulness of synthesised compounds for the electropolymerization. The structures and properties of synthesised molecules were determined theoretically. All examined molecules possess the planar three-ring fragment with negligibly different structural parameters.

1.LIU Y., LIU M., *New Journal of Chemistry*, 2002, 26, 180-183.

2.ZHAN X., LIU Y., ZHU D., LIU X., XU G., YE P., *Chemical Physics Letters*, 2002, 362, 165-169.

3.AFZALI A., BREEN T. L., KAGAN C. R., *Chemistry of Materials*, 2002, 14,1742-1746.

17:05 - 17:20

oral

Structure of alumina and zirconia nanoparticles synthesized by the Karlsruhe Microwave Plasma Process

Sabine Schlabach¹⁾, Vinga Szabó¹⁾, Dieter Vollath¹⁾, Adelina Braun²⁾, Rolf Clasen²⁾

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Due to their small particle size, light scattering in nanomaterials with particle sizes below ca. 40nm is very small. Therefore, new materials for optoceramics based on nanosized particles of zirconia, alumina-coated zirconia, alumina, and alumina doped with some additives are very promising. These materials were synthesized using the Karlsruhe Microwave Plasma Process [1,2]. The focus of this work is the synthesis and the characterization with respect to particle size and phase composition.

Zirconia based nanopowders with particle sizes below ca. 10nm crystallize in the cubic phase [3]. In case of the bare zirconia particles, the monoclinic phase is observed for larger particles (>10nm). Coating the zirconia nanoparticles with alumina prevents mainly the formation of the monoclinic zirconia phase. The alumina coating is amorphous. Similar like the alumina coating, alumina nanoparticles are amorphous; sometimes, small amounts of the cubic gamma-phase are observed.

Alumina nanoparticles were doped with chromia, ferric ironoxide and magnesia in concentrations up to about 6mol-%. Magnesia and chromia exhibited no influence on phase formation and color of the alumina powders. Additions of ferric ironoxide lead to a slightly yellow coloring of the alumina powders. Amounts larger than 6mol-% ferric ironoxide seem to favor the formation of the cubic gamma-phase.

Greenbodies from these nanopowders are translucent or transparent. Additionally, the transformation temperature to the stable alpha-phase of alumina can be reduced significantly by dopants [4].

[1] Vollath D, Sickafus KE, *Nanostruct.Mater.*1992, 1, 427.

[2] Vollath D, Szabo DV, *Nanostruct.Mater.*1994, 3, 927.

[3] Vollath D, Forker M, Hagelstein M, Szabo DV, *Mat.Res.Soc.Symp.Proc.*2001, 634, B7.7.1.

[4] Appel S, Clasen R, Schlabach S, Xu B, Vollath D, 26th Ann.Conf. *Advanced Ceramics & Composites, Materials and Structures, Amer.Ceram.Soc.*2002, 609.

Thursday, September 18th

Morning session

Main Building, Small Hall (Mała aula)

09:00 - 10:30

Zirconia pressure sensors: from nanopowders to device

Robert R. Piticescu¹, **Marko Hrovath²**, **Darko Belavic³**, **Ana Ionascu⁴**, **Barbara Malic²**, **Adrian M. Motoc¹**

9:00 - 9:30

invited oral

1) *Institute for Non-ferrous and Rare Metals, 102 Biruintei Blvd., Pantelimon 73957, Romania*

2) *Jozef Stefan Institute, Jamova 39, Ljubljana, Slovenia*

3) *Hipot Hyb, Sentjernej, Slovenia*

4) *IPEE SA, Albesti 14, Curtea de Arges, Romania*

Yttria doped zirconia nanopowders have been obtained using the hydrothermal procedure starting from soluble inorganic costs. The mechanisms and kinetics of the process have been studied to obtain high purity powders with crystalline size in the range 4 to 22 nm and specific surface near 200 m²/g.

These powders have been used to obtain membranes with controlled thickness and densities over 95% from the theoretical value by the tape casting technique using organic binders, dispersants and surfactants. The influence of the additives and sintering regime on the density and microstructure of membranes has been studied. Different types of ruthenates pastes were used to obtain thick resistive films on the zirconia membranes and interactions between the substrate and membranes were studied. The gauge characteristics of the device are finally discussed and possibilities for applications as mechanical pressure sensors with high sensitivity.

Morphology control of PbS nanocrystals by a novel hydrothermal process

Yujue Ji¹, **Deren Yang¹**, **Hui Zhang¹**, **Xiangyang Ma¹**, **Jin Xu¹**, **Duanlin Que¹**

9:30 - 9:45

oral

1) *Zhejiang University, State Key Lab of Silicon Materials (ZJU), Zhe Da Lu 38, Hangzhou 310027, China*

As an important IV-VI semiconductor, PbS has attracted considerable attention due to its special small direct band-gap energy (0.41eV) and a large exciton Bohr radius (18 nm), which permit quantum size confinement effect to be clearly evident even for relatively larger particles or crystallites. Due to its distinguished properties, the control of the size and morphology of PbS nanocrystals has been a primary subject. Various methods have been used for the preparation of low-dimensional PbS nanostructures. Among them, hydrothermal method has been widely used to prepare low-dimensional PbS nanostructure due to its simplicity, high-efficiency, low-cost, and more practice. Previously, PbS nanostructures have been prepared by hydrothermal method in the presence of surfactant or solvothermal method. However, The control of size and morphology of PbS nanocrystals is difficult and complicated. Herein, we report a thioglycolic acid (TGA) assisted hydrothermal method to control the morphology of PbS nanocrystals. Star-shaped, rod-like and cubic-shaped PbS nanocrystals have been obtained by TGA-assisted hydrothermal process using different sulphur and lead source. X-ray diffraction (XRD) reveals that the PbS nanocrystals are of the cubic rock-salt structure and well crystallized. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) observations show that the morphology of PbS nanocrystals is easily controlled according to sulphur, lead source, and pH value. Furthermore, the mechanism and the critical role of the TGA-assisted hydrothermal synthesis on PbS nanostructures have been preliminary presented.

Synthesis of one-dimensional chalcogenides by a novel hydrothermal process

Hui Zhang¹, **Deren Yang¹**, **Yujue Ji¹**, **Xiangyang Ma¹**, **Jin Xu¹**, **Duanlin Que¹**

9:45 - 10:00

oral

1) *Zhejiang University, State Key Lab of Silicon Materials (ZJU), Zhe Da Lu 38, Hangzhou 310027, China*

During the past decade, one-dimensional (1D) nanostructured materials have sparked a worldwide interest because of their unique electronic, optical, and mechanical properties and their potential applications in nanodevices and functional materials. Recently, Qian et al. has reported a solvothermal synthesis of chalcogenides using ethylenediamine as the solvent [1-2]. However, it is inevitable to use toxic, dangerous and expensive solvent in the solvothermal process. Addressing this issue, we have presented a thioglycolic acid (TGA) assisted hydrothermal method to prepare one-dimensional chalcogenides, which is milder, simpler, more practical, and more environmentally friendly in comparison with the solvothermal method.

We have successfully prepared CdS nanorods, Bi₂S₃ nanowires, and SnS₂ nanobelts using this method. We believed that the formation of the complexed chalcogenide clusters during the hydrothermal process is the key factor to the growth of one-dimensional chalcogenide. Based on this idea, we have introduced TGA-assisted hydrothermal method to prepare FeS nanostructure. The X-ray diffraction pattern (XRD) and Transmission electron microscopy image (TEM) indicate that the FeS nanorods have been prepared by TGA-assisted hydrothermal process using Na₂S as sulphur source. Furthermore, the different sulphur source and the ratio of Fe/S have greatly affected on the morphology and structure of FeS. The mechanism for the TGA assisted hydrothermal synthesis of FeS nanostructures has been preliminary presented.

Reference

(1)Xie, Y. Huang, J. Li, B. Liu, Y. Qian, Y. Advanced Materials 2000, 12, 1523.

10:00 - 10:15

Processing of Ceramic Nanopowders and Nanocomposites
Henryk Tomaszewski¹⁾

oral

1) *Institute of Electronic Materials Technology (ITME), Wolczynska 133, Warsaw 01-919, Poland*

The potential for ceramic nanocomposites to offer significantly enhanced mechanical properties is generally known since the first work of Niihara published in 1991. However achieving these properties needs carefully done colloidal processing, because ceramic nanopowders are naturally prone to agglomeration. The work presented here is concerned with the processing of zirconia/alumina nanocomposites via aqueous and alumina/silicon carbide nanocomposites via nonaqueous colloidal route. The effect of pH of aqueous alumina and zirconia suspensions on properties of suspension and centrifuged green bodies was studied. A correlation between surface electric charge of grains and agglomerate size, viscosity of suspension and porosity of green compacts was found. In the case of nonaqueous route alumina and silicon carbide suspensions in iso-propanol were investigated. Electrostatic surface charge of grains was changed by addition of chloroacetic acid and determined indirectly by the mass of powder deposited on electrode during electrophoresis. Different behaviour of SiC nanopowder than alumina was observed and mechanism of charge creation is proposed on the base of DLVO theory. The effect of grain charge on preventing agglomeration of the silicon carbide powder is presented on SEM micrographs of sintered nanocomposites.

10:30 - 11:00

Coffee break

Main Building, Main Hall (Duża aula)

10:30 - 10:45

Nanocalorimetry: thermal process studied in Nan-technological scale
R. Karmouch¹⁾, J-F. Mercure¹⁾, F. Schiettekatte¹⁾, S. Roorda¹⁾

oral

1) *Laboratoire René-J.-A. Lévesque, Université de Montréal (MTL), C.P. 6128, succ. centre-ville, Montréal, Québec, Montreal H3C 3J7, Canada*

A promising technique for the study of the thermal processes on a microscopic scale is the nanocalorimetry. This method of analysis is a derived version from differential calorimetry (DSC) adapted to systems implying of the energy processes of the order of the nanojoule. Our system is based on calorimeters manufactured using the modern techniques of microfabrication.

The latter consist of a membrane of Si₃N_x of a few tens of nm thickness on the surface of which was evaporated a metal strip, which will be used to heat a sample at speeds reaching (100 000 K/s) by simple induction of a current. Thus minimizing the losses by radiation and thermal conduction, measurement is taken under conditions quasi-adiabatic.

The fabrication of these ultrasensibles calorimeters starts with the deposit of a very thin layer of Si₃N_x (90 nm thickness), by LPCVD on the two faces of a silicon wafer. One of the faces will be served to deposit upon a layer of 50 nm of platinum, which will be useful as heater and thermosenseurs, whereas the other it will be etched to open a window in silicon until the thin membrane.

The subject that we want to explore using this ultrasensible technique is of great importance in the manufacture of electronic systems, that is to say the behavior of the defects in silicon. It can also be useful in the study of a large variety of phenomena: phase transitions in the nanoparticles, biological materials.

11:00 - 12:30

Morning session - continued

Main Building, Small Hall (Mała aula)

11:00 - 11:15

Synthesis of a zero-birefringence polymer doped with an inorganic birefringent crystal
Hisanori Ohkita^{1,2)}, Akihiro Tagaya²⁾, Yasuhiro Koike^{1,2)}

oral

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2) ERATO, Japan Science and Technology (ERATO/JST), K2 Town Campus E-building, 144-8, Ogura, Saiwai-ku, Kawasaki 212-0054, Japan

Birefringence induced by the orientation of polymer main chains during an injection-molding or extrusion processing restricts the application of polymers to optical devices that require focusing by lenses or maintaining the polarization states of incident light. We have proposed the "random copolymerization method" and the "anisotropic molecule dopant method" to prepare zero-birefringence polymers. Negative orientational birefringence of poly(methylmethacrylate) based type polymers was compensated by both the methods. However, polymers which have high glass transition temperature (T_g), such as polycarbonate, generally exhibit positive orientational birefringence that has not been eliminated yet. In this report, we proposed the "birefringent crystal dopant method" to compensate the birefringence of polymers by homogeneous doping with an inorganic birefringent crystal which has needle-like shape. In this method, the crystal needs to have opposite orientational birefringence to that of polymers. Strontium carbonate (SrCO_3) crystal was selected and synthesized for this method, with a particle length of 50-200nm and an aspect ratio of 2-5. SrCO_3 was doped into poly(methylmethacrylate-co-benzylmethacrylate)=78/22(wt./wt.) films with a thickness of approximately 40 μm . The copolymer films were uniaxially drawn at 130°C and 4mm/min. The positive birefringence of heat-drawn copolymer films at a wavelength of 633nm was compensated by doping with 0.3wt.% of SrCO_3 . Transmittances of blank film and SrCO_3 doped film at a wavelength of 633 nm were 92.6 % and 92.5 %, respectively. Furthermore, the glass transition temperatures of blank film and SrCO_3 doped film were 109.4 °C and 109.6 °C, respectively. These results confirmed that SrCO_3 compensated the positive birefringence of optical polymers without losing transparency and thermostability.

Strongly scattering fine dispersed media as active elements of small-size tunable sources of coherent emission

11:15 - 11:30

Eugene O. Tikhonov²⁾, Vasil P. Yashchuk¹⁾, Olga A. Prygodjuk¹⁾, Volodymyr I. Bezrodny²⁾

oral

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Strongly scattering luminescence media are alternative sources of coherent emission. Up to now these media have been mainly of scientific interest, because the features of this lasing were unascertained, and this limits possible practical use. Particularly, method of lasing wavelength tuning was not found. We have found these objects could present a practical interest. Particularly, investigated samples are able to generate even being optically thin due to surface influence. It is proved by the fact that lasing disappears when the surface influence is precluded by immersion. But in the absence of the scattering particles in the sample lasing is not observed. Thus, main origin of the lasing is the scattering by fine dispersed particles.

Lasing spectrum shifts versus the sample thickness and depend on the immersion presence which removes surface reflection. It also depends on the dye and scattering particle concentration. In the aggregate all these factors give the wide possibility to vary the lasing wavelength within the wide spectral range (according to our experiments wavelength tuning within $\Delta\lambda=20\text{nm}$ was realized). It gives a possibility to produce a tunable source of stimulated emission where the wavelength is changing by simple movement of the active element within the excitation beam without any additional adjustment.

Metallopolymer nanocomposites: preparation and properties

11:30 - 12:00

Gulzhian I. Dzhardimalieva¹⁾, Nina D. Golubeva¹⁾, Aleksei M. Bochkina¹⁾, Aleksander S. Rozenberg¹⁾, Anatolii D. Pomogailo¹⁾

invited oral

1) Institute of Problems of Chemical Physics Russian Academy of Sciences, Institutskii pr., 18, Chernogolovka 142432, Russian Federation

The materials based on metal nanoparticles have found many applications in chemistry, physics and biology. The nanoscale association of inorganic colloids with polymers affords hybrid materials that combine the properties of both components. In such a way, a catalytic, optical and electronic features of inorganic colloids might be combined with the characteristics of polymers that offer many advantages in processing. The main problems in this field focus on the development of effective methods for the synthesis and stabilisation of metal nanoparticles.

In this paper we consider the methods including simultaneous formation of the polymer matrix and metal nanoparticles. One of them is based on the thermal transformations of metal-containing monomers which includes dehydration, solid phase polymerization, and decomposition of the product which proceed sequentially at varied temperature ranges. The thermolysis resulted in the synthesis of metal nanoparticles with narrow size distribution (the mean particle diameter of 5-10 nm) in the polymer matrix formed in situ. The topography of the initial compounds and metallopolymer composites obtained as well as their composition are analyzed by optical and electron microscopy. The initial stage of nanoparticle nucleation in metallopolymer system was studied using the EXAFS spectroscopy. The novel approach for synthesis of noble metal polymer-immobilized catalysts via frontal polymerization of acrylamide complexes into surface of inorganic oxides is elaborated. Electron microscopy studies indicate that such polymer-inorganic composites contain Pd nanoparticles which are homogeneously distributed in the polymer matrix.

The hybrid inorganic polymer nanocomposites on the base of titanium oxide and methacrylate polymers containing transition metal ions were obtained and characterized using sol-gel synthesis.

The catalytic, magnetic and other properties of the metallopolymer nanocomposites are also discussed.

12:00 - 12:15

Interfacial effects in molecular polymer-polymer composites

Anatolii D. Pomogailo¹⁾

oral

1) *Institute of Problems of Chemical Physics Russian Academy of Sciences (IPCP RAS), Institutskii pr., 18, Chernogolovka 142432, Russian Federation*

In recent years the assortment of composite materials produced from industrially important polymer blends or alloys has much expanded. Blending of two or more thermoplastics, an intricate physicochemical process which proceeds under the action of mechanical forces and high temperatures, is an efficient method for preparation of novel materials with desired properties. The molecular design of multicomponent polymeric systems is a rather complicated problem. In this connection, the considerable attention should be given to the structural and chemical modification of ingredients (compounds). To achieve compatibility means to minimise the interfacial stress and to improve the interfacial adhesion. A way to design novel materials with improved properties involves the preparation of microheterogeneous compositions with controllable structural inhomogeneity.

The main ways of improving the compatibility of polymer-polymer composites containing polyolefins as one component are considered. The so-called reactor blends and the synthesis and structure of diblock and conducting composites are described. The important role of compatibilisation of polymers of different nature with the use of a third component is pointed out. The formation mechanism of molecular compositions, the structure and properties of the intermediate layer and the main fields of application of synthetic polyolefin-polymer compositions are discussed. One of them is the formation of hybrid polymer-inorganic nanocomposites. It consists in the formation of macromolecular metal complexes via functional groups of a polyolefin component followed by the reduction to obtain metal nanoparticles. It is important that by this way one may synthesize the nanoparticles in the surface layer of functioning polyolefins. This leads to the formation of the conducting layer on the surface of films or fibers of polyolefins.

12:15 - 12:30

Thermodynamic properties of nano- and micro- structured perovskite-type compounds

Speranta Tanasescu¹⁾, **Cornelia Marinescu**¹⁾, **Florentina Maxim**¹⁾

oral

1) *Institute of Physical Chemistry "I.G.Murgulescu" of the Romanian Academy (IPC), Splaiul Independentei, Bucharest 77208, Romania*

The acceptor doped micro- and nanostructured lanthanum perovskites form an important and interesting object of study owing to their technologically useful electric, magnetic and catalytic properties. Besides the determination of their electrical and structural characteristics, the knowledge of the thermodynamics of the new materials is very important in order to evaluate and optimize the properties for utilization in different applications.

In the present paper the thermodynamic properties of some doped nano- and micro- structured lanthanum manganites of general formula $\text{La}_{1-x}\text{M}_x\text{Mn}_{1-y}\text{Me}_y\text{O}_3$ (where $\text{M}=\text{Ca}$; $\text{Me}=\text{Al}$; $0.3 < x < 1$; $y=0; 0.05$) have been studied within the temperature range of 1073-1273 K and in reducing atmosphere (10^{-5} Pa). The relative partial molar free energies, enthalpies and entropies of oxygen dissolution in the perovskite phase, as well as the equilibrium partial pressures of oxygen have been obtained by using a solid electrolyte galvanic cells method. The influence of the oxygen stoichiometry change on the thermodynamic properties was examined using the data obtained by a coulometric titration technique coupled with EMF measurements.

The research was focused on emphasising the modifications in the thermodynamic properties connected with the nanocrystalline state. The results obtained for the nanocrystalline ceramics are discussed being related with the significant changes in the overall defect concentrations.

12:30 - 14:00

Lunch break

Symposium G

Bulk and Graded Nanometals

Symposium organisers

- **Yuri Estrin**, Department of Materials Science and Technology, Clausthal University of Technology, Germany
- **Dorote Juul Jensen**, Materials Research Department, Riso National Laboratory, Roskilde, Denmark
- **Terence G. Langdon**, Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, U.S.A.
- **Zbigniew Pakiela**, Faculty of Materials Science & Engineering, Warsaw University of Technology, Poland
- **Maria Richert**, Faculty of Non-Ferrous Metals, University of Mining and Metallurgy, Krakow, Poland
- **Michael Zehetbauer**, Institute of Materials Physics, University of Vienna, Austria
- **Ruslan Valiev**, Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Russia

Proceedings

The papers submitted to the conference will be published in SOLID STATE PHENOMENA.

Monday, September 15th

13:55 - 14:00

Opening address

14:00 - 15:30

Afternoon session

14:00 - 14:30

Unconventional Deformation Mechanism in Nanometals?

Helena Van Swygenhoven¹⁾

invited oral

1) ASQ division- Materials and Research, Paul Scherrer Institute (ASQ-PSI), PSI-Villigen, Villigen 5232, Switzerland

The reduction of the grain size to the nanometer range has led to many interesting material properties, including those involving structural and mechanical behaviour. Basic assumptions of dislocation theory may no longer be appropriate in nanocrystalline metals and new mechanisms that may come into play at these very small dimensions need to be explored and studied.

Large-scale molecular dynamics simulations of structural and mechanical properties of nanocrystalline Ni samples do provide intriguing insights into the effects of grain size and grain boundary (GB) structure. The simulations suggest a deformation mechanism intrinsic to the nanosized GB network where the GB structure plays a central role consisting of an interplay between (1) GB sliding accommodated by GB and Triple Junction migration and (2) dislocation emission and absorption in GBs, both at the origin of the formation of local shear planes that facilitates plastic deformation. The proposed mechanisms are interpreted in terms of experimental results such as work hardening and characteristic features on the fracture surface.

The synergies provided by molecular dynamics computer simulations and classical experimental investigations have resulted in the development of a very successful new experimental approach: in-situ profile analysis at the Swiss Light Source.

References

Hasnaoui, A., Van Swygenhoven, H. & Derlet, P. M, Science 2003

Van Swygenhoven, H. & Derlet, P. M, Phys. Rev. B 64, 224105-9 (2001)

Van Swygenhoven, H., Derlet, P. M. & Hasnaoui, Phys. Rev. B 66, 024101-8 (2002).

14:30 - 14:45

Size Effects during Nanoindentation: Molecular Dynamics Simulations in Nanocrystalline Gold

oral

Abdellatif Hasnaoui¹⁾, **Peter M. Derlet**¹⁾, **Helena Van Swygenhoven**¹⁾

1) ASQ division- Materials and Research, Paul Scherrer Institute (ASQ-PSI), PSI-Villigen, Villigen 5232, Switzerland

Mechanical properties of thin films are to a great extent measured by indentation techniques. By scaling down both the indenter size and the grain size, size effects are evoked originating from the nature of the nc-structure below the indenter and from the ratio between the indenter size and the grain size.

Large-scale molecular dynamics simulations are employed to investigate the deformation properties of a thin nanocrystalline (nc)-Au film. Two deformation mechanisms are observed in the nc-structure: (1) dislocations emitted under the indenter and absorbed in neighbor grain boundaries and (2) grain boundary sliding.

In earlier work [1] it was observed that GBs act as sinks for dislocation nucleated under the indenter. In the present work it is shown that the sink efficiency of the grain boundaries is highly dependent on the stress distribution in the GBs and that dislocations can be as well absorbed as be repelled by the GBs.

By changing the size of the indenter it is shown that the characteristic features of the indentation curve are dependent on the ration between the indenter and the grain size by means of the number of grain that are below the indenter. Other size effects on as well the elastic as the plastic behavior resulting from the curvature of the indenter are also discussed.

[1] D. Feichtinger, P. M. Derlet, and H. Van Swygenhoven, Phys. Rev. B 67, 024113 (2003).

14:45 - 15:00

Excellent Mechanical Properties of Nickel Processed by High Pressure Techniques

oral

Nikolay A. Krasilnikov⁴⁾, **Zbigniew Pakiefa**³⁾, **Krzysztof Kurzydowski**³⁾, **Witold Lojowski**²⁾,

Ruslan Valiev¹⁾

1) *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation*

2) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

3) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

4) *Ulyanovsk State University (UlGU), L.Tolstoy, 42, Ulyanovsk 432700, Russian Federation*

It was shown that Nickel processed by Equal Channel Angular Pressing (ECAP) followed by cold rolling and annealing displays quite unique mechanical properties. This permits to produce in a controlled way nickel samples with high mechanical strength and ductility. The mechanical properties ranged from UTS of 1270 MPa, with elongation to rupture of about 6%, to UTS of 900 MPa, and elongation to rupture up to 12%. The high strength properties of the material results from the additive strengthening contributions of grain boundaries in ultra fine grains and dislocation substructures. The grain boundaries and, in particular degree of their nonequilibrium state influence primarily ductility and strength of the material. The material was deformed with the formation of shear bands of diameter much larger than the grain size, and it seems that its ductility is connected with a deformation mechanism involving collective relative displacement of grain groups, with extensive grain boundary sliding. The produced high strength Ni with good ductility has a potential for practical applications, particularly, for MEMS.

Deformation strengthening anisotropy of FCC-solid solution of substitution and penetration at variation of stress-strain state indexes and stacking fault energy

15:00 - 15:15

oral

Natalya B. Efros²⁾, Dmytro Kuzmenko¹⁾, Dmytro Kolesnikov¹⁾, Boris M. Efros²⁾

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

2) *Donetsk Physics & Technology Institute of National Academy of Sciences of Ukraine (DonPhTI NASc), 72, R.Luxemburg, 83114 Donetsk, Ukraine*

In the paper the peculiarities of variation of strength and plastic characteristics of nitrogen-contented FCC polycrystals in dependence on stress-strain indexes (indexes of stress rigidity ν and Lode Nadai μ_σ) and /or the stacking fault energy (SFE).

The anisotropy of deformation hardness in dependence on ν and μ_σ indexes variations have been revealed. Moreover at lower of critical strain degree (ϵ_{cr}) the value of deformation hardness ΔH_μ at compression is higher than at tensile and apparently that is. caused by character of texture evolution connected with twinning and, consequently with rise of Taylor factor at compression. Above ϵ_{cr} the inversion of character variation of strain hardness is observed: ΔH_μ value is higher at tension than at compression). Apparently that is caused by induction by external stress field of additional dislocation splitting (Copley - Kera effect) by means of the SFE decreasing and, consequently, by more active development of planar slipping and mechanical twinning at plastic deformation. Superposition of pressure at deformation by compression and tension raises the value of strain strengthening anisotropy ΔH_μ . Interaction of twinning with slipping and twinning with twinning is the main reason of austenite strengthening as a result of fine structure of deformation twinning in FCC solid substitution solution what results in the increasing of pseudo twinning phase strengthening

Besides this it was shown that with the growth of nitrogen concentration and therefore decreasing of SFE the characteristics of plasticity and fracture rigidity of FCC crystals are increased. That, in generally, connects with peculiarities of dislocation structure evolution, and, namely, with the difficulty formation of fragmented structure - critical structure of fracture.

Batch hydroextrusion influence on structure and strengthening of nanostructured nickel

15:15 - 15:30

oral

Natalya B. Efros¹⁾, Vladimir A. Ivchenko²⁾, Boris M. Efros¹⁾

1) *Donetsk Physics & Technology Institute of National Academy of Sciences of Ukraine (DonPhTI NASc), 72, R.Luxemburg, 83114 Donetsk, Ukraine*

2) *Institute of Electro-Physics Ural Division of Russian Academy of Sciences, Russian Federation*

The deformation refinement of structural elements is one of the most effective methods for raising of strengthening characteristics, because it is not accompanied by material brittleness concomitant to the most another strengthening methods. The more structural element dispersion at least in two structure sizes and, consequently, more the value of relative extension of their boundaries, the more material strengthening must be. At present there are many methods of refinement of structural elements for different materials.

In this work we have studied the influence of structural element size on the value of deformation strengthening of nano-metals at deformation by batch hydroextrusion method where Ni was used as an example. It has been shown that grain boundary component of strengthening is stipulated by the dislocation density rise at deformation. It was obtained the interrelation between the strengthening deformation value and structural element size which coincides with Hall-Petch relation for small values of strain degree

The results of structural variations and properties of nano-crystalline nickel are presented. At values of logarithmic deformation $\epsilon > 8$ the ultra-grain structure is formed and the appearance which is caused by action of intensive plastic deformation under high pressure conditions.

Firstly, with the aid of field ion microscopy the evidence of high density defects' presence in atom structure of nano-structural nickel has been obtained. In particular, in the structural element volume the ultra-dispersed (1-10nm) block structure has been revealed. The dislocation nature of observed block boundaries has been established.

15:30 - 15:50

Coffee break

15:50 - 17:35

Afternoon session - continued

15:50 - 16:20

Structural refinement and coarsening in deformed metals

Niels Hansen¹⁾, Xiaoxu Huang¹⁾, Xing Qingfeng¹⁾

invited oral

1) Center for Fundamental Research: Metal Structures in Four Dimensions, Materials Research Department, Riso National Laboratory, Roskilde DK-4000, Denmark

Deformation of metals at increasing strain leads to a microstructural evolution which follows a continuous refinement down to the nanometre dimension. The increasing structural refinement generally leads to an increase in strength and a decrease in ductility. However these trends are reversed when the deformed microstructure is coarsened for example during a heat treatment. Optimisation of mechanical properties therefore relies on characterization and modelling of process structure-property relationships.

The behaviour of metals deformed to large strains by processing such as cold rolling, accumulative roll-bonding and equal channel angular extrusion is illustrate through changes in their microstructure and mechanical properties with increasing strain. In a further step the deformed metals have been heat treated from low temperatures coarsening slight structural modification to temperatures where significant structural coarsening and possibly recrystallisation takes place. The structural changes are quantified based on TEM observations and related to changes in tensile properties.

16:20 - 16:50

Factors affecting the accuracy and precision of quantitative description of materials microstructure

Jan Cwajna¹⁾

invited oral

1) Silesian University of Technology, Department of Materials Science, Krasińskiego 8, Katowice 40-019, Poland

The required properties of structural criteria for materials quality assessment are discussed. An example of the application of various 3D computer simulated models of polycrystalline materials microstructure for assessment of accuracy and versatility of hitherto used methods for the determination of grain volume distribution parameters is presented. It shows that none of these methods ensured the required accuracy and versatility of grain size evaluation at acceptable labour consumption. The new empirical relationships between parameters of distribution of plane section area and grains volume were determined. They are the basis of simple method for grain size estimation.

The importance of proper selection of morphological and stereological parameters was shown on the ground of the improvement of quantitative description of sintered carbides microstructure. New empirical relationships between structural factors and properties of these materials are presented.

The effect of sample preparation technique, image acquisition and image analysis method on the precision of plane section area of grains and particles was analysed. The methodological conditions of obtaining repeatable results of quantitative description of microstructure of selected metallic materials, ceramics and composites were established. The application of area weighted distribution of parameters describing grains or particles size as well of "structural maps" presenting diversification of microstructure on transverse and longitudinal cross sections of metallurgical products in the study of inhomogeneous microstructures are presented. They enable to increase the clarity and usefulness of quantitative description of materials microstructure.

16:50 - 17:05

Orientation Correlations in Metal Substructures from the Micrometre to Nanometre Range

Dorote Juul Jensen¹⁾, Jake Bowen¹⁾

oral

1) Riso National Laboratory, Materials Research Department, Roskilde, Denmark

By deformation from low to high strains, the deformation microstructure typically refines from the micrometre to the nanometre range by subdivision of the original grains. During this subdivision, geometrically necessary boundaries (GNBs) and incidental dislocation boundaries (IDBs) develop [1]. Large misorientations evolve across the GNBs while lower misorientations are observed across the IDBs [1]. An important question is, therefore, if the distribution of crystallographic orientations in the deformation microstructure becomes randomised, or if orientation correlations exist. This is, in particular, relevant after deformation to very large strains.

In the present presentation, possible orientation correlations are investigated in aluminium deformed up to strains of 10. The investigation is based on SEM/EBSP as well as TEM/Kikuchi pattern analysis. The results are compared to results from an investigation of recrystallised samples and effects of processing procedure are discussed.

[1] N. Hansen et al. These Proceedings

Crystal refinement by cold rolling in NiTi shape memory alloys

Guru Prasad Dinda¹⁾, Harald Roesner¹⁾, Gerhard Wilde¹⁾

1) Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany

17:05 - 17:20

oral

Repeated cold-rolling with intermediate folding of sheet samples represents a technique to obtain severe plastic deformation that avoids excessive heating at the internal interfaces and that proceeds without the simultaneous action of a high pressure in the range of several GPa. In the present work, bulk nanocrystalline Ni₅₀Ti₅₀ samples with average grain sizes below 10 nm have been synthesized at ambient temperature using two different processing pathways that start from a layered array of individual elemental sheets and alternatively, starting from the prealloyed NiTi intermetallic compound. The development of the microstructure during the different deformation routes was investigated by X-ray diffraction and scanning and transmission electron microscopy as a function of the deformation level. The development of the microstructure during cold-rolling - including the early and the late stages of deformation-induced nanocrystal formation - is analyzed in terms of the major thermodynamic and mechanical properties that govern the intermixing during the deformation treatment. Additionally, the microstructure refinement is discussed with regard to a crystal-to-amorphous transition that can occur during intense mechanical deformation. This work is supported by the DFG under contract no. WI 1899/1-2.

Quantitative analysis of grain boundaries in HPT iron.

Yulia Ivanisenko²⁾, Ruslan Valiev¹⁾, Hans-Jörg Fecht²⁾

1) Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation

2) Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany

17:20 - 17:35

oral

High Pressure Torsion (HPT) is a well established method to produce the ultrafine grained materials - nano- and submicrocrystalline. However conclusions about the presence of high angle misorientations between grains in the HPT-processed materials are usually based on the analysis of selected area diffraction patterns obtained in TEM, and represent therefore rather quantitative than qualitative description.

The given report represents the results of the Orientation Imaging Microscopy investigation of the grain boundaries in HPT-processed Armco-iron and after subsequent annealing. The as-processed mean grain size is 150 nm. It was found that in the as-processed state about 85% of grains are surrounded by the high angle boundaries, but character of misorientations distribution differs from the random one because of the high fraction of low angle boundaries. Low angle boundaries are situated mainly inside of grains. Annealing in temperature range 100-450°C does not lead to a notable changes in the misorientations spectra and only after the accomplishment of recrystallization at 500°C the misorientations distribution approaches to the random one.

In the annealing temperature range of 100-450°C recovery of non-equilibrium grain boundaries and dispersion of the low angle boundaries occur. The small grains with high angle boundaries act as a numerous nuclei for the recrystallization that takes place at the temperature of 500°C. Further increase in the annealing temperature leads to a rapid grain growth.

The possible mechanisms of formation of the observed misorientation spectra in the HPT iron are discussed.

Poster session

19:00 - 21:00

Nanostructured Ni₃Al intermetallic alloy processed by severe rolling at liquid nitrogen temperature and annealing

PAWEL JÓZWIK¹⁾, ZBIGNIEW BOJAR¹⁾, JERZY BYSTRZYCKI¹⁾, WOJCIECH PRZETAKIEWICZ¹⁾ poster

1) Military University of Technology (WAT), Kaliskiego 2, Warsaw 00-908, Poland

19:00 -

G-1

Refining grain size to the nanocrystalline level has been suggested as a way of improving strength while enhancing ductility and toughness. However, there is a serious problem with production of fully-dense bulk nanostructured materials. Conventional methods of powder consolidation cannot be employed for nanophase materials, because they involve high temperature (at least $0.6T_m$, T_m is the melting temperature) for extended times.

The present work is a comprehensive overview of our efforts to produce of nanostructured bulk Ni_3Al intermetallic alloy by rolling at liquid nitrogen temperature to a high value of percentage cold work and subsequent annealing. This thermomechanical treatment results in a bimodal grain size distribution, with micrometre-sized grains embedded inside a matrix of nanocrystalline and ultrafine grains. Changes of microstructure during processing and mechanical properties of nanostructured bulk intermetallics are shown and discussed.

19:00 -

poster

G-2

Morphology and properties of oxide films formed on the nanocrystalline 316L austenite steel

Anna Druzycza-Wienciek¹, Michal Wozniak¹, Krzysztof J. Kurzydowski¹

1) Warsaw University of Technology, Faculty of Materials Science and Engineering, Woloska 141, Warsaw 02-507, Poland

Passive oxide films on austenite steels (with chromia being major component) render generally high corrosion resistance of these materials in atmospheric air, steam and some acids. At a microscopic level, this resistance depends on the quality of the layers, their tightness, brittleness and recovery processes and to a large degree is influenced by microstructure of the substrate.

The paper presents the results obtained on nanocrystalline austenitic stainless steel processed by High Pressure Torsion (HPT). The aim of the project was to investigate properties of the oxide layers formed on the material subjected to Severe Plastic Deformation (SPD) in comparison to standard micro-metre grain size specimens. To this end AFM investigations have been carried of the surface of specimens subjected to HPT and subsequent annealing. TEM studies of the substrate and measurements of tribological properties have also been made. The results of all these experiments are discussed in terms of the characteristics of nanocrystalline austenitic stainless steels.

19:00 -

poster

G-3

Metal plasticity during structure formation by twist extrusion

Yan Y. Beygelzimer², Boris M. Efros², Dmitry V. Orlov¹

1) Donetsk State Technical University, Artioma str. 58, Donetsk 340000, Ukraine

2) Donetsk Physics & Technology Institute of National Academy of Sciences of Ukraine (DonPhTI NASC), 72, R.Luxemburg, 83114 Donetsk, Ukraine

Severe plastic deformations at last time attract intent attention of researchers that work in field of physical materials science. It's a powerful tool for transformation of materials structures. Basic structure forming regularities during the plastic deformation are determined by the parameters of initial structural material state, temperature-velocity deformation conditions, and mechanics of deformation process. The last is specified by scheme of external kinematics and force action on deformed volume (by the deformation scheme, tool geometry, conditions of contact friction etc.) and determine the series of basic process parameters: value of accumulated deformations intensity and deformations velocities in any spots, orientation of macroshear spatial evolution, configuration of deformed volume changes, homogeneity of stress and strain distribution etc. Each of these parameters influence in the corresponding way on formation of structure and properties of work materials.

There are two ways are used now for deriving severe plastic deformations without forming. The first way is torsion of disc-shaped blanks in Bridgmen anvils and the second one is equal channels corner extrusion of cylindrical and prismatic blanks.

A new method for deriving severe plastic deformations by hydromechanical extrusion of prismatic specimens through a twist channel die is offered in this paper. All the cross-sections of the channel that is orthogonal to the extrusion axis are invariable along this axis. An inclination of twist line changes along the matrix height and on initials and final section it's equal to zero. The peculiarities of channel geometry bring that the form of specimen doesn't change. It allows multiple extrusion of one for the purpose severe deformations accumulation.

19:00 -

poster

G-4

Hydrostatic extrusion and nanostructure formation in an aluminium alloy

Malgorzata Lewandowska², Halina Garbacz², Wacław Pachla¹, Krzysztof Kurzydowski²

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

2) Warsaw University of Technology, Faculty of Materials Science and Engineering, Woloska 141, Warsaw 02-507, Poland

Severe plastic deformation has become a popular method for processing ultrafinegrained microstructures. The most widely used deformation methods are: equal channel angular pressing (ECAP), torsion under compression (TUC) and multi-step forging (MSF). Large plastic strain may also be achieved by a hydrostatic extrusion which results in ultrafine and homogenous structure in the entire volume of the material. The aim of this work was to show that hydrostatic extrusion can be used to obtain nanostructured materials.

The material used in this study was an Al-4Cu-1Mn-0.5Mg aluminium alloy. The material in the form of 30 mm diameter rod was fully recrystallized with the mean grain size of 50 μm . The specimens were subjected to the hydrostatic extrusion under high pressure. The final diameter was 3 mm. The microstructure of the alloy was examined using light and electron transmission microscopy and image analysis. Microhardness measurements and tensile tests were performed to characterize the mechanical properties.

Microstructure observations by transmission electron microscope have revealed the grain refinement down to nanometer scale. Quantitative analysis of the grain size has shown its uniform distribution with mean equivalent diameter of 70 nm. Microhardness measurements suggest that the microstructure is homogenous on the cross section of the rod. Results of tensile tests indicate also significant increase in the flow stresses.

The Superplasticity of Hard Magnetic Alloy Fe-23wt.%Co-30%Cr with Sub-microcrystalline Structure

Alexander V. Korznikov¹⁾, Zbigniew Pakieła²⁾, Galia F. Korznikova¹⁾, Krzysztof Kurzydłowski²⁾

1) *Institute for Metals Superplasticity Problems, Russian Academy of Sciences, UFA, Russian Federation*

2) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

19:00 -

poster

G-5

Fe-23wt.%Co-30%Cr alloy is a precipitation hardening alloy used industrially as a hard magnetic material. Magnets are usually produced from this alloy by casting, as mechanical deformation and machining are hampered by the presence of brittle intermetallic σ phase in the temperature range 700-1050°C. However it is known, that variation of the size and morphology of intermetallic phases in commercial alloys enhances plasticity. Previously it was reported, that severe plastic deformation at room temperature of Fe-23wt.%Co-30%Cr alloy by high-pressure torsion results in a partial transformation of the initial phases and the formation of a nanocrystalline structure. The nanocrystalline samples exhibited increased strength and plasticity in comparison with coarse grained samples. The drawback of the samples processed by means of high-pressure torsion is their small thickness (less than 1 mm) whereas deformation at elevated temperatures in superplastic conditions enables to produce massive samples.

The purpose of this work was to study the evolution of the structure and mechanical properties of the Fe-23wt.%Co-30%Cr alloy during superplastic deformation at elevated temperatures. Maximum value of elongation (about 700%) was found at strain rate about 10^{-3}s^{-1} in the temperature range 900-950°C corresponding to $\gamma+\sigma$ phase domain. Tensile tests at different heating rates before deformation revealed significant increase of elongation at high value of heating rate. This increase can result from a less thickness of quasi-eutectoid lamellae formed at high heating rate. Subsequent deformation leads to transformation of lamellar quasi-eutectoid to a microduplex structure with a mean size of grains of both phases comparable to the lamellae thickness. Thus, the higher was the rate of preliminary heating, the smaller the mean size of γ and σ grains (less than 1 μm), what in turn increased plasticity of material.

X-ray Analysis of Cu Subjected to SPD

Igor V. Alexandrov¹⁾, Ascar R. Kilmametov¹⁾, Rafael M. Mazitov²⁾

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2) *Institute of Molecules and Crystal Physics, Ufa Scientific Centre of Russian Academy of Sciences, 151 Prospekt Oktyabrya, UFA 450075, Russian Federation*

19:00 -

poster

G-6

Recent investigations revealed that bulk nanostructured materials exhibit very attractive properties caused by their specific structure. So, it is very important to conduct thorough study of its peculiarities.

In the present research the bulk nanostructured Cu samples were investigated by the X-ray diffraction technique in the temperature interval from 85 K to 295 K. The samples were prepared by the severe plastic deformation (SPD) method performed by the high pressure torsion technique.

The obtained X-ray data were analyzed in framework of the Debye quasiharmonic approximation. A decrease in the Debye temperature up to 19 - 23 % and an increase in static and dynamic atomic displacements up to 45% and 48% correspondently were revealed. The linear heat expansion coefficient increased more than 3 times in comparison with the initial well annealed state. The shift of the phonon maximum spectrum to a long-wave part was discovered. The obtained experimental data are discussed in framework of the two-phase structural model composed of the perfect bulk lattice phase and nonequilibrium grain boundary phase.

Microstructure and mechanical properties of binary Al-Li alloys processed by ECAP

Jarosław Mizera²⁾, Malgorzata Lewandowska¹⁾, Aleksandr Korznikov³⁾, Krzysztof Kurzydłowski¹⁾

19:00 -

poster

G-7

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

2) *Faculty of Mat. Sci. Eng., Warsaw Technical Univ., Warsaw, Poland*

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

Microstructure and mechanical properties of binary Al-Li alloys processed by equal channel angular pressing (ECAP) were investigated. The study was carried out on two model Al-0,7wt.%Li and Al-1,6wt.%Li alloys. Specimens in the form of rods were subjected to severe plastic deformation up to strain values of 4,6 and 9,2.

A significant grain refinement, from the grain size of 300 μm in an initial state in both alloys to the grain size below that 1 μm has been observed in deformed material. A quantitative analysis of the microstructure and the mechanical properties in both initial and deformed state was also performed. It has been found that the yield stress increases from 40 MPa in the initial state to 190 MPa after deformation. The effect of lithium content on microstructure refinement and mechanical properties was also investigated.

19:00 - **Ductility of Nanocrystalline Materials Processed by Severe Plastic Deformation**

poster **Zbigniew Pakieła¹⁾, Michal Miskiewicz¹⁾, Krystian Paradowski¹⁾, Anna Druzycka¹⁾, Krzysztof Kurzydłowski¹⁾**

G-8 *1) Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

Nanocrystalline materials are often examined in the form of small specimens. Problems arising in testing such specimens can be classified into 3 groups: a) difficulty in the machining and handling the specimens; b) applying loading and c) strain measurements.

The present study focused attention on the mechanical properties of nanocrystalline metals measured in tensile tests. The specimens were obtained via two severe plastic deformation (SPD): a) high-pressure torsion (HPT), b) cold rolling and c) equal channel angular pressing (ECAP).

Force-displacement curves have been recorded and strain field distribution within gauge sections and in specimen heads measured. Interpretation of the experimental curves is carried out in terms of the true and apparent values of the flow stress and elongation to fracture.

19:00 - **Modelling of the Influence of the Grain Size Distribution on the Grain Growth in Nanocrystals**

poster **Tomasz Wejrzanowski¹⁾, Krzysztof Kurzydłowski¹⁾**

G-9 *1) Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

The paper presents results of grain growth modelling in single-phase polycrystalline materials based on the Hillert mean field theory [1]. For the context of grain growth in nano-polycrystals this theory has been modified to take into account transient states and the effect of grain size distribution on the kinetics of the grain growth. Both, global and local mean size of grains as well as effect of spatial fluctuations in the size of grains has been studied. The results show that the structure homogeneity and the mobility of the grain boundaries have a significant impact on the grain growth process. Relevance of the results to the processes taking place in some examples of nano-metals [2-4] is discussed.

1. M. Hillert. On the theory of normal and abnormal grain growth. *Acta metal.* 13, 1965, 227.

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3. M. A. Fortes. Grain growth kinetics: The grain growth exponent. *Materials Science Forum Vols. 94-96*, 1992, 319-324.

4. T. Sakuma, Y. Yoshizawa. The grain growth of zirconia during annealing in the cubic/tetragonal two-phase region. *Materials Science Forum Vols. 94-96*, 1992, 865-870.

19:00 - **Strain localization in nanocrystalline iron after Severe Plastic Deformation**

poster **Małgorzata Sus-Ryszkowska¹⁾, Grzegorz Dymny²⁾, Zbigniew Pakieła¹⁾, Michal Miskiewicz¹⁾, Krzysztof Kurzydłowski¹⁾**

G-10 *1) Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

2) Warsaw University of Technology, Faculty of Mechatronics, Warsaw, Poland

In the present work technically pure iron was subjected to severe plastic deformation (SPD) by equal channel angular pressing (ECAP) which brought about significant refinement of microstructure. Characteristic inhomogeneous, banded structure was observed in micro- and macro-scale. Hardness investigations at macro-, micro- and nanometrical level and tensile tests of micro-samples were carried out. The fracture surface observations show that failure mode depends on microstructure banding. Plastic strain localization in tensile micro-samples was observed via multiple-twin beam reticular interferometry method. Plastic behaviour of the material has also been modelled by Finite Element Method (FEM).

Mechanical properties nanostructured iron obtained by different methods of Severe Plastic Deformation

Malgorzata Sus-Ryszkowska²⁾, Zbigniew Pakieła²⁾, Ruslan Valiev¹⁾, J. W. Wyrzykowski²⁾, Krzysztof Kurzydłowski²⁾

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19:00 -

poster

G-11

Severe plastic deformation was used to obtain technically pure iron in nano-crystalline form. Two methods were employed to produce a total cumulated deformation of $\epsilon = 12$: (a) high pressure torsion (HPT) and (b) equal channel angular pressing (ECAP). Microstructure and mechanical properties were investigated of as deformed material. The grain size after deformation was found to be about 100 nm. This is 3 orders of magnitude less than that of the initial. Significant increase in tensile strength, yield stress, micro- and nano-hardness was also observed. However, materials obtained by HPT and ECA exhibit significantly different mechanical properties. This can be explained in terms of the relative number of high-angle grain boundaries produced by different deformation routes.

The influence of heat treatment on electrical resistivity of high-purity and boron-doped nanocrystalline Ni3Al

Saida R. Idrisova¹⁾, Alexandr V. Korznikov¹⁾

1) *Institute for Metals Superplasticity Problems, Russian Academy of Sciences, UFA, Russian Federation*

The influence of boron on the structural stability of nanocrystalline Ni₃Al intermetallic compounds was investigated by comparing a high-purity material with a boron-doped (0.1 wt.%) compound. The nanocrystalline structure was obtained by severe shear deformation by torsion under quasi-hydrostatic pressure. Residual electrical resistivity was used to characterise the material evolution during thermal treatments in the temperature range 293-1313 K. After severe plastic deformation the materials were disordered, with a small crystallite size of about 20 nm, similar in both materials. During isochronal anneals the evolution of the microstructure, the long-range ordering and the recovery of the residual electrical resistivity took place at higher temperatures in the boron-doped compound, i.e. the thermal stability of the cold-worked structure was higher.

The evolution of the residual electrical resistivity on annealing is characterised by the existence of three main stages, which are qualitatively similar in the two materials, but differ by their amplitudes and by the temperatures at which they occur: first a small resistivity increase (stage A), a strong decrease (stage B), then a domain of weak, reversible increase followed by a slight decrease (stage C). In the following, these stages will be used as a guide for describing the structural and property changes which were determined. The 77 K and room-temperature resistivities displayed variations which were qualitatively the same as those of the residual (4.2 K) resistivity.

19:00 -

poster

G-12

X-Ray investigations of the natural and artificial White Etching Layer

Marcin Wojdyr^{1,3)}, Stanisław Gierlotka¹⁾, Yulia Ivanisenko²⁾, Witold Lojkowski¹⁾

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

2) *Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany*

3) *Faculty of Physics, Warsaw University of Technology, Koszykowa 75, Warsaw 00-662, Poland*

19:00 -

poster

G-13

The structure of the White Etching Layer (WEL) and the mechanism of its formation still remains a matter of discussion. We analyzed X-Ray diffraction data on the original WEL and WEL-like structures obtained by high pressure torsion. Data analysis was performed by the whole-pattern fitting method. The natural WEL shows clear asymmetry of the diffraction lines, which can be attributed to either tetragonal martensite structure or to the formation of a ferrite nanophase with a lattice parameters larger than that of the raw material. X-Ray patterns of the natural WEL were fitted with the two models. The quality of the fit clearly favors the model with two cubic phases over the model with a single tetragonal phase. Artificial WEL samples could be fitted with a single cubic phase model. Several alternative methods were simultaneously used to extract information on crystallite size and internal stress. The analysis of the natural WEL shows that the secondary cubic nanophase is formed of the crystallites of the average size of 8 nm essentially free of any strain. The lattice parameter 0.2881nm corresponds to the 0.26wt% concentration of the dissolved carbon. In the artificial WEL samples the crystallite size decreases down to 9nm at the maximum shear stress of 430, while the internal strain gradually increases. There is however no

change of the lattice parameter. This means that no carbon from cementite dissolves in the lattice of ferrite. It must therefore be located in the grain boundaries and dislocation cores. We conclude that the high pressure torsion does not accurately reproduce the natural process of the WEL formation. It involves an immediate severe deformation the sample while natural process takes numerous subsequent cycles of modest deformation with simultaneous local heating of the deformation area. Such a process may lead to the gradual diffusion of the carbon atoms from the grain boundaries into the crystal lattice of ferrite.

19:00 - **Durability of wheel surface layer in railway wheelsets**

poster **Laura Perchuc¹⁾**

1) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

G-14

It is clear that the spin at the tread of drive wheels appear always. Under operation conditions a phenomenon of macro slip and side slipping is especially undesirable and harmful, since it causes an excessive wear of rolling wheel surfaces owing to generation of such defects as spallings, shellings, flat places etc. The better knowledge about the process of formation of martensite around a wheel flat, and about the ensuing spalling, will facilitate money-saving improvements in material of wheel and will enable to increase safety.

Wheel surface microstructure in steel (of the type UIC 812 3, R7T) for railway wheels was investigated. In paper was shown results of study of the optical metallography, SEM, TEM observations of the real wheel surface microstructure. Structural investigations were performed on metallurgical microscope Neophot 1 and scanning electron microscope Philips XL30 and transmission electron microscopes (Philips EM300 and Jeol JEM 1200 EXII).

19:00 - **Crystallization kinetics of Al_{95-x-y}Mm_xNi₅(Co,Fe)_y series (x = 4, 5; y = 2, 3)**

poster **Javier S. Blazquez¹⁾, Hristo Dimitrov¹⁾, Jerzy Latuch¹⁾, Tadeusz Kulik¹⁾**

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wotowska 141, Warsaw 02-507, Poland*

G-15

Effect of small changes of the composition on the crystallization process of a quaternary AlMmNi(Co,Fe) alloy series (Mm = mischmetal) obtained in amorphous state by melt-spinning technique, is studied using differential scanning calorimetry (DSC) and X-ray diffractometry (XRD). Devitrification process takes place in several steps for all the studied alloys. Nanocrystalline microstructure appears during the first transformation stage detected by DSC. This microstructure has been characterized by XRD as nanocrystals of α -Al phase embedded in an amorphous matrix. Average grain size, D is about 15-20 nm (estimated using Scherrer formula) and crystalline volume fraction, X_C = 50-80 %. The highest X_C was found for the lowest Mm containing alloy. Annealing above the second and third transformation stages produces fully crystallized samples. At this stage only equilibrium phases were detected: α -Al, orthorhombic Al₁₁Mm₃ and orthorhombic Al₃Ni. Kinetics of nanocrystallization was studied in terms of non-isothermal approaches (Kissinger, Augis-Bennet, Gao-Wang and Ozawa methods). Activation energy of nanocrystallization, E_a ranges from 1,5 to 2,7 eV. The highest values were found for the alloys with the lowest Al content. Combining Gao-Wang and Ozawa methods, an idea of the value of Avrami exponent, n, at the early (Gao-Wang) and final (constant value in Ozawa) stages of nanocrystallization is obtained: n decreases from 1-1.5 to 0.5.

19:00 - **Bulk nanostructured Al-Mm-Ni-(Fe,Co) alloys produced by high-pressure hot compaction of amorphous powders**

poster **Hristo Dimitrov¹⁾, Jerzy Latuch¹⁾, Tadeusz Kulik¹⁾, Petr Kubecka²⁾, Peter Svec²⁾**

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wotowska 141, Warsaw 02-507, Poland*

G-16

2) *Institute of Physics, Slovak Academy of Sciences, Dubravska cesta 9, Bratislava 842-28, Slovakia (Slovak Republic)*

Amorphous and nanocrystalline ribbons can be obtained in quaternary Al-RE-TM1-TM2 systems (RE - rare earth metal, TM1 and TM2 are transition metals). Replacement of RE by Mischmetal (Mm) reduces the cost of the material, as Mm is cheaper than pure rare earth elements. Bulk nanostructured Al-Mm-Ni-(Fe,Co) alloys were produced by ball-milling of amorphous ribbons followed by high-pressure (7.7 GPa) compaction at elevated temperatures. Amorphous ribbons of Al₈₇Mm₅Ni₅(Fe,Co)₃ alloys were produced by melt-spinning technique. Nanocrystallization took place not during additional annealing, but during short-time heating at temperatures close to the onset crystallisation temperature during pressing. X-ray diffractometry and differential scanning calorimetry were used for characterisation of the crystallization process and its products.

Mechanical properties of nanostructured compacted samples, represented by their microhardness, were higher than those of amorphous ribbons.

19:00 - **A physical criterion for the grain subdivision during severe plastic deformation**

poster

Nariman A. Enikeev¹⁾, Tatiana S. Orlova²⁾, Igor V. Alexandrov¹⁾, Alexei E. Romanov²⁾

1) *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation*

2) *Ioffe Physico-Technical Institute (Ioffe), Polytechnicheskaya, 26, St.-Petersburg 194021, Russian Federation*

G-17

It is well known that severe plastic deformation (SPD) leads to considerable structure refinement in the processed materials. The obtained nanostructured materials are characterized by equiaxial grains of small size and high-angle grain boundaries. These features are responsible, in particular, for their outstanding mechanical properties [1].

However, there is still no unified theoretical approach to describe mechanisms which control the structure refinement at SPD. To assist solving this important problem, in the present work it is suggested to use a physically-based energetic criterion for grain subdivision which is formulated in terms of disclination theory. This model is based on the assumption that during SPD grain splits into a number of substructure elements in order to adjust the total system energy by decreasing the local contribution related to individual grains. Using disclination approach allowed to calculate the energy of a probe grain caused by its deformation under given deformation conditions. The grain believed to be divided in a number of subgrains if the split configuration possessed less energy than initial one. The capabilities of the proposed model for the structure refinement description (decrease in grain size and misorientation growth) are demonstrated for the case of deformation by equal-channel angular pressing. It is planned to implement the developed criterion with self-consistent viscoplastic model [2] in order to describe deformation behavior and microstructure evolution during SPD (equal-channel angular pressing).

[1] R.Z.Valiev, R.K.Islamgaliev, I.V.Alexandrov, Bulk nanostructured materials from severe plastic deformation, Prog. Mater. Sci., vol. 45, 103-189 (2000).

[2] I. J. Beyerlein, R. Lebonsohn, Carlos Tome, Modeling of texture and microstructural evolution in the equal channel angular process, Mater. Sci. Engng A. 345, 122-138 (2003).

Development of in-situ technology for producing nanostructural materials by intensive shear deformation under high pressure.

19:00 -

Bilousov Mykola¹⁾

poster

1) *A.Galkin Donetsk Institute for Physics&Technology (DonPTI), Roza Luxemburg 72, Donetsk 83114, Ukraine*

G-18

The development of in-situ technology for formation submicro- and nanocrystalline structures by intensive shear deformation under high pressure is a necessary conditions for producing nanomaterials with the given properties. The idea of in-situ technology consists in simultaneous registration of internal properties of samples, external deforming parameters and corresponding cubmicrostructural changing with following establishing of connection between them.

The results of researches Cu, Al, Fe (solids and powders) have shown, that the curve of dependence of stress of compression from shear deformation had stage character, it is marked as: a) a stage of elastic behaviour; b) a stage of intensive plastic deformation with fragmentation of elements of structure (structural - kinetic changes); c) a stage of structural hardening and secondary elastic behaviour.

Parameters of a crystal lattice monotonously decreased with growth of a compressing pressure. Width and intensity of X-ray lines unmonotonically changed, that correlates with the beginning of fragmentation of elements of structure, that are confirmed by unmonotonous changing of temperature, electroresistance and increasing of intensity of acoustic issue. At a pressure more then 20GPa considerably has increased diffusing dispersion. The quantitative processing X-ray diffraction peak by profile analysis using the Multiple Whole Profile Fitting Method has shown presence of nanodimensional elements- 80-200 nm. The formation of elements with nanostructures was facilitated at additional imposing shear components. Is shown, that the proceed of intensive shear plastic deformation under high pressure at a stage of fragmentation.

Mechanical properties of aluminium wires produced by plastic consolidation of fine grained powders.

19:00 -

Henryk Dybiec¹⁾, Paweł Kozak

poster

1) *AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland*

G-20

The paper will present results of mechanical tests and grain structure investigation of aluminium wires obtained by plastic consolidation of aluminium powders produced by atomisation from liquid state. The powders production by this method could be potentially adopted to obtain ultra fine/nano structured materials by rapid solidification and/or subsequent milling. Conversion of material from powder to bulk state with retaining of powders fine structure and minimizing of porosity are problems resolving of which is crucial for such technological rout of nanometals production. An influence of condition of plastic consolidation by hot extrusion on mechanical properties of consolidated products is a main target of presented investigation. The standard mechanical properties of extrusion products have been determined and grain structure has been inspected. The results have been compared to extrusion products of conventional bulk aluminium. It has been stated substantial increase of properties and refining of grain structure in materials consolidated from powders and negligible porosity of consolidated products.

19:00 - **Synthesis of nanoprecipitates of vanadium carbides in the alloys with ferrite matrix**

poster **Edward Fras¹⁾, Jan Głownia¹⁾, Edward Guzik¹⁾**

1) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland

G-21

The main purpose of this research is investigation of possibility of carbides nanoparticles precipitation in the alloys with ferrite matrix. Those alloys permit to obtain the optimum plasticity and strength parameters. Investigations provided with cast, heat treated multielements Fe- C alloys with small content of vanadium. Heat treatment was carried out in muffle furnace with a programmable controller. Specimens were encapsulated in vacuum in silica tube to prevent scalling and decarburization. Samples were heated up to austenitizing temperature and held in this temperature, cooled to about 450 °C and then air cooled to room temperature. The TEM examination of the samples revealed that there was a wide range of submicron precipitates in the range of about 10-50 nm. It is assumed that these particles have been precipitated from austenite and ferrite during cooling. Selected area diffraction patterns of these precipitates showed that they had a cubic structure and suggested that formula of these molecules the most probably is VC_{0,78}.

19:00 - **Hardness and XRD Investigation of Ti/Nb and TiN/NbN Superlattices Prepared by Magnetron Sputtering**

poster **Paweł Kałdonek¹⁾, Andrzej Wolkenberg¹⁾, Zygmunt Nitkiewicz¹⁾, Adam Tokarz¹⁾, Hanna Wrzesińska²⁾**

1) Technical University of Częstochowa, Armii Krajowej, Częstochowa, Poland

2) Institute of Electron Technology, Warszawa, Poland

G-22

Various hard coatings have been widely used to improve the performance of tools and to extend tool life. Superlattices have very interesting properties, which are unattainable in bulk materials. The primary interest is in the mechanical properties with the secondary goal of improving the tribological properties of the surface. Among the different methods, the magnetron sputtering was found to be a good method to deposit high-quality superlattice coatings on tool materials because of easy control of composition modulation wavelength, crystal structure and stoichiometry of each layer.

In this work magnetron sputtering method is presented. By this method we have obtained Ti/N and TiN/NbN superlattices. We used as the substrate Si (100) wafers. Additionally 300 nm TiN and 300 nm NbN thin films evaporated onto Si wafers were used for comparing with our superlattices. X-ray diffraction method was used for the structural investigations (both substrate and deposit) and evidence of layered (or not) deposit structure. The hardness of composite

(Si + multilayer) was measured using the Vickers indenter with 5g load.

The production of a well defined multilayered structure was confirmed

(by XRD) for TiN/NbN superlattice grown onto Si substrate. This superlattice exhibited pure cubic structure with main visible (111) and (200) peaks. The hardness of the composite (substrate + deposit) had the highest value of 45 GPa.

Value of hardness of Ti/Nb superlattice is lower- 30 GPa. The hardness of the TiN/NbN superlattices deposited onto Si substrate had higher values than hardness of Si.

In this work the possibility of deposition of the superlattices onto Si wafers has been checked. The hardness results, especially for TiN/NbN superlattice, are very promising for improving of the wear resistance. The coatings demonstrates application prospects in the cutting tool industry.

19:00 - **Structure and chemical composition of nanocrystalline stainless steel powders obtained by ball milling and duplex stainless steels obtained by their sintering**

poster **Agnieszka Szymanska¹⁾, Krzysztof Sikorski¹⁾, Jan Kazior*²⁾**

G-23

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

2) *Institute of Materials Science and Metal Technology, Cracow University of Technology, Cracow, Poland*

The article illustrates the influence of ball milling of the 316L and 434L stainless steel powders (medium size of grains was about 135µm) as well as their mixes (75 wt. % of 434L + 25 wt. % of 316L; 50 wt. % of 434L + 50 wt. % of 316L and 25 wt. % 434L + 75 wt. % 316L) on their structure. Medium size of the grains (about 20 nm) were obtained after 110 hours of milling. The powders that obtained were pressed isostatically and sintered under hydrogen atmosphere. The samples were then characterized using an optical microscope equipped with a computer image analyser as well as in scanning electron microscope (SEM), equipped with EDS for local chemical analysis.

Microstructure of Ni-P thin layers obtained by a chemical method

Katarzyna Konopka¹⁾, Witold Zieliński¹⁾, Dariusz Oleszak¹⁾, Anna Wyszynska¹⁾, Maria Trzaska¹⁾, Krzysztof Kurzydłowski¹⁾

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

The present paper is focused on the microstructure of the Ni-P thin layers crystallized from the amorphous state. The layers in this study have been produced by a chemical reduction method. They have been crystallized in order to obtain mixed amorphous-crystalline and polycrystalline microstructures. During the very early stages of crystallization there is possibility to produce nanocrystalline materials.

Three microstructures obtained by annealing at the temperatures 270 and 320°C, i.e. below the peak of crystallization (370°C) are described. Earlier results of the same authors showed that nanocrystals appear in Ni-P layers. The X-ray study, calorimetric analysis and microstructure observations carried out on transmission electron microscope in this study revealed the transition from amorphous to crystalline microstructure and changes in the morphology of grains. Crystals with different sizes have been observed, some of them in the nano-metres range. Some kind of correlation has been found between the P concentration in amorphous Ni-P material and the microstructure at the front of crystallization. The crystallization starts in the area exhibiting lower concentration of the phosphorous. The appeared crystals in this region have an elongated shape and form bands separated by amorphous material.

19:00 -
poster
G-24

Novel ceramic matrix composites with metal phase particles in the range from nano to micro - size

Katarzyna Konopka¹⁾

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland*

The present paper is focused on the cerami-metal composites obtained via different technologies which leads to different microstructures. Microstructures of composites differed in terms of size and distribution of metal phase.

Composites analysed in paper were produced by the following methods: (a) infiltration of porous ceramic by metal, (b) infiltration under high pressure and (c) sintering of ceramic powder coated by metal films. The microstructures were investigated using scanning and electron transmission microscopy.

Different methods of composite fabrication result in specific distribution and size of metal phase. Pressureless infiltration of porous ceramic by liquid metal is driven by capillary force and make possibility to produce microstructure with percolation of metal phase in ceramic matrix. The size of metal phase in composite depends on the size of pores. The size of pores influence also the process of infiltration. Since ceramic preforms with small size of pore are not fully infiltrated. This method is useful to produce composite with size of metal phase in the range of micrometers.

Infiltration under high pressure produces microstructure with metal phase in the range from nano to micrometers. Moreover, it allows to achieve the nanometrical size of ceramic grains.

In the case of ceramic powders covered by metal, compression and hot pressing preserves nanometrical size of metal. Moreover, the grain growth of ceramic grains is suppressed.

19:00 -
poster
G-25

The influence of nickel layer with nanosize grains on aluminium-ceramic

19:00 -
poster

interface structure**Anita Olszówka-Myalska¹⁾**

G-26

1) Silesian University of Technology, Department of Materials Science, Krasińskiego 8, Katowice 40-019, Poland

The structure of bonding between aluminium matrix and ceramic particles in composite materials obtained by hot pressing at the temperature range of 550-640°C in Degussa press was examined by SEM and TEM methods.

Alumina particles were coated with nickel layer by a chemical method before consolidation with aluminium matrix. The nickel layer observed by SEM consisted of three zones with different grain size of each zone. The zone directly on alumina was continuous and formed of nanograins (10nm and less).

The thermal behaviour of alumina particles coated with nickel and mixture of aluminium powder with alumina particles coated with nickel were examined by differential thermal analysis (DTA) using GDTD 16 analyser produced by SETARAM. The heating rate was set at 13°C and argon was flowed into processor. The exothermal peak considered with nickel layer and aluminium matrix interaction were noticed at the temperature above aluminium melt point.

After the hot pressing in vacuum of mixture of nickel coated alumina particles and aluminium powder, different structure of ceramic-metal bonding was observed in composite material with the temperature increase. The interface was characterised on polished samples by SEI and X-ray line scans. For the TEM investigation thin foils were prepared for observation in bright field, and microanalysis and electron diffraction were performed.

On the particle-metal border there were three processes noticed considered with nickel diffusion:

- a) dissolution of nickel layer in aluminium matrix;
- b) transformation of nickel layer into a nickel aluminides layer;
- c) formation of single particles from Al-Fe-Ni system.

The presence of particles from Al-Fe-Ni system on the interface can be explained as a result of iron impurity in aluminium powder of 99.7 % purity.

Al₂O₃-Fe functionally graded materials fabricated under magnetic field**Artur Oziebło¹⁾, Katarzyna Konopka¹⁾, Ewa Bobryk²⁾, Krzysztof Kurzydłowski¹⁾, Mikołaj Szafran²⁾**

19:00 -

poster

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

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

G-27

This paper describes a technology of obtaining the ceramic-metal composites with a gradient of metal particles concentration. Such composites combine properties of ceramics and metals, due to gradient of metal concentration it can be used in a wider range of applications both as a structural and functional material.

This paper includes the results of investigation of graded Al₂O₃-Fe composites, obtained by slip casting. The gradient of iron concentration was induced by magnetic field.

Microstructure, of the specimens have been investigated using a optical and scanning electron microscopy. Quantitative analysis of microstructures have been carried out with the help of image analyser. The obtained results prove the possibility to produce Al₂O₃-Fe functionally graded materials under the magnetic field.

The influence of temperature and pressure on possibility of obtaining Al₂O₃/Ni-P nanocomposite through hot pressing process.**Jakub Michalski²⁾, Katarzyna Konopka²⁾, Stanisław Gierlotka¹⁾, Krzysztof Kurzydłowski²⁾**

19:00 -

poster

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

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

G-28

The influence of temperature and pressure on Ni-P nanoparticles during sintering of Al₂O₃/Ni-P nanocomposites under high pressure was investigated in this paper. The fine-grained alumina powder was covered with Ni-P nanoparticles of 20-50 nm sizes by electroless nickel plating. The material was sintered in two temperatures: 900 and 1400°C, using pressures above 5 GPa. It was found that sintering in such conditions gave a possibility to maintains nanometrical size of Ni-P particles. In the case of 1400°C the metallic phase melted and non-uniform grain growth of ceramic was observed. Hot pressing at 900°C allowed for the metal to remain in a solid state and ensured a uniform microstructure of the nanocomposites, characterized by uniform distribution of nanometrical Ni-P grains in the ceramic matrix. In this temperature the grain growth of the ceramic was not observed. The results are discussed in terms of developing a technology for ceramic-metal composites in various applications.

Investigation of the microstructure of SiC-Zn nanocomposites by microscopic methods: SEM, AFM and TEM**Anna Swiderska-Sroda²⁾, Jan Kozubowski⁵⁾, Agnieszka Maranda-Niedbala³⁾, Ewa Grzanka^{1,2)},**

19:00 -

poster

G-30

Bogdan F. Palosz²⁾, Adam Presz²⁾, Stanislaw Gierlotka²⁾, Svetlana Stelmakh²⁾, Grzegorz Kalisz^{2,5)}, Christian Lathe⁴⁾

1) *University of Warsaw, Department of Physics, Hoza 69 Str., Warsaw 00-681, Poland*

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3) *Institute of Physical Chemistry Polish Academy of Sciences, Kasprzaka 44/52, Warsaw 01-224, Poland*

4) *GeoForschungsZentrum Potsdam, Telegrafenberg A17, Potsdam D-14473, Germany*

5) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

Bulk SiC-Zn nanocomposites with about 20% volume fraction of metal were fabricated through a high-pressure infiltration process under pressures in the range of 2-8 GPa and temperatures in the range of 400-1000°C. Ceramic powders used in the experiments were loosely agglomerated by Van der Waals forces. The dimensions of the agglomerates were in the micrometer range with individual particles 10-30 nm in diameter. Investigations of the microstructure were performed at different resolution levels using scanning, transmission, and atomic force microscopy techniques. The samples were mechanically polished and ion-etched. The observations of the mechanically polished surfaces show a nano-dispersed mixture of two phases with some zinc-rich regions. AFM shows a smooth surface with the roughness on the order of the SiC grain size (10-30 nm). After subsequent ion etching the SEM images of the samples show homogenous surface composition while AFM topographs indicate surface irregularities: periodically spaced 100 nm high hillocks. Apparently ion etching removes selectively the zinc-rich areas exposing inhomogeneity in the bulk of the material. Grain size, grain size distribution, and dispersion of the phases were investigated with TEM technique. The observations indicate a presence of homogeneous (on a sub-micron scale) distribution of two phases with parallel metal streaks across the sample volume. HRTEM images show thin metal layers separating SiC grains. The grain size of the ceramic component remains equal to that of the initial powder. The grain size of Zn was found to depend on the SiC particle size and the composite fabrication conditions.

Synthesis of metal-ceramic nanocomposites by high-pressure infiltration

Stanislaw Gierlotka²⁾, Bogdan F. Palosz²⁾, Anna Swiderska-Sroda²⁾, Ewa Grzanka^{1,2)}, Grzegorz Kalisz^{2,3)}, Svetlana Stelmakh²⁾

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2) *High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland*

3) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

Infiltration by a melt is an attractive but rarely utilized technique of synthesis of composite materials. When applying pressures in the GPa range the technique allows for a synthesis of multi-phase nanomaterials. The process is done in a toroid high-pressure high-temperature cell at pressures up to 7.7 GPa and temperatures up to 2000°C. The nanoporous matrix is prepared by compacting nanopowders of high-hardness materials such as Al₂O₃, SiC, or diamond. The infiltrating material can be any substance with the melting temperature (at a given pressure) below the processing temperature. When the infiltrant melts the pressure forces it to fill the pores in the matrix. The resulting composite contains a continuous network of solidified injected material with embedded grains of the ceramic powder. The crystallite size of the secondary phase depends on the grain size of the ceramic powder used and can be as low as 10 nm. The technique proved successful with metals (Mg, Sn, Zn, Al, Ag, Cu), semiconductors (Si, Ge, GaAs, CdTe), and ionic glasses as the injected material. Under certain p-T conditions the infiltrating material can chemically react with the matrix. In such cases the interfaces between the nanograins of the two components become chemically bonded which improves mechanical properties of the material. High-plasticity of some metals under pressure allows for their infiltration below the melting point. Depending on the matrix-infiltrant system and the processing conditions the technique may produce materials serving different objectives. The process can produce nanograins of a given material embedded in an ambient matrix. Such composites are used to study electrical, optical and magnetic properties of nanoparticles of metals and semiconductors. Chemically-bonded composites are primarily the subject of investigations of the effect of the crystallite size on the material mechanical properties.

19:00 -
poster
G-31

Micromechanical properties of nanometer-thick layers of implanted alumina.

Jacek Jagielski^{2,3)}, Anna Piatkowska³⁾, Zdzislaw Librant³⁾, Pascal Aubert⁴⁾, Caroline Legrand-Buscema⁴⁾, Jerzy Piekoszewski^{1,2)}, Zbigniew Werner²⁾

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One of the limitations of the use of ceramic materials for the construction of mechanical parts is their rather high friction coefficient. Taking into account that ceramics are used mainly in parts exposed to extreme working conditions (high temperatures, aggressive environment, vacuum etc.) the use of liquid lubricants for friction reduction is excluded. The method of choice is thus such modification of surface layer of ceramic which, keeping its chemical

19:00 -
poster
G-32

inertness and temperature resistance almost unchanged, allows one to effectively reduce the friction forces. In the presented work we examined two methods of friction reduction; (i) formation of thin surface layer of reduced hardness (to take advantage of Tabor and Bowden concept of adhesive friction in layered materials) and (ii) introduction of nanometer-sized precipitates of solid lubricants to the surface layer of material. The samples were implanted with various doses of inert ions (Ar or Kr) or with soft metal ions (In or Ag). Both methods lead to significant friction reduction, the best results being obtained for alumina implanted with In ions when the friction coefficient was reduced by a factor of about 3. The measurements of nanohardness revealed substantially lower hardness and very high plasticity of the irradiated layers.

The results obtained are analysed in terms of the level of radiation damage caused by ion implantation and the depth distribution profiles of implanted ions. The sample surface was inspected by Scanning Electron Microscopy (SEM) technique supplemented with the elemental analysis. The analysis of the experimental data allowed us to explain the obtained results within the frames of the adhesive friction theory.

Composition and mechanical properties of graded Ti-Cr-N coatings, deposited by CAVD

19:00 -
poster

Vladimir V. Uglov²⁾, Viktor M. Anishchik²⁾, Sergey V. Zlotski²⁾, Sergey N. Dub¹⁾, Viktor A. Ukhov³⁾

G-33

1) *V. Bakul Institute for Superhard Materials, NAS, 2 Avtozavodskaya, Kiev 254074, Ukraine*

2) *Belarusian State University (BSU), F. Skaryna av. 4, Minsk 220050, Belarus*

3) *Belmicrosystems, Korzhenevsky, Minsk 220064, Belarus*

The Ti-Cr-N coatings were formed by the method of condensation from a plasma phase in a vacuum with ion bombardment of sample surfaces with combined Ti and Cr plasma flows in a residual nitrogen atmosphere. Graded systems were deposited by varying arc currents of Ti and Cr cathodes. The thickness of formed coatings is about 0,5 mkm. The element and phase composition, nanohardness were studied by Auger electron spectroscopy (AES), X-ray diffraction analysis (XRD) and nanoindentation. AES investigations showed that the systems graded by composition and structure were formed: Cr bonding layer and the main layer of alternate composition in which the concentration of Ti and Cr is increasing or decreasing with changing the thickness of the layer. XRD measurements show that multiphase systems with the dominant nitride phases of Ti₂N and Cr₂N were formed. The nanohardness of Ti-Cr-N coatings depends on the concentration distribution of the main elements. The hardness of graded coatings (~ 37GPa) is larger than the hardness of the coatings with a constant composition (~ 24 GPa), which are formed under similar conditions. The interdependence of element and phase composition with mechanical properties of graded Ti-Cr-N coatings is discussed.

Amorphous bulk alloys from Al-Mm-Ni system

19:00 -
poster

Maciej Kowalczyk¹⁾, Tadeusz Kulik¹⁾, Jerzy Latuch¹⁾

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wotowska 141, Warsaw 02-507, Poland*

G-34

Recent studies have shown that amorphous alloys from Al-based ternary systems, containing lanthanide metal and late transition metal can be fabricated. Such materials are characterized by good mechanical properties. The most common way of production those alloys is melt spinning, giving a ribbon as a final product. It is possible to replace lanthanide metal by mischmetal. The mischmetal used in this study contains: Ce-50,3 % at., La-43,5 % at., Pr-5,9 % at., Nd-0,3 % at. This is a way to reduce the costs of the raw materials because Mm is several times cheaper than pure lanthanide elements.

This study has two main objectives. First, to check the possibility to replace yttrium in the Al-Y-Ni system alloys by mischmetal, without losing the structure and mechanical properties. Second goal was to produce the bulk amorphous material.

Several alloys from Al-Mm-Ni system, were investigated. The as quenched ribbons were milled to powder and then semi-isostatically compacted at elevated temperature to bulk material. After every step of this investigation, the XRD and DSC measurements were undertaken to distinguish eventual changes occurring during the process (eg. producing the ribbon, milling, compacting). Mechanical properties were characterized by Vickers microhardness.

The results of the studies show the possibility to produce bulk amorphous materials from the above mentioned system in three-step production cycle. The microhardness values are good or even better compared to Al-RE-Ni alloys. Microhardness depends not only on the chemical composition of the alloy but also on the temperature of the compacting process.

Phase transformations in steels subjected to ball milling

19:00 -
poster

Dariusz Oleszak¹⁾

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Wotowska 141, Warsaw 02-507, Poland*

G-35

As the most widely applied material in the world, steel has been well studied for over hundred years. Many processing methods have been applied to improve its structure and properties. To control the properties of steels, the

cold work such as rolling and drawing was performed together with other treatments. However, the grain size of such treated steels is generally at the range of micrometers. It is expected that further improvement of steels can be achieved by obtaining nanostructure by heavy cold work, e.g. ball milling of powders.

The present study is devoted to the obtaining nanocrystalline structure in ball milled powders of various steels. For carbon steels the formation of nanoferrite and dissolution of carbides was observed. The obtained nanoferrite was characterized by grain size, strain and lattice parameter calculations. In the case of austenitic stainless steel subjected to ball milling, strain-induced transformation leading to the martensite formation was observed. The thermal stability of the obtained nanostructures was also studied.

The phase transformation Co-Al alloys by ball milling and mechanical alloying

Kirill V. Tretjakov²⁾, Valeri K. Portnoy²⁾, Viktoria I. Fadeeva²⁾, Jerzy Latuch¹⁾

1) *Warsaw University of Technology, Faculty of Materials Science and Engineering, Włocławska 141, Warsaw 02-507, Poland*

2) *M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation*

19:00 -

poster

G-36

The structural transformation in pure cobalt and in powder mixture Co - 3, 10, 20 and 50 at. % Al during mechanical alloying (MA) were studied by X-ray diffraction and differential scanning calorimetry (DSC). Additionally, the influence of deformation under ball milling on cobalt modifications and on arc melted alloys with similar compositions were investigated.

The solid solutions Cofcc(Al) were formed during MA by diffusion mechanism. The CoAl of Co-50 at. % Al (type CsCl) composition was formed by explosive regime without the intermediate stage of chemical interactions.

Two cobalt modifications, i.e., face-center-cubic (fcc) and hexagonal close-packed (hcp) phases usually coexist at room temperature. It was found that initial stage of MA was accompanied by structural transformation of fcc cobalt into equilibrium hcp modification. The long milling lead to the solution of aluminum in cobalt and structural transformation of hcp Co to fcc Co (Al). The fcc Co structure of after following annealing in DSC up to 720°C is conserved.

The substructure parameters changes (block size and microstrain) and lattice parameters of fcc and hcp cobalt modifications were studied. The milling of CoAl + Al mixture leads to amorphous phase formation. The milling of mixture CoAl + Co results in the formation of fcc Co(Al) solid solution.

Besides, the evaluation of probability of stacking faults formation in Co and in Co(Al) solid solution were monitored. The phase transformation of Co (hcp to fcc) induced by ball milling was caused by the accumulation of structure defects (stacking fault).

Similar investigations of substructure evolution were carried out for equilibrium alloys with the same compositions during transformation into nanocrystalline state under the ball milling.

The temperature stability of MA alloys was studied by DSC method and annealing.

The work was supported by RFBR (projects 03-02-06043 and 02-02-16154).

The electrocrystallization of nanonickel

Benigna Szeptycka¹⁾, Anna Anna Gajewska-Midziałek¹⁾, Daniela Derewnicka¹⁾

1) *Institute of Precision Mechanics, Duchnicka 3, Warsaw 01-796, Poland*

19:00 -

poster

G-37

The micro- and nanostructure, microhardness and wear resistance was investigated for d.c. - plated Ni electrodeposits prepared from high- and low-concentrated Watts electrolytes, also with KCl instead of NiCl₂, without or with organic compounds on Cu substrate, which was subsequently removed from the deposits. Increase of current density and temperature caused increase of grain dimensions and decrease of wear resistance. With the change of the type of bath, particularly with organic compounds, the deposit grain size was found to decrease markedly, until obtain of deposits with an average grain size of about 20 and 50 nm. In this set of investigations the wear resistance decreased with decreasing grain size. In discussing the factors controlling the deposit grain size, it is concluded that content in the bath of the organic compounds may have a decisive role.

Wettability and reactivity between molten aluminum and selected oxides

Natalia SOB CZAK¹⁾

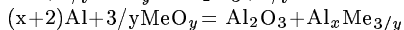
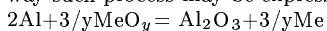
1) *Foundry Research Institute (FRI), 73 Zakopianska Street, Cracow 30-418, Poland*

19:00 -

poster

G-38

Nanostructured composites and functionally graded materials based on Al-Al₂O₃ or Al_xMe_y-Al₂O₃ systems can be produced in situ by liquid phase processing due to reacting molten aluminum with reactive oxide. In general way such process may be expressed by the following oxy-redox reactions:



where MeO_y is an oxide, which is reactive to Al, and according to widely accepted opinion it should be wettable by molten Al; Al_xMe_{3/y} is intermetallic phase.

The paper presents the experimental results on wettability and reactivity in different couples with simple oxide (Al/SiO₂, Al/ZnO, Al/B₂O₃, Al/TiO₂, Al/ZrO₂, Al/CoO, Al/NiO), binary oxides of SiO₂-Al₂O₃ system and complex oxides of SiO₂-Al₂O₃-Fe₂O₃-TiO₂-CaO-MgO type.

The analysis of factors affecting wetting and transformation kinetics in Al/reactive oxide couples has been done taking into account structural, physical and mechanical properties of initial oxide reactant and morphology of resulting microstructures.

Analysis of Nanometals' Morphology by Scanning Probe Microscopy

19:00 - **Alexei N. Lachinov²⁾, Victor M. Kornilov²⁾, Rafael M. Mazitov²⁾, Igor V. Alexandrov¹⁾, Ruslan Valiev¹⁾**
poster

G-40

1) *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation*

2) *Institute of Molecules and Crystal Physics, Ufa Scientific Centre of Russian Academy of Sciences, 151 Prospekt Oktyabrya, UFA 450075, Russian Federation*

The surface morphology of six metals and alloys were studied using scanning tunneling microscopy technique. Cu, Ni, Ti, Ni₃Al, TiNi were subjects for investigations. All samples were subjected to severe plastic deformation (SPD) by the torsion straining technique under various conditions. At that, hydrostatic pressure reached 6 GPa, and number of turns was up to 5. Scanning tunneling microscopy measurements were performed at room temperature and on the air before and after SPD. The development of the ultrafine grain structure as a result of SPD was observed. This result was confirmed by x-ray diffraction investigations. All samples showed very complicated morphology of metal surfaces depending from SPD conditions. The evolution of the nanograins' morphology was studied as a function of annealing conditions as well.

Multi-layer composite based on amorphous materials and quasicrystals, deposited by laser ablation

19:00 -

poster

G-41

Waldemar Mroz²⁾, Miroslaw Jelinek⁵⁾, Lukasz Major¹⁾, Jerzy Morgiel¹⁾, Artur Prokopiuk²⁾, Michal Wozniak³⁾, Koichi Kasuya⁴⁾

1) *Institute of Metallurgy and Materials Sciences, PAS (IMIM PAN), Reymonta 25, Krakow 30-059, Poland*

2) *Institute of Optoelectronic Military University of Technology (MUT), Woloska, Warsaw 02-507, Poland*

3) *Faculty of Material Sci. Eng., Warsaw Univ. Technology, Warsaw, Poland*

4) *Department of Energy Science, Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, Midori-ku, Yokohama 226, Nagatsuta 4259, Japan*

5) *Institute of Physics, Academy of Sciences of the Czech Republic, Na Slovance 2, Prague 182-21, Czech Republic*

The PLD method allows to produce multi-layers materials. Interest in pulsed laser deposition (PLD), as a technology producing thin layers of complex materials is growing rapidly in recent years. Condensation from the vapor phase at rapid solidification favors the nano-structure formation in the thin layers.

First attempts have been made to produce multi-layers composite. Amorphous material is treated as a matrix. Materials, which were used for coatings, were:

- amorphous material: Ti25at.% Zr20at.% Ni27.5at.% Cu27.5at.%,

- quasicrystals: Al65at.%Cu23at.%Fe12at.%.

Amorphous materials have high elasticity and in the compression test they exhibit plastic features. This is the reason that these materials are used as a matrix.

As a hard phase, quasicrystals have been deposited. Quasicrystals have a periodic structure, however they have strong diffraction peaks. They are characterized by icosahedral symmetry (5- rotation axis symmetry).

Amorphous and quasicrystalline thin layers were fabricated by pulsed laser deposition (PLD) using an excimer, ArF laser (Lambda = 193 nm). Scanning electron microscopy (SEM) and atomic force microscopy (AFM) observations have been done to examine the topography and chemical composition of coatings. Texture analyses were carried out for the deposited amorphous as well as for the quasicrystalline substrate. Application of the pseudo-position sensitive detector in texture examination allows drawing the pole figures of residual stresses.

Production of nano-wires on semiconductor surfaces by ion bombardment

19:00 -

poster

G-42

Johan B. Malherbe¹⁾, Herbert W. Kunert¹⁾

1) *Department of Physics. University of Pretoria., University str., PRETORIA 0084, South Africa*

Low energy (typically in the keV regime) ion bombardment of some semiconductor materials can lead to the development of regularly spaced ripples on the surfaces, which with suitable doping procedures can be employed as nano-wires. Although these ripples are reminiscent of those induced by the action of wind on sand dunes or by water currents on the seabed, the mechanisms are different. The formation of the bombardment-induced ripples

depends primarily on the material substrate. The average distance between the ripples, i.e. ripple wavelength is also a function of ions species, ion energy, angle of incidence, ion areaic dose and substrate temperature. In this paper, some of the reported results on the ripples produced by noble gas, oxygen and nitrogen ion bombardment on selected semiconductor materials will be tested against Bradley-Harper theory. It will be shown that some of the ripple properties can be explained by this analytic theory. For the others, one will have to resort to the more advances theories, which can only be solved numerically.

Kinetic behaviour of free and localized on dislocations nano-scale liquid Pb inclusions in aluminum

19:00 -

Sergei Prokofjev^{1,3)}, Victor M. Zhilin¹⁾, Erik Johnson^{2,3)}, Mogens T. Levinsen⁴⁾, Ulrich Dahmen⁵⁾

poster

1) *Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka, Mosco 142432, Russian Federation*

G-43

2) *Riso National Laboratory, Materials Research Department, Roskilde, Denmark*

3) *Oersted Laboratory, Niels Bohr Institute, Copenhagen University, Copenhagen, Denmark*

4) *Niels Bohr Institute, Copenhagen University, Copenhagen, Denmark*

5) *National Center of Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, United States*

A kinetic behaviour of nanosized liquid Pb inclusions in crystalline aluminum was investigated in-situ using transmission electron microscopy (TEM). The studies are carried out at 720 K. The observations show that initially all Pb inclusions in crystalline Al are trapped by dislocations. Their motion is spatially confined by the field in the close proximity of the dislocations. They show 1D random motion along the dislocations and 2D chaotic vibrations in transversal plane. Eventually the inclusions detach from the dislocations, and move in free random mode for some time before their disappearance on free surface or coalescence with other inclusions.

The transversal vibrations of the inclusions are due to the effect of dislocation which operates like an elastic string. The later is confirm by the agreement of the rigidity of the dislocation found experimentally with an estimation based on the elastic model of the dislocation. This model is supported by observations of repulsion of inclusions trapped by one dislocation one from other and from fixed ends of the dislocation that is due to their non-correlated vibrations in a transversal plane.

To determine the diffusion coefficients of the free inclusions the Einstein equation is used. The dependence of the diffusion coefficient on their size indicates kinetic processes on Pb/Al interface control their mobility. It is shown that the Einstein equation can be applied correctly to determine the diffusion coefficients of longitudinal motion of trapped inclusions. A comparison of the diffusion coefficients obtained of the free and trapped inclusions indicates the same underlying microscopic mechanism and the small effect (in contract to that of grain boundaries) of the dislocation. The mobility of Pb inclusions is likely controlled by diffusion along their surface, if the diffusion regime holds, or by the process of dissolution of Al-matrix crystal at the Al/Pb interface in the kinetic regime.

Tuesday, September 16th

Morning session

11:00 - 12:30

Developing a Superplastic Forming Capability in Nanometals

Cheng Xu¹⁾, Minoru Furukawa²⁾, Zenji Horita³⁾, Terence G. Langdon¹⁾

11:00 - 11:30

1) *Departments of Aerospace & Mechanical Engineering and Materials Science University of Southern California, Los Angeles, United States*

invited oral

2) *Department of Technology, Fukuoka University of Education, Munakata, Fukuoka 811-4192, Japan*

3) *Department of Materials Science and Engineering, Faculty of Engineering, Kyushu University, Fukuoka 812-8581, Japan*

Equal-Channel Angular Pressing (ECAP) is a processing procedure in which a sample is pressed through a die constrained within a channel that is bent through an abrupt angle generally equal to, or very close to, 90°. An extensive set of experimental data has now established that ECAP processing is remarkably successful in significantly refining the grain size of metallic alloys to the submicrometer or nanometer range. Additional advantages of ECAP processing include the ability to produce large bulk samples containing no residual porosity and a potential for varying the processing conditions in order to produce samples having different distributions of the grain boundary misorientations. This paper describes the ECAP technique and other similar processing procedures and shows that processing in this way provides a potential for achieving high superplastic ductilities at elevated temperatures. In addition, since these high ductilities are achieved at exceptionally rapid strain rates, typically about two orders of magnitude faster than in conventional superplasticity, there is a potential for making use of these materials

in industrial superplastic forming operations. Examples are presented both for special alloys produced in the laboratory and for alloys available from commercial sources.

11:30 - 12:00

oral

Grain Boundary Microstructural Control in Nanocrystalline Nickel by Magnetic Annealing

Tadao Watanabe¹⁾, Tsurekawa Sadahiro¹⁾, Palumbo Gino²⁾

1) Graduate School of Engineering Tohoku University, Department of Nanomechanics, Sendai 980-8579, Japan

2) Integran Technology Inc., Toronto, Canada

How to improve the thermal and microstructural stability in nanocrystalline materials is a key subject for development of high performance and more reliable nanocrystalline advanced materials. It is well known that abnormal grain growth can easily take place and produce the heterogeneity of grain boundary microstructure in nanocrystalline materials. Most recently, it has been found that the annealing in a magnetic field can restrict abnormal grain growth, resulting in the formation of homogeneous grain structure in nanocrystalline nickel samples whose initial grain size was about 30nm, produced by electrolytic deposition. The grain boundary microstructures in magnetically annealed nickel specimens were analyzed by the orientation imaging microscopy (OIM) with a FE-SEM-EBSP system. It has been found that the thermal and microstructural stability can be improved drastically by magnetic annealing. The DSC analyses were also made to reveal the origin of the improvement of thermal stability of the grain boundary microstructures in nanocrystalline nickel. The result of DSC analyses for annealed in a magnetic field suggests a change of the grain boundary energy during annealing in a magnetic field. A new approach to the grain boundary engineering by the application of a magnetic field is demonstrated to be unique and promising to design and development of high performance and thermally stable advanced nanocrystalline materials.

12:00 - 12:15

oral

Grain growth in nanocrystals: effect of triple junction mobility

Vladimir Y. Novikov¹⁾

1) Moscow Inst. of Steel & Alloys (MISA), Treptower Str. 74 d, Hamburg 22147, Germany

Thermal stability of microstructure is of crucial importance for nanocrystalline materials (NM). Grain growth in NM is usually inhibited, apparently by nano- and micropores, second phase particles, and segregations as well as by vacancy redistribution accompanying a decrease in the specific area of grain boundaries (GB). Since triple junctions can also impede the movement of GB, their decreased mobility should affect the growth of an individual crystallite provided the distance between the junctions is small, which is the case in NM.

In this work, the effect of triple junctions on grain growth in two-dimensional polycrystals was investigated by computer simulation. The energy and mobility of all the GB were assumed identical, the mobility of triple junctions being supposed the same but different from that of GB. Parameter L accounting for the effect of triple junctions is the distance between the neighbouring junctions along the moving GB multiplied by the mobility ratio (MR) of the triple junction and the GB. As the grains in a polycrystal are adjacent to their neighbours of different sizes, L at a given MR is different for different grain pairs and varies in a wide range depending on the width of grain size distribution.

It was shown that in polycrystals with the initial $L < 300$, grain growth is retarded in comparison to the case that its kinetics is controlled by the GB mobility only. In a polycrystal with the initial $L < 0.3$, the time dependence of the average grain size becomes linear at the initial stage. A reduction in MR (and, thus in L) increases the duration of the linear stage and decreases its slope. The simulation results are in agreement with some experimental data. So it can be supposed that this is a result of a decreased mobility of triple junctions.

12:15 - 12:30

oral

Bulk nanostructured TiNi shape-memory alloys: unusual structures and unique properties

V. G. Pushin²⁾, Ruslan Valiev¹⁾

1) Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation

2) Institute of Metal Physics, Ural Division of Russian Academy of Sciences, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation

We presented here a new shape memory materials based on alloys of four practically most important systems, namely, Ti-Ni, Ti-Ni-Fe, Ti-Ni-Co and Ti-Ni-Cu. For the first time, high-strength nanostructured shape-memory TiNi-based alloys were obtained using several different methods of severe plastic deformation (SPD), including multiple torsion straining under high pressure, multiple equal-channel angular pressing, multiple rolling and drawing, and melt spinning (MS). The formation of amorphous, nano- and submicron-grained crystalline structures was shown to be possible upon cold SPD and superrapid MS-quenching. It was established that a special place among these techniques belongs to the process of controlled nanocrystallization during annealing of amorphous alloys.

The main characteristic features of physical and mechanical properties, including shape-memory effects in nanostructured alloys, have been analysed in comparison with those for usual course-grained TiNi-based alloys. Some surprising results (unusual structures, and their influence on MT, mechanical properties and unique shape-memory

effects) are discussed.

Lunch break	12:30 - 14:00
Afternoon session	14:00 - 15:30
Recent progress in developing nanostructured SPD materials with unique properties Ruslan Z. Valiev¹⁾ <i>1) Institute of Physics of Advanced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation</i>	14:00 - 14:30 invited oral
Effect of Non-conventional Large Deformations on Microstructure and Properties of Metals Krzysztof Kurzydłowski²⁾, Maria Richert¹⁾, Jan Richert¹⁾, Józef Zasadziński¹⁾, Malgorzata Sus-Ryszkowska²⁾ <i>1) University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland</i> <i>2) Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, Warsaw 02-507, Poland</i>	14:30 - 14:45 oral
"Perspective of nanomaterials production by cyclic extrusion compression method of exerting unconventional, large plastic deformations." Maria Richert¹⁾, Sonia Hawrylkiewicz¹⁾, Jan Richert¹⁾, Józef Zasadziński¹⁾ <i>1) University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland</i>	14:45 - 15:00 oral
Aluminium and copper monocrystals and also AlMg5 and AlCu4Zr alloys have been deformed by the cyclic extrusion compression method (CEC) in the range of true strains $j = 0,4 - 14$. It has been found that about 50% of the volume fraction of aluminium alloys samples, deformed to the true strain $j = 14$, shows features of nanostructure. This kind of microstructure consists of nanograins with the average dimension of about 150 - 200 nm, having large misorientation. The investigations indicate that mutually crossing of microbands is the main mechanism of formation of nanograins. In the areas of mutually crossing microbands, the nanograins have been formed, showing large misorientation angles. In aluminium and copper monocrystals also about 50% of investigation subgrains shown large misorientation, however their size was much larger than the typical nanograin size. In aluminium monocrystals it showed dimension of about 600 nm. In copper monocrystals subgrains had average dimension of about 200 nm and were more closed to the typical nanosize grains. The aluminium alloys, deformed to the true strain of about 14 revealed very high level of microhardness (about 100 mHV), comparabled with the level of properties, characteristic for nanomaterials produced by other methods. The level of microhardness of copper monocrystals after the 14th of true strain by the CEC method, was comparabled to the microhardness of copper obtained by the condensation - evaporation method. The obtained results indicate that using the CEC method it is possible to produce the bulk nanomaterials, especially from the aluminium alloys, which reveal the unconventional large level of mechanical properties.	
Experimental studies and modeling of SPD materials Igor V. Alexandrov¹⁾	15:00 - 15:15 oral

1) *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks St.12, UFA 450000, Russian Federation*

Recent results of experimental studies and computer modeling of severely plastically deformed (SPD) pure metals are presented. Various structural levels are considered. The questions related to the influence of the high-pressure torsion and equal channel angular pressing parameters on the structure evolution and nanostructured states development at SPD are discussed. Specific features of the developed nanostructures and novel properties of the SPD processed bulk metals are analyzed.

15:15 - 15:30

oral

The peculiarities of macrostructural evolution of Fe-Mn alloys after severe plastic deformation under pressure

Sergei V. Gladkovsky³, Boris M. Efros¹, V. P. Pilyugin²

1) *Donetsk Technical-Physical Institute NASU, R. Luxemburg 72, Donetsk 83114, Ukraine*

2) *Institute of Metal Physics, Ural Division of Russian Academy of Sciences, 18 S.Kovalevskaya str., GSP-170, Ekaterinburg 620219, Russian Federation*

3) *Ekaterinburg, Urals State Technical University, 19, Mira srteet, Ekaterinburg 620002, Russian Federation*

Influence of severe plastic deformation (SPD) under high pressure on martensitic phase transformations and hardening of Fe-Mn alloys has been studied. The named alloys with wide concentration content of manganese (from 0 to 55 wt.%), having in initial state bcc, hcp and fcc (stable and metastable austenite) phases after thermoplastic treatment and quenching from 1370 K in water were as the objects under investigation. SPD under high and ultra-high pressure was performed by the means of hydroextrusion techniques, torsional shear under pressure and usage of diamond anvil cell (DAC). The ranges of pressure varies from 0 to 60 GPa and the deformation degree does not exceed 10 units of true deformation, accordingly.

The influence of manganese contents (8-20-26-40 wt.%) and hydroextrusion parameters on $\gamma\gamma$

The complex study of properties and structures of Fe-Mn-alloys, deformed in DAC, including the nanocrystalline structures, by methods of microhardness measurements, transmission electron microscopy, X-ray diffraction analysis), Moessbauer effect and metallography has been carried out. The obtained results of complex investigation of studied alloys at nanocrystalline state have revealed the strong dependence of phase solid transformations on parameters of SPD under pressure and initial phase, structure state and concentration of Mn at alloys.

15:30 - 15:50

Coffee break

15:50 - 17:20

Afternoon session - continued

15:50 - 16:20

invited oral

Processing metals by severe plastic deformation

Andrzej Rosochowski¹

1) *University of Strathclyde (Strath), 75 montrose Street, Glasgow G1 1XJ, United Kingdom*

Severe plastic deformation (SPD) is one of the methods of obtaining sub-microcrystalline structure in different crystallographic systems (e.g. aluminium, iron, magnesium). SPD causes the creation of micrometer and sub-micrometer sized subgrains in the original coarse grains of the material. The mechanism responsible for this effect is still under investigation, however, it is believed that short and long-range intersecting shear bands, produced by plastic deformation, play a major role. Sufficiently large deformation leads to a distinct structure of dislocation-free and highly misoriented fine grains.

The structural changes caused by SPD are reflected in improved mechanical properties of metals. The reported effects include increased hardness and yield stress, both featuring tendency to saturation. Other research revealed increased ductility and toughness as well as improved damping and magnetic properties. The fine grain structure leads to superplastic behaviour of materials at lower temperatures and yet with higher deformation rates than for other superplastic materials.

Various aspects of structural changes caused by SPD have been the main research topic so far. Less attention has been paid to the mechanics of material flow and practical aspects of SPD. Anticipating commercialisation attempts, this work addresses the processing issues. First, a brief review of SPD processes will be undertaken. Next, the three most popular SPD processes of high pressure torsion (HIT), cyclic extrusion compression (CEC) and equal channel angular pressing (ECAP) will be analysed in more detail. This analysis will include strain distribution and the role of hydrostatic pressure and friction. These, in turn, will be used to address tooling concerns. Appropriate billets and feeding systems will also be considered. Survey of possible applications will show the routs of commercialisation. Finally, future developments of SPD will be suggested.

16:20 - 16:35

oral

Computer simulation of SPD processes

Alexander V. Spuskanyuk¹⁾

1) *Donetsk Technical-Physical Institute NASU, R. Luxemburg 72, Donetsk 83114, Ukraine*

Computer simulation has been engaged to investigate the peculiarities of new severe plastic deformation processes: twist extrusion and equal channel multi-angular extrusion. During the twist extrusion, a workpiece is extruded through the helical die with prismatic input and calibrating output channels. The unique feature of this method is the twisting deformation in the beginning and in the end of helical die. Equal channel multiangle extrusion is performed by extruding a workpiece through the constant cross-section channel with several changes of metal flowing direction, i.e. with several zones of localized severe shear deformation.

Computer simulation of the deformation and structure forming has been performed by combined use of FEM modeling for detailed study of the stressed-strained state evolution and original micromechanical multiscale (macro-, meso- and microlevels) computer model of polycrystalline aggregate to study the microstructure modification with emphasis on grain refinement. The latter treats the material representative volume element as a multilevel system of elements with self-similar structure. Deformation of this system is numerically investigated within the framework of self-consistent field by means of cellular automata approach.

Correct correspondence of the simulation results with the experimental data for number of materials has allowed predicting of material structure and properties' evolution during processing, which made it possible to control and enhance the listed technologies.

New possibilities of achieving ultra - fine grained microstructure in metals and alloys employing MaxStrain technology

16:35 - 16:50

Roman M. Kuziak²⁾, Wladyslaw Zalecki²⁾, Maciej Pietrzyk¹⁾, Stanisław Węglarczyk¹⁾

oral

1) *University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland*

2) *Institute for Ferrous Metallurgy (IMZ), Karola Miarki, Gliwice 44-100, Poland*

In the paper, the deformation experiments conducted on the MaxStrain system aimed at achieving ultra fine grained structure in selected metals and alloys are described. The MaxStrain system provides an alternative way of developing such a structure compared to other methods that involve the effect of severe plastic deformation on grain refinement in bulk samples, such as the equal channel angular pressing. The part of the sample (10 mm in length) is compressed by two synchronised anvils to maintain its integrity. The sample is rotated by 90 degrees between the consecutive deformations, and it may be allowed to expand along its axis during deformation or may be restrained at the ends requiring the lateral deformation in the deformation zone. In this way, a total strain of 20 can be accumulated in the structure causing a substantial grain refinement.

In the experiments, the samples were cold deformed 10, 20 and 40 times with constant strain (0.2, 0.4 and 0.6) for all deformations. Constant strain rate of 1/s was applied for all deformation schedules.

The deformed structure of the samples was investigated by means of the Light and Transmission Electron Microscopy. Also, the hardness measurement in the deformed zone was conducted. The mean grain size of the samples was around 150 nm, and the hardness tripled in the samples compared to the annealed characterised by a large grain size material.

The microstructure and hardness of the samples deformed employing MaxStrain system are comparable to the ones obtained in the samples deformed using equal channel angular pressing. However, the MaxStrain technology is more flexible compared to the other methods of plastic working in terms of the deformation parameters. Also, MaxStrain system allows sophisticated heat treatment operations to be applied during and after the deformation sequence.

Twist extrusion: theory and technique

16:50 - 17:05

Dmitry Orlov¹⁾, Yan Y. Beygelzimer¹⁾, Sergey Synkov¹⁾, Alexey Reshetov¹⁾

oral

1) *Donetsk Phys.&Tech. Institute of the NAS of Ukraine (DonPhTI), R.Luxemburg, Donetsk 83114, Ukraine*

Twist Extrusion (TE) is a new effective technique of severe plastic deformations (SPD). The SPD allows to accumulate high strain degrees in workpieces and obtain materials with ultrafine grained structure. The latter attract high interest of investigators working in the field of materials science, in particular, due combination of high strength

and ductility.

It is analyzed technical aspects of workpieces' treatment by Twist Extrusion (TE) in the report. It is shown relations for determination of basic parameters of the process.

In previous our works basically was discussed peculiarities of deformation during twist extrusion, investigated mechanics of the process, analyzed structures obtained. In this report we put your attention on technology aspects of the twist extrusion. Also, we show engineering relations for basic TE parameters determination.

It is described installation for both cold and warm TE, showed results of experimental investigations of influence of twist extrusion on structure and properties of some materials.

17:05 - 17:20 **SPD by twist extrusion and spread extrusion**

oral **Yan Beygelzimer¹⁾**

1) Donetsk Phys.&Tech. Institute of the NAS of Ukraine (DonPhTI), R.Luxemburg, Donetsk 83114, Ukraine

One of the most effective methods for obtaining bulk ultrafine-grained materials is severe plastic deformation that is typically done using equal channel angular extrusion. In this work I describe and analyze new methods for obtaining severe plastic deformations proposed by mine. These methods is based on direct extrusion and was called "Twist Extrusion" (TE) and "Spread Extrusion" (SE). The first one is basically realize simple shear and the second one is pure shear.

The main idea of the TE is to extrude a prism bulk through a die with a twist channel. The channel cross-section that is orthogonal to the extrusion axis is constant along this axis. In the general case, it is possible to have channels of quite arbitrary cross-section.

The main idea of the SE is to extrude a prism bulk through a die with a spread-shrink channel. The channels cross-sectional area that is orthogonal to the extrusion axis is constant along this axis.

I argue that in some cases both these methods and their combinations have certain advantages.

Thursday, September 18th

09:00 - 10:30 **Morning session**

9:00 - 9:15 **Nanostructured high-strength aluminum alloys containing nano-size quasicrystalline particles**

oral **Yu. V. Milman¹⁾**

1) Institute for Problems of Materials Science, 3, Krzhizhanovsky, Kiev 03142, Ukraine

Nanostructured aluminum-based alloys of Al-Fe-Cr-Ti system containing quasicrystalline nano-size particles as a reinforcing phase were produced in the form of powder or ribbons by water atomization or melt spinning techniques, respectively. Rods were compacted from the powders and some ribbons, and their mechanical properties were determined. Microstructures and phases in powders, ribbons and compacted rods were investigated using TEM and X-ray diffraction. Several alloys produced had high strength (about 300 MPa) at temperatures up to 300 °C. The quasicrystalline particles were stable for coarsening and decomposition at temperatures up to 400 °C.

It was shown that quasicrystalline particles of i-phase 50 - 100 nm in diameter provide effective reinforcement for Al alloys. A process of water atomization may be used as effective method for producing powders of Al alloys containing quasicrystalline i-phase. Rods containing of nanostructured aluminum matrix and quasicrystalline nano-size particles may be obtained from these powders after compacting (extrusion, forging etc.).

Small additions of Sc to alloys of AlFeTiCr, AlCrCo and AlCrCeCo (which contain i-phase) result in increasing hardness.

X-ray line broadening analysis has shown that significant phonon and phason distortions occur in i-phase particles of AlFeCrTi ribbons, which correspond to the presence of defects in these particles as observed in TEM dark field images. The concentration of defects in nano-size particles of the i-phase in Al matrix is appreciably higher than in the i-phase of a model alloy that corresponds to the stoichiometric concentration of the i-phase.

9:15 - 9:30 **Nanocrystallization and mechanical properties of the amorphous Ni-Zr-Ti -Al-Cu -Si ribbons.**

oral

Tomasz Czeppe¹⁾, E. Vasileva¹⁾, J. Dutkiewicz¹⁾

1) Institute of Metallurgy and Materials Sciences, Polish Academy of Sciences, Reymonta 25 St, Krakow, Poland

In the paper the results concerning ribbons produced by the melt spinning on the rotating disc from the Ni₅₆Zr₁₈Ti₁₆Al₃Cu₂Si₅ alloy are presented. The ribbons were obtained at two different circumferential velocities and both revealed amorphous structure, what was confirmed by X-ray and the transmission electron microscopy (TEM) analysis. Differential scanning calorimetry (DSC) and differential thermal analysis (DTA) were used to determine basic parameters like glass transition temperature T_g , the onset of the primary crystallization T_{x1} and liquidus temperature T_{lq} . The range of the supercooled liquid $\Delta T_x = T_{x1} - T_g$ was determined as well as the reduced glass transition temperature $T_g/T_{lq} = 0.638$, related to the GFA. The DSC was also used to determine the different stages of the isothermal crystallization of the ribbons and the activation energy for the crystallisation. The microstructure after nanocrystallization was observed using TEM and X-ray diffraction. The structure of ribbons was amorphous below T_g temperature. It was investigated after isothermal crystallization at temperatures: 560, 570, 580, 590 and 600°C. It was concluded that in agreement with works on NiTiZrSi alloys, the first crystallizing phase is the B2 NiTi(Zr). The average size of crystallites was 15 nm in diameter after isothermal crystallization during 20 min at 600°C. It was observed that even small particles of this phase might transform to martensite. The microhardness of the ribbons was measured at room temperature after different stages of the isothermal crystallization. It was found that the room temperature microhardness essentially increases during nanocrystallisation. The increase of the grains from nano- to the microcrystalline size has decreased microhardness down to about 1100 μ HV.

Microacoustical study of elastic properties and microstructure of new generation of bulk metallic glasses

9:30 - 9:45

Vadim M. Levin¹⁾, Yulia S. Petronyuk¹⁾, Nikolai P. Kobelev²⁾

oral

1) Institute of Biochemical Physics RAS (IBCP), Kosygin 4, Moscow 119991, Russian Federation

2) Chernogolovka, Moscow region, Institute of Solid State Physics, RAS, Russian Federation

Ultrasonic measurements of elastic properties are traditional for studying structural transitions in metallic glasses. In last few years glassy state was obtained for massive specimens of a new generation of multicomponent alloys - bulk metallic glass (BMG) of Pd-Ni-Cu-P and Zr-Ti-Cu-Ni-Be systems.

We have applied the microacoustical technique for measuring local elastic properties and imaging internal microstructure of BMG samples. Short probing pulses of focused high-frequency ultrasound penetrate into the specimen as convergent beams of longitudinal and transverse waves. Echo pulses caused by reflection at the specimen face and backside are employed for measuring local values of sound velocities, both longitudinal and transverse ones. Echo signals reflected from details inside the specimen are employed for imaging its bulk microstructure. The BMG specimens were produced by the quenching method. Different states (glassy state, super-cooled liquid (SCL) and crystallized states) were obtained by annealing of the samples under normal and high pressure and at different temperatures near the glass transition and onset crystallization. Local values of longitudinal and transverse wave velocities and local elastic properties (bulk and shear moduli; Poisson coefficient) have been measured for an as-quenching specimen and for samples annealed at different temperature. Decreasing the longitudinal wave velocity (~6%) and bulk elastic modulus (~20%) has been observed in transition from the glassy state to SCL. High attenuation was observed in the crystallized BMG sample. Acoustic images (C-scans) evidence that coarse grain microstructure is inherent to BMG sample at this state.

Microstructure selection in severely deformed Al-base alloys

9:45 - 10:00

Gerhard Wilde¹⁾, Guru Prasad Dinda¹⁾, Rainer Hebert²⁾, John H. Perepezko²⁾

oral

1) Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany

2) University of Wisconsin-Madison, Department of Materials Science and Engineering, 1509 University Avenue, Madison WI 53706, United States

Many Aluminum base glass forming alloys require extremely high cooling rates for vitrification and often do not display a clear glass transition signal upon reheating. This marginal glass formation behavior is related mainly to high nucleation rates coupled with growth limitations that prevent a complete crystallization during quenching. The same kinetic control also provides the basis for the development of a high number density (10^{22} m^{-3}) of nanocrystals (diameter about 10-20 nm) during a primary crystallization reaction - the so-called nanocrystallization - upon reheating. With alternative synthesis routes based upon solid state alloying resulting from severe plastic deformation of initially crystalline multilayers, e.g. by repeated cold rolling and folding, the kinetic pathways to glass formation can be altered to avoid nanocrystallization reactions in the marginal glass forming alloys. In other systems, a deformation-induced nanocrystal synthesis can be observed during repeated cold rolling of amorphous ribbons. The microstructure selection in dependence of the processing pathway was monitored by X-ray diffraction and electron microscopy techniques. Additionally, modulated-temperature calorimetry has been used to detect the calorimetric signature of the glass transition on melt-quenched samples directly. The results are discussed with respect to the origin of the nanocrystallization, especially the formation of nanocrystal precursors during rapid melt quenching. Moreover, the development of the microstructure during cold-rolling and the comparison of the compositional ranges that allow the synthesis of vitreous product structures by severe plastic deformation or rapid

quenching are analyzed in terms of the major thermodynamic - and mechanical properties that govern intermixing during the deformation process. The support of the DFG (WI 1899/1-2) is gratefully acknowledged.

10:00 - 10:15

oral

Nanocrystallisation in light-weight amorphous alloys

Shantanu V. Madge²⁾, Nancy Boucharat²⁾, A. Lindsay Greer¹⁾, Gerhard Wilde²⁾

1) *University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, United Kingdom*

2) *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany*

Al-base marginally glass-forming systems such as $\text{Al}_{92}\text{Sm}_8$ and $\text{Al}_{88}\text{Fe}_5\text{Y}_7$ that have been vitrified by rapid quenching often crystallize via the initial formation of large number densities of Al-nanocrystals at temperatures that are below the respective glass transition. Calorimetric investigations and quantitative TEM analyses concerning the so-called nanocrystallization reaction indicate that the primary transformation results from diffusion-limited growth of quenched-in nuclei. Quite surprisingly, some easy glass-forming alloy systems that permit the synthesis of vitreous bulk samples by melt-quenching, such as $\text{Mg}_{65}\text{Cu}_{25}\text{Y}_{10}$, also form comparably large number densities of nanocrystals during the initial stages of the devitrification process. However, as opposed to the marginally glass-forming Al-base alloys, the nanocrystal formation in bulk glass-forming alloys occurs in the undercooled melt, i.e. at temperatures that are above the respective glass transition temperatures. Here, we compare the nanocrystal formation process and its kinetics for different groups of alloy systems in order to analyze the mechanism and the major determining parameters that govern the phase formation sequence and the nanoscaled microstructure evolution in light-metal base glass-forming alloys.

10:15 - 10:30

oral

Thermally Controlled Nanocrystallization in Amorphous Al Alloys

Joe Hamann²⁾, William S. Tong²⁾, Harald Rösner¹⁾, John H. Perepezko²⁾, Gerhard Wilde¹⁾

1) *Forschungszentrum Karlsruhe, Institute of Nanotechnology, P.O.B. 3640, Karlsruhe D-76021, Germany*

2) *University of Wisconsin-Madison, Department of Materials Science and Engineering, 1509 University Avenue, Madison WI 53706, United States*

In many amorphous Al-alloys, primary crystallization is the key reaction that controls the synthesis of high strength nanostructured bulk volumes comprised of a high density (10^{21} - 10^{23} m^{-3}) of nanocrystals (7 - 20 nm) within a residual amorphous matrix. The primary crystallization kinetics, in response to pre-crystallization annealing treatments, are assessed in specific sample types that serve as model systems to evaluate primary nanocrystallization reactions. Differential scanning calorimetry (DSC) studies on powders and melt spun ribbon (MSR) samples based upon thermal cycling and annealing below the glass transition, T_g , demonstrate a strong sensitivity of the primary crystallization onset and reaction enthalpy to thermal history and the as-quenched state. Although the sensitivity of conventional DSC is not sufficient to detect the isothermal heat evolution at 130°C , the integrated effect of the heat evolution is clearly detected by DSC as a reduction in primary crystallization enthalpy during continuous heating. Varying heating rates in continuous heating calorimetry measurements demonstrates that a significant amount of crystallization occurs during continuous heating through the primary crystallization following annealing. The kinetics behavior highlights the important role of the as-synthesized amorphous structure, the reaction pathways and transient conditions and their interplay on the evolution of nanoscale microstructures during primary crystallization. The support of the DFG (WI 1899/1-2) and the ARO (DAAD 19-02-1-0245) is gratefully acknowledged.

10:30 - 11:00

Coffee break

11:00 - 12:30

Morning session - continued

11:00 - 11:30

invited oral

Bulk Fe-Al-C nanoalloys made by mechanically alloying with subsequent spark plasma sintering and their mechanical properties

Yoritoshi Minamino¹⁾, Yuichiro Koizumi¹⁾, Nobuhiro Tsuji¹⁾, Takanori Tanaka¹⁾, Naoko Hirohata¹⁾, Kiyoshi Mizuuchi²⁾, Yoshihira Ohkanda²⁾

1) *Department of Adaptive Machine Systems, Osaka-university, 2-1, Yamadaoka, Suita, Osaka, Suita 565-0871, Japan*

2) *Osaka Municipal Technical Research Institute, Osaka 536-8553, Japan*

Fe_3Al intermetallic compound has attractive properties as high temperature structural materials, such as high strength, good oxidation resistance, low-cost and so on. However, their commercial application has been limited because of their poor toughness and limited workability. The combination of grain ultra-refinement and phase control is one of the most promising methods to improve their mechanical properties. In this work, we will present the nanostructure and superior mechanical properties of bulk Fe-Al-C nanoalloys made by mechanically alloying (MA) with subsequent spark plasma sintering (SPS).

Four kinds of nanocrystalline Fe-25at%Al-Xat%C (X=1,2,4,6) alloy powder were produced by MA from Fe powder, Al powder and methanol, and were subsequently consolidated by SPS with 32MPa up to 1273K. These compacts have the relative densities of 99.97% (1at%C) to 99.7% (6at%C). The calculations of the relative density variation during the SPS suggest that the compact is consolidated mostly by diffusion, besides the plastic yield and power-law-creep. The structure of most compacts consists of nanocrystalline Fe_3Al -phase of about 80nm with

nano κ -carbides ($\text{Fe}_3\text{AlC}_{0.5}$) and small amount of large α -grains of about $1\mu\text{m}$ with coherently precipitated nano κ -carbides. Their structures were maintained even at 973K. The mechanical properties were measured by the compression test at R.T. to 973K. These bulk nanoalloys perform the superior mechanical properties at R.T. to 973K (e.g. yield strength of 2150MPa and rupture strain of 0.14 for Fe-25Al-2at%C compact at R.T.) when compared with the ordinary Fe_3Al (e.g. 380MPa and 0.12 for Fe-28Al). They also show no environmental embrittlement caused by some gaseous species in air, which is one of fatal problems for the ordinary Fe_3Al .

Formation and Processing of Nanocrystalline Materials by Electrical Discharge Mechanical Milling

11:30 - 11:45

Andrzej Calka¹⁾, D. Wexler¹⁾

oral

1) Faculty of Engineering, University of Wollongong, Wollongong NSW 2522, Australia

A novel device for mechanical milling, incorporating high voltage, low current electrical discharges was constructed and its application for materials processing investigated [1]. In this paper we present recently obtained results on nanostructural phase formation using this method. Milling experiments were performed under different discharge conditions and resulted in completely different reaction paths for the same reacting species. The effects of spark discharge milling condition on formation of nanocrystals were studied in a large number of different materials. It was found that nanocrystallisation of metallic glass ribbons occurs over very short milling times and is accompanied by the formation of fine particles with narrow particle size distributions. The effect of spark milling on phase transformations was also studied. Strong effects of the introduction of spark discharge conditions on nucleation and interface-controlled transformations were observed.

Results of investigations involving the spark discharge milling of carbon and boron under various atmospheres will be presented. Samples, including activated carbon, toluene and crystalline boron were prepared by both conventional milling techniques and electrical discharge assisted milling. X-ray diffraction revealed a range of nano-fragments present in the milled products, in addition to conventional amorphous and crystalline phases. Results of preliminary transmission electron microscopy characterisation of these fragments will be presented.

[1] A. Calka and D. Wexler, Mechanical milling assisted by electrical discharge', Nature, 419 (6903): 147-151 SEP 12 2002.

Nanocrystalline TiAl-V intermetallics hot pressed from mechanically alloyed powders

11:45 - 12:00

Jan M. Dutkiewicz¹⁾, Wojciech Maziarz¹⁾

oral

1) Institute of Metallurgy and Materials Sciences, Polish Academy of Sciences, Reymonta 25 St, Krakow, Poland

The TiAl-V alloys compositions were chosen from the gamma-phase stability region up to maximum 20 at. % vanadium. The elemental powders were initially blended to desired composition of Ti-52.7Al-5V, Ti-55.4Al-10V, Ti-57.7Al-15V and Ti-60.4Al-20V (numbers indicate at. %). The milling was performed in a high energy planetary mill Fritsch Pulverisette P/4 resulted in formation of the almost fully amorphous structure after 40 hours. The formation of Al(Ti, V) solid solution was observed after 20 hours of milling. Longer milling caused amorphization of most of the material and formation of metastable phase in the alloy with the highest content of vanadium. TEM observation of powders after 40 hours of milling allowed to identified 50 nm wide nano-layers within individual particles. The origin of layered structure was explained as resulting from the mechanical alloying process, not from the thin foil preparation. The electron diffraction and bright field techniques revealed the amorphous and partially nanocrystalline structure within a milled powders. The local chemical analysis and elemental mapping allowed to show a high homogeneity of individual particles.

Uni-axial hot pressing at 700°C under 200 MPa of milled powders allowed to obtain a bulk samples with density of about of 94 % of theoretical one. The X-ray diffraction revealed the two phases $\gamma+\alpha_2$ structure. TEM studies allowed to identify the nanometer range grains structure with a mean size of about 20-30 nm. The diffraction pattern has shown a ring like pattern without preferred orientation. Some larger grains has shown a parallel aligned defects causing streaks in the diffraction pattern indicating basal Ti3Al plane defects. Their existence indicate disordering within intermetallic grains in spite of 700°C annealing.

Lunch break

12:30 - 14:00

Afternoon session

14:00 - 15:30

Size-Dependent Phenomena of Interdiffusion in Nanomaterials

Ludmila Paritskaya²⁾, Yuri Kaganovskii²⁾¹⁾, Valerij Bogdanov

14:00 - 14:30

1) Bar-Ilan University, Department of Physics, Israel

2) Karazin Kharkov National University, Department of Crystal Physics, Ukraine

invited oral

Interdiffusion between two nanomaterials (NMs), A and B, occurs mainly inside the wide network of GBs, which are simultaneously the sources and sinks of vacancies. Partial GB diffusion fluxes of both A and B components directed to one another govern the conditions for low-temperature interdiffusion. The inequality of A and B GB partial diffusion coefficients initiates the GB Kirkendall and Frenkel effects and low-temperature phase formation in the grain interiors without participation of bulk diffusion. These phenomena are accompanied by generation and relaxation of stresses and degradation of nano-scale structure. The kinetics and mechanisms of these processes are controlled by the interdiffusion along stationary and migrating GBs. The size-dependent kinetics of GB interdiffusion phenomena in NMs can be described using two dimensionless parameters: δ/l for stationary GBs (l is the size of structural element, δ is the GB width) and λ/l for migrating GBs ($\lambda = (sD_b \delta / V_b)^{1/2}$ is the characteristic diffusion length, D_b is the GB diffusion coefficient, V_b is the GB velocity, s is the segregation factor). The criteria, which allow predicting the minimum temperature of low-temperature phase formation depending on GB diffusivity, the velocity of GB migration, and time dependence of the size of structural element have been formulated and confirmed experimentally for different nano-objects. The mechanisms and models of GB interdiffusion phenomena in NMs have been proposed for analysis of experimental data.

Interdiffusion in Au-Ni Micro- and Nanolayers

14:30 - 14:45

oral

Marek Danielewski²⁾, Robert Filipek²⁾, Anna Rakowska²⁾, Krzysztof Sikorski¹⁾

1) Warsaw University of Technology, Faculty of Materials Science and Engineering, Wólowska 141, Warsaw 02-507, Poland

2) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland

Interdiffusion in nano- and micro- (500nm - 60 μ m) Au-Ni layers deposited on different substrates is studied using EDS X-Ray technique. Quantitative X-ray microanalysis was applied and intensities of the characteristic X-ray lines (NiK α , AuM α , SiK α and WM α) were measured up to 30 kV accelerating voltage. Thin layers (500 nm) were analyzed by electron beam perpendicular to their surface. The mean concentration of these elements in each layer were calculated using CAMECA correction program XTFML based on the Pouchou and Pichoir method (PAP) for in-depth analysis of multilayer samples. Bulk layers (up to 60 μ m) were investigated on their cross-sections using standard EDS technique with ZAF-4 correction.

Theoretical analysis using the Darken method for multicomponent systems is applied for modelling interdiffusion in the Au-Ni system. Computer simulations, in which intrinsic diffusivities of the Au and Ni are functions of composition, are presented and compared with experimental results.

The aim of this work is: i) to reveal the limits of the Darken method in describing the mass transport in Au-Ni layers, ii) to check an applicability of in depth X-Ray analysis for obtaining the concentration profiles in nanometre thickness multi-layers.

Nanocrystalline Thermoelectric Alloys: Processing, Chemistry and Characterization

14:45 - 15:00

oral

Muhammet S. Toprak¹⁾, Yu Zhang, Mamoun Muhammed¹⁾

1) Materials Chemistry Division, Dept of Materials Science and Engineering, Royal Institute of Technology (KTH), Brinellvaegen 23, 2tr., Stockholm SE10044, Sweden

A novel chemical alloying method has been developed for the fabrication of nanocrystalline thermoelectric alloys Cobalt antimonide, CoSb₃, and Bismuth telluride, Bi₂Te₃. The method combines a solution chemical method and thermal processing under controlled heating conditions. The components have been co-precipitated from a solution and the precursor consisted of a solid solution of the different intermediate compounds with different crystallinity. Calcination and hydrogen reduction of the precursor at moderate temperature, 350°C, resulted in the alloying of these elements to obtain the nanocrystalline thermoelectric material. Crystallization of the amorphous precursor as well as the processing route has been investigated by following the changes in the chemistry of the material from the precursor to the final alloy. Detailed microstructure investigation by electron microscopy has also been presented.

Nanomaterials produced by ablation and pulsed laser deposition

15:00 - 15:15

oral

Bogusław Major¹⁾

1) Institute of Metallurgy and Materials Sciences, PAS (IMIM PAN), Reymonta 25, Krakow 30-059, Poland

Interest in pulsed laser deposition (PLD), as a technology producing thin layers of complex materials is growing exponentially in recent years. Condensation from the vapour phase at rapid solidification favours the nano-structure formation in the thin layers. Deposition of virtually any materials, from pure elements to multicomponent compounds, on various substrate, makes PLD very suitable technology in fabrication of thin layers of nano-structure type for wide range of application. Examinations on the PLD layers of FeAl and Ni₃Al intermetallics produced using an excimer KrF are presented. The thickness of deposited layers was dependent on substrate temperature and ion energy. Nano-structure to fine-grained micro-structure in the range from 50 to 1000 nm was formed in deposited thin layer in respect to the deposition conditions. The stoichiometry transfer was under examination in the case of the ablated FeAl and Ni₃Al targets. Titanium nitride thin layers were fabricated by PLD using a

Nd:YAG laser on both metallic (ferritic steel and metallic titanium) and non-metallic (polyurethane) substrates by ablation of pure titanium in nitrogen environment. Deposition of the BN from the h-BN target was performed with application of an excimer ArF laser. Complex structure examinations of microstructure, texture and residual stresses were performed using XRD, SEM, TEM and AFM.

Physics of growth of thin films deposited by laser ablation

Waldemar Mróz¹⁾

1) *Institute of Optoelectronic Military University of Technology (MUT), Wotowska, Warsaw 02-507, Poland*

15:15 - 15:30

oral

The pulsed laser deposition (PLD) is the new method for producing multi-component and composite surface layers. The short interaction time (tens of nanoseconds), equivalent to the growth time of deposited layer causes that the PLD method is one of the most perspective methods to produce nano-structure protective coatings. This method is commonly used for deposition of bio-materials, ceramics, and intermetallic layers on metallic alloys, but also can be used to produce thin films on plastics. Thin films can be grown by different type of lasers, from CO₂ ($\lambda = 10.6 \mu\text{m}$) and Nd:YAG ($\lambda = 1.06 \mu\text{m}$) lasers, to the large family of excimer lasers, with shortest commercially available wavelength $\lambda = 157 \text{ nm}$ (F₂ lasers). According to the laser wavelength used for material ablation and power density on the target, different mechanisms of absorption can dominate. Mechanisms of absorption influence on the ion energy, quantity and sizes of droplets, as well as on the quantity and sizes of large parts of material (debris) deposited on the layers. Influence of different laser parameters and substrate surface quality on the topography and morphology of deposited layers will be illustrated by experimental results obtained for the deposition of Ni₃Al and FeAl intermetallics.

Experimental conditions like laser fluence, substrate temperature and gas pressure also influence on the process of thin film deposition. To illustrate the above mentioned dependencies, the boron nitride (BN) layers deposited with the KrF laser

($\lambda = 248 \text{ nm}$) onto silicon substrates will be used. The BN layers have been deposited on the substrates under different temperatures, from room temperatures to the temperature of 700 °C, and for nitrogen atmosphere with the pressure changed from 5 Pa to the 40 Pa. The laser fluence in our experiments was changed between 5 and 14 J/cm².

Coffee break

15:30 - 15:50

Afternoon session - continued

15:50 - 17:50

High Pressure School 2003 (5th)

Vth High Pressure School on High Pressure Methods for Biotechnology

Organisers

- **Witold Lojkowski**, High Pressure Research Center, Polish Academy of Sciences (UNIPRESS) Warsaw 01-142, Poland
- **Jacek Arabas**, High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Warsaw, Poland
- **Wojtek Dzwolak**, High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Warsaw, Poland

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- Contract: No. G1MA-CT-2002-04055

High Pressure Research Center "UNIPRESS", Polish Academy of Sciences:

- SPUB MSN and other

Proceedings

The papers submitted to the conference will be published in the electronic Journal of the High Pressure School.

Saturday, September 13th

09:10 - 11:25

Morning session

9:10 - 10:10

High Pressure Bioscience: From Molecules to Cells

Karel Heremans¹⁾

invited oral

1) *K. U. Leuven, Dept. of Chemistry and Biological Dynamics, 200 D Celestijnenlaan B-3001, Leuven-Heverlee B-3001, Belgium*

Life as we know it is connected to liquid water. The chemistry of life is characterized by reactions of a limited number of biomolecules. Extreme conditions of a chemical nature are e.g. water activity, salinity, acidity, etc. In general organisms cope with these external conditions by maintaining normal internal conditions. Extreme conditions of a physical nature require different mechanisms.

Proteins can be denatured/unfolded by high hydrostatic pressure as well as by high and low temperatures. The observation that stability diagrams for bacteria, viruses and more complex systems are similar to those obtained for proteins suggests that proteins are the primary target in the inactivation of organisms.

The temperature-unfolding of proteins induces intermediates that show a strong tendency to form intermolecular aggregates. Because of its important role in a number of diseases, the mechanism of the formation of these aggregates gets closer attention. It turns out that pressure may be a good parameter to induce conformations of proteins that have a less stronger tendency for aggregation. This makes pressure an ideal tool to study the mechanism of fibril formation in detail.

Although pressure- as well as temperature-induced effects in biopolymers are closely connected with water, pressure-induced amorphization has been observed in inorganic substances, liquid crystals, synthetic polymers and starch. This gives some new directions for the interpretation of the observed effects in water-soluble biopolymers.

A good overview of these and other aspects may be found in contributions to a special issue on "Frontiers in High Pressure Biochemistry and Biophysics" edited by C. Balny et al. *Biochem. Biophys. Acta* (2002) 1595, 1-402.

10:10 - 10:55

Pressure Effects on Conformational Equilibria of Proteins and Rotational Isomers in Aqueous Solutions

Yoshihiro Taniguchi¹⁾

invited oral

1) *Ritsumeikan University (Rits), Kusatsu 525-8577, Japan*

Molecular conformational equilibrium is the most central concept in the chemistry and biochemistry of chain molecules. This equilibrium is occasionally affected by environmental conditions such as temperature, pressure and solvents. Such structural flexibility of molecules plays an important role in chemical and biological phenomena in liquid phase. In particular, the equilibrium in water is of vital importance for biological systems.

The pressure effect on the equilibrium in liquids can give information of volume differences between trans/gauche of rotational isomers or native/unfolded states of proteins. The volume properties are important to understand the intermolecular interaction between the solute and solvent molecules and the molecular mechanism. It is accepted that the volume changes for trans/gauche equilibrium of rotational isomers in non-polar solvents are less than -5 cm³/mol and for native /unfolded states of proteins in aqueous media less than -100 cm³/mol.

Recent development of high pressure Fourier transform infrared spectroscopy combined with resolution enhancement techniques and Raman spectroscopy is able to detect the signal of each rotational isomer in dilute aqueous solution and the secondary structure of the pressure induced structure changes of proteins in water. In this study, the effect of pressure on the conformational equilibrium between rotational isomers of halo-acetone and proteins in aqueous media has been studied by the Fourier transform infrared and Raman spectroscopies. On the base of both observed volume changes of simple chain molecules and proteins, the molecular mechanism on the pressure induced conformational changes will be discussed.

10:55 - 11:25

Comparative study of solvation pressure and hydrostatic pressure in biological and molecular systems

Hannes Hubel¹⁾, Nancy W. van Uden¹⁾, David J. Dunstan¹⁾

oral

1) *Physics Department, Queen Mary, University of London, Mile End Road, London E1 4NS, United Kingdom*

Liquids do exert pressure on small, immersed particles, such as nanotubes^[1]. The magnitude of this solvation pressure is closely related to a parameter called the cohesive energy density (CED)^[2]. We report the equality of

solvation pressure and external pressure in the case of molecular and biological samples.

Raman spectra of chloroform were measured as a function of concentration in various solvents, under hydrostatic pressure applied by a diamond anvil cell and in the vapour phase. It was found that the peak shifts due to solvation pressure matched the shifts obtained by hydrostatic pressure for some vibrational modes of chloroform.

Starch grains were subjected to different solvation pressures by placing them in water-ethanol mixtures. The pressure induced gelation of starch grains was then observed as a function of applied pressure. It was found that the sum of CED and gelation pressure is constant^[2]. Here we report data on water-ethanol-glycerol mixtures, again the sum of CED and applied pressure remained constant, except for high glycerol concentration.

Protein unfolding was also studied under external applied pressure. The proteins were immersed in ethanol-water, glycerol-water solutions and a hydrostatic pressure was applied. The unfolding of the protein was recorded using tryptophan fluorescence. Preliminary results suggest a strong correlation between the internal pressure of the solution and the unfolding pressure in the regime of low ethanol-glycerol concentration.

[1] J. R. Wood et al., J. Phys. Chem. B 103 (1999), 10388

[2] N.W.A. van Uden et al., Journal of Physics: Condensed Matter 15 (2003), 1577

Break

11:25 - 11:45

Morning session - continued

11:45 - 14:15

Recent advances in high-pressure synthesis of macrocyclic organic compounds

11:45 - 12:30

Janusz Jurczak¹⁾

invited oral

1) Institute of Organic Chemistry, Polish Academy of Sciences, Kasprzaka, Warsaw, Poland

Present-day methods of organic synthesis are mostly based on chemical modification of substrates, reagents, and catalysts. Nevertheless, frequent use has recently been made of new, nonconventional techniques, such as high-pressure procedure. Some important problems of organic synthesis have been recently solved using the high-pressure technique. Among them, preparation of synthetic macrocyclic molecular receptors is one of the most important examples.

In this presentation, we would like to show the progress in the high-pressure synthesis of macrocyclic compounds, such as diazocoronands, bi- and tricyclic cryptands in both achiral and nonracemic chiral forms. Some examples of such molecular receptors will be also shown.

Toward understanding compressibility of hydrogen-bonded solids

13:30 - 14:15

Andrzej Katrusiak¹⁾

invited oral

1) Adam Mickiewicz University (UAM), Grunwaldzka 6, Poznan 60-780, Poland

Any material transforms at varied thermodynamical conditions, and pressure can be very efficient in inducing such transformations. For predicting the transformations of any substance - their types, directions and magnitudes - one must refer to the microscopic structure of molecules and their interactions. The intermolecular forces were classified long time ago, and it is well known that structures of organic compounds are dominated by van der Waals and electrostatic interactions and by hydrogen bonds. The substances with structures governed by these interactions are abundant in Nature, especially in biosphere, and many chemical, physical, or biological processes involve transformations of hydrogen bonds, also in the living tissue. High-pressure x-ray diffraction is an ideal method for investigating the effects of pressure on the structural transformations of the crystals. Several examples of such experimental studies on organic crystals, and a general overview of the rules governing transformations of hydrogen-bonded solids will be presented. These rules can be further extended to systemize monotonous transformations, and also the anomalous transformations at first-order (discontinuous) or second-order (continuous) phase transitions, as well as for closed and open systems (which do not change, or which change their stoichiometry, respectively). Although the precise high-pressure diffraction experiments were carried out for relatively simple crystal structures, the same rules can be applied for much more complicated materials, to ferroelectrics, superconductors, and even proteins and viruses. Therefore the high-pressure studies of hydrogen-bonded crystals are of general and technological importance.

Sunday, September 14th

Morning session

10:00 - 11:30

10:00 - 10:45

invited oral

Patterns in the Phase and Structural Behavior of Lyotropic Lipid Mesophases, Model Biomembranes and Proteins at High Pressure

Roland H. Winter¹⁾

1) *University of Dortmund, Physical Chemistry I, Otto-Hahn Straße 6, Dortmund D-44221, Germany*

By use of X-ray and neutron scattering, densitometry and calorimetry as well as fluorescence, NMR and FT-IR spectroscopy, the temperature and pressure dependent structure, dynamics and phase behavior of a variety of lipid and model biomembrane systems was studied. Hydrostatic pressure was used as a physical parameter for studying the stability and energetics of lyotropic lipid mesophases, but also because high pressure is an important feature of certain natural membrane environments. Neutron scattering in combination with the H/D contrast variation technique and fluorescence microscopy was used to the study of lateral organization of phase-separated lipid mixtures. We also present data on the effect of incorporating polypeptides on the structure and phase behavior of lipid bilayers. By using the pressure-jump relaxation technique in combination with time-resolved synchrotron X-ray diffraction, the kinetics and mechanisms of different lipid phase transformations was also investigated. The technique has also been applied for studying other soft-matter and biomolecular phase transformations, such as protein unfolding. We present data on the pressure-induced unfolding and refolding of small proteins. The data are compared with the corresponding results obtained using other trigger mechanisms and are discussed in the light of recent theoretical approaches of protein folding.

10:45 - 11:30

invited oral

High pressure, a new tool to investigate protein structural changes

Claude Balny¹⁾

1) *Institut National de la Santé et de la Recherche Médicale (INSERM), 1919, route de Mende, Montpellier 34293, France*

One major recent challenge in the field of life science is to understand the protein folding because many diseases involve protein misfolding. There are several ways to investigate protein structural changes. This is possible using either a physical parameter such as the temperature or chemical compounds, both perturbing the system under study.

A new approach could be high hydrostatic pressure, a physical parameter that has been recently qualified by Ernst (Nobel winner) as a invaluable tool for exploring and comprehending biological function (1). High pressure causes complete or partial denaturation of proteins because the protein-solvent system for the denatured state occupies a smaller volume than that for the native state. In a similar manner, the effect of pressure on dissociation of oligomeric proteins or nonspecific aggregates is to favor states that present a smaller specific volume. These effects are thought to arise from a combination of several consequences such as loss of packing defects existing in the native structure or hydration variations. Pressure perturbation depends solely on the volume change of the system under study; in contrast, temperature involves changes in both the volume and the total energy. While denaturation by temperature is often irreversible, pressure-denaturation is, within a limited range, reversible. Consequently, hydrostatic pressure provides an elegant alternative as it induces reversible phenomenon and avoids the addition of external chemical agents.

This method has been successfully used for different pressure-induced effects, such as reversible or irreversible folding and unfolding, conformational changes with ligand binding and oligomeric dissociation. More recently, alternative prion structural changes have been revealed by high pressure.

Together with the basic concepts, all these aspects will be evoked through specific examples.

1) Balny C, Masson P, Heremans K, *BBA-PROTEIN STRUCT MOL ENZYMOL* 1595, 3-10, 2002

11:30 - 11:50

Break

11:50 - 15:20

Afternoon session

11:50 - 12:35

invited oral

Pressure and protein aggregation.

Laszlo Smeller¹⁾

1) *Institute of Biophysics and Radiation Biology, Semmelweis University of Medicine, Budapest, Hungary*

Denaturing effect of pressure is well known. The influence of pressure on intermolecular forces became interesting topics only recently, when the role of protein aggregates in the development of conformational diseases was established.

Another approach to the aggregation comes from the protein folding problem. It was already clear in the study of disease related aggregation, that correctly folded proteins cannot form aggregates, unless there is a serious destabilizing factor, lowering the stability of the native state. From the point of view of the folding, aggregation can occur among the misfolded proteins.

Pressure is used in study of protein aggregation for two reasons. First we used pressure unfolding to be able to study the refolding. The second aim was to study the effect of pressure on the intermolecular forces, by pressurizing

protein aggregates of different kind.

Our studies show that refolding of the pressure unfolded protein can only be partial. Apart the folded protein molecules, a significant amount of molecules with intermediate conformation is formed. This effect has been demonstrated for several proteins including myoglobin, lipoxigenase, lysozyme, indicating the general feature of this behavior. In case of horseradish peroxidase the destabilized intermediate was however detected only if the disulfide bridges were cleaved prior to the pressure treatment.

Pressure induced dissociation of oligomers is known. This would suggest pressure-disaggregation. Our observations indicate that the aggregation process consist of several successive steps, which are probably governed by a very slow kinetics. The few first steps of this aggregation process can be reversed by pressure, but the well formed (probably restructured) aggregates persist the pressure treatment.

We suggest a new phase diagram containing metastable states, that expands the capabilities of the conventional elliptic T-p phase diagram of the proteins in order to explain aggregation phenomena.

Why does high pressure prevent protein aggregation ? An FTIR/DSC/PPC case study on insulin.

12:35 - 13:05

Wojtek Dzwolak¹⁾

oral

1) High Pressure Research Center, Polish Academy of Sciences (UNIPRESS), Sokolowska 29/37, Warsaw 01-142, Poland

High pressure is known to possess a modulating effect on protein aggregation - both in terms of inhibition and triggering of this process. It is not yet clear, though, which particular stage on an aggregation pathway is addressed and affected by pressure. Pressure Perturbation Calorimetry (PPC), Differential Scanning Calorimetry (DSC) and time-resolved FTIR have been employed to investigate aggregation of bovine insulin at low pH. As moderately high pressure of 30-40 MPa has been implicated to possess a strongly inhibiting effect on insulin aggregation, we were seeking to explain these changes monitoring volumetric effects on the aggregation pathway. PPC results show that the stage of actual, exothermic aggregation (seen in FTIR spectra as the appearance of non-native β -strands) is preceded by formation of the molten globule state and its unfolding. The unfolding is accompanied by a volume expansion of approx. 0.2 %. High pressure FTIR showed that under hyperbaric conditions the aggregation is blocked already at the first stage. Namely high-pressure prevents unfolding of insulin.

High Pressure for Food Processing

14:05 - 14:50

Volker Heinz¹⁾

invited oral

1) Department of Food Biotechnology and Food Process Engineering, Berlin University of Technology, Königin-Luise-Str. 22, Berlin D-14195, Germany

High pressure processing is a tool which more and more is reaching industrial relevance in food processing since there are now technical systems available which allow the treatment of considerable quantities (more than 2 tons per hour) at pressures up to 800 MPa. There is a long tradition of using high pressure treatment for sterilization or pasteurization of foods even if it was limited to scientific investigation or only small scale processing.

The main reason for its application is the lethal effect of high pressure on microorganisms. The metabolism of many living entities is adapted to atmospheric pressure at earth's surface and an increase can disturb vital metabolic reactions or the reproduction. However, it is also known that deep sea is the habitat of a variety of organisms which obviously can withstand hydrostatic pressures up to 110 MPa. Some bacteria even survive in the vicinity of submarine hydrothermal vents where they are simultaneously exposed to elevated pressures and temperatures up to 120°C. Since proteins are involved in most vital functions in living organisms, the survival of those extremophilic bacteria must be due to highly efficient stabilization mechanisms such as van der Waals contacts, hydrophobic effects, intramolecular hydrogen bonding or disulphide bridges. To chose proper conditions which can eliminate significant amounts of microbes and which are technically feasible and economically efficient in the same way is the goal of food process engineering.

An overview will be given covering the current practice of industrial high pressure technology. Related to food processing, the technical requirements for a broader application will be discussed and future research needs will be identified.

Pressure Modulating Effect on ATP-ase Activity

14:50 - 15:20

Natalya V. Shishkova¹⁾, Lyudmila I. Donchenko²⁾, Vladimir I. Barbashov¹⁾, Maria D. Vdovychenko²⁾, Anatolii V. Stepura²⁾

oral

1) Donetsk Physics & Technology Institute of National Academy of Sciences of Ukraine (DonPhTI NASc), 72, R.Luxemburg, 83114 Donetsk, Ukraine

2) Scientific Research Institute for Traumatology & Orthopaedics of Donetsk State Medicine University (DonIT&Or), 106, Artema St, Donetsk 83048, Ukraine

Na⁺ / K⁺ -ATP-ase (EC 3.6.1.37) is the integral protein of plasmatic membranes of cells for majority of animal

tissue. It is a more intensively studied the vector enzymes of biological membranes.

We have studied the high hydrostatic pressure influence on ATP-ase activity variation.

The human blood stabilized by heparin was subjected to high pressure in the range 25-150 MPa at chilling temperature.

The Mg^{2+} and Na^+/K^+ ATP-ases activities was studied by evaluation of phosphor content in hemolysates of erythrocytes at their incubation with ATP by spectro-photometry method at 756 nm.

It was revealed the progressive increase of Na^+/K^+ ATP -ase and decrease of Mg^{2+} -ATP-ase activities with high pressure value enhancement into the studied pressure range.

The obtained results will be discussed.

Monday, September 15th

Perspectives of nanotechnology

Wednesday, September 17th

11:00 - 12:30

Morning session

11:00 - 11:45

Challenges for Polish Research in European Industry

Marcel Van de Voorde¹⁾

invited oral

1) University of Technology Delft, Rotterdamseweg 137, DELFT 2628 AL, Netherlands

The lecture gives a vision in materials research at the university, in the research institute and the industry, in Europe. The importance of a strong relation between Academy and industry will be highlighted and mechanisms for collaboration be suggested.

It will be demonstrated that without sufficient investment (people and budget) in - basic and applied - materials research; new ideas, discoveries and innovations necessary for the continued growth and competitiveness of a company or a country are less likely to be forthcoming. For an industrial country as Poland it becomes of vital importance to focus on "revolutionary" research leading to breakthroughs: new materials, innovative processes etc.

It is not evident for a research community or for industry to pinpoint areas for future research assuring industrial successes. The new nano-world offers for materials science and technology plenty of opportunities for universities/research centres to become "centres of excellence" in Europe and for industry to become "centres of technology and innovation". Poland should not miss this challenge of becoming an attractive pole for European scientists and a most modern industrial nation with a flourishing economy, in the future.

The lecture will explore possibilities for research, which make the field of "nanomaterials" attractive to students - of all disciplines - and of interest, and importance for the scientist and its career. Ideas for the development of technology parks around universities/ centres of excellence and the creation of innovative SME's will be elaborated. Routes for the application of nanomaterials research in industry will also receive attention.

This lecture will be easy to follow for policy-makers, industrialists, scientists and students

11:45 - 12:30

Nanotechnology in Medicine

Leonard Fass¹⁾

invited oral

1) GE Medical Systems, 352 Buckingham Avenue, Slough SL1 4ER, United Kingdom

Nanotechnology will play an important role in future applications in medicine.

New materials made from nanocomposites will be used to make high performance X Ray tubes with stronger and lighter anodes. Low friction nanolayers will be developed for higher speed bearings and field emitters will be used to make cathodes.

Magnetic nanolayers will be used to develop smaller, higher field strength open MRI systems or for separation and testing of bio-molecules.

Molecular and Cellular Imaging will utilize functionalised nanoparticles with specific magnetic and optical properties.

Future medical diagnosis systems using nanotechnology including In-vivo, in-vitro sensors, hand-held, wireless diagnostics and wearable, wireless monitors

constructed through the use of micro-nano electronic packaging and organic electronics & systems for miniaturized displays, sensors and transducers.

A systems approach to nanotechnology is key to the development of medical applications.

Biocompatible, biodegradable bio-sensors & actuators will be used to measure bodily functions.

Nano biosensors will have applications in the emergency room for real time,

in-situ reading of biochemical activity, cellular level optical imaging

and in sensor guided precision surgical tools.

In the doctors office it will be possible to obtain a total blood analysis in minutes, a rapid, accurate disease diagnosis and a patient specific disease treatment through the use of molecular recognition and high density nano-arrays.

At home the use of biosensors, wireless communications, self powered devices and high-resolution displays will allow simple patient administered diagnostic tests and automatic transmission of outpatient data from home to the doctor.

Nanotechnology will help overcome current limitations of power supply to cochlear and other implants.

Nanotechnology will play a key role in the development of tissue engineering.

12:30 - 14:00

Lunch break

Afternoon session

14:00 - 15:30

High Performance Nanoscale Materials and Bulk Metallic Glass

Hans-Jörg Fecht¹⁾

14:00 - 14:30

1) Ulm University, Albert-Einstein-Allee 47, Ulm 89081, Germany

invited oral

The correlation between the micro- and nanostructure of a material and its physical and chemical properties is the key issue in materials development. Considerable progress has been achieved recently by the development of new processing technologies (advanced solidification techniques, mechanical alloying etc.) and new multi-component alloys (bulk metallic glass, nanocomposites, advanced intermetallics etc.) with superior mechanical, magnetic and tribological properties.

A short overview will be given concerning new trends in the development of bulk metallic glasses. Glasses are generally produced from the highly undercooled liquid state. For metallic systems it is therefore essential to effectively control heterogeneous nucleation sites in the undercooled liquid. This has recently led to the development of bulk metallic glasses, e.g. samples with volumes of several cubic centimeters. These new materials with a complex multicomponent chemistry are characterised by deep eutectic temperatures and excellent magnetic and mechanical properties. Most recent results on glass forming melts suggest a scenario where the glass transition in a metallic alloy is not a phase transition in the classical sense but kinetic freezing triggered by an underlying entropic instability. Possible applications are wear resistant bulk materials, high-strength sporting equipment, electronic casing, high performance magnetic coils etc. Furthermore, the deformation processes relevant for nanoscale materials (grain boundary sliding) and bulk metallic glasses (formation of shear bands) is also being extended to develop wear resistant coatings and surfaces.

Participation of Accession Candidate Countries to the FP6 - Priority 3

Antoaneta Folea¹⁾

14:30 - 15:00

1) European Commission, Brussels, Belgium

invited oral

(no abstract available)

Nanotechnology start-ups in Europe

Ottília Saxl¹⁾

15:00 - 15:30

1) The Institute of Nanotechnology (IoN), 6 The Alpha Centre, Stirling FK9 4NF, United Kingdom

invited oral

In Europe, nanotechnology research is taking place in Universities in the main, actively supported by large scale Government and EU funding. Commercialisation is taking place through small companies, spinning out from the research base. The characteristics of these companies is discussed and reference made to how larger companies are approaching the opportunities and threats posed by nanotechnology. Recommendations as to how smaller countries such as Poland can maximise their potential for success will also be covered.

Coffee break

15:30 - 16:20

Synthesis of inorganic nanoparticles and their application on ceramic substrates

Giovanni Baldi

15:50 - 16:20

invited oral

At the moment scientific research is being carried out on nanoparticles (particles with dimensions below 500 nm up to 1 nm) for their synthesis and for their application in industrial processes. Many applications can be thought of for nanoparticles materials, from catalysis to bioinorganic systems, from textile to pharmaceutical fields etc. Basically all the improvements which resulted when microparticles were developed can now be extended to nanoparticle based products with the difference that the physical and chemical properties of materials often change when in nanometric form. The most critical aspects encountered are the limits involved in using the reactions to obtain controlled particle shapes and dimensions in very short ranges.

In Advanced Research Laboratory of Gruppo Colorobbia a project for synthesis and application of inorganic nanomaterials started in 2001, two main different synthetic path were considered: the glycolic synthesis and the reverse emulsion synthesis. Nanomaterials specifically developed can be used to improve the properties of ceramic products like as porcelain stoneware or glasses surfaces reducing at the same time the environmental impact. Examples are in the "quadricromia" inkjet application. Another promising field of investigation is the coating of tiles to have "glass like", functionalized surfaces: researches of a coating of surfaces to obtain special surface effects as superhydrophobic (Lotus effect), superhydrophilic, self-cleaning properties are in progress. The materials in nanometric scale appear suitable for many application in the ceramic field were the improvement of chemical physical properties of surfaces leads to new functionalized materials. This will be reflected in a increment in the quality of life for manufacturers and customers. The industrial scale-up and the application appears difficult, nevertheless the

challenge is to conquer new markets with innovative products.

16:20 - 17:20

Afternoon session - continued

16:20 - 16:50

Nanomaterials for lighting

Martin Zachau¹⁾, Armin Konrad¹⁾

invited oral

1) OSRAM GmbH (OSRAM), Munich, Germany

Today a variety of non-luminescent nano materials are used for lighting applications. Nano silica and nano alumina synthesized by flame pyrolysis are commercially available. Nano silica is a highly reactive source in the production of Si-based phosphors for fluorescent lamps or LEDs. Nano silica is also used for fluidization, adjusting tixotropy and as a scattering pigment in LEDs. A thin layer of densely packed nano silica or nano alumina between glass and phosphor layer serves as a selective UV reflector and/or a barrier to mercury diffusion /1,2/. Nano alumina is added to the phosphor layer to improve adhesion. Finally non-luminescent nano materials may be used to form a thin conformal coating around phosphor particles that protects the phosphor particles and improves the maintenance of fluorescent lamps or LEDs.

Luminescent nano materials have been the subject of extensive research in the last 15 years. The small and large organic molecules that are used in OLED and LEP displays may be viewed as nano phosphors. Their efficiency and stability, however, still excludes their use in lighting applications. Potentially more stable are inorganic nano phosphors. Widely known are the semiconducting nano phosphors such as CdSe /3/ or InP /4/, ZnS:Mn /5/, as well as rare-earth doped LaPO₄:Ce,Tb /6/. We have ourselves investigated nano Y₂O₃:Eu in detail /7/. For particle sizes below 20 nm, the optical properties of nano phosphors change, e.g. the exciton energy increases and perhaps even more important, scattering decreases. We will discuss the use of inorganic nano phosphors in fluorescent lamps and LEDs.

/1/ R. Hoffmann et al., US 43440116 A (10.8.82)

/2/ H. Verhaar et al., WO 96/60451 (29.2.96)

/3/ C. B. Murray et al., Science 270 (1995) 1335

/4/ D. V. Talapin et al., J. Phys. Chem. B 106 (2002) 12659

/5/ A. A. Bol et al., Phys. Rev. B 58 (1998) 15997

/6/ K. Riwotzki et al., J. Phys. Chem. B 102 (19

/7/ A. Konrad et al., J. Appl Phys. 86 (1999) 3129

16:50 - 17:20

Structural funds in Poland and Polish Research Potential in Nanotechnology

Andrzej Siemaszko¹⁾

invited oral

1) Institute of Fundamental Technological Research, PAS (IPPT PAN), Świętokrzyska 21, Warsaw 00-049, Poland

Poland is facing enormous opportunities related to the absorption of structural funds. It is expected that about 0.5 billion euro could be allocated to initiatives supporting research and technological development in years 2004 - 2006. Important initiative should be a development of Center of Nanotechnology in Warsaw which could integrate the existing potential in this field and which through the development of research infrastructure as well as development of human resources could become a leading research institution in Poland. This center, together with a national-wide nanotechnology network should carry out the long term advanced research as well as to coordinate all Polish activities in the field of nanotechnology. Contributing to the development of the European Research Area, they should be strongly involved in activities of European Nanotechnology Networks of Excellence.

An important step towards development of the Center and nanotechnology network will be to carry out the mapping of excellence activities evaluating the existing RTD potential and its spatial distribution. In parallel, there will be the foresight activities defining the most prospective directions of development of nanotechnology in Poland. These exercises are included into the strategy of Ministry of Research and Information Technology.

19:00 - 21:00

Poster session

Pre-School of Symposium B

Development of methods for Characterisation of the Microstructure of Novel Materials

Introduction

On 14 September 2003 a Pre-school with a series of tutorial lectures for PhD students and young scientists will be organized.

The idea of organizing a pre-school is to give tutorial lectures concerning the main techniques used in materials characterization. These lectures should provide the scientific background for understanding the recent achievements in development of the methods being presented by invited lecturers during the Symposium B and the scientific results presented in oral and poster presentations. The Pre-school is addressed, mostly but not only, to the PhD -students and young scientists. There is not provided any additional fee for participation in the pre-school. In registration form, please indicate the intention of participation in the pre-school. It helps us to make a choice of proper lecture room.

The scientific programme of the Pre-school will offer tutorial lectures and the programme of Symposium will include invited general lectures, oral presentations, poster sessions and panel discussions.

Organisers

* Krystyna Lawniczak-Jablonska, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Sunday, September 14th

09:30 - 11:00

Morning session conducting by Rajmund Bacewicz

Main Building, room 208

9:30 - 10:15

Synchrotron Radiation - Sources and Properties

Krystyna Lawniczak-Jablonska¹⁾

invited oral

1) Institute of Physics, Polish Academy of Sciences, al. Lotnikow 32/46, Warsaw 02-668, Poland

Synchrotrons are modern, intense sources of continuous electromagnetic radiation emitted in the wide range of energy. This emitting range runs through the infrared, by the visible and vacuum ultraviolet up to soft and hard x-ray radiation. The radiation is emitted by the charged particles with trajectories curved in the magnetic field. Almost 99% of particle energy is transferred to the emitting radiation, therefore the process is very effective as compare with x-ray tube. The description of the construction and operation of third generation facilities and short overview of their locations will be given. The idea of insertion devices will be discussed and introduction to the X-ray optics and synchrotron radiation detectors will be provided. Next the properties of the synchrotron radiation in comparison with other sources of radiation will be presented. A few examples of beamline construction and advantages of use of synchrotron radiation in natural science will be shown.

10:15 - 11:00

The Diffraction Phenomenon In Radiation

Ake Kvick

invited oral

The phenomenon of diffraction has been widely used in research on atomic and atomic structure for last century . The lecture will outline the basic principles of diffraction using electromagnetic radiation (X rays) or particles (neutron). The lecture will also cover the basis for the extension to synchrotron radiation and outline the present developments of new sources of X rays such as the free electron lasers. Examples of leading edge experiments and comparisons between different techniques as well as current trends in instrumentation will be included. A comparison between different techniques will be provided.

11:00 - 11:30

Coffee break

11:30 - 13:00

Morning session - continued- conducted by Bronislaw Orłowski

Main Building, room 208

11:30 - 12:15

An introduction to X-ray Absorption Fine Structure Spectroscopy

Sakura Pascarelli¹⁾

invited oral

1) European Synchrotron Radiation Facility (ESRF), 6 Rue Jules Horowitz, GRENOBLE F-38043, France

In this tutorial I will give a very basic introduction to X-ray Absorption Fine Structure (XAFS) Spectroscopy. I will start by the definition of the absorption coefficient and its measurement. Then I will describe the physical origin of the fine structure and illustrate its sensitivity to the local chemical and structural environment of the absorber atom. I will then show how the Extended X-ray Absorption Fine Structure (EXAFS) can be interpreted to extract quantitative structural information in the framework of the gaussian approximation (low disorder). Finally I will discuss typical applications, the complementarity with Diffraction Methods and the advantages of 3rd generation sources for XAFS Spectroscopy. I will end the tutorial by showing an example or two of recent experiments carried out at the ESRF.

12:15 - 13:00

VUV photoemission using synchrotron light: a tool for characterising surfaces and interfaces occurring in OLEDs

invited oral

Jacques Ghijsen²⁾, **Robert L. Johnson**¹⁾, **Andreas Elschner**⁴⁾, **Norbert Koch**³⁾

1) Institute for Experimental Physics, University of Hamburg, Hamburg, Germany

2) FUNDP-LISE, 61 rue de Bruxelles, Namur B-5000, Belgium

3) Humboldt University of Berlin, Institute of Physics, Newton Street 15, Berlin 12489, Germany

4) H.C. Starck GmbH, c/o Bayer AG Uerdingen, Krefeld, Germany

In principle, organic light-emitting diodes (OLEDs) just need one layer of appropriate material sandwiched between two metal electrodes in order to create light when the device is polarized. Real life is more complicated: in order to create a long-lived (> 10,000 hr) and efficient device (tens of lm/W), it is necessary to use more complex structures involving additional layers. Just as with more conventional inorganic devices, it is important to monitor the interfaces in order to identify possible reactions between the materials involved, to check for interdiffusion, and to determine the alignment of electronic levels.

Photoelectron spectroscopy is a well-suited technique for addressing these questions. Because the interfaces encountered in such devices often involve a metal on one side and an oligomer or a polymer on the other side, it is in many cases convenient to use a tuneable light source in order to balance properly the photoemission cross sections of the different levels involved in the valence band.

The use of the technique will be illustrated by considering the case of pentacene and low-work-function metals (samarium and alkali metals), in the cases of the growth of a metal film on pentacene as well as the deposition of pentacene on metal. This will be put in parallel with a study of interfaces with high-work-function materials, such as gold or poly(3,4-ethylenedioxythiophene/polystyrenesulfonate, and three different conjugated organic materials: N,N'-bis-(1-naphthyl)-N,N'-diphenyl-1,1biphenyl-4,4'-diamine, para-sexiphenyl, and pentacene.

Lunch

13:00 - 14:30

Afternoon session conducted by Wojciech Szuszkiewicz

14:30 - 16:45

Main Building, room 208

Neutron scattering in magnetic materials

14:30 - 15:15

Radosław Przeniosło¹⁾, Izabela Sosnowska¹⁾

1) Warsaw University, Institute of Experimental Physics, Hoża 69, Warsaw, Poland

invited oral

The basic properties of the neutron are discussed in the context of the applications of neutron scattering in studies of the magnetic properties of condensed matter. A general cross section for magnetic neutron scattering from unpaired electrons is given. The characteristic features of the cross section are discussed in terms of its sensitivity to the spin correlations, both in space and time. The elastic neutron scattering provides information about the arrangement of the magnetic moments in an atomic scale. These orderings may be paramagnetic, short-range order spin-glass type, or long range order like: ferromagnetic, ferrimagnetic, antiferromagnetic or modulated. Elastic scattering provides also important information about the coherence length of the magnetic ordering and it may be also used to study magnetic ordering in 2 dimensional planes as well as orderings along 1 dimensional chains. The kinetic energy of thermal and cold neutrons is comparable to the energy of elementary excitations in condensed matter between about few to hundreds of meV. Inelastic neutron scattering, associated with changes of both, momentum and kinetic energy of the neutron, can be used in studies of elementary excitations in solids (e.g. magnons). Inelastic neutron scattering studies of magnetic excitations of the electronic and nuclear spin systems are discussed.

X-ray imaging

15:15 - 16:00

Jürgen Härtwig¹⁾

1) European Synchrotron Radiation Facility (ESRF), Grenoble BP220-38043, France

invited oral

A review will be given over X-ray imaging techniques used at modern synchrotron sources (and not only there). Their principles, optical elements, physical basics will be presented. With different emphasis elements of X-ray optics, X-ray microscopy, radiography, micro-tomography and X-ray diffraction imaging (X-ray topography) will be discussed.

Structural and chemical characterization of crystals by TEM

16:00 - 16:45

Wolfgang Neumann¹⁾

1) Humboldt University of Berlin, Institute of Physics, Newton Street 15, Berlin 12489, Germany

invited oral

Transmission electron microscopy (TEM) is a powerful tool to get a detailed insight into the materials characteristics. To elucidate the correlations between crystal structure and properties, including the nanochemistry (chemical composition and bonding), of advanced crystalline materials besides the imaging techniques of transmission electron microscopy, particularly high-resolution transmission electron microscopy (HRTEM), the microanalytical techniques of energy-dispersive X-ray spectroscopy (EDXS), electron energy loss spectroscopy (EELS) as well as microdiffraction and nanodiffraction techniques (e.g., convergent beam electron diffraction - CBED) can be applied. Moreover, dedicated sub-techniques are used to image the element distribution along a line (EDXS profile, series of EEL spectra) or two-dimensionally (X-ray mapping, energy-filtered TEM) at a lateral resolution of some nanometers (EDXS) or even on the sub-nm scale. In addition, the chemical bonding of the elements present in the transmitted volume is characterized by analysing electron energy loss near edge structures (ELNES). This additional information is available owing to the relatively high energy resolution of the EELS technique of about 0.5 - 1 eV. For a reliable interpretation of TEM images, especially HRTEM images corresponding image simulations have to

be carried out.

The fundamentals of the different TEM methods will be outlined. Furthermore, the potential of combined use of imaging and analytical TEM will be demonstrated for various modern crystalline materials of different dimensions.

NanoCentre Pre-School

Nanocrystalline Materials: Fabrication Structure, Modelling and Applications

Organisers

* NanoCentre, Warsaw University of Technology, Faculty of Materials Science & Engineering

Sunday, September 14th

10:00 - 11:30

lectures

10:00 - 10:45

Bulk and Graded Materials**Krzysztof Kurzydłowski¹⁾**

invited oral

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

(no abstract available)

10:45 - 11:30

Materials for Medical Applications**Małgorzata Lewandowska-Szumiel^{1,2)}**

invited oral

*1) Medical University of Warsaw, Department of Biophysics and Human Physiology; Department of Transplantology & CTB, Chałubińskiego 5, Warszawa 02-004, Poland**2) Department of Biophysics and Human Physiology, Department of Transplantology & CTB, Medical University of Warsaw, Warszawa, Poland*

(no abstract available)

11:30 - 12:00

Coffee break

12:00 - 13:30

Afternoon session

12:00 - 12:45

Recent progress in developing bulk nanomaterials using SPD**Ruslan Z. Valiev¹⁾**

invited oral

1) Institute of Physics of Advanced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation

(no abstract available)

12:45 - 13:30

Microanalysis-orientation, disorientation and microtexture determined by transmission electron techniques (TEM)**Maria Richert^{1,2)}, Sonia Hawrylkiewicz¹⁾, K. Chruściel, J. Długopolski, A. Baczmański**

invited oral

*1) University of Mining and Metallurgy, 30 Mickiewicza, Krakow, Poland**2) AGH University of Science and Technology (AGH), al. Mickiewicza 30, Krakow 30-059, Poland*

(no abstract available)

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