The seminar is organized by

the Czechoslovak Association for Crystal Growth and
the Slovak Expert Group of Solid State Chemistry and Physics

under the auspices of

the Institute of Physics, Academy of Sciences of the Czech Republic and
the Slovak Society for Industrial Chemistry

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The 22nd Joint Seminar “Development of Materials Science in Research and Education” (DMSRE22) will be held in Lednice at Lednice spa during 3-7 September 2012. The first Joint Seminar in these series was held at Gabčíkovo in the Slovak Republic in 1991. DM-SRE22 is organized by the Czechoslovak Association for Crystal Growth and the Slovak Expert Group of Solid State Chemistry and Physics under the auspices of the Institute of Physics AS CR, Faculty of Chemical and Food Technology SUT Bratislava, and Slovak Society for Industrial Chemistry.

The Seminar is intended to bring together a unique combination of scientists across a multidisciplinary spectrum and provides an ideal forum for the presentations and discussions of recent developments and achievements in all theoretical and experimental aspects of preparation processes, characterization and applications of materials in bulk, thin film, nanocrystalline and glassy states.

The scientific sessions cover the following topics of materials science:
- Trends in development of materials research
- Education of materials science at the universities
- Information about the research programmes of individual institutions
- Information about equipment for preparation and characterisation of materials
- Results of materials research


This booklet contains the abstracts of all contributions, which reached us before August 7. The authors are responsible for the technical and language quality of the contributions.

The conference will run from Monday afternoon, September 3, till Friday noon, September 7 in Lednice spa.

Dear colleague, we welcome you to the DMSRE22 Joint Seminar and we hope you will enjoy your stay in Lednice spa.

On behalf of the organizers,

Zdeněk Kožišek, Karel Nitsch, and Marian Koman
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Monday, 3 September 2012

11:15 – 13:50: Registration
   Location: Hotel Langue

14:00 – 14:10: Opening
   Location: Lecture Hall

14:10 – 15:50: Monday Session I
   Location: Lecture Hall   chairperson: Zdeněk Kožíšek

14:10 – 14:50: L. Koudelka:
   Phosphate and borophosphate glasses: structure–properties–application

14:50 – 15:10: Petr Mošner:
   Structure and properties of TeO\textsubscript{2} containing borophosphate glasses

15:10 – 15:30: Ivana Rösslerová:
   Investigation of thermal behavior of lead molybdate-phosphate glasses by various thermoanalytical methods

15:30 – 15:50: Vladimír Kucek:
   Nonstoichiometry and physical properties of GaGeTe

15:50 – 16:15: Coffee break

16:15 – 17:15: Monday Session II
   Location: Lecture Hall   chairperson: Miroslav Kučera

16:15 – 16:35: Martin Hanuš:
   Optimization of scintillation properties of epitaxial garnet films doped by rare-earth ions

16:35 – 16:55: Jiří A. Mareš:
   Scintillating properties of rare earth aluminum garnets

16:55 – 17:15: Maksym Buryi:
   Electron Paramagnetic Resonance study of Lu\textsubscript{2}SiO\textsubscript{5} and Y\textsubscript{2}SiO\textsubscript{5} scintillators doped by cerium
17:15 – 17:35: Miroslav Menšík:  
Resonant and non-resonant components of the rate of excited state decay

18:00 – 19:00: Dinner

---

**Tuesday, 4 September 2012**

08:40 – 10:20: **Tuesday Session I**

*Location: Lecture Hall*  
*chairperson: Zdeněk Kožíšek*

08:40 – 09:20: Marian Koman:  
*Ligand ronicol, which brings together and divides*

09:20 – 09:40: Blažena Papánková:  
*Determination of large zero-field splitting parameters.*

09:40 – 10:00: L’ubor Dlháň:  
*Iron oxide minerals deposited in the human spleen*

10:00 – 10:20: Maroš Martinkovič:  
*Solderability of ceramic materials*

10:20 – 10:45: Coffee break

10:45 – 11:45: **Tuesday Session II**

*Location: Lecture Hall*  
*chairperson: Petr Mošner*

10:45 – 11:05: Kateřina Rubešová:  
*Thin films of lithium niobate prepared by sol-gel method*

11:05 – 11:25: Vit Jakeš:  
*Optimized sol-gel synthesis of LiNbO₃ for PLD targets*

11:25 – 11:45: Jakub Cajzl:  
*Erbium doped lithium niobate - study of its luminescence properties and structure*

12:00 – 13:00: Lunch
14:00 – 15:40: **Tuesday Session III**

*Location: Lecture Hall*  
*chairperson: Blažena Papánková*

14:00 – 14:20: Zdeněk Kožíšek:  
*New approach to crystal nucleation from solution on active centers*

14:20 – 14:40: Jan Kulveit:  
*Formation of structured nanophases in halide crystals*

14:40 – 15:00: Karel Nitsch:  
*Study on glass transition temperature of meta-phosphate glasses*

15:00 – 15:20: Miroslava Rodová:  
*DSC and TMDSC studies on glass transition temperature of alkali rare earth metaphosphate glasses*

15:20 – 15:40: Robert Král:  
*Study of temperature field measurement at vertical Bridgman method in static and dynamic arrangement*

15:40 – 16:05: **Coffee break**

16:05 – 17:05: **Tuesday Session IV**

*Location: Lecture Hall*  
*chairperson: Maroš Martínkovič*

16:05 – 16:25: Jaromír Drápal:  
*Regional Materials Science and Technology Centre in Ostrava*

16:25 – 16:45: Kateřina Rubešová:  
*Material science education at ICT Prague*

16:45 – 17:05: Petr Holzhauser:  
*History and Presence of International Chemistry Olympiad*

17:05 – 17:45: **CSACG meeting**

18:00 – 19:00: **Dinner**

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**Wednesday, 5 September 2012**

08:40 – 10:20: **Wednesday Session I**

*Location: Lecture Hall*  
*chairperson: Marian Koman*

08:40 – 09:20: Alexander Kromka:  
*Diamond Films Deposited by Oxygen-Enhanced Linear Plasma Chemistry*
09:20 – 09:40: Štěpán Potocký:
Diamond structures grown from polymer composite nanofibers

09:40 – 10:00: Vít Jirásek:
Modeling of thermal stress induced during the diamond-coating of AlGaNGaN high electron mobility transistors

10:00 – 10:20: Tibor Ižák:
Growth rate enhancement and morphology engineering of diamond films by adding CO₂ or N₂ in hydrogen rich gas chemistry

10:20 – 10:45: Coffee break
Location: Lecture Hall

10:45 – 11:45: **Wednesday Session II**
Location: Lecture Hall  
chairperson: Zdeněk Potůček

10:45 – 11:05: Karel Král:
Power-law photoluminescence decay in quantum dots

11:05 – 11:25: Zdeněk Remeš:
Photoluminescence and infrared absorption spectroscopy of the aminated nanocrystalline diamond surface

11:25 – 11:45: Pavla Řýparová:
Antibacterial studies on nanocrystalline diamond thin films

12:00 – 13:00: Lunch

14:00 – 18:00: Joint Meeting

18:30 – 22:00: Conference Dinner and Night Discussion
Location: Winevault

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**Thursday, 6 September 2012**

08:40 – 10:20: **Thursday Session I**
Location: Lecture Hall  
chairperson: Jaromír Drápala

08:40 – 09:20: Jan Polák:
Growth of profiled sapphire single-crystals by EFG method

09:20 – 09:40: Martin Klejch:
Growth of sapphire single-crystal fibres and their applications
09:40 – 10:00: Zdeněk Bureš:  
*Preparation and potential applications of silver and gold nanoparticles*

10:00 – 10:20: Jan Hostaša:  
*Transparent Yb:YAG ceramics, layered in situ composites*

10:20 – 10:45: Coffee break

10:45 – 11:45: **Thursday Session II**  
*Location: Lecture Hall*  
*chairperson: Jiří Luňáček*

10:45 – 11:05: Roman Yatskiv:  
*Thermally stable semimetal graphite n-type ZnO Schottky contact*

11:05 – 11:25: Jan Grym:  
*Porous III-V semiconductors*

11:25 – 11:45: Petr Kostelník:  
*Optimization of the bond and etch-back silicon-on-insulator manufacturing processes*

12:00 – 13:00: Lunch

14:00 – 15:40: **Thursday Session III**  
*Location: Lecture Hall*  
*chairperson: Lubor Dlháň*

14:00 – 14:20: Jiří Kotlan:  
*Dielectric properties of calcium titanate prepared by plasma spraying techniques*

14:20 – 14:40: Karel Dušek:  
*Influence of Humidity on Voids Formation Inside the Solder Joint*

14:40 – 15:00: Pavel Mach:  
*Current Loading of Adhesive Joints Aged in Environment with High Humidity*

15:00 – 15:20: Ivana Pilarčíková:  
*Affect of Carbon Black Modification on Electrical Properties of Carbon Black - Polymer Composites*

15:20 – 15:40: Pavel Žák:  
*Methods of Accelerated Growth of Tin Whiskers*

15:40 – 16:05: Coffee break
Thursday, 6 September 2012

16:05 – 17:25: **Thursday Session IV**

**Location:** Lecture Hall  
**Chairperson:** Jiří A. Mareš

16:05 – 16:25: Alexej Sveshnikov:  
*Preliminary study of polyvinyl alcohol nanotextile produced by electrospinning*

16:25 – 16:45: Iveta Klicmanová:  
*Electrospinning of polyvinyl alcohol for application in civil engineering*

16:45 – 17:05: Vítězslav Vydra:  
*Effect of gamma irradiation on pore-space of hardened cement paste*

17:05 – 17:25: Sárka Hošková:  
*Effect of sucrose as a retarder on some physico-chemical properties of cement paste*

18:00 – 19:00: Dinner

Friday, 7 September 2012

08:40 – 10:20: **Friday Session I**

**Location:** Lecture Hall  
**Chairperson:** Zdeněk Kožíšek

08:40 – 09:20: Zdeněk Potůček:  
*Luminescence Properties and Application of Impurity Ion Probes in Perovskite-type Oxides*

09:20 – 09:40: Jiří Luňáček:  
*Surface plasmon resonance sensor using spectral interference*

09:40 – 10:00: Michal Kamradek:  
*The Influence of Chloride Ions on Electrocatalytic Activity of Conducting Polymer/Pt Composite*

10:00 – 10:20: Tomáš Hlášek:  
*Comparison of various wet chemical methods used for cobaltite thermoelectrics synthesis*

10:20 – 10:45: Coffee break

**Location:** Lecture Hall
10:45 – 11:45: **Friday Session II**  
*Location: Lecture Hall*  
*chairperson: Zdeněk Kožíšek*

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<td>Estimation of grain deformation of polycrystalline materials</td>
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<td>11:05</td>
<td>Pavlína Ruleová</td>
<td>Preparation and characterization of $Bi_{2-x}Sr_xSe_3$ Single Crystals</td>
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<td>11:25</td>
<td>Jiří Stuchlík</td>
<td>Nanoparticles in hydrogenated silicon</td>
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12:05 – 13:05: Lunch
Preparation and potential applications of silver and gold nanoparticles

Zdeněk Bureš and Miroslav Vlček

Department of General and Inorganic Chemistry, Faculty of Chemical Technology, University of Pardubice, Studentská 573, 532 10 Pardubice, Czech Republic

Silver and gold nanoparticles can have multiple applications in electrochemistry, spectroscopy, surface enhanced Raman scattering, medicine, biosensing as probes for the detection of proteins and DNA molecules, sequence analysis, catalysis, as well as antistatic and antibacterial materials, etc., due to their unique physical and chemical properties. Nanoscale particles, since they have large surface areas, can easily agglomerate to form either small clusters or larger particles to minimize the total surface or interfacial energy of the whole system. Van der Waals attractive forces which tend to minimize the total surface energy of the system are responsible for agglomeration of fine particles. It is necessary to ensure repulsive interparticle forces to prevent the agglomeration of these particles. The water-based methods are the most frequently used for preparation of silver (AgNPs) and gold nanoparticles (AuNPs). Various polymers, surfactants, and coordinative ligands, which are able to protect the nanoparticles from their agglomeration, to acquire highly stable and well-dispersed nanodispersed metals, are mainly used as stabilizers in water-based methods.

In this investigation various reduction methods were used for preparation nanodispersed metal nanoparticles especially silver (AgNPs) and gold nanoparticles (AuNPs). Method using sodium borohydride Na[BH₄] and glucose as a reducing agent was found as a suitable method for AgNPs preparation. Method starting from H[AuCl₄] which is reduced by ascorbic acid was successively applied for synthesis of long term stabil colloidal AuNPs.

This work was supported by the grant P204/11/0832 from the Czech Science Foundation and by the grant 0021627501 from the Czech Ministry of Education, Youth and Sports.
Electron Paramagnetic Resonance study of Lu$_2$SiO$_5$ and Y$_2$SiO$_5$ scintillators doped by cerium

Maksym Buryi$^{1, 2}$, Valentyn Laguta$^1$, and Martin Nikl$^1$

$^1$Institute of Physics AS CR, Cukrovarnická 10/112, 162 00 Praha 6, Czech Republic
$^2$Czech Technical University in Prague, Faculty of Nuclear Sciences and Physical Engineering, Břehová 7, 115 19, Praha 1, Czech Republic

Trivalent cerium ions act as effective luminescent centers in Lu$_2$SiO$_5$:Ce (LSO:Ce) and Y$_2$SiO$_5$:Ce (YSO:Ce) scintillating crystals. These materials satisfy the requirements for gamma rays detection. They possess high density, high light yield (about 26000 ph/MeV), short decay time (about 40 ns) and find application in positron emission tomography. It is essential to get better understanding of Ce$^{3+}$ distribution inside the dielectric matrix of YSO/LSO and its properties. Ce$^{3+}$ substitutes for two lutetium (yttrium) sites: the first site CeI is surrounded by seven oxygen ligands and the second CeII by six oxygen ligands. Besides, each site contains two magnetically inequivalent Ce$^{3+}$ positions.

The aim of the present work was to study the cerium distribution in YSO/LSO crystals and to determine spectroscopic parameters of Ce$^{3+}$ ions by means of EPR.

Single crystals of Lu$_2$SiO$_5$: 0.044 mol.% Ce and Y$_2$SiO$_5$: 0.043 mol.% Ce were grown by the Czochralski method using the iridium crucible in argon atmosphere. YSO/LSO crystallize in monoclinic structure, space group C2/c. Crystals were cut in three orthogonal planes (a*b), (bc) and (a*c). The axis a* was deflected from the crystallographic axis a by the angle of 32 deg. Some measurements were also performed with powders. The spectra were acquired on Bruker X/Q-band E580 FT/CW ELEXSYS spectrometer at microwave frequency 9.5 GHz and at temperatures 5-15 K. Powder spectra were measured by using the echo detected field-swept EPR. Angular dependencies of Ce$^{3+}$ spectra were measured by conventional continuous wave EPR.

Ce$^{3+}$ ion has electron spin S=1/2. Therefore only three g-tensor parameters are required in order to describe its EPR spectrum. Due to the low symmetry of Ce$^{3+}$ positions in the lattice (C1), correct principal g-factors could be obtained solely from powder spectra. From the fitting of CeI powder spectra, the following g-factors were determined: (i) LSO: $g_1=0.561$, $g_2=1.685$, $g_3=2.238$; (ii) YSO: $g_1=0.545$, $g_2=1.609$, $g_3=2.319$. Proper orientations of g-tensor principal axes with respect to the crystallographic directions for each inequivalent position of Ce$^{3+}$ in lattice were determined from angular variations of the resonance lines measured in three crystallographic planes. The population of two Ce sites was calculated from the integral intensities of corresponding EPR lines. In particular, in LSO the population of CeII is only 3%.

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Erbium doped lithium niobate - study of its luminescence properties and structure

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Nowadays, there are available various laser-active ion doped materials. These materials are of great attention in photonics and optoelectronics because they have high potential in the future branch of optical communications systems. Single- and poly-crystalline materials are often doped with rare-earth ions for the purposes of various applications in optics and photonics. Among other materials, lithium niobate doped with erbium (Er:LiNbO₃) is unique due to its combination of special properties which makes it possible for simultaneous modulation and amplification of light (with wavelength around 1.5 µm).

This work is focused mainly on the relationship between fabrication techniques of thin erbium doped layers and the luminescence properties in the area around 1.5 µm, where the position of erbium in the crystal is a key factor. Various techniques were used for the erbium doping of LiNbO₃, i.e. erbium ion implantation, thermal diffusion of erbium from metal (Er) and oxide (Er₂O₃) layer, erbium doping from the melt at different temperatures, and electric field-assisted erbium doping from melt. Because lithium niobate is a crystal material there is also an importance in orientation of the structure, so due to that experiments were done with different crystallographic cuts. The focus was also placed on the study of subsequent annealing process of the doped samples at the different temperatures. Prepared samples were measured for the erbium concentration depth profiles (RBS method), luminescence properties in the region from 1440 to 1600 nm, lithium depletion in the surface area (NDP method), and the degree of ordering of the structure (RBS/Channeling). The prepared samples contains from 1 to 24 at. % of erbium in the depth ranging from 100 nm to 1 µm depending on the used technology of preparation. Overall, the results showed strong relationship between luminescence and the degree of ordering of the crystal structure that surrounds erbium ions. This relationship is substantially influenced by the selected erbium doping technology as well as by the post-doping annealing. For the complete understanding of the position of erbium in the LiNbO₃ structure, the theoretical model of Er:LiNbO₃ structure and the computer geometry optimizations were done.

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Thin films of lithium niobate prepared by sol-gel method

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Lithium niobate thin films doped by f-elements are widely used in waveguiding technologies. The films are produced by various methods – e.g. an ion implantation to a monocrystalline substrate, a pulsed laser deposition or a sol-gel technique. The sol-gel preparation of doped lithium niobate mainly uses alkoxide solutions. The metal alkoxides derived from primary lower alcohols are commercially available; however, they are highly reactive and water sensitive. Therefore less reactive derivatives must be prepared – e.g. the alkoxides of 2-methoxyethanol. These alkoxides are more stable in time and are less sensitive to hydrolysis. Their higher stability is caused by lowered polarity of metal-oxygen bond in 2-methoxyethoxide that moreover plays the role of bidentate ligand and thus spatially hinders metal centre from hydrolysis.

In this work, we present various syntheses of 2-methoxyethoxides of Li and Nb. Also alkoxides of Er and Yb were prepared and used at doping with these laser active ions. Precursor solutions were further used for the synthesis of mixed alkoxide LiNb(OCH₂CH₂OCH₃)₆. The solutions were applied at thin films preparation using dip- or spin-coating techniques. Li/Nb solutions were coated on sapphire (0001) substrates. Many parameters of coating techniques were optimized – e.g. spin speed, pull-out speed, the volume of spin-coated solution, the regime of the following thermal treatment etc.

We prepared the monophase polycrystalline thin films of LiNbO₃ oriented according to the sapphire substrates – i.e. LiNbO₃ (006) layers. We established dependence of thin film thickness on deposition parameters and on the number of coatings. Different recrystallization treatments were compared in order to enhance the microstructure and crystallinity of deposited films.

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Iron oxide minerals deposited in the human spleen

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There is growing information about a pathological biocrystallization of iron oxides in the human organs like brain and spleen. Several minerals of the nanosize have been detected so far in extracted and lyophilized tissues: magnetite \(\text{Fe}_3\text{O}_4\), maghemite \(\gamma\)-\(\text{Fe}_2\text{O}_3\), hematite \(\alpha\)-\(\text{Fe}_2\text{O}_3\) and ferrihydrite \(\text{5Fe}_2\text{O}_3.9\text{H}_2\text{O}\). The deposits can be visualized by the scanning/transmission electron microscope and identified by magnetometry and Mössbauer spectroscopy. Three samples extracted from the human spleen are under investigation: a reference sample from the undiagnosed individual (1), a sample with diagnosis for hemosiderosis (2), and a sample of patient suffering from hereditary spherocytosis (3). The SQUID magnetometry proves that all three samples carry a paramagnetic signal but with different extent. The sample 2 shows paramagnetism, and below the bifurcation point \(T < 50\) \(\text{K}\) it switches to an ordered phase. The sample 3 is paramagnetic in the whole temperature range (2 – 300 \(\text{K}\)); the field-cooled magnetization and zero-field cooled magnetization experiments show that the Curie temperature \(T_C\) lies above the room temperature. As a consequence, the magnetic hysteresis exists for 3 even at the room temperature that has been proven experimentally. Probably the deposits of magnetite/maghemite are origins of this magnetic behavior. The Mössbauer spectra at \(T = 5\) \(\text{K}\) were deconvoluted into a doublet (D) and three sextets (S1 – S3). The sextet S1 shows parameters close to that identified for hematite. The other two sextets represent ferrihydride core and surface Fe atoms. The doublet can be ascribed to small ferrihydride and/or magnetite particles. It is noteworthy that the sample 3 exhibits higher deposits of hematite (S1) than the reference 1. On concluding: (i) the TEM photograph confirms the presence of the magnetite among the iron-oxide deposits; (ii) the magnetometry confirms the presence of the hysteresis loop even at the room temperature and the magnetic response different from the ferritin; (iii) the Mössbauer data are compatible with the presence of a number of iron-oxide minerals in the samples under study.

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Regional Materials Science and Technology Centre in Ostrava

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The objective of the project “Regional Materials Science and Technology Centre (RMSTC)” is to build a research infrastructure of laboratories and create research teams that will develop, prepare and optimise the properties of advanced materials and technologies for their preparation for the application sphere. From the viewpoint of material the activities are aimed at preparation of highly pure materials, special alloys, bio-medical materials, development of materials for high temperature applications, preparation of materials by progressive powder metallurgy technologies, preparation of nano-crystalline materials based on non-ferrous metals, their alloys and steels processed by severe plastic deformation. The project solves the issues of the processes running in the liquid phase, both in steel and in materials based on non-ferrous metals, in order to improve their structural perfection with impact on the service properties. In the area of modelling the activities of the RMSTC are focused on the physical and mathematical modelling of the processes of materials forming, including forging and application of the latest research and development findings in the field of technology of forming the components. Within solution of research and development activities of the RMSTC the findings obtained at solution of the research plans are further developed and applied at solution of the issues that are realisable in the application sphere, and which offer high innovation potential at solution of specific tasks in practice.

The structure of the RMSTC centre was also subordinated to the envisaged outputs into the industrial sphere with a strong application potential. Organisational structure of the centre is divided into five departments:

1. Department of preparation of materials (laboratory of pure metals and technological laboratory for preparation of special alloys).
2. Department of powder technologies (laboratory of magnetic and ceramic materials, laboratory of friction composites).
3. Department of forming processes (laboratory of processes of severe plastic deformation, laboratory of materials with ultra fine-grained structure prepared by forming, laboratory of modelling and optimisation of the forming technologies).
4. Department of evaluation of materials properties (laboratory of structural analysis, laboratory of mechanical properties, laboratory of chemical analyses, laboratory of surface analysis and corrosion, laboratory of physical properties).
5. Department of experimental verification of technologies and applications (laboratory for experimental verification of technologies for the production of new materials and laboratory of modelling of processes in liquid and solid phases).

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Influence of Humidity on Voids Formation Inside the Solder Joint

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Void is defined such as blow hole in the solder joint. Voids may degrade the mechanical and conductive properties of the solder joint and thus decrease the reliability [1]. Voids are caused by the amount of outgassing flux that gets entrapped in the solder joint during reflow [2]. The outgassing substance is generally produced by the evaporation of the solvent in the solder paste and the rheological additives in the solder paste that may evaporate in the heating process during reflow [3]. The outgassing substance may also be generated by the metallization of the substrate, component or the solder powder surface during the fluxing reaction in the reflow process. Previous studies have indicated that the reflow process and the solder material are the most significant factors that affect void formation [4]. Article deals with influence of humidity on voids formation inside the solder joint. Experimental part was carried on testing samples, where we used three types of solder pastes (Sn$_{62}$Pb$_{36}$Ag$_{2}$, Sn$_{96.5}$Ag$_{3}$Cu$_{0.5}$ and Sn$_{95.5}$Ag$_{4}$Cu$_{0.5}$), three types of reflow technology (hot air, vapor phase and infra radiation) and three types of humidity environments. PCB together with SMD resistors, were placed in three different types of humidity environments (dry - humidity was less than 5%, ambient - average humidity was around 60 % and humid environment - 100 % humidity) for 500 hours. After that we took the components from environments and we prepared samples – the solder paste was applied on the solder pads using a screen printing process, components were placed on PCB and then the solder paste was reflowed. The inspection was made on X-Ray and following diagnostic was made by image analysis with special software. We evaluate the number of voids together with their total area of all voids inside each solder joint. Results indicate that number of voids together with their total area mainly depends on type of used solder paste. Dependence of used reflow process on voids formation is minor and the influence of components humidity on voids formation wasn’t observed.

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Increasing number of papers has been devoted to the preparation of porous III-V semiconductors and their potential applications [1]. On the other hand, epitaxial growth on porous substrates has been rarely investigated and the results of these investigations are far from giving deep understanding of the phenomena taking place at the porous substrate/epitaxial layer interface.

There is a limited number of III-V semiconductor substrates which are available at acceptable quality and cost. Restriction to lattice-matched systems would greatly limit the number of applications. Development of vapor phase growth techniques allowed to precisely control the layer thickness and uniformity on the atomic level. Still, when the critical layer thickness is exceeded, misfit dislocations are created having negative impact on the performance, reliability and lifetime of semiconductor devices [1]. A number of defect engineering approaches are available to gain control over the generation of defects during heteroepitaxial growth. One of the approaches consists in the growth on a porous substrate to accommodate elastic strains at the heteroepitaxial interface [2]. An essential step in successful application of porous substrates in epitaxial growth is to achieve control over their properties: the surface roughness, pore size, orientation, density and depth.

We report on the electrochemical preparation of InP, GaAs and GaP porous substrates, their heat treatment in As rich environment and their overgrowth by metalorganic vapor phase epitaxy and liquid phase epitaxy. The goal is to demonstrate that porous substrates are capable of accommodating strain at the interface with lattice mismatched epitaxial layers.

The pore etching was carried out in an electrochemical cell containing a fluoride-iodide and chloride aqueous electrolytes [3] using a three-electrode configuration. The porous structures before and after the epitaxial growth were characterized by Nomarski differential interference contrast microscopy (NDICM), scanning electron microscopy (SEM), atomic force microscopy (AFM), low temperature photoluminescence (PL), x-ray diffraction (XRD) and transmission electron microscopy (TEM).

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Optimization of scintillation properties of epitaxial garnet films doped by rare-earth ions

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Scintillators are unique systems for detection of high energy particles and photons. Rare-earth doped aluminum garnets are considered perspective scintillators. They have high density, fast scintillation response, high quantum efficiency and good chemical and mechanical stability. Thin cerium doped aluminum garnet films are promising especially for X-ray imaging and scintillation screens with micrometer resolution [1] but these materials can be also used for luminescent illumination (fluorescent lamps, white LEDs [2]). In this work we studied single crystalline rare-earth doped garnet films prepared by isothermal liquid phase epitaxy (LPE). The films were grown from the flux onto various substrates, such as lutetium (LuAG), yttrium (YAG) aluminum garnet and gadolinium gallium garnet (GGG). The films were grown from the flux at temperatures around 1000 °C. The rare-earth doped garnet films prepared from the PbO-B2O3 flux exhibit good optical, emission, structural, and surface properties. The major drawbacks of the PbO flux are divalent and tetravalent Pb and Pt impurity ions, which come from the flux and cannot be eliminated and which can evoke energy loss due to nonradiative transitions [3]. We have recently demonstrated that the Ce-doped garnet films prepared from the lead-free BaO-BaF2-B2O3 flux exhibit excellent optical and emission properties comparable to the Czochralski grown bulk crystals [4]. However, the epitaxial films grown from this flux were sensitive to stress produced by the viscous BaO flux remaining on the sample surface and the films with large lattice mismatch were difficult to grow. In this work our attention was focused on the study of multicomponent Ce³⁺-doped \((Y_{3-x}Gd_x)(Al_{5-y}y Ga_y Sc_z)O_{12}\) garnet systems, where \(x, y = 0 - 5, z = 0 - 2\). The aim of this research was the study of Gd, Ga and Sc substitution on the emission and scintillation properties aluminum garnets. Optical, luminescent, radioluminescent, and scintillation characteristics of films were measured by the methods of time-resolved emission spectroscopy. The structure and chemical composition was measured by the X-ray diffraction and electron probe microanalysis techniques, respectively. Detailed discussion and summary of emission and scintillation properties of epitaxial films and comparison with bulk single crystals will be presented.

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Comparison of various wet chemical methods used for cobaltite thermoelectrics synthesis

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The figure of merit for thermoelectrics is defined as dimensionless \( ZT = S^2T/\lambda \rho \). From this perspective, the optimal combination of electrical and thermal conductivity and Seebeck coefficient corresponds to semiconductors. Thermoelectric materials such as Bi-based alloys, lead telluride or Si-Ge alloys are already commercially used in thermoelectric power generation and refrigeration. However, other materials are synthesized, as well, with aim to enhance thermoelectric performance. For example, so-called “phonon glasses-electronic crystals” (e.g. skutteridites - CoSb₃, clathrates - NaₓSi₁₃₆) are materials with lowered thermal conductivity.

Oxide materials are in the spotlight of researchers due to their low toxicity, stability up to high temperatures and also low price. The oxides do not reach as high figure of merit as alloys materials but they have large perspectives. Besides doped ZnO or layered hybrid oxides (e.g. doped Srᵣ₋₁TiₓOₓ), misfit cobalt oxides with general formula \([M_m A_2 O_{m+2}]^{R.S.} [CoO_2]_y^{HEX.}\) are developed. Apart from the “mother” phase NaₓCoO₂, Ca₃Co₃.₉₅Oₓ (CaCo-34) and Bi₂Sr₂Co₁.₈Oₓ (Bi-222) phases are the most studied.

In this contribution, we present comparison of various water based sol-gel methods for CaCo-34 and Bi-222 thermoelectrics preparation. In the Bi-222 case, Bi³⁺ behavior in water solution (easy hydrolysis) had to be taken into account at sol-gel system selection. We compare the sol-gel prepared samples with the samples synthesized using solid state process. Various gel decomposition atmospheres and thermal regimes were applied; the phase composition and grain size of precursors was established. The decomposition of gels under nitrogen (during which amorphous carbon matrix is formed) and subsequent annealing in oxygen produces much smaller grains of precursors. The temperature dependence of electrical resistivity, thermal conductivity and Seebeck coefficient was measured. The calculated figure of merit and the bulk density of the final samples were compared for all sol-gel methods used.

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History and Presence of International Chemistry Olympiad

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The idea to organize international competition was born in 1968 in former Czechoslovakia. Only three countries – former Czechoslovakia, Poland and Hungary – had participated. Till nowadays the International Chemistry Olympiad (IChO) has grown to monumental dimensions. In 2011 about 273 competitors from 70 countries have participated on the 43rd IChO in Ankara.

In the Czech Republic the students for the national representation are selected on the base of National round of Chemistry Olympiad (January) and two preparatory seminars. The best 16 students from national round are invited for one-week theoretical seminar (traditionally organized in March at ICT Prague), where they have theoretical lectures and tests thematically based on preparatory tasks provided by the host country. The best 8 students (results from both national round and theoretical seminar) are invited for another one-week practical seminar (traditionally organized in April at Charles University, Faculty of Natural Sciences), where students work in laboratory on practical problems (also mainly preparatory tasks). Finally the best 4 students (averaged results from all three events) are selected to represent the Czech Republic at IChO.

The IChO goes ahead in July in different countries (in 2012 the 44th IChO takes place July 21 – 30 in Washington DC at University of Maryland). The national team consists of 4 students and 2 mentors. The mentors’ most important role is the careful translation of competition tasks to national language, communication with international Jury and arbitration of the points assignment. The Czech team is traditionally successful, from the origination of the Czech Republic students were awarded by 27 bronze, 23 silver and 11 gold medals.

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Chemical admixtures are used to alter the properties of concrete for many years. The most important admixtures are accelerators and retarders, compounds that accelerate or retard setting or hardening, to decrease the water quantity needed to obtain a given degree of workability, or to entrain in order to increase the resistance of the concrete from freezing.

The effect of retarding admixtures on the hydration of Portland cement has been extensively studied. Several theories have been proposed to explain how admixtures affect the hydration clinker compounds. These can be conveniently summarized as adsorption, precipitation, complexation, and nucleation. This problem is very complex.

Many organic compounds have the properties of retarder-saccharides, hydroxycarboxylic acids, lignosulfonates are examples. They effect in low concentrations. Most of them contain one or more groups in which oxygen atoms are attached to adjacent carbon atoms, such as HO=C-C=O, or otherwise able to approach each other closely. Adsorption on either a clinker phase or hydration product might cause retardation, in the latter case through interfering with growth.

The effect of sucrose on the early hydration of Portland cement paste by determining of Ca$_2^+$ ions, calorimetry and differential thermal analysis (DTA) has been studied in this contribution. Compressive strengths of samples in the presence of different concentration of sucrose at 3, 7, 14, 28 and 90 days were determined.

The concentration of free Ca$_2^+$ ions in hydrated cement paste during the first two hours of cement hydration for cement paste and heat of hydration by SETARAM calorimeter at 30$^\circ$C in the presence and absence of sucrose as a retarder was measured. The resulting curves for both measurement methods show a significant difference, which is slowing down the cement hydration reaction by adding sucrose. DTA and compressive strengths curves also are changed considerably in presence sucrose.

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Transparent Yb:YAG ceramics, layered in situ composites

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Transparent YAG (yttrium aluminum garnet) ceramics doped with rare earth ions are interesting above all for applications in solid-state lasers, when doped with ions such as Nd\(^{3+}\), Yb\(^{3+}\) or Er\(^{3+}\). Yb:YAG allows significantly high dopant concentrations thanks to the similar size of Y\(^{3+}\) and Yb\(^{3+}\) ions; its simple electron structure, low thermal loading, and the possibility of pumping by InGaAs laser diodes makes Yb:YAG an ideal active laser material for high power applications. Recent development of high power solid-state lasers requires also more specific and complicated architectures, such as layered structures with varying dopant concentration for better thermal management and thermal lensing control and applications like thin-disk lasers. This work reports on the feasibility of fabrication of architectures with controlled Yb content via subsequent uniaxial pressing, followed by cold isostatic pressing and solid state reaction sintering in high vacuum with TEOS as sintering additive. Samples of undoped YAG and 5 and 10 at.% Yb:YAG were prepared and characterized. Further, the fabrication of a transparent ceramic sample with layers of undoped and 10 at.% Yb-doped YAG is shown. Contrary to single crystal processing, where fabrication of such composite materials would require bonding of separately grown crystals, ceramic processing allows in situ fabrication.

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Growth rate enhancement and morphology engineering of diamond films by adding CO$_2$ or N$_2$ in hydrogen rich gas chemistry

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Synthetic diamond film is very promising material for many applications in tribology, electronics, biotechnology or bioelectronics. These applications are determined not only by their intrinsic properties (high hardness, high thermal conductivity, chemical stability, biocompatibility, etc.) but also by chemical purity and morphology. Crystallographic morphology, especially nano- or micro-sized dimension of crystal, strongly influences electrical properties and consequently the suitability to realize final electronic devices (sensors, electron emission or high power devices, etc.). In some case, the fabrication of such device requires larger thickness (up to 5-10 $\mu$m) which usually require long time deposition (17 h). Thus, the main technological demand is enhancing the growth rate which reduces not only the process time but also production cost. Furthermore, shorter deposition process is more favourable for microelectronic industry to minimize temperature sensitive processes (diffusion of atoms, cracking, stress evolution, etc.).

In this study, we demonstrate significant enhancement of the diamond growth rate by addition of carbon dioxide (CO$_2$) and/or nitrogen (N$_2$) to the hydrogen rich gas mixture (CH$_4$+H$_2$). Diamond films and/or structures were grown by microwave plasma enhanced chemical vapor deposition system with ellipsoidal cavity resonator. Surface morphology of diamond films was analysed by Scanning Electron Microscopy. The chemical composition, i.e. presence of sp$^3$ (diamond) and sp$^2$ (non-diamond phases) bonded carbon was determined from Raman measurements provided at two different excitation wavelengths of 325 nm and 442 nm, respectively. We observed that increasing of CH$_4$ concentration in the gas mixture up to 8% resulted in increased growth rates (up to 900 nm/h) almost linearly. This is due to the higher density of CH$_3$ radicals, which are known as a dominant precursor for diamond growth.

Unfortunately, diamond film quality decreased due to the dominance of sp$^2$ carbon phases, as confirmed by Raman measurements. We found that increasing of CH$_4$ concentration increased also spontaneous nucleation. Adding of N$_2$ and CO$_2$ into CH$_4$/H$_2$ gas mixture enhanced growth rates. Nitrogen shifts the growth from micro- to nanocrystalline character. At methane concentration of 5% CH$_4$, nitrogen further shifts the growth in formation of diamond nanowire structures. Adding of CO$_2$ to the gas mixture improved the film quality. Thus, higher methane concentration should be used at keeping good enough film quality. The extended region of diamond formation in Bachmann ternary diagram as well as the changes in plasma chemistry and growth kinetics will be discussed too.

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Optimized sol-gel synthesis of LiNbO₃ for PLD targets

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The optical properties and possibility of their tuning by doping make lithium niobate one of the promising materials for possible photonic applications nowadays. Its use is not limited only to bulk, thin layers have been widely studied as well. We use pulsed laser deposition (PLD) technique for thin films preparation. In this method, it is essential for the target to have the maximal bulk density possible.

We tested the precursor powders prepared by sol-gel method – Pechini polyesternification using citric acid and ethyleneglycol as organic matrix components. The prepared precursors were decomposed afterwards using various temperatures (800, 600 and 400 °C) and atmospheres (air, N₂/O₂). Out of several combinations tested, the decomposition at 800 °C in nitrogen followed by oxidation at 400 °C in oxygen was found to be the best way to achieve minimal particle size of powder precursor resulting in denser targets, a prerequisite for the successful deposition of thin layers via PLD.

This optimized process was used for preparation of four targets of different composition: pure LiNbO₃, LiNbO₃ doped with Er³⁺ ions (1 and 3 % w/w) and LiNbO₃ co-doped with Er³⁺ and Yb³⁺ ions (0.5 % w/w each cation). Each target contained 10 % w/w of Li⁺ ions in excess due to lithium losses during the PLD processing. The phase composition, thickness, roughness and optical properties of prepared layers were measured. In comparison to layers deposited from commercially available monocrystalline target, the layers prepared from our polycrystalline targets showed better characteristics.

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Modeling of thermal stress induced during the diamond-coating of AlGaNGaN high electron mobility transistors

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A thermally-induced stress during the microwave-plasma-enhanced chemical vapor deposition of a thin nanocrystalline diamond (NCD) films on GaN/AlGaN heterostructures and their subsequent cooling off was simulated in the CFD-ACE+ software. The samples intended to use in HEMT (High Electron Mobility Transistor) devices were prepared by two different methods: (a) continuous diamond film deposition followed by selective etching and (b) the selective growth of patterned diamond films. The finite-element method on the axisymmetric geometry was used to calculate the thermal deformations of these two types of samples. The qualitative dependencies of the deformations on the diamond film thickness and the substrate material were found. The lowest stresses were found on the Si substrate, thanks to its lowest thermal expansion. The simulation results of the structure with continuous and selectively-grown patterned film were compared.

This work occurred in frame of the LNSM infrastructure and was also supported by the project of the National Science Foundation under the No. P108/12/G108.
Conducting polymer/Pt (CP/Pt) composites found significant application in the field of fuel cells where they are considered to be an alternative electrode material. Platinum serves as an electrocatalyst and poisoning of its surface can lead to decrease of the fuel cell efficiency. Despite the chloride ions sorption on platinum surface is well known effect, practically only the chlorocomplexes of platinum, $\text{H}_2[\text{PtCl}_6]$, $\text{K}_2[\text{PtCl}_6]$, $\text{H}_2[\text{PtCl}_4]$, $\text{K}_2[\text{PtCl}_4]$, $\text{PtCl}_4$, are used for CP/Pt synthesis. Together with the fact that chlorides are common impurity in drinking water there is a risk of decrease of electrocatalytic activity of composite in fuel cell. Yet the systematic study of this effect has not been done. The aims of this study are: (i) to find suitable, chloride free and commercially available platinum compounds which can be used for CP/Pt composites synthesis, (ii) to study the influence of the precursor on platinum particles morphology and (iii) to measure electrocatalytic activities of CP/Pt composites for methanol oxidation reaction (MOR) and for hydrogen oxidation reaction (HOR) and determine the influence of chloride ions on these activities. Composites based on polyaniline (PAni) and polypyrrole (PPy) were studied. The films were prepared electrochemically. Glassy carbon rotating disc electrode, area 0.196 cm$^2$, served as a support. PAni was synthesized using cycling voltammetry, PPy was prepared potentiostatically at the potential 1 V vs. SHE. Platinum particles were deposited galvanostatically and potentiostatically under different conditions from $\text{K}_2[\text{PtCl}_4]$, $\text{K}_2[\text{Pt(ox)}_2]$, $[\text{Pt(NH}_3)_4](\text{NO}_3)_2$ and $\text{Pt(NO}_3)_2$ water solutions. The samples with different amounts of platinum were characterized by scanning electron microscopy and by means of image analysis. Subsequently the electrocatalytic activities for MOR and HOR under different chloride concentration were measured. The activities for MOR were measured using cyclic voltammetry, activities for HOR were obtained by Koutecky-Levich analysis of potentiostatic polarization curves. It was found that the only compounds from which the platinum can be deposited on the PAni and PPy films are $\text{K}_2[\text{PtCl}_4]$ and $\text{K}_2[\text{Pt(ox)}_2]$. The morphology of obtained Pt particles differs strongly. The precursors $[\text{Pt(NH}_3)_4](\text{NO}_3)_2$ and $\text{Pt(NO}_3)_2$ are not suitable for Pt depositin on CP films. It was also found that chloride ions strongly decrease the electrocatalytic activity. Concentration of chloride anions $10^{-2}$ mol dm$^{-3}$ leads to decrease of activity for HOR of about 50–70%. The influence of the chloride ions on the HOR on the PPy/Pt composites is dependent on the electrode potential. The influence of the chloride ions on the MOR is even more pronounced, in the presence of above stated chloride ions concentration the platinum surface is almost completely blocked and the MOR doesn’t take place.
Growth of sapphire single-crystal fibres and their applications

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Sapphire, the single-crystal of Al₂O₃, is a versatile material with plenty of applications due to its material properties. Thanks to perfect crystalline structure, it maintains its unmatched characteristics even when processed into very thin filaments (with diameters ranging from 100 to 1000 µm). In this paper we deal with preparation and characterization of bare sapphire fibres and their applications.

The production of sapphire fibres in Crytur is achieved via the “edge-defined film-fed growth” (EFG) technique. The principle of this process is that the melt rises through a die from the heated crucible into a small basin of defined shape from which the filament is drawn. We used molybdenum dies to produce fibres 200–500 µm thick and 1500 mm long. The crystals were pulled at a rate of 60–300 mm/h. The two principal designs of the dies are given in this paper. We further discuss and show the possible defects in the crystal lattice and fibre integrity typical for the EFG method, including the cause of such troubles. The major imperfections are micro-bubbles, growth stripes, facets, surface defects and impurities incidence. These defects were studied using optical microscopy with 500 × magnification. The attenuation of fibres was measured with pulsed Er:YAG laser. The measurement was conducted for different fibre lengths 50–160 mm. The wavelength of the laser radiation was 2.94 µm and input energy ranged between 10–110 mJ.

Sapphire optical fibres can be prepared in such a quality by the EFG process which allows their usage in optical applications with working range up to few meters. The disadvantage of sapphire fibres prepared by EFG over traditional silica-made ones are their higher loss, therefore their length is limited. Sapphire fibres can be used in medical equipment, namely in the delivery of Er:YAG laser beam at 2.94 microns for minor skin and dental surgeries. They are biologically inert, non-toxic and have high laser damage threshold. The resistance of sapphire can be employed in spectrometric and pyrometric measurements in harsh environments, e.g., inside chemical reactors, furnaces, combustion engines and in other extreme conditions. These are the two current major applications of sapphire fibres. However, other sapphire features, e.g., thermal or electrical, might be utilized as well. Sapphire fibres prepared by EFG method exhibit extraordinary mechanical durability and maintain their shape and strength even for very high temperatures – up to 2000 °C (the melting point is 2053 °C). They are hard but still show some flexibility and can be bent at a radius 200 × the filament diameter.

Electrospinning of polyvinyl alcohol for application in civil engineering

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The goal of this paper is to create a complex overview of a process of electrostatic spinning. The principle of this process, pros and cons of existing methods, parameters of the process and used materials are discussed. Special attention is paid to Nanospider technology, which has been used in our experiments.

In experimental part of the paper a determination of a surface density of textile is described. The textile is composed of a supporting textile (a spun bond) made of polypropylene and nanofibers (nanofiber textiles) made of polyvinyl alcohol. We have measured the weight per square area for three types of samples: the supporting textile (the spun bond), the supporting textile (the spun bond) with the nanofibers of polyvinyl alcohol, and the nanotextile by itself. The purpose of the experiments was to determine the most appropriate type of samples for further experiments.

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Solderability of ceramic materials

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The issue of solderability of ceramic materials consists mainly in the fact, that the ceramic materials are non-wettable for commercial solders, owing to their ionic and covalent bond amongst the atoms. There exist several ways to ensure wettability of ceramic material surface. One of them for example consists in coating of ceramic material with a metallic layer. More perspective way seems to be application of soldering alloys which are alloyed by small amount of elements providing high affinity to some component(s) of ceramic material. The main group are the so-called active solders which contain from 1 to 5 weight % Ti, eventually another active element as Zr and/or Hf [1]. The solders alloyed by a small amount of lanthanoids are another group of solders that can wet the ceramic material. Content of lanthanoids usually varies from 0.1 up to 2 wt. % [2]. The last group consists of solders containing indium in amount from 20 to 100 wt. % [3]. The aim of work was to select a suitable solder from each group, to analyse the interactions between the solder and ceramic substrate and to determine the strength of fabricated soldered joints. To improve the wettability, soldering in combination with an active power ultrasound was employed. Experiments were performed with Al2O3 ceramics. For comparison, also reference Cu substrate material with 4N purity was used. The soldering alloy type Sn2Ti, Sn3.5Ag4Ti(Ce, Ga), Sn2La and Bi25In18Sn were used. EDX analysis was applied for the study of interactions on the boundary of soldered joints. Different mechanisms of joint formation were revealed. In case of solders containing 2 to 4 wt. % Ti it was found out that the active element in solder reacts with the surface of ceramic material in soldering process at formation of a reaction layer. The reaction layer allows wetting of ceramic material. The bond is of diffusion character. In case of In containing solders, the dominant role in joint formation is played by indium. Indium exerts a high affinity to oxygen and it is therefore supposed that it is combined with oxygen from the air in soldering process at formation of complex indium oxides which subsequently enter into reaction with the surface oxides of ceramic material. The bond is of quasi-adhesive character. Considerably higher shear strength was achieved with Ti containing solders. This was caused on one hand by higher strength of diffusion bond with the surface of ceramic substrate and, on the other hand by higher cohesion strength of soldering alloy proper.

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Ligand ronicol, which brings together and divides

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The pyridylmethanols [2-pyridylmethanol (2-pyme), 3-pyridylmethanol (3-pyme), as well ronicol (ron) and 4-pyridylmethanol (4-pyme)] have interesting properties in biological systems. The neutral ligand ronicol in coordination compounds prefer definite types of structure. The ronicol is usually N,O-bridging ligand and its complexes are coordination polymers resulting in 1-D chains or 2-D sheets, but only for a few complexes, it is terminal ligand [Cu(3-Mesal)\(_2\)(3-pyme)\(_2\)(H\(_2\)O)].(H\(_2\)O) [3-Mesal = 3-methylsalicylate] with monomeric structure and [Cu(3-MeOsal)\(_2\)(3-MeOsal)\(_2\)(3-pyme)\(_4\)] [3-MeOsal = 3-methoxysalicylate] with dimeric structure. The different transitional variations in this rich group of compounds will be discussed. A large number of tetranuclear copper(II) complexes Cu\(_4\)OX\(_6\)L\(_4\) (where X = Br, Cl and L = monodentate O-, S-, N-, P- and Sb- donor atom ligands) have been prepared and structurally studied. Recently, new groups of tetranuclear complexes with nitrogen donor ligands of pyridine type have been prepared. The neutral ligands are 4-Mepy – 4-methylpyridine and ronicol (3-Mepy – 3-methylpyridine).

The Cu\(_4\)OCl\(_6\)(4-Mepy)\(_4\) complex crystallizes in the triclinic crystallographic system. Independent part of the unit cell contains four symmetrically independent molecules of the same composition. The Cu\(_4\)OBr\(_2\)Cl\(_4\)(4-Mepy)\(_4\) complex crystallizes in the triclinic crystallographic system too. The unit cell contains eight symmetrically independent molecules with following compositions: four molecules of Cu\(_4\)OBr\(_1\)Cl\(_5\)(4-Mepy)\(_4\), two molecules of Cu\(_4\)OBr\(_2\)Cl\(_4\)(4-Mepy)\(_4\) and two molecules Cu\(_4\)OBr\(_3\)Cl\(_3\)(4-Mepy)\(_4\).

The crystal structure of [Cu\(_4\)OCl\(_6\)(ron)\(_4\)] consists independent monomeric molecules [Cu\(_4\)OCl\(_6\)(ron)\(_4\)]. The molecule contain the structural units Cu\(_4\)OCl\(_6\) in the central oxygen atom is tetrahedrally coordinated to four copper atoms, which are bridged in pairs by six chloride atoms in trigonal bipyramidal coordination. The fifth coordination sites of Cu(II) atoms are occupied in axial positions by nitrogen atoms of ronicol ligands and do not form polymeric structure.

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Optimization of the bond and etch-back silicon-on-insulator manufacturing processes

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We have studied the bond and etch-back silicon on insulator (BESOI) manufacturing processes. The top silicon layer, called device layer, was studied from its epitaxial growth on 2 μm thick SiGeB etch-stop layer, through 850 °C / 12 h bond strengthening annealing and selective etching, to the final chemical-mechanical planarization (CMP) treatment. We have found that the device layer thickness is reduced during the annealing and selective tetramethyamonium hydroxide and HNA (mixture of hydrofluoric, nitric and acetic acids) etching processes. This thickness reduction was found to be 0.592 μm, independently on the original device layer thickness. We have also found that the wafer surface is covered by a thin silicon suboxide layer after the HNA etching. The layer can be however easily removed by CMP with stock removal higher than 0.1 μm. Such process also polishes the wafer surface to prime quality micro-roughness. For studied BESOI process we therefore propose additional epitaxial growth of 0.8 μm to the thickness target of the lightly doped epitaxial layer (future device layer) and using CMP stock removal of 0.2 μm to obtain BESOI wafer with superior device layer surface micro-roughness and thickness uniformity.

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Dielectric properties of calcium titanate prepared by plasma spraying techniques

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In recent decades plasma spraying has become a well accepted technology as the coating method for metallic and ceramic materials and has been used in a variety of fields. Several works on dielectric properties of CaTiO₃ ceramics have been published, but there is a lack of research comparing plasma sprayed samples produced with different techniques. One series of samples was produced using a high-throughput water-stabilized plasma spray system WSP [1]. Spray distance SD was set to 350 mm and to 450 mm. A conventional atmospheric plasma-spray system, gas-stabilized plasma gun GSP F4 [2], was used for spraying of the other set of samples.

Capacity and loss factor of specimens was measured in the frequency range from 20 Hz to 5 MHz. Relative permittivity was calculated from measured capacities and specimen dimensions [3]. WSP samples have very high relative permittivity at frequencies lower than 1 kHz. The permittivity is strongly frequency dependent and it does not drop below 130. The GSP sample has lower relative permittivity, and was also more stable versus the tuned frequency. The most significant is high loss factor of the deposit from a spraying distance SD 450 mm. Sample SD 350 has lower and less frequency-dependent losses. At about 4 MHz the value of both samples is the same, about 0.1. The GSP sample has rather frequency-dependent loss factor, however more than 10 times lower than WSP SD 450 – only about 0.01 at 5 kHz.

Porosity was determined on light microscopy images of cross-sections via image analysis software Lucia G (Laboratory Imaging, Praha, Czech Republic). There is relation between porosity and relative permittivity. High porosity represents lower relative permittivity because volume of pore is full of unknown gas which should have lower relative permittivity than pure CaTiO₃. Also there takes place different microstructure of WSP and GSP layers. All these facts affects dielectric parameters.

Phosphate and borophosphate glasses: structure–properties-application

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Phosphate and borophosphate glasses present an important group of glass materials having several commercial applications. Their research is still continuing and new materials based on these glasses have been prepared. Therefore studies of their structure and properties are reported on international conferences on glasses worldwide. Special phosphate glasses show promising usefulness as fast ion conductors, waveguides, optical switches, fibres, etc. Nevertheless, the applications of these glasses are often hampered by their low chemical durability. The addition of trivalent oxides together with substitution of alkali oxides by divalent oxides can improve their chemical durability. Our studies in recent years were aimed at the stabilization of phosphate glasses with B$_2$O$_3$ combined with ZnO or PbO. Such materials offer better chemical durability than alkali phosphate or borophosphate glasses. Several studies were also devoted to mixed MO-Me$_2$O borophosphate glasses with M = Pb and Zn and Me = Li, Na and K. We studied also the modification of zinc phosphate and borophosphate glasses with higher-valent oxides, e.g. Sb$_2$O$_3$, Bi$_2$O$_3$ or TeO$_2$ and transition metal oxides TiO$_2$, Nb$_2$O$_5$, MoO$_3$ and WO$_3$.

For structural studies we applied Raman and infrared spectroscopy combined with 31P and 11B MAS NMR spectroscopy. The aim of these studies is to identify basic structural units in these glasses and to investigate structural changes with changing glass composition and to relate structural changes with changes in the properties of glasses. Study of thermal properties of glasses is carried out using a variety of thermoanalytical techniques (DTA, DSC, dilatometry and heating microscopy thermal analysis).

The most important applications of these glasses present laser glasses, glasses for the deposition of radioactive wastes, and glass solders. Nd-doped laser glasses used in high-power systems are predominantly phosphate-based with near metaphosphate composition. These glasses are characterized by large stored energy, efficient energy extraction, resistance to laser-induced damage and mature manufacturing technology. For the storage of radioactive wastes glasses containing about 40% Fe$_2$O$_3$ a 60% P$_2$O$_5$ were proposed due to their high chemical durability and the ability to include a high content of radioactive oxides like Na$_2$O, Cs$_2$O, SrO, UO$_2$ or Bi$_2$O$_3$. These oxide dissolve in the glass melt without a significant effect on its chemical durability.

Zinc borophosphate glasses or SnO-ZnO-P$_2$O$_5$ glasses were proposed for the application in solders replacing lead-based solders. Both glasses reveal a sufficient chemical durability, low values of the glass transition temperature and a low coefficient of thermal expansion.

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New approach to crystal nucleation from solution on active centers

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Standard model of nucleation on active centers is based on Avrami approach, when the total number of nuclei formed on active centers is determined by the equation, which governs the depletion of the active centers [1]. Nucleation rate per active center enters the Avrami kinetic equation as a parameter and it is usually computed from the standard kinetic model of nucleation, when the number of active centers does not change in time. We present a new approach to the nucleation on active centers, when the number density of nuclei, nucleation rate, and the total number of nuclei are determined from the standard kinetic model. Exhaustion of active centers is taken into account through the boundary condition.

As a model case we consider the crystal nucleation from solution. The total number of nuclei in Avrami model reaches the number of active centers faster in comparison with numerical solution of kinetic equation. In our model, nucleation rate and the number density of nuclei reach an extremum and then decrease to negligible value with time. At lower initial superstauration and lower number of active centers a decrease in supersaturation is very slow, but it is faster at higher number of active centers.

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Power-law photoluminescence decay in quantum dots

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In this work the experimentally observed effect of a slow decay of the photoluminescence is studied theoretically in the case of quantum dots with an indirect energy band gap. The slow decay of the photoluminescence is considered as decay in time of the luminescence intensity, following the excitation of the quantum dot sample electronic system by a short optical pulse. In the presented theoretical treatment the process is studied as a single dot property. The inter-valley deformation potential interaction of the excited conduction band electrons with lattice vibrations is considered in the self-consistent Born approximation to the electronic self-energy. The theory is built on the non-equilibrium electronic transport theory. The time dependence of the photoluminescence decay is estimated upon using a simple effective mass approach. The numerical calculation of the considered model shows the power-law time characteristics of the photoluminescence decay in the long-time limit of the decay. Relation of the numerical results to the experimental data is to be discussed. We demonstrate that the non-adiabatic influence of the interaction of the conduction band electrons with the lattice vibrations provides a mechanism leading to the power-law time dependence of the photoluminescence intensity signal. This theoretical result emphasizes the role of the electron-phonon interaction in the nanostructures.

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Study of temperature field measurement at vertical Bridgman method in static and dynamic arrangement

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This work deals with study of the temperature field measurement at simulated crystal growth by the vertical Bridgman method. Its aim is to understand influence of growth conditions (temperature gradient in the furnace and pulling rate) on shape and position of crystal/melt interface and finding suitable growth conditions for preparation of high quality single crystals of lead halides and ternary alkali lead halides. As a model compound, a lead chloride was chosen for its availability in high purity and for more accessible information about its material constants. The temperature field in our experimental setup was measured by four thermocouples inside specially prepared quartz ampoule. Thermocouples moved in four capillaries asymmetrically positioned along the ampoule axis. Two experimental configurations were used: static and dynamic. During the static one, the ampoule was held at fixed position. After formation of the crystal/melt interface, the temperature was measured by pulling the thermocouples up through the capillaries by step of 1 mm. In this way, the temperature was sampled from the bottom crystal part, through the crystal/melt interface, the melt and the atmosphere over the melt in the total length of 65 mm. At the dynamic arrangement the ampoule was pulled down by a pulling rate of 3 mm/hour. When it reached the same position in the furnace as in the static arrangement, the temperature field was measured in the same manner as for the static measurement [1]. Obtained results of the temperature field were displayed in the first instance in 2D diagram as “isolevel” temperature curves, assuming radial symmetry of measured positions inside the ampoule. For further study these data were recomputed into isothermal curves by linear approximation [2]. From obtained results, we determined position and shape of the crystal/melt interface. The shape of the crystal/melt interface depended on the temperature gradient in the furnace and the volume ratio of crystal and melt. At the temperature gradient of 35 K/cm and a small volume of the crystalline phase the crystal/melt interface was markedly convex. At the dynamic setup, influence of the ampoule movement caused changing of the interface shape to planar one. Temperature data were used as boundary conditions for a numerical model to determine melt flow and temperature field (isotherms) in the studied system using COMSOL Multiphysics 4.1 software.

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During the last twenty years, diamond has attracted increasing attention of many research and industrial groups worldwide due to an excellent combination of intrinsic properties (mechanical, chemical, and radiation resistance, thermal conductivity, optical transparency, wide electrochemical window, biocompatibility, etc.). However, several optimistic predictions of early applications failed on account of technological limitations (such as n-type diamond doping, low temperature growth, and heteroepitaxial growth over large areas). In the last 5 years, the large area diamond growth by microwave plasma CVD process became attainable, however, growing electronic-grade material remained a challenge. Our present work focuses on the large area growth (up to 20x30 cm$^2$) of diamond thin films by linear antenna microwave plasma CVD process from CO$_2$ containing gas mixtures. We will show that adding CO$_2$ to the hydrogen rich gas mixture (H$_2$+CH$_4$) has a significant influence not only on the plasma gas chemistry (conc. of atomic hydrogen) but also on the diamond growth kinetic (growth rate). Furthermore, we show that grain size (nano- or micron-sized), roughness, and chemical purity (ratio of sp$^3$ to sp$^2$ bonded carbon) can be tailored by adjusting the process conditions. The presence of CO$_2$ in the gas mixture is identified as crucial for obtaining functional field effect transistors (FETs). Our first model assumed that CO$_2$-grown diamond films are not suitable for fabrication of hydrogen terminated FET devices due the presence of recombination centers for holes induced by hydrogen termination. This limitation seems to be valid only for a certain CO$_2$ concentration. We have overcome this technological limitation and we will show for the first time a functional FET devices working on hydrogen–terminated diamond films grown by linear antenna microwave CVD plasma from CO$_2$ containing gas mixtures.

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Nonstoichiometry and physical properties of GaGeTe

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In the GaTe - Ge system there is GaGeTe compound with peritectic melting point 1073 K. GaGeTe crystallizes in hexagonal crystal lattice (space group R3m) with layered structure with distinct cleavage planes perpendicular to the trigonal c-axis. According to [1] its lattice parameters are a=4.05 Å, c=34.65 Å. In the literature, there are studies of crystal structure [2] and an investigation of chemical bonding using Raman spectroscopy [3] only. The aim of this contribution is to present original information on transport properties of Ga$_{1+x}$Ge$_{1-x}$Te and GaGeTe$_{1-y}$ series. The products of synthesis were identified by X-ray diffraction; phase purity and microstructure were examined by EDX/SEM. Samples for measurement of transport properties were prepared using hot-pressing. They were characterized by measurement of electrical conductivity, the Hall coefficient, and the Seebeck coefficient over a temperature range 80–480 K and of thermal conductivity over a temperature range 300–580 K. All samples show p-type conductivity. We discuss the influence of stoichiometry on the phase purity of the samples and on free carrier concentration and hence on their thermoelectric properties. The investigation of thermoelectric properties shows that the power factor of these samples is low compared to state-of-the-art materials at room temperature but increases distinctly with temperature [4].

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Formation of structured nanophases in halide crystals

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We examined the problem of impurity nucleation in a solid matrix on several alkali halide crystals. While the studied NaCl and KCl form simple ionic crystals, when impurities are introduced and the crystals subjected to heat treatment, complex aggregation phenomena do occur. In the theoretical part of the work, we tried to follow the aggregation of these impurities using computational modeling approach. We examined the possibilities and based the model on interionic potentials (IOPs) lattice modeling standing in the model hierarchy (and computational demands) between ab-initio approaches such as DFT models and classical nucleation theory.

We determined formation energies for clusters, treated as defects, starting from single impurity-vacancy dipole and small aggregates to possible configurations of larger clusters.

Experimentally, we studied the system using optical spectroscopy. The advantage of this method is in the possibility of following nanocluster growth in the bulk, without destroying the sample. Experimental results coincide with model. While the study was limited to alkali halide system, similar behaviour can be observed in other systems.

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Surface plasmon resonance sensor using spectral interference

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This paper presents a novel surface plasmon resonance (SPR) sensor based on white-light spectral interferometry. The experimental arrangement employs a white-light source (bandwidth from 350 nm to 1000 nm), a polarizer and a birefringent quartz crystal for SPR excitation. SF10 Kretschmann SPR sensor prism is inserted into the beam path and one measures both the corresponding SPR phase change and reflectivity at a suitable fixed angle of incidence across the visible spectrum. The prism (sensing surface) is coated with gold film of 44 nm nominal thickness and the incident angle is approximately 58°. In our case of the SPR effect, the useful phase information is encoded in the spectral interferogram. Thereby, p- and s-polarized spectra interfere among themselves and the interferograms are obtained by using an analyzer when only p-polarized waves are affected by the SPR effect.

Spectral interference signals are then used in corresponding phase retrieval [1]. In order to calculate the SPR induced differential phase change, a windowed Fourier transform (WFT) [2] was adopted to extract the phases from two spectral interferograms, one corresponding to the reference material and the other corresponding to the analyte.

The pilot measurements were tested with water as an analyte and air as a reference material and the measurement results were compared with the results of theoretical models. The first results show good agreement between a calculated resonance wavelength and a resonance wavelength determined from reflectivity measurements. The second results show good agreement for phase change measurements. The proposed technique offers a much simpler experimental arrangement than was recently presented in a paper [3].

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Current Loading of Adhesive Joints Aged in Environment with High Humidity

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The electrical resistance of conductive joints formed of electrically conductive adhesives changes with external influences such as the temperature, humidity, mechanical stress, electrical current [1-2] and many others. The goal of the work was to examine influence of electrical current pulses of high intensity on two groups of adhesive joints: on the joints, loaded with the current after their forming and on the joints first aged in environment with high humidity and then loaded with the current pulses. The pulses amplitude, width and frequency were set so that the joints were only minimally heated. The measured results were mutually compared and discussed.

Formulation of adhesive was based on bis-phenol epoxy matrix and was filled with silver flakes with concentration 75 % (by weight). Adhesive joints were formed by adhesive assembly of resistors with the resistance of 14 mΩ on a test PCBs. These boards made four point measurements of the joints resistivity possible. Humidity ageing was carried out at the 98 % RH for 100, 200 and 300 hours. The rectangular current pulses with the amplitude of 10 A, 20 A and 50A were used for loading of the adhesive joints. The width of the pulses was 5 µs, the frequency was 50 Hz. The lost power was, for the standard resistance of 20 mΩ of an adhesive joint, 12 mW for the amplitude of pulses 50 A and 0,5 mW for the amplitude of pulses 10 A. This value is negligible from the point of view of possible additional curing of adhesive. The time of loading with the current pulses was 60 min.

It was found that the current pulses can change the resistance of the adhesive joints. The resistance first increased with the amplitude of current pulses and then fall down, or fall immediately down after application of current pulses of minimal current intenzity. The changes were small (up to 10 %) for the joints, which were not aged in humidity. Higher changes (up to 37 %) were found for the joints aged in humidity. It supports assumption about the increase of silver ions mobility in adhesive depending on moistening of the adhesive matrix.

Scintillating properties of rare earth aluminum garnets

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Rare earth aluminum garnets, RE₃Al₅O₁₂, where RE are Lu, Y or their mixture in asked ratio, doped with Ce³⁺ or Pr³⁺ are efficient scintillators characterized by high or medium light yield (L.Y.) up to 27000 ph/MeV [1,2]. These scintillators are tested and used in various applications such as medical imaging (PET, PEM, radiography etc. [3]) or in 2D X-ray micro-radiography [1]. Crystalline samples of above mentioned aluminum garnets can be prepared as the single crystals grown by the Czochralski or Bridgman methods [4,5], as epitaxial layers grown by the liquid phase epitaxy (LPE) and in the form of ceramics [7]. In particular the epitaxial layers seem to be very promising medium materials regarding their good scintillating properties and cost [5,6]. Scintillation properties of garnet structure materials were investigated by pulse height spectroscopy using a HPMT photomultiplier [2]. Scintillating properties of garnet structure materials can be influenced by (i) garnet sample composition, by (ii) annealing at different temperatures and atmospheres and by (iii) effective doping if energy transfer is present. The main goal of this report is to present short summary of scintillating properties (L.Y. and energy resolution) of various aluminum garnet structure materials depending on the above described modifications (i)-(iii).

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Final properties of formed steel or another alloy pieces are affected by production, at first conditions of working. For all that it is needful to know detail structure changes of plastic deformed material caused by working - forming, grinding, drilling etc. Recently the deformation is in a general way based on its macroscopic effects, but these are not corresponding fully with microscopic structural changes [1-2]. The deformation of parts leads to grain boundaries deformation in volume of parts or in its surface layers. On metallographic cut it is possible to observed grain boundaries orientation which was caused by grain boundaries deformation. If prior knowledge of axes of orientation are known (as it is in most of mechanical working processes), stereological measurement of degree of grain boundaries orientation is relatively simple [3]. There are only three principal schemes of elementary bulk deformation, which very good classified strain in an analysed place of a formed body. They are basic indicators at analysis and evaluation deformation state caused by an external load. In an undeformed state the structure is isotropic, the grains have isometric dimension and grain boundaries are not oriented. In case of plastic deformation of this isotropic structure the grains will occurred anisometric dimension and grain boundaries are oriented. Grain boundary orientation can be observed in various planes in dependence on deformation. Direction of grain boundary orientation is the same as direction of deformation. If a deformation scheme is known, the anisotropic microstructure is decomposed into isotropic, planar and linear oriented components using stereology methods. However, orientation is not the same as deformation. A model of conversion of grain boundary orientation degree to deformation was based on similar comparison orientation – deformation of an idealized grain shape. From three basic equations – definition of deformation, definition of degree of orientation and invariability of volume the dependence of true strain to degree of orientation was solved. This model of conversion of grain boundary degree orientation to grain deformation is independent on initial dimension of the grain – strain depends only on shape of grain and not dependents on a grain dimension. The method also does not requires knowledge of the parameter of structure with zero value of initial deformation, which in most of cases is unknown and in the whole volume of pieces is not the same.

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Resonant and non-resonant components of the rate of excited state decay

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Hybrid organic-inorganic nanostructures are known for their suitable optoelectronic applications in photovoltaics and light-emitting diodes. The organic part usually provides a large oscillator strength providing thus good optical properties, while the inorganic one exhibits a good charge transport. Therefore, it is the aim of this work to study the resonant energy (charge) transfer in such a system.

Inorganic materials of the III-N-V-type are used in optical communications. It was shown that in quantum dots based on III-V semiconductors the electron-phonon coupling is responsible for the electronic relaxation process [1,2]. Simultaneously, we expect that the organic part in the hybrid organic-III-N-V system similarly increase the electronic transfer due to its strong coupling between excited electrons and vibrational modes.

In our model we studied a charge (energy) transfer in a two-level (donor-acceptor) system. Particularly, we calculated time-dependences of the population transfer with respect to the parameters of the electron-vibrational mode couplings.

We proved that the donor population dynamics contains at least two strongly oscillating components as well as the decay kinetics. We assigned the oscillatory frequencies of the donor population to the particular vibronic transition frequencies of the energy of the system. Moreover, we proved that decay rates exhibits resonances for the integer value of the ratio between the energy difference of the donor and acceptor and the vibrational energy. We discussed the model in a great detail.

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Structure and properties of TeO$_2$ containing borophosphate glasses

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Lithium and calcium borophosphate glasses doped with TeO$_2$ were prepared and studied in the compositional series (100-x)[0.5Li$_2$O-0.1B$_2$O$_3$-0.4P$_2$O$_5$]-xTeO$_2$ and (100-y)[0.5CaO-0.1B$_2$O$_3$-0.4P$_2$O$_5$]-yTeO$_2$ within the concentration range of x=0-60 and y=0-30 mol% TeO$_2$. The structure of the glasses was studied by Raman spectroscopy. Differential thermal analysis, thermodilatometry and heating microscopy have been used to study thermal behaviour of the glasses. According to the Raman spectra, TeO$_2$ is incorporated in the structural network in the form of TeO$_3^-$ and TeO$_4^{2-}$ structural units. The ratio of TeO$_4$/TeO$_3$ units increases with increasing TeO$_2$ content in both compositional series of glasses. DTA studies showed that all of glasses crystallize on heating within the temperature region of 400-700°C and the surface nucleation mechanism prevails over the internal one. The contribution of the surface crystallization mechanism over the internal one decreases with increasing TeO$_2$ content. Compounds formed by crystallization were BPO$_4$, LiPO$_3$, Li$_4$P$_2$O$_7$, Ca$_2$P$_2$O$_7$, Ca$_4$P$_6$O$_{19}$ and TeO$_2$. Glass transition temperature and crystallization temperature decrease with increasing TeO$_2$ content as well as the thermal stability of glasses, whereas thermal expansion coefficient increases. The addition of TeO$_2$ to the starting lithium and calcium borophosphate glass also resulted in a gradual increase of chemical durability of the glasses.

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Study on glass transition temperature of meta-phosphate glasses

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Glass arises by cooling of a melt or a high temperature solution with rate greater than the critical one in the so-called temperature range of glass transition. Glass formation is not any momentary process as crystallization is, but it is a consecutive one during which the melt viscosity increases, the melt structure starts rearranging and the values of enthalpy, entropy and volume decrease. The adequate temperature range of glass formation is characterized by the temperature of the glass transition - \(T_G\), which as the midpoint of the temperature range of glass transition is determined by thermal analysis methods. Unfortunately, \(T_G\) determined in that way depends on the measurement conditions (heating or cooling and their rates) and conditions of glass preparation (thermal history of the glass). These facts disqualify application of \(T_G\) as a material constant giving fundamental information about condition of glass preparation. Therefore methods to reduce negative influence of measuring conditions on the \(T_G\) determination were searched for. The first researches who were interested in relations between \(T_G\) and heating rate during DSC measurements were probably Kovacs [1] and Lasocka [2]. However, the great achievement was attained by developing new extensions to conventional DSC - the Temperature-Modulated Differential Scanning Calorimetry (TMDSC) performed by the TA Instruments [3,4], the Pyris StepScan DSC Software (Perkin-Elmer) and the temperature modulated DSC named TOTEM by Mettler-Toledo [5].

These methods enable one to separate the measured heat flow into the temperature dependent reversing (thermodynamic) component and the time dependent nonreversing (kinetic) one. Using these methods, both \(T_G\) and \(\Delta C_P\) values were obtained, which were independent of their conditions of measurement (heating or cooling) and glass preparation and thermal history of the glass as well. The values of \(T_G\) and \(\Delta C_P\) only depend on the glass chemical composition and accordingly the \(T_G\) value determined in such a way can be considered as an actual material constant for the glass of given composition.

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Determination of large zero-field splitting parameters.

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A high-spin Co(II) complexes (3d⁷, S = 3/2) have been investigated by temperature-dependent FAR infrared spectroscopy. A series of mononuclear Co(II) complexes was chosen for which the structures have been resolved by X-ray diffraction and the zfs parameters have been accurately determined by the SQUID magnetometry [1-3]. ZFS is usually characterized by two parameters, termed the axial (D) and rhombic (E) zero field splitting constants. Determination of the D-parameter is traditionally performed by the electron spin resonance (when D is low enough) and the magnetometry (when D is large enough). However, other techniques can be applied as well, like calorimetry, inelastic neutron scattering, magnetic circular dichroism, Mossbauer spectroscopy, or the FAR-infrared spectroscopy. The values of D vary from 10⁻¹ to 10² cm⁻¹ [4].

The FT-IR spectroscopy is a useful tool for determining the zero-field splitting when the D-values are large. In Co(II) complexes there are six lowest energy levels (Kramers doublets) arising from the splitting of the parent ⁴T₁g(O₉h) term due to the tetragonal crystal field and the spin–orbit coupling. Therefore there are 15 allowed electronic transitions among them. Five transitions from the ground state determine remaining 10 transitions among the excited states so that the IR data should be selfconsistent.

One of these transitions refers to the zero-field splitting Δ(¹Γ₆→¹Γ₇) = 2D + 2E and its intensity progressively decreases on temperature lowering. The peak maximum corresponds to the vertical transition. A decomposition of the vibrational envelope (when resolved) to Lorentzian-shaped primitive functions yields the on-set of the absorption peak, that refers to the 0–0 transition, giving rise to the corrected value D′. These data are not far from those determined by fitting the magnetic data (temperature dependence of the magnetic susceptibility, and the field dependence of the magnetization)[5].

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Affect of Carbon Black Modification on Electrical Properties of Carbon Black - Polymer Composites

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In this work the carbon black (CB) was used as the filler in polymer matrix. Primary CB particles (30-50 nm) form larger agglomerates which can build up the continuous electrically conductive path when the concentration of CB reaches the critical value (percolation threshold). The dependence of conductivity on CB concentration shows the sharp rise the composite transforms from insulator to conductor. The aim of this work is microscope (optical and AFM) observation and the measurements of electrical properties of the thin plate (thickness 0.2 mm) composites CB (modified) – polystyrene (PS) prepared from the solution of PS in toluene. The modification of CB was based on ion extraction from CB in distilled water. UV irradiation was added for some samples. The extraction of ions was verified by the measurements of the conductivity of water used and by atomic absorption spectroscopy. Three series of the samples were studied. 1. Composites with dry CB. 2. Composites with CB (DCB-deionised CB) after ion extraction in distilled water. 3. Composites with DCB irradiated by UV lamp during the extraction. The concentration of CB was 1-5 %w in all series. The electrical measurements were carried out in the condenser with micro shift in the frequency region 0.1 – 1000 kHz for temperatures 25 – 80 °C. The separation polyethylene foil was used for higher conductive samples. The impedance – phase measurements are presented directly or in the terms of complex permittivity, where real part is relative permittivity and imaginary one represents losses. The differences in the structure and electrical properties between the samples of composites contained different modified (or non-modified) CB will be discussed in more detailed.
Growth of profiled sapphire single-crystals by EFG method

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Sapphire - the single crystal of aluminum oxide (Al$_2$O$_3$) - belongs to one of the most important artificially produced materials due to its unique physical and chemical properties. Sapphire has a high refractive index and a broad transmission band spanning the ultraviolet, visible and infrared bands. Sapphire is very hard material (Moh’s 9) and has an extraordinary mechanical strength up to its melting point 2053 °C, very high thermal conductivity, high electric resistivity and outstanding chemical resistance even to strong acids, bases and fluorination agents. All this makes sapphire a much sought-after material in industry and science. Typically sapphire is grown as bulk crystal by Verneuil, Czochralski or Kyropoulos methods. However crystal machining is difficult, in comparison to other materials due to its high hardness. The first growth of profiled single crystal sapphire was published in 1971 by La Belle and Mlavsky (TYCO laboratories) [1]. The EFG (Edge-defined Film-fed Growth) method is in principle similar to older one (1953) of A. V. Stepanov for growth of shaped single crystals of metals. Main advantages of the EFG method are growth of near net shape which demands zero or small need of machining decreasing production costs, possibility of growth of complicated shapes and large scale profiles, higher growth rate and also automated process control. Sapphire profiles can be prepared by EFG method in various shapes - rod, tube, plate, ribbon, etc. Although EFG method was primarily applied for sapphire production, is today used also for metals, silicon and other oxides compounds. Review of modern modifications of EFG and related methods was published. The EFG method is based on rising of Al$_2$O$_3$ melt by capillary action up to the top of the orifice. Thanks to the wettability of metal surface of the orifice, the 0.1 mm thick layer of the melt arises. The meniscus of the melt is then touched by the seed crystal at proper melting temperature. The edges of orifice define the geometrical shape of crystal. Seed crystal is then slowly pulled from the melt and the final profiled crystal is grown. Profiled sapphire is produced commercially by several companies in USA, Japan and Russia, Crytur is the only EU producer. Sapphire profiles are produced in form of fibre diameter 100-500 µm, rods with diameter 0.5 - 20 mm. Capillaries have minimal outside diameter 0.9 mm and inside diameter 0.6 mm. Tubes have maximum diameter 85 mm and maximum length up to 1800 mm. The largest sapphire plates have dimensions 300 x 600 x 8 mm. Sapphire profiles are used in various industrial and scientific applications demanding extraordinary material properties. High pressure, high temperature, together with harsh chemicals are the right reason to use sapphire tube instead of quartz or corundum ceramics. Although is EFG method known more than 40 years, is there still many new aims and the next development is necessary.

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Diamond structures grown from polymer composite nanofibers

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Diamond exhibits a unique combination of properties while keeping its high biocompatibility [1]. The growth of homogeneous and continuous films is always preceded by the nucleation/seeding method. Presently, the ultrasonic seeding seems to be the most progressive and universal technique with nucleation densities up to $10^{11}$ cm$^{-2}$. Seeding by a composite polymer-diamond powder was shown suitable for seeding of mechanically soft and/or unstable substrates [2]. Needle-less electro-spinning allows industrial-scale production of nanofibers with high productivity, scalability and quality of nanofibers.

In this work we present the new approach for diamond nucleation which implements nanofiber composites produced by textile industry. In this approach, ultra-dispersed diamond nanoparticles (UDD) (5 – 10 nm) are dissolved in PVA solution which is then deposited by electro-spinning method onto the substrate material (glass, silicon, paper, etc.). Due to used high voltage (20 – 60 kV), charged liquid “jets” are extracted in the direction to substrate, during this extraction they are gradually “dried” and collected onto the substrate as fiber composites (PVA fibers with immersed diamond nanoparticles) in their solid state. Based on SEM images, the diameter of fabricated fiber composites varied from 50 nm up to 200 nm and their length was up to hundreds of micrometers. Then the fiber composites were exposed to the microwave plasma CVD process using following process parameters: microwave power 900 – 3000 W, gas pressure 30 – 50 mbar, 1 % methane diluted in hydrogen gas mixture. After the CVD process, samples were characterized by SEM, AFM, and UV micro-Raman spectroscopy and by FTIR measurements. All the techniques confirmed diamond character of the fibrous structures. The morphology varied from porous-like 3D structures to single isolated nanofibers in diameter <50 nm. Combined effect of seeding by the UDD particles and GPa pressure difference across the fibre interface (given by fibre radii) will be discussed together with applications of these diamond nanofibers and porous structures in a tissue engineering, sensor applications, and self-standing flexible diamond structures.

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Luminescence Properties and Application of Impurity Ion Probes in Perovskite-type Oxides

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Study of ferroelectric and structural phase transitions in ABO$_3$ oxides with perovskite-type structure is of great importance to successful improvement and optimization of their functional properties that can be tailored for wide range of applications by doping with suitable admixtures. Due to high sensitivity of luminescence parameters to small changes in host material luminescent ions with zero-phonon emission lines offer delicate probes for detection of phase transitions and local lattice distortions in dielectric materials. A luminescent probe method is particularly advantageous in the study of small size and confine geometry effects on properties of ferroelectric and related materials performed mainly on ultrathin films, fine-grained ceramics, and nanopowders because of a whole range of inconveniences connected in this case with the application of conventional methods for investigation of ferroelectric phase transitions.

The luminescence properties of trivalent rare-earth ions and transition-metal ions with 3d$^3$ electron configuration in perovskite-type ABO$_3$ oxides will be reviewed with respect to their possible application as a luminescent probe in the detection of phase transitions. Special attention will be paid to SrTiO$_3$ because of very unusual and large temperature shift of the zero-phonon R-line of photoluminescence of octahedral Cr$^{3+}$ and Mn$^{4+}$ centers observed in SrTiO$_3$:Cr and SrTiO$_3$:Mn crystals [1, 2]. An antiferrodistorsive structural phase transition from the cubic to tetragonal phase results in the splitting of the R-line to the doublet. Moreover the temperature shift of the average position of the R-lines was found in the tetragonal phase proportional to the reciprocal permittivity of the pure SrTiO$_3$ and to the square of TO$_1$ soft phonon mode frequency. Such temperature behavior of the R-lines makes it possible to study ferroelectric phase transitions induced in SrTiO$_3$ by appropriate impurities or perturbations [3]. Recent study of the temperature behavior of the Cr$^{3+}$ emission R-line in SrTiO$_3$:Cr nanocrystalline powders revealed conservation of quantum paraelectric properties down to a particle size of about 10 nm but in small enough SrTiO$_3$ nanoparticles clearly indicated an emergence of a low-temperature ferroelectric phase transition [4].

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Photoluminescence and infrared absorption spectroscopy of the aminated nanocrystalline diamond surface

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Nanocrystalline diamond (NCD) coatings deposited on non-diamond substrates such as glass at a relatively low temperatures below 600°C provide economical alternative to single crystal diamond showing most of the diamond excellent properties. We have significantly reduced the non-diamond content by the deposition of NCD under the optimized growth conditions on carefully selected substrates followed by the post-growth etching and cleaning. The diamond surface exhibits an extraordinary variety of properties at the atomic level and the functionalized diamond surface differs significantly in surface properties such as electrical conductivity, electron affinity, wettability and chemical reactivity. We have already demonstrated the usability of the FTIR spectroscopy and fluorescence microscopy to detect the surface coverage of the oxidized NCD surface with a relatively thick amino-organosilane polymer. However, the FTIR sensitivity was rather low due to the low reflectivity of NCD/silicon or NCD/glass interface. Therefore, we have recently developed the NCD coating deposition on the highly reflective protected gold substrates.

In this paper the microwave based surface wave-discharge in linear antenna configuration is applied for the growth of diamond thin films on commercially available protected gold on glass mirrors to enhance the infrared optical reflectivity of NCD layers. To demonstrate the high sensitivity of the optical methods for the surface characterization, the oxidized NCD surface is aminated by the wet amino-organosilane treatment. The amination of the NCD surface is of high interest as it would enable the straightforward binding of a large variety of functional molecules such as bioactive compounds or polymer building blocks by amide formation, reductive amination, nucleophilic attack or direct condensation reactions. Atomic force microscopy (AFM) reveals the macroscopically homogeneous amino-organosilane coverage consisting of the insulated microscopic organosilane clusters with submicron height partly covering the NCD surface. The grazing angle reflectance Fourier transform infrared (GAR FTIR) spectroscopy in the mid infrared region detects with high sensitivity to the characteristic molecular vibrations of the functional groups. From the signal to noise ratio follows that the GAR FTIR is a potentially suitable method for monolayer detection. The presence of the amino groups on the partly aminated NCD surface is detected under UV excitation by the photoluminescence spectroscopy in blue-green spectral region after applying fluorescamine reagent.

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DSC and TMDSC studies on glass transition temperature of alkali rare earth metaphosphate glasses

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Glass transition temperature $T_G$ as the midpoint of the temperature range of glass transition offers fundamental information important for glass preparation. When $T_G$ is determined by convectional DSC, it depends on the measurement conditions (heating/cooling and their rates) and conditions of glass preparation (thermal history of the glass). However, if the temperature modulated DSC (TMDSC) is used the heat flow is independent of conditions during the $T_G$ measurement and of thermal history of the glass. $T_G$ values only depend on the glass chemical composition [1-3].

Our contribution deals with the glass transition study on meta-phosphate glasses with a composition of LiGd(PO$_3$)$_4$ and LiY(PO$_3$)$_4$ using both the convectional DSC and TMDSC. These glasses were developed and tested when searching for scintillating materials to be used in the detection of gamma- and x-rays [5]. The glassy samples were prepared by a direct synthesis of Li$_2$CO$_3$, YPO$_4$ (GdPO$_4$), CePO$_4$ and some excess of P$_2$O$_5$ at 1200°C [4]. Two groups of powdered samples of particle sizes smaller than 96 µm and between 96 and 106 µm and bulk glassy samples in the form of both side polished round plates 3.6 mm in diameter and 1.6 mm in height were used. Glass transition temperature was studied using non-isothermal conventional DSC at heating and cooling rates of 2, 5, 8, 10, 18, 25, and 40 K/min from room temperature to 550°C. Using TMDSC, the temperature of glass transition was only scanned at heating rates of 2 and 5 K/min. The aim of this contribution was to determine the glass transition temperature of all glassy samples depending on scanning rates using both DSC and TMDSC and to compare the $T_G$ values obtained by the TMDSC with those obtained by means of Černošek’s procedure [5] and to discuss the results obtained. The procedure is based on application of the two-phase exponential association kinetics equation and extrapolation of the $T_G$ values measured by the DSC to infinity.

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Investigation of thermal behavior of lead molybdate-phosphate glasses by various thermoanalytical methods

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Phosphate glasses are able to form relatively large glass-forming regions with molybdenum oxide and tungsten oxide. These glasses offer prospective applications for their electrochromic properties and a high ionic conductivity. Phosphate glasses belong also among possible candidate materials for the vitrification of nuclear wastes with molybdenum because they possess high waste loading capacities. Our previous study of glasses from the ternary system PbO-MoO$_3$-P$_2$O$_5$. [1, 2] was realized with Raman spectroscopy and $^{31}$P NMR spectroscopy. A large glass-forming region was observed in this system and in the glass series Pb(PO$_3$)$_2$-MoO$_3$. glasses were obtained within the concentration range 0-70 mol% MoO$_3$. For all these applications it is necessary to characterize these glasses by several techniques, to investigate their structure and properties and also to study their thermal behavior.

Presented study is devoted to the investigation of thermal behavior of glasses from the ternary system PbO-MoO$_3$-P$_2$O$_5$. by several thermoanalytical methods. Thermal properties of these lead molybdate-phosphate glasses were studied by differential thermal analysis (DTA), heating microscopy thermal analysis (HMTA) and dilatometry (DT). Characteristic temperatures obtained from these three methods were compared and discussed. Crystallization products, obtained by annealing of powdered glasses at high temperatures, were identified by X-ray diffraction analyses. In the glasses of the Pb(PO$_3$)$_2$-MoO$_3$. series with a high MoO$_3$. content crystalline compound of Pb(MoO$_2$)$_2$(PO$_4$)$_2$. is formed. Crystallization of this compound was studied by the Day’s method [3] and the prevailing mechanism of surface nucleation was revealed. Heating microscopy was found as a useful technique especially for the study of glass behavior above the glass transition temperature.

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Material science education at ICT Prague

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Institute of Chemical Technology Prague (ICT) is the biggest educational institution of its kind in Central and Eastern Europe. Apart from education, research in material science forms the large deal of its activity.

Education in chemistry (alchemy) in the lands of the Czech Crown is as old as the Charles University. In times prior to the reign of Rudolph II, the great admirer of alchemy, an empirical approach started to take shape. In 1546, Georgius Agricola wrote De re metallica libria dealing with methods used in mining and metallurgy, thus beginning the transformation of alchemy into chemistry. Industrial development in the 18th century led to the foundation of Czech Estates Polytechnic Institute which included also the department of chemistry. This department was part of the Prague Polytechnic until 1920 when Institute of Chemical Technology and Engineering was formed within the Czech Technical University. Complete independence was achieved in 1952 when ICT was created in the form we know it today. Nowadays, ICT comprises four faculties: Faculty of Chemical Technology, Faculty of Environmental Technology, Faculty of Chemical Engineering and Faculty of Food and Biochemical Technology. The core of materials research and education in this field lies in all study degrees at Faculty of Chemical Technology.

The study in ICT is structured into 3 years of bachelor’s and 2 years of master’s program. Bachelor study offers 12 programs with 28 branches. Out of these, 3 study programs deal with material science: Applied chemistry and materials, Biomaterials for medicine and Nano- and microtechnologies in chemical engineering. In master study, students can apply for 16 study programs (35 branches), where there is one material program: Chemistry of materials and material engineering. This study program is divided into five branches: Inorganic non-metallic materials, Materials for electronics, Metallic materials, Polymer materials and Nanomaterials.

Apart from lectures and seminars in specialized subjects, the students have specialized laboratory practice during both bachelor and master studies. There they familiarize themselves with advanced preparative, analytical and measuring techniques that are being used in respective departments in the research and application of materials. ICT has of course a subsequent PhD study program where the students usually continue in research they started in their diploma thesis.

At this presentation, the typical study course of materials engineering program will be demonstrated. All special subjects and lab trainings (including equipment used) will be presented.
Preparation and characterization of Bi$_{2-x}$Sr$_x$Se$_3$ Single Crystals

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Layered semiconductors such as Bi$_2$Se$_3$ belongs to a class of material used in thermoelectric (TE) applications. TE devices have ability to convert heat energy directly into electricity and can be used as alternative source of "green"energy. TE materials with tetradymite structure (Bi$_2$Se$_3$, Bi$_2$Te$_3$, Sb$_2$Te$_3$) are degenerate semiconductors with high concentration of native defects producing free carriers. For Bi$_2$Se$_3$, selenium vacancies are dominant defects giving rise electron doping of the order $10^{19}$ cm$^{-3}$. Doping with electropositive element as strontium could lead to a decrease of concentration of native defects and consequently to a decrease of free carrier concentration and has a beneficial effect on transport parameters.

This lecture is focused on preparation and characterization of single-crystalline Bi$_2$Se$_3$ doped with strontium. Single crystals were grown from high purity elements using self-flux method. The samples were characterized by x-ray diffraction analysis and optical emission spectroscopy with induction coupled plasma (ICP-OES) was used for determination of actual content of Sr in the samples. Transport properties involved measurement of Seebeck coefficient $S$, Hall coefficient $R_H$ and electrical conductivity $\sigma$ in the temperature range 80 K to 470 K. It was found that the substitution of Sr atoms for Bi atoms in the Bi$_2$Se$_3$ crystal lattice leads to a decrease in the free carrier concentration. This effect is explained within a model of the point defects in the crystal lattice of Bi$_{2-x}$Sr$_x$Se$_3$. From measured data the power factor PF and thermoelectric figure of merit ZT were calculated.
Antibacterial studies on nanocrystalline diamond thin films

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Diamond thin films are recognized as promising material for life science and regenerative medicine due to its excellent biocompatibility and tunable surface properties by grafted molecules. Use of diamond films as antibacterial coating is less known and still evokes discussions. In this work we present a study on antibacterial properties of hydrogen and oxygen terminated diamond thin films (H- and O-diamond). As a negative reference sample is used glass and copper is used as a strong positive sample. Gram-negative bacteria - E. Coli is used as a modeling organism. The bacterial culture was grown in whole media at 37 ºC on specimens (H-diamond, O-diamond, glass and copper) with a specific area around 20 cm². The antibacterial properties were determined by the number of bacteria colonies after 24 hours of incubation. Experiments on bacteria growth on copper, the well-known member of antibacterial materials, confirmed its killing effect on all bacteria. Oxygen grafted diamond films had a minimal impact on bacterial growth and achieved results are comparable with reference samples (glass). In comparison to the reference sample, the hydrogen grafted diamond film effectively reduced the number of bacteria by 60%. This observation confirms that in specific case (i.e., proper surface termination on atomic level), diamond can be classified as antibacterial material.

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Nanoparticles in hydrogenated silicon

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Solar cells based on photovoltaic diode structures made from hydrogenated amorphous (a-Si:H) and microcrystalline (µc-Si:H) silicon thin films with the energy conversion efficiency about 10% have been developed in the last 35 years as an economical alternative to the solar cells based on sliced crystalline silicon ingots. However, silicon thin film conversion efficiency still needs to be improved to be competitive with the crystalline silicon solar cells with the efficiency about 20%. One of the possible ways to improve the cells efficiency is to prepare silicon thin films with embedded nanoparticles to enhance the light absorption in the near infrared region.

In the Department of Thin Films and Nanostructures at the Institute of Physics ASCR, we developed the radio frequency plasma enhanced chemical vapor deposition (RF PE CVD) of hydrogenated silicon thin film in glow discharge of Silane diluted in Hydrogen. In the last years we have started to investigate the properties of nanoparticles embedded in the silicon matrix. We have observed that some nanoparticles can indeed be embedded in the silicon to create the quantum dots, whereas others lead to catalytic growth of silicon nanowires (Si-NW).

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Preliminary study of polyvinyl alcohol nanotextile produced by electrospinning

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The understanding of physical properties of nanotextiles and parameters, controlling the process of their production, is very important for future application of this kind of material in all branches of technology. In this paper we present a preliminary study of polyvinyl alcohol nanotextiles, produced by a modification of electrospinning method – a so-called Nanospider technology. The homogeneity of the material is studied by means of measurement of a surface density. The analysis of polypropylene spun bond, used in our experiments as a substrate for PVA nanotextile, shows that for future research it would be desirable to use a material with higher homogeneity. The theoretical model for the filtering efficiency of nanotextile is proposed. Within the frames of this model an expression for the transparency of the multi-layer nanotextile on the parameters of the technological process is obtained. The research forms a basis for future detailed study of this new and prospective type of material.

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Effect of gamma irradiation on pore-space of hardened cement paste

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There is a wide range of degradation factors that may potentially impact the lifespan of so-called concrete nuclear-safety structures in Nuclear Power Plants (NPP). The limited experimental material connected with γ-irradiation as degradation factor [1] concluded that the least dose of γ-irradiation producing measurable damage of concrete is $10^8$ Gy. Such massive doses cannot be reached in case of NPP’s under the normal operation. Typically, during its lifetime the concrete in NPP receives a dose about $10^6$ Gy. However, our research suggests that even substantially lower doses of γ-irradiation can lead to remarkable changes in the material.

In our experiments hardened cement paste (HCP) has been exposed to γ-irradiation up to a dose of 1 MGy and then the changes of properties of the porous space were studied by mercury porosimetry and adsorption of nitrogen.

It was found that the concentration of nanopores in the hardened cement paste increases and the concentration of pores with a diameter in the range of 10 – 50 nm decreases due to γ-irradiation. The most significant change induced by irradiation is the specific pore area. It can be interpreted as coarsening of an originally smooth surface of the pores. It corresponds to our hypothesis of so-called “radiation carbonation” of the HCP: the crystals of calcite growing during the carbonation decrease diameter of the pores and increase their surface. There is, however, another effect of γ-irradiation on HCP: the γ-irradiation causes the radiolysis of water and this leads to drying of samples and can therefore result in the shrinkage of HCP. Our experiments really show that the relative change of mass of the control samples was only 1 %, but the relative change of mass of the irradiated samples was up to 6 %. Therefore, the shrinkage due to drying may also explain an observed decrease of the mean (average) pore diameter, the specific volume of pores and porosity. It can also explain (contrary to the “radiation carbonation” hypothesis) that the bulk density and the skeleton density did not change their values due to irradiation. Nevertheless, it cannot explain an increase in the pore surface.

Our experimental results can be most likely interpreted by two independent phenomena: the shrinkage due to radiation drying and radiation carbonation.

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Thermally stable semimetal graphite/n-type ZnO Schottky contact

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In recent years, increased interest in the application of ZnO in new optoelectronic and microelectronic devices has emerged [1,2]. One of the crucial tasks, which has come into play, is understanding and control of ZnO electrical contact properties. High quality ohmic and Schottky contacts (SC) are a prerequisite for the fabrication of high performance ZnO based devices. Moreover, high quality Schottky contacts are crucial vehicles to study electrically active defects in semiconductor materials by capacitance-based defect characterization techniques such as capacitance-voltage measurement (C-V), deep-level transient spectroscopy (DLTS), and admittance spectroscopy (AS). In fact, only rather limited number of studies exist utilizing capacitance-based defect characterization techniques to study electrical active defect in single crystalline ZnO [3], reflecting the challenge to prepare high-performance Schottky contact. We report the formation of a Schottky contact by printing colloidal graphite on an O- and Zn-face ZnO substrate [4]. A significant face effect with higher quality barriers achieved on O-face compared to Zn-face was observed. The extracted values of the barrier height and of the ideality factor from I-V characteristics were 0.89 eV and 1.0 for O-face and apparent 0.60 eV and 2.53 for Zn-face, respectively. This significant variation was explained by differences in the defect nature of the Zn-face and O-face surfaces of ZnO which was confirmed by photoluminescence (PL) measurements. In the case of O-face ZnO substrate, the current transport through the Schottky barrier was described by thermionic emission model and extracted values of the barrier height followed the Schottky-Mott relation. Using the Schottky-Mott relation together with the assumption that Fermi levels of the semiconductors are not pinned, we calculated the graphite contact work function $\phi = 5.09$ eV. This value is in a good agreement with theoretical and experimental values reported in the literature [5]. Temperature I-V measured of semimetal graphite/ZnO SC showed that TE was dominant in current transport at RT and above. We demonstrated that low-energy deposition colloid graphite on ZnO substrate can be good candidates for replacing conventional metal contact, which degrade easily at high temperature by diffusing into semiconductor and irreversibly forming undesirable ohmic contact.

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Methods of Accelerated Growth of Tin Whiskers

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Unalloyed tin electroplating has a long history of whisker formation that has resulted in present reliability risks for electronic equipment [1]. Tin whiskers can grow between adjacent conductors of different potential, causing transient or permanent electrical shorts. In addition, the whiskers can break loose, causing mechanical damage in slip rings, optical components or MEMS.

The objective of our work is to investigate methods for tin whisker growth, which can further enable measuring the effectiveness of mitigation strategies. To achieve this objective, a set of experiments was conducted using bright and matte tin platings on brass and bronze coupons and hot-solder dipped bronze coupons. Two sets of experiments were conducted: static stress test (permanent bending, permanent compression) and dynamic bending test (one-sided bend).

The results clearly show a high reliability risk when layers of pure tin are used. However, the risk does also occur when tin-rich solder alloys are used. The tin whiskers have been grown under these conditions. Furthermore, it happened in a relatively very short time. The first signs of tin whisker growth were identified after 380 hours of exposure to dry heat. According to the evidence of the experimental data, the absence of Cu underlayer slows first whiskers occurrence nearly twice. The presence of Cu underlayer is supposed to accelerate diffusion of Cu atoms into the tin layer and the formation of intermetallic compounds. Another risk is caused by brighteners in galvanic bathe if layers from bright tin are deposited. The extreme risk is brought by brighteners consisted of organic compounds with large molecules containing a greater number of inter-linked benzene nuclei. The results also proved that tin whiskers can also grow on the surface of lead-free solders. Hot dip applied lead-free solder used in the dynamic stress test failed on all tested samples (Sn99Cu1, Sn95.5Ag3.8Cu0.7, Sn97Cu3), as well as a the reference sample with electroplated pure tin. The Whiskers grow after load of 84 000 bending cycles.

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