### <u>DMS – RE 2013</u>

The twenty-third joint seminar

# DEVELOPMENT OF MATERIALS SCIENCE IN RESEARCH AND EDUCATION

BOOK OF ABSTRACTS OF THE 23 rd JOINT SEMINAR

September 9 – 13, 2013 Kežmarské Žľaby

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The twenty-third joint seminar

# DEVELOPMENT OF MATERIALS SCIENCE IN RESEARCH AND EDUCATION

September 9 – 13, 2013 Kežmarské Žľaby

Organized by

Slovak Expert Group of Solid State Chemistry and Physics Czech and Slovak Association for Crystal Growth Faculty of Chemical and Food Technology STU Faculty of Materials Science and Technology STU Crystallographic Society Slovak Society for Industrial Chemistry Regional Committee of Czech and Slovak Crystallographers

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### FOREWORD

The seminar "Development of Materials Science in Research and Education" is already the twenty third in the series started at Gabčíkovo in 1991 by the initiative of the Czech and Slovak Association for Crystal Growth and the Slovak Expert Group of Solid State Chemistry and Physics.

The objective of this meeting is to offer an opportunity to Czech and Slovak teachers and scientists as well as guests from other countries who are working in the field of Materials Science to present their recent results and experience and to exchange new ideas and information.

The scientific session will cover the following topics on 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 workshop is aimed at creation of a stimulating atmosphere of cooperation and at the support of patient dissemination of scientific idea s and propagation of materials science in education.

Organizers

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### PROGRAM

# Monday, 9 September 2013

10:00	_	13:30	Registration Location: Hotel Langue
13:00	_	13:45	Lunch
14:00	_	14:10	Opening Location: Lecture Hall
14:10	_	15:40	Monday Session I Location: Lecture Hall (chairperson: B. Papánková)
14:10	_	14:30:	Z. Kožíšek:
14:30	_	14:50:	Limits of the applicability of classical nucleation theory Karel Nitsch: Preparation and characterization of Er doped potassium ytterbium lanthanum phosphate glasses
14:50	_	15:10:	Miroslava Rodová:
15:10	_	15:30:	Study on crystallization kinetics of the molten glassy $Er:LiGd(PO_3)_4$ Petra Zemenová: Study of activation energy of $Er:LiY(PO_2)_4$ glass
15:30	_	15:50:	Alexey Sveshnikov: Statistics of heterogeneous nucleation
15:50	_	16:20	Coffee break
16:20	_	17:40	Monday Session II Location: Lecture Hall
			(chairperson: Z. Kožíšek)

16:20 – 16:40: Petr Kalenda: *Crystallization of lead tungstate-phosphate glasses* 

16:40	_	17:00:	Tomáš Hlásek:
			Preparation of ytterbium and erbium 2-methoxyethoxides applicable at the sol-gel synthesis of RE garnets
17:00	_	17:20:	Martin Plaček:
			The growth of whiskers on thick layers of lead-free solders
17:20	_	17:40:	Ondřej Marcinka:
			Whiskers growth on lead-free solder under dynamic stress
18:10	_	19:00	Dinner
19:30	_	22:30	Welcome Party

## **Tuesday, 10 September 2013**

08:00 – 8:45 Breakfast

09:00	—	10:00	Tuesday Session I
			(chairperson: M. Koman)
09:00	_	09:20:	Ivana Pilarčíková:
			Strategy for Obtaining Quality Applicants to College Engineering Programmes
09:20	_	09:40:	Kateřina Rubešová:
			Innovation of general and inorganic chemistry education in ICT Prague - Operational Programme Prague - Adaptability
09:40	_	10:00:	Kristýna Králová:
			Niels Steensen and his contributions to crystal growth and crystal- lography
10:00	_	10:30	Coffee break
10:30	_	12:10	Tuesday Session II
			Location: Lecture Hall
			(chairperson: I. Pilarčíková)
10:30	_	10:50:	Zdeněk Bureš:
			Preparation, characterization and microbiological properties of nanocopper, nanogold and nanosilver
10:50	_	11:10:	Blažena Papánková:
			Single-molecule magnets progress

11:10	_	11:30:	Ľubor Dlháň:
			Magnetism of Finite Magnetic Chains of Fe(III)
11:30	_	11:50:	Katarína Lacková:
			Pentacoordinated Cu(II) compounds with tricyanomethanide anion
11:50	_	12:10:	Marian Koman:
			New tetrameric copper(II) complexes.
12:30	_	13:15	Lunch

Location: Lunchroom

14:00	_	15:40	Tuesday Session III
			Location: Lecture Hall
			(chairperson: M. Behulová)
14:00	_	14:20:	Jaromír Drápala:
			Thermodynamic and experimental study of tin – zinc – aluminum
			ternary system
14:20	_	14:40:	Maroš Martinkovič:
			Modelling of deformed grains in polycrystals with regular polyhedra
14:40	_	15:00:	Michal Madaj:
			Modelling of ternary alloy systems using polythemal and isothermal sections
15:00	_	15:20:	Jan Kulveit:
			<i>Heterogeneous nucleation on heterogeneous substrate in lattice Ising model</i>
15:20	_	15:40:	Vladimír Jorík:
			<i>Beta-glucan complexes: Characterization by X-Ray powder diffrac- tion</i>
15:40	_	16:10	Coffee break
16:10	_	17:50	Tuesday Session IV
			Location: Lecture Hall
			(chairperson: J. Drápala)
16:10	_	16:30:	Mária Behúlová:
			Thermally induced microstructure transition in the tool steel of lede- burite type
16:30	_	16:50:	Robert Král:
			Study and modeling of temperature field and melt flow in simulated crystal growth of PbCl <sub>2</sub> by vertical Bridgman method

16:50	_	17:10:	Ladislav Nádherný:
			Phase diagram of Zn-Mn-O system for diluted magnetic semicon-
			ductor study
17:10	_	17:30:	Pavlína Ruleová:
			Preparation and transport properties of layered semiconductor -
			$Bi_2O_2Se$
17:30	_	17:50:	Martina Mahuliaková:
			HGMS using permanent magnets for separation submicron particles
18:00	_	18:45	Dinner

## Wednesday, 11 September 2013

- 08:00 8:45 Breakfast
- 09:00 17:00 Panel discussion and joint meeting
- 18:00 18:45 Dinner

### Thursday, 12 September 2013

08:00 – 8:45 Breakfast

09:00	_	10:00	Thursday Session I
			Location: Lecture Hall
			(chairperson: Z. Potůček)
09:00	_	09:20:	Petr Mošner:
			Application of thermoanalytical methods to the study of glasses
09:20	_	09:40:	Ivana Rösslerová:
			Viscosity and thermal behavior of zinc phosphate glasses
09:40	_	10:00:	Maryna Vorokhta:
			Structure and properties of $GeO_2$ modified borophosphate glasses.
10:00	_	10:30	Coffee break

10:30	_	12:10	Thursday Session II
			Location: Lecture Hall
			(chairperson: P. Mošner)
10:30	_	10:50:	Zdeněk Potůček:
			Photoluminescence of Erbium Doped K <sub>1-x</sub> Li <sub>x</sub> TaO <sub>3</sub> :Er Crystals
10:50	_	11:10:	Kateřina Dragounová:
			Luminescence Spectroscopy of Impurity Ions in Nominally Pure
			SrTiO <sub>3</sub> Crystals
11:10	_	11:30:	Zuzana Onderišinová:
			Optical and luminescent properties of $Ce^{3+}$ doped GdLuAG and
			GdYAG garnet scintillators
11:30	—	11:50:	Karel Král:
			Photoluminescence of hybrid quantum dots
11:50	—	12:10:	Jiří A. Mareš:
			Scintillation properties of Pr-doped garnets
12:30	_	13:15	Lunch
			Location: Lunchroom

14:00	_	15:20	Thursday Session III
			Location: Lecture Hall
			(chairperson: Ľ. Dlháň)
14:00	_	14:20:	Milena Luňáčková:
			Spectral interferometry for surface plasmon resonance sensing of aqueous solutions
14:20	_	14:40:	Vít Jirásek:
			DFT calculations of vibrational spectra of diamond and silicon sur- faces with different surface orientation
14:40	_	15:00:	Zdeněk Remeš:
			The comparison of the optical spectra of carbon coatings prepared by magnetron sputtering and microwave plasma enhanced chemical vapor deposition and measured by the absolute photothermal deflec- tions
15:00	_	15:20:	Josef Sedláček:
			Spark Plasma Sintering of Dielectric Ceramics $ZrSnTiO_4$
15:20	_	15:50	Coffee break

15:50	_	16:50	Thursday Session IV
			Location: Lecture Hall
			(chairperson: J. Luňáček)
15:50	_	16:10:	Pavel Mach:
			<i>Electrical properties of electrically conductive adhesives thermally aged under mechanical stress</i>
16:10	_	16:30:	Miroslav Kučera:
			Liquid phase epitaxy of rare earth garnets and silicates for scintilla- tors
16:30	_	16:50:	Vít Jakeš:
			Determination of oxygen content in mixed cobalt oxides
19:30	-	22:30	Farewell party

## Friday, 13 September 2013

08:00 – 8:45 Breakfast

09:00	—	9:40	Friday Session I
			Location: Lecture Hall
			(chairperson: M. Koman)
09:00	_	09:20:	Martin Klejch:
			High pressure durability of sapphire capillaries
09:20	_	09:40:	Lukáš Válek:
			Direct wafer bonding for SOI
09:40	_	9:50	Closing
09.50	_	10.10	Coffee break
07.00		10.10	e en e e ant

## ABSTRACTS

# Thermally induced microstructure transition in the tool steel of ledeburite type

Jana Mesárošová, Mária Behúlová, and Peter Grgač

Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava, Paulínska 16, 917 24 Trnava, Slovak Republic

Tool steels of ledeburite type form a group of alloys produced mainly by powder metallurgy using the gas atomization of melt followed by consolidation of rapidly solidified particles. They are characteristic by the high wear resistance resulting from the carbide phases of solidification origin which are present in their microstructure [1-6].

The paper deals with the microstructure development in the hypoeutectic tool steel Ch12MF4 with the chemical composition of 2.37% C, 12.06 % Cr, 1.2% Mo, 4.0% V and balance Fe [wt. %] in the process of nitrogen gas atomisation. At quasi-equilibrium conditions, the solidification of this steel starts by the crystallization of austenite dendrites. It proceeds by two eutectic reactions during which two types of morphologically different carbides (MC and  $M_7C_3$ ) develop [6-7]. In rapidly solidified powder particles, three main types of solidification microstructures were identified- dendritic, cellular and compound, representing the mixture of dendritic and cellular microstructures [6-10]. The scheme and microstructures documented the process of microstructure transition from dendritic to compound and cellular by the thermally induced procedure of dendrites fragmentation and following spheroidization of dendrite fragments in rapidly solidified powder particles are presented and discussed.

The research has been supported by the Scientific Grant Agency of the Slovak Republic (VEGA) within the project No. 1/1041/11 and the project ITMS 26220120048 under the Research and Development Operational Programme funded by the ERDF.

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# Preparation, characterization and microbiological properties of nanocopper, nanogold and nanosilver

Zdeněk Bureš<sup>1</sup>, Jiří Palarčík<sup>2</sup>, Jarmila Vytřasová<sup>3</sup>, and Miroslav Vlček<sup>1</sup>

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The aim of the work was to synthetise Gold (AuNPs), Silver (AgNPs) and Copper nanoparticles (CuNPs) using different methods, characterization and estimation of their biological activity in vitro. Ascorbic Acid, Glucose, Sodium Citrate and Sodium Borohydride were used as reducing agents in order to prepare metal nanoparticles of various sizes from their salts (Silver Nitrate, Tetrachloroauric Acid, Copper Sulfate). The dispersions were characterized by DLS (Dynamic Light Scattering) and  $\zeta$ -potential measurement. Antibacterial activities of the prepared dispersions were tested qualitatively using the inhibition zone method (application of disc with bio-active substance on inoculated agar plate in Petri dish). CuNPs, AuNPs and AgNPs dispersions were applied on the agar media with Staphylococcus aureus, Escherichia coli, Saccharomyces cerevisiae and Candida albicans resp. glabrata. The dispersions inhibited the growth of these microbes studied. It was found that all these nanodispersed metals have significant bacteristatical effect, but no bactericidal effect.

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.

### Magnetism of Finite Magnetic Chains of Fe(III)

Ľubor Dlháň<sup>1</sup>, Roman Boča<sup>2</sup>, Beáta Vranovičová<sup>2</sup>, and Vladimir Kokozay<sup>3</sup>

<sup>1</sup>Institute of Inorganic Chemistry (FCHPT),, Slovak University of Technology, Radlinskeho 9, SK-812 37 Bratislava, Slovakia

<sup>2</sup>Department of Chemistry, FPV, University of SS Cyril and Methodius, Trnava, Slovakia <sup>3</sup>Department of Inorganic Chemistry, Taras Shevchenko National University of Kyiv, Ukraine

Finite chains of Fe(III) eventually decorated by heteroatoms like Co(III) and Cu(II) have been studied by X-ray crystallography and SQUID magnetometry. The catena-[Fe(III)nCo(III)m] complexes under study cover systems with the following core:  $1 = Fe_2Co_4$ ,  $2 = Fe_2Co_2$ ,  $3 = Fe_4Co_4$ ,  $4 = Fe_6Co_6$ , and  $5 = Fe_2Cu_2$ .

The SQUID magnetometry has been conducted by taking the temperature dependence of the magnetic susceptibility at small field of B0 = 0.1 T, and field dependence of the magnetization until B = 7 T at T0 = 2.0 and 4.6 K, respectively.

The exchange interactions in these O-bridged FeIII systems are of an antiferromagnetic nature with different negative coupling constants according to the actual topology. Attempts to correlate the coupling constants J with the structural parameters are presented.

This work was supported by Slovak grant agencies (VEGA 1/0052/11, 1/0233/12, APVV-0014-11).

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### Luminescence Spectroscopy of Impurity Ions in Nominally Pure SrTiO<sub>3</sub> Crystals

Zdeněk Potůček, Zdeněk Bryknar, and Kateřina Dragounová

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Properties of perovskite-like ABO<sub>3</sub> oxides advantageous for wide range of applications are very sensitive to content of impurities and can be successfully improved by doping with suitable admixtures. A favorite model material for study of doping effect on properties of these materials is strontium titanate (SrTiO<sub>3</sub>). Unfortunately, transition metal ions typically contaminate SrTiO<sub>3</sub> crystals during growth process and strongly influence a whole range of their properties such as optical transmission, electric conductivity or photorefractive properties. Consequently proper understanding of doping induced phenomena observed in SrTiO<sub>3</sub> crystals necessarily requires considering possible effect of present unavoidable impurities. In this context, one of the most convenient methods to obtain needed knowledge on transition metal impurity centers in SrTiO<sub>3</sub> crystals is luminescence spectroscopy because of high sensitivity to low concentrations of impurity ions and possibility to determine their properties in diverse valence states. Therefore, we performed a study of photoluminescence of a usual nominally pure SrTiO<sub>3</sub> single crystal grown by the Verneuil technique. Photoluminescence emission and excitation spectra were examined at temperatures between 12 and 300 K within the 350 - 1600 nm and 250 - 1000 nm spectral range, respectively.

Three structured impurity related emission bands with relative intensities dependent on excitation wavelength were revealed in the low-temperature emission spectra of photoluminescence of the nominally pure SrTiO<sub>3</sub> crystal studied. The emission bands consisted of pronounced zero-phonon line accompanied by well developed vibrational sidebands. The emission bands with zero-phonon line peaking at 12595 and 13827 cm<sup>-1</sup> at 12 K were observed earlier on SrTiO<sub>3</sub> crystals doped by Cr and Mn, respectively, and attributed to <sup>2</sup>E  $\rightarrow$ <sup>4</sup>A<sub>2</sub> transition in Cr<sup>3+</sup> and Mn<sup>4+</sup> (3d<sup>3</sup>) impurity ions substituted for octahedral coordinated Ti<sup>4+</sup> ions [1, 2]. The third emission band with zero-phonon line peaking at 8642 cm<sup>-1</sup> at 77 K was to our knowledge detected in SrTiO<sub>3</sub> crystals for the first time. Two excitation bands centered near 15800 and 20400 cm<sup>-1</sup> were found below the SrTiO<sub>3</sub> absorption edge in the excitation spectrum of this infrared luminescence at 77 K. The origin of responsible luminescence centers will be discussed taking into consideration probable vanadium impurity in the crystal studied and analogy of spectral features of the observed emission and excitation spectra with photoluminescence of V<sup>3+</sup> (3d<sup>2</sup>) ions in sixfold oxygen coordination [3].

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# Thermodynamic and experimental study of tin – zinc – aluminum ternary system

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Six binary Sn–Zn and twenty four ternary Al–Sn–Zn alloys with various content of elements were prepared experimentally. The alloys were studied metallographically after casting and after long-time annealing in vacuum. X-ray structural and chemical micro-analysis (EDX, WDX) of the phases was measured. Temperatures and latent heats of characteristic phase transitions (liquidus, solidus, invariant reactions etc.) were obtained with use of the DTA method. Experiments were performed with Setaram SETSYS 18TM experimental laboratory system for thermal analysis. Resulting experimental data were compared with data of known Sn–Zn binary system and Al–Sn–Zn ternary system. For the modelling of phase equilibria from critically assessed data software packages were used MTDATA, PANDAT and Thermo-Calc. Experimental results of the studied alloys from the point of view to measured structural and physico-chemical characteristics were compared with thermodynamic calculations. The result will be present in the form of isothermal and poly-thermal sections of the ternary Al–Sn–Zn system.

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### Preparation of ytterbium and erbium 2-methoxyethoxides applicable at the sol-gel synthesis of RE garnets

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Among rare earth (RE) ions, the doping of either Er3+ or Yb3+ is often used in optical materials. When optical materials are prepared by sol-gel process, the dopants are added in different forms depending on the chosen method. However, thanks to the low concentration (tenths of %), the form of starting compound has negligible effect on the quality of a final film. Different situation occurs when rare earth ions are a major part of a host material, such as RE aluminium garnets - e.g.  $Yb_3Al_5O_{12}$  has been established as a suitable luminescent oxide. In this case, an inappropriately chosen starting RE compound can cause cracking of the thin films during the thermal treatment of deposited gel layers. An alkoxide sol-gel process is a powerful way at functional oxides preparation and 2-methoxyethoxide metal derivatives are successfully used at films deposition due to their lower sensitivity to hydrolysis.

There are only a few works about the preparation of RE 2-methoxyethoxides; moreover, none of them describes further use of the alkoxide in a sol-gel process. Our aim was not to isolate individual 2-methoxyethoxides but to prepare intermediate solution without any necessary purification, directly applicable at the preparation of Yb/Er/Al alkoxide solution.

Different synthesis methods of preparation were used. The formation of alkoxides was studied by IR spectroscopy. Especially, the changes in the C-O terminal and bridging stretching modes (that are typical for metal alkoxides) were monitored.

Firstly, ytterbium or erbium powders were heated at  $126 \,^{\circ}$ C under reflux in 2-methoxyethanol in the presence of HgCl<sub>2</sub>. Even though the heating time overcame 10 days, the quantitative dissolution was not achieved. When electrolysis was applied, the metals dissolved in 2-methoxyethanol readily. However, as the electrolysis was proceeding, the surface of metal pieces was corroded so that the powder of unreacted metal shed to the solution. Therefore, after electrolysis, it was necessary to filter the suspension. Moreover, the method produces "waste" metal powder and becomes uneconomical. The exchange reaction between i-propoxides and 2-methoxyethanol was fully successful; arising i-propanol was easy to distil out. The ease of this reaction is caused by the sum of several factors - e.g. the lower boiling point of i-propanol or slightly lower pKa of 2-methoxyethanol. The exchange reaction between acetates and 2-methoxyethanol was also studied. In this case, we can confirm an exchanging reaction also by the detection of released acetic acid. Unfortunately, the acid under reflux in the used alcohol forms ester which cannot be distilled out (b.p. 145 °C). The present ester could cause the cracking of a prepared film.

Solutions from exchange reactions are suitable for thin film preparation without any purification. We are going to study the influence of the ester presence on a film microstructure in the further work.

### Determination of oxygen content in mixed cobalt oxides

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Thermoelectric materials allow the conversion of heat to electricity and vice versa. However, this ability is not being used as much as expected, due to low thermoelectric efficiency (so called "figure of merit"). Therefore, the research has focused lately on finding new materials with higher figure of merit and also stable at elevated temperature. Among the most studied groups of such materials are mixed cobalt oxides (cobaltites), where cobalt exists in various oxidation states ( $Co^{3+}$  and  $Co^{4+}$ ). The electron state of cobalt directly affects the thermoelectric properties of the material. The cobalt heterovalency is naturally exhibited in the oxygen content in studied material. In this work, the possibility of oxygen content determination was studied using titration methods (iodometry, cerimetry), UV-VIS spectrophotometry and the thermogravimetric reduction of the sample in hydrogen atmosphere. As model samples, thermoelectric cobaltites LaCoO<sub>3</sub>, La<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3-x</sub> and Ca<sub>3</sub>Co<sub>3.95</sub>O<sub>9+x</sub> were used.

The use of titration methods, in which the sample must be transferred into solution, is limited due to high oxidation power of  $\text{Co}^{3+}$  (with  $\text{Co}^{4+}$  being even stronger oxidizer). This causes water to oxidize during dissolution process. For iodometric determination, several manners of sample dissolution were tested. The sample was dissolved in HCl or HBr of variable concentration; iodide was added either before dissolution started or after the complete dissolution. In neither of these cases, iodine was titrated with thiosulphate. This fact suggests that the higher oxidation states of cobalt oxidized water preferentially to iodide. Next, we tried to decrease the oxidation power of  $\text{Co}^{3+}$  by complexation. Our aim was to lower the reduction potential into the water stability area in Pourbaix diagram and simultaneously to keep it high enough to be able to oxidize iodide to iodine. As complexing agents, iminodiacetic acid (IDA) and triethylentetraamminehexaacetic acid (HEDTA) were used. Neither in this case iodine was detected. The second titration method used was cerimetry. The same methods of sample dissolution were tested but the presence of  $\text{Co}^{3+}$  in the solution was not confirmed – the titration agent consumption equaled to the amount of Fe<sup>2+</sup> added to the solution. This means that all the  $\text{Co}^{3+}$  was reduced during the dissolution.

Apart from titration methods, spectrophotometry was used, based on the absorbance measuring of complex cobalt forms  $[Co(SCN)_4]^{2-}$  and  $[Co(edta)]^-$ . These measurements proved to be inaccurate due the impossibility of proper calibration caused by the complex composition of the samples (other cations also react with the complexants). The last method used was the direct reduction of the sample in hydrogen atmosphere at elevated temperature. In such case, the sample is reduced to elementary cobalt and the respective oxides of the remaining cations. From the mass change before and after the reduction, the oxygen content was calculated. From the methods tested in this work, this analysis proved to be the most reliable.

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### DFT calculations of vibrational spectra of diamond and silicon surfaces with different surface orientation

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First principle calculations based on the DFT-B3LYP theory were used for modelling of selected features in infrared vibrational spectra of diamond and silicon surfaces. Different functional groups predominantly on the (111) surface were studied. The calculated results are discussed together with the experimental FTIR spectra and spectral databases of simple gas and liquid molecules. A special emphasis is devoted to the C=O groups of functionalized diamond nanoparticles, which are usually separated from the other groups in the FTIR spectra. Compared to the isolated molecules in a gas phase, the peak positions of the C=O stretch modes of the surface-bonded carboxyl groups are shifted to lower frequencies by  $50-90 \text{ cm}^{-1}$ . but they lay, by coincidence, in the range of liquid-phase carboxylic acids (1700-1730 cm<sup>-1</sup>). The anhydrides and lactones, which are formed by the condensation of two -OH groups, are calculated in the range 1730-1850 cm<sup>-1</sup> and the bands are very broadened as their surface density increases. Further, the easily-observable in the spectra and surface-orientation dependent Si-H bonds on the purely hydrogenated silicon surface were calculated. It was shown that, unlike SiH<sub>3</sub> groups, the vibrations of SiH and SiH<sub>2</sub> groups exhibit characteristic frequency shifts in dependence on their position on either flat surface or on a step or edge. In comparison with the flat surface Si-H stretch, the SiH mode for H atoms closest to the step of the ideally terminated (111) is shifted in a very different extent to higher frequencies due to closer mutual distance of H atoms and their repulsion. On the other hand, the modes for H atoms at the (111)/(111) edge of a pyramidal grain are shifted towards lower frequencies (up to 22 cm<sup>-1</sup>). A large diversity of the pure surface modes (except the SiH<sub>3</sub> group) was found on the (100) surface. Due to the different kinds of (100) surface reconstruction, the SiH modes are spread in the range 2060-2150 cm<sup>-1</sup>.

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### Beta-glucan complexes: Characterization by X-Ray powder diffraction

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The complexes of  $\beta$ -glucan with complex former substances (CF) were prepared in water or dimethylsulfoxide medium. Nutraceuticals, i.e. folic acid, boswellic acids, ascorbic acid, coenzyme Q10, quercetin and curcumin were used as CFs. Although the CFs are crystalline,  $\beta$ -glucan appears to be amorphous and prepared  $\beta$ -glucan:CF complexes, too. For this reason was introduced a new assessment method and the X-Ray powder diffraction (XRPD) characterization of  $\beta$ -glucan:CF complexes using the line profile analysis. This method can help to demonstrate experimental evidence for the formation of novel  $\beta$ -glucan:CF complexes.

 $\beta$ -glucan possesses a polymer structure resulting in very broad overlapped peaks.  $\beta$ glucan: CF complexes retain the character of  $\beta$ -glucan polymer structure and that also a character of powder diffraction pattern. In order to analyze their powder diffraction patterns in the entire measurement range from 1.5 to  $61.5^{\circ} 2 \theta$ , GSAS package in "rawplot" mode was used [1-2]. A line profile analysis was applied to inspect subtle differences in diffraction patterns. Similar XRPD patterns of all  $\beta$ -glucan complexes, virtually formed by two the very broad peaks, exclude the possibility their simple comparison. By the means of line profile analysis we were able to deconvolute each powder pattern into four peaks unambiguously characterized by their peak position and integral breadth ( $\beta$ ). The results of line profile analysis of  $\beta$ -glucan:CF complexes confirm the formation of new phases. In addition, from the XRD was found that formation of complexes runs for the total consumption of the initial CFs. No diffraction peaks of CFs were found in powder diffraction patterns of  $\beta$ -glucan:CF complexes. The XRPD pattern of the physical mixture was quite different from the XRPD pattern of the complex and showed sharp diffraction peaks corresponding that of the CF. Generally, these observations indicated that CF and  $\beta$ -glucan were not present in the complex as unbound forms.

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### Crystallization of lead tungstate-phosphate glasses

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The crystallization of PbO-WO<sub>3</sub>-P<sub>2</sub>O<sub>5</sub> ternary glasses was studied in two compositional series of  $(100-x)[0.5PbO-0.5P_2O_5]$ -xWO<sub>3</sub> glasses (A series) and  $50PbO-(50-y)P_2O_5$ -yWO<sub>3</sub> glasses (B series). The crystallization products of the annealed glass powders were evaluated using X-ray diffraction and Raman spectroscopy.

In the (A) series  $(100-x)[0.5PbO-0.5P_2O_5]$ -xWO<sub>3</sub>: glasses were prepared with 0-60 mol% WO<sub>3</sub> [1]. Glasses were crystallized in the powder form by annealing above their crystallization temperatures determined by DTA. The results of the X-ray diffraction analysis of the crystallized glasses revealed the formation of several crystalline phosphate compounds and in the samples with a high WO<sub>3</sub> content also the formation of a new crystalline compound of Pb(WO<sub>2</sub>)<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> at the glass composition with x = 50 mol% WO<sub>3</sub> [2]. Raman spectra of the polycrystals and glass of the same composition showed on the close relations between both forms.

The mechanism of crystallization of the glass composition  $Pb(WO_2)_2(PO_4)_2$  was evaluated using Day's method [3]. Raman spectra of the crystallized samples confirmed the results of the X-ray diffraction measurements and provided information on the thermal stability of the glasses and formation of the glass-crystalline phases by the annealing of glass samples.

In the (B) series:  $50PbO-(50-y)P_2O_5-yWO_3$  with 0-30 mol% WO\_3 all glasses crystallize on heating. Crystalline phases formed by annealing cover not only various lead phosphates, but also the compounds  $PbWO_4$  and  $Pb(WO_2)_2(PO_4)_2$  and a new unidentified ternary lead tungstate-phosphate compound formed by annealing the glass with y = 30 mol% WO\_3.

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### High pressure durability of sapphire capillaries

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Sapphire, the single crystal of aluminium oxide, is a desired material for many demanding applications thanks to its unique physical and chemical properties, e.g., high mechanical durability, excellent optical properties, chemical stability against most substances and high melting point [1]. Employing the "Edge-defined Film-fed Growth" (EFG) method, profiled crystal are manufactured, mostly in the form of tubes [2]. Sapphire capillaries are successfully used for X-ray diffraction studies in non-ambient environments [3].

We investigated the strength of sapphire by a pressure burst test. Single crystal capillaries can withstand inside pressure reaching, or even exceeding, 100 MPa. A handy formula can be derived to estimate the maximum pressure achievable inside a general tube before it bursts [4]:  $p_b = \sigma_{\max} \cdot (K^2 - 1)/(K^2 + 1)$ , where  $\sigma_{\max}$  is the material ultimate tensile strength and Kis the ratio of outer/inner diameter of the cylindrical vessel. A hydrostatic testing apparatus was used for the measurement, using water as the pressurized medium. Two sizes of sapphire capillaries were tested in the experiments, namely 1.0/0.6 and 1.6/1.1 (outer/inner diameter in mm). They were closed on one end by a sapphire cap grown via the EFG technology and on the other end sealed by epoxy adhesive in a conical flange fitting in the testing device. The movement of the piston of the burst machine first filled the capillary with water and then was increasing its pressure until destruction of the tube. The thinner ones exhibit an average burst pressure of 117 ( $\pm$  19) MPa, the thicker ones could be pressurized up to 154 ( $\pm$  26) MPa. From this data, the strength of CRYTUR, spol. s r.o. EFG sapphire tubes was determined,  $\sigma_{\max} = 328 (\pm 54)$  MPa. This value is consistent with published data [1] and can be used for computing the burst pressure of any tube diameter using the above stated formula.

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### New tetrameric copper(II) complexes.

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Structural features of tetrameric copper(II) complexes can be divided into six major categories: (I) Cu4( $\mu$ 4-0) tetrahedron; (II) central Cu4O4 cubane type; (III) bifolded dimers; (IV) chain type; (V) step-like structure and (VI) unique structures [1]. Recently, we analyzed almost fifty examples of Cu4OX6L4 complexes X = Cl, Br and L = N, O donor ligands) [2]. In this paper are analyzed two crystal structures of two Cu(II) tetramers: Cu16( $\mu$ 4-0)4Br7Cl17(4-Mepy)16 (1) and Cu4OCl6(ron)4 (2) (4- Mepy = 4- methylpyridine; ron = ronicol (3- methanolpyridine)). The X-ray parameters of the complex (1) show that there are crystallographically four independent tetramers: CuCl3ON, CuCl3OO, CuCl3OCl and CuBr3ON. The crystal structure of (2) complex consists of three penetrating polyhedra, namely, OCu4 tetrahedrons, OCl6 octahedron and four CuOCl3N trigonal bipyramids. Such type of structure is suitable for better reasoning and understanding the donor-acceptor and electron-transfer process in copper proteins.

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### Limits of the applicability of classical nucleation theory

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Clusters of a new phase are formed within supercooled or supersaturated parent phase due to density fluctuations and after reaching of a certain critical size are able to grow up to macroscopic sizes. Supercritical clusters are called nuclei of a new phase. Basic growth unit of nucleus is atom, molecule, or repeating unit of polymer chain (monomer). Classical nucleation theory is based on the kinetic equations describing formation of the clusters by step-by-step process within Markovian approach under the following assumptions: (i) the number of monomers does not change in time and (ii) the work of formation of cluster can be approximated within capillarity approximation. Under these restrictions the analytical formula to nucleation rate and the number of supercritical sizes were derived. Is is easy to use this approaches and that is why it is widely used in analysis of experimental data. Nevertheless, in some cases the assumptions (i) and (ii) are not fulfil and that is why the predictions of theory fails.

We analyze the classical approach to nucleation with numerical solution of kinetic equations and with the experimental data in various systems (formation of droplets from supersaturated system, nucleation in glasses, and the polymer crystallization). Is is shown that it is appropriate to have data about nucleation kinetics for the nucleation rate and the size distribution of nuclei near its critical size. In some cases it is necessary to modify the work of formation of clusters. In small volumes the depletion of the parent phase plays important role and even no stationary value is reached. In that case the numerical solution of kinetic equation helps to predict experimental data [1, 2].

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### Photoluminescence of hybrid quantum dots

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Electronic excitation energy transfer is studied theoretically in the system of the hybrid quantum dots using the excitonic representation of the excited states of two quasi-zero dimensional subsystems between which the excitation energy is transferred as an irreversible kinetic process. The electron-phonon interaction is used to avoid the energy conservation problem in considering the uphill and downhill excitation transfer processes. The theory is studied with utilizing a simplified model of two interacting quantum dots. The theoretical results are presented together with numerical calculations. The presently considered calculation could be relevant to various cases of the electronic excitation energy transfer between quasi zero dimensional nanostructures.

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# Study and modeling of temperature field and melt flow in simulated crystal growth of PbCl<sub>2</sub> by vertical Bridgman method

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Influence of growth conditions i.e. temperature gradient in the furnace and the pulling rate, on the position and the shape of the crystal/melt interface and thus on the quality of grown crystal at the simulated vertical Bridgman growth were studied by experimental methods and by numerical modeling. Experimental methods were based on the temperature field measurements in a quartz ampoule filled with lead chloride (PbCl<sub>2</sub>) under zero pulling rate (stationary arrangement) and pulling rates of 1 and 3 mm/h (dynamic arrangement). Obtained temperature data describe the temperature field in the ampoule i.e. position and shape of isotherms including the position and shape of the crystal/melt interface at 500°C (m.p. PbCl<sub>2</sub>) [1].

Modeling was performed using commercially available COMSOL Multiphysics 4.1 software. Its application the temperature field and heat transfer (in solid, liquid and gaseous state), and melt flow i.e. direction and velocity of melt flow in PbCl<sub>2</sub> and surrounding atmosphere were computed. Experimentally obtained temperature data were used as boundary conditions for their computation. In stationary numerical model the PbCl<sub>2</sub> filled quartz ampoule attached into a nickel holder and surrounded by external air atmosphere were taken into account. In the dynamic model, with identical geometry as in the stationary, the latent heat and the pulling rate were incorporated into the heat capacity term. Simulation of the latent heat release near the crystal/melt interface was achieved by adding a smoothed Dirac function as described by Marin [2].

This contribution deals with the analysis of the temperature field and modeling of simulated crystal growth by the vertical Bridgman method. Its goal is to optimize growth process and find suitable growth conditions for crystal growth of attractive optical materials such are ternary alkali lead halides. These materials doped with rare earth elements are considered to be suitable low phonon host for infrared solid state lasers [1, 3].

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## Niels Steensen and his contributions to crystal growth and crystallography

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Danish scientist and bishop Niels Steensen was born in Copenhagen in 1638. His family expected him to take over the business of his father, a goldsmith, but Steensen entered the University of Copenhagen to pursue the medical studies. As a student he began, in accordance with the academic customs of his time, to use Latin name Nicolaus Stenonis. The commonly used name Steno is probably an incorrect derivation from this [1]. Steensen is considered to be one of the last polymaths, i.e. scientists who focused on a wide range of scientific disciplines. His research deals not only with medicine and anatomy, but also with geology, crystallography, crystal growth and other natural sciences. Steensens contribution to science is of a great importance. In the first place he suggested a modern approach to the natural sciences. In his early work, so called *Chaos - manuscript*, Steensen criticizes the contemporary state of the natural sciences as too conservative, related to the ancient authors and the Bible and closed to scientific progress. According to Steensen science must be based on logic and individual research made with accuracy and precision. His motto was: "Experimental exactitude, reproducible results". Steensens contribution to crystal growth and crystallography was published in his geological studies De solido intra solidum naturaliter contento dissertationis prodromus (Preliminary discourse to a dissertation on a solid body naturally contained within a solid) in 1669. Steensens observations concerning crystals are impressive, if we realize that he made them in the time, when no analytical methods were used. He has recognized, that crystals do not grow from "inside" like flowers and other living organisms, but from "outside" as a result of material deposition on the surface of the existing crystal. He has also disproved a common belief that all crystals were made from ice, and divided crystals into different groups according to parent material. It was also Steensen, who first discovered that the interfacial angles of quartz crystals were the same regardless of the size or shape of the crystal [2]. This principle was later named after him and has been known as Steno's law or Steno's law of constant angles. The last Steensens conclusion of great importance relates to the reciprocity of crystal growth and its dissolution. It took over 300 years before modern research on the crystal growth confirmed these observations [3].

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## Liquid phase epitaxy of rare earth garnets and silicates for scintillators

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Ce3+-doped YAG and LuAG single crystal are widely used for scintillation applications. Thin epitaxial films represent an interesting alternative to the bulk single crystals for 2D imaging scintillation screens with submicron resolution. Compared to the Czochralski grown single crystals, the epitaxial films can be grown from the flux at much lower growth temperatures and consequently with lower structural disorder and lower concentration of intrinsic defects.

Here we studied the effect of flux composition and the growth conditions on the structural, emission, and scintillation properties of epitaxial garnet and orthosilicate films. The films, typically simple garnets Ce:Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>, multicomponent garnets such as Ce:(GdYLu)<sub>3</sub>(AlGa)<sub>5</sub>O<sub>12</sub>, or structured silicates Tb:Y<sub>2</sub>SiO<sub>5</sub>/(CeY)<sub>2</sub>SiO<sub>5</sub> were grown on various substrates by the isothermal dipping liquid phase epitaxy. The films were characterized by the XRD, emission spectroscopies, decay kinetics, and by scintillation response using the alpha particles. A variety of fluxes have been used with the goal to reduce the fluxrelated defects and impurities and to obtain high light yield from the films. An advantage of the LPE technology is significantly higher Ce content in LuAG films (by a factor of 2-6 depending on the used flux) compared to the Czochralski grown single crystals.

Detailed discussion of the growth methods and summary of scintillation properties of our epitaxial films will be presented.

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## Heterogeneous nucleation on heterogeneous substrate in lattice Ising model

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In most heterogeneous nucleation studies, the substrate is assumed to be homogeneous. We investigate effects of surface inhomogeneity on nucleation barrier and rate in the simple case of lattice Ising model, using Monte Carlo (MC) methods. Simulations show that even if we keep average surface energy fixed, nucleation barrier can be altered considerably by variance of surface energy on small scales.

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#### Pentacoordinated Cu(II) compounds with tricyanomethanide anion

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The shape of coordination polyhedra (SCP) in the case of five-coordination is one of the current problems in coordination chemistry. With the aim of establishing possible reasons for different SCP in related compounds, our research group have previously prepared and studied the structures of five-coordinated copper(II) complexes of the general formula  $[Cu(L)_2X]Y$ , where L is 1,10-phenanthroline (phen), 2,2'-bipyridine (bpy) or 1,3-diaminopropane, X is  $N(CN)_2^-$  or  $ONC(CN)_2^-$  and Y is 1- anion [1-2]. The obtained results showed that the preferred SCP for compounds with phen is close to trigonal bipyramid, whereas SCP for bpy compounds is close to tetragonal pyramid.

It is known that tricyanomethanide anion (tcm,  $C(CN)_3^-$ ) can coordinate similarly as  $N(CN)_2^-$  and  $ONC(CN)_2^-$  anions [3]. Thus, to verify the findings about SCP, we have attempted to prepare compounds of general  $[Cu(L)_2C(CN)_3]Y$  formula (L = bpy or phen and Y = Br<sup>-</sup>, Cl<sup>-</sup>, ClO<sub>4</sub><sup>-</sup> and NO<sub>3</sub><sup>-</sup>) and checked their SCP. We have prepared five coordinated compounds containing required cations:  $[Cu(bpy)_2C(CN)_3]C(CN)_3$  (1),  $[Cu(phen)_2C(CN)_3]C(CN)_3$  (2),  $[Cu(bpy)_2C(CN)_3]ClO_4$  (3),  $K[Cu(phen)_2C(CN)_3]_3C(CN)_3(ClO_4)_3$  (4) and  $[Cu(phen)_2C(CN)_3][Cu(phen)_2H_2O]C(CN)_3(ClO_4)_2 \cdot H_2O$  (5) and another five coordination compounds with exchanged anions:  $[Cu(phen)_2NO_3]C(CN)_3$  (6),  $[Cu(bpy)_2Cl]C(CN)_3$  (7),

compounds with exchanged anions:  $[Cu(phen)_2NO_3]C(CN)_3$  (6),  $[Cu(bpy)_2CI]C(CN)_3$  (  $[Cu(phen)_2CI]C(CN)_3 \cdot H_2O$  (8),  $[Cu(bpy)_2Br]C(CN)_3 \cdot H_2O$  (9) and  $[Cu(phen)_2Br]C(CN)_3 \cdot 0.5H_2O \cdot 0.5EtOH$  (10).

The structures of the prepared compounds are stabilized by  $\pi$ - $\pi$  interactions and, moreover, hydrogen bonds were also observed in the structures containing water molecules.

Prepared compounds were characterized by infrared spectroscopy, elemental and X-ray analysis. SCP of cations in all prepared compounds was characterized by the parameters  $\tau$  [4] and  $\Sigma$  [5].

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## Spectral interferometry for surface plasmon resonance sensing of aqueous solutions

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This paper deals with a polarimetric setup to measure the refractive index change of aqueous solutions. The experimental method is based on surface plasmon resonance (SPR) in Kretschmann configuration combined with spectral interferometry. The experimental setup consists of a white-light source, a linear polarizer, a birefringent quartz crystal, a SF10 coupling prism covered by a gold layer (thickness 44 nm) and a linear analyzer. The angle of incidence is approximately 60 degrees. The attenuated total reflection at the prism base serves for the excitation of surface plasmon waves (SPWs). The output spectral interference signals affected by the SPWs are recorded by a spectrometer [1]. In order to calculate 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 (air) and the second to the analyte (NaCl aqueous solution). The shift of phase curve is related to the analyte refractive index change.

The refractive index of the NaCl aqueous solutions (0, 2 and 5 percent) was measured by the Abbe refractometer at a wavelength of 589.3 nm and compared with theoretical one, calculated by the Lorentz-Lorenz equation [3]. The resonance wavelengths as extremes of the SPR-induced differential phase changes retrieved from spectral interference signals were compared with the resonance wavelengths determined from spectral reflectance measurements [4]. A good agreement between the values was confirmed. Comparison of the experimental dependencies with the theoretical ones will be a subject of a subsequent work.

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#### Electrical properties of electrically conductive adhesives thermally aged under mechanical stress

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Electrically conductive adhesives (ECAs) are composite materials consisting of insulating matrix and electrically conductive filler. Epoxy resin is mostly used as a matrix and silver flakes as filler. ECAs are used for assembly of some types of electronics components, because some types of components must not be soldered because they do not have adequate resistance to temperatures required for soldering[1]. It was examined six types of electrically conductive adhesives of the epoxy type. Four adhesives of the one-component type and two adhesives of the two-component type. The curing temperature of adhesives was in the range of 140–180 o"C and the curing time in the range of 10-100 min in dependence on the type of adhesive. Measurements were performed on adhesive joints formed on test printed circuit boards by surface assembly of resistors with the zero resistance (jumpers). The resistance, noise and nonlinearity of the current-voltage characteristic of the joints were measured. The layout of the test boards made four-point measurement of the joints resistance possible, a precision LCR Meter HP 4284A was used for this measurement. The noise voltage was measured using a lock-in applifier PAR 124 A added with a differential pre-amplifier PAR 114, the measuring frequency was 373 Hz, the DC current 100 mA. Nonlinearity of the currentvoltage characteristic was measured using an intermodulation method. A spectral analyzer HP 8560 added with a piezoelectric filter was used for this measurement. Electrical properties of the joints were measured after adhesive assembly at first. Then the boards with assembled jumpers (there were 7 jumpers on one test-board) were warped using a special fixture. This way the mechanical load was inbuilt into every adhesive joint. The level of this load was calculated. All following experiments were provided on the warped boards. The joints properties were again measured after the deflection of the boards and then the boards were placed into a furnace with the controlled temperature and aged at the temperature of 120 °C for 300 hours. After the thermal ageing the electrical properties were again measured. Percentag changes of the joints resistance, noise and nonlinearity were evaluated. It was found that the mechanical load causes increase of the joint resistance, the reason is that the contacs among filler particles worsen this way. It was also found that the thermal ageing decreases the contanct resistance. It can be caused by the decrease of the adhesive volume caused by additional curing of adhesive and improvement of contacts among filler particles. Percentage changes measured for noise and nonlinearity of the current-voltage characteristics were mostly substantially higher then the percentage changes of the resistance. It means that these parameters are more sensitive to changes in adhestive than the adhesive resistivity.

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## Modelling of ternary alloy systems using polythemal and isothermal sections

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The ternary alloy phase diagram is formed by an equilateral triangle, the vertices of which represent pure components. A temperature axis rises perpendicularly from each vertex of the concentration triangle of this basis. Individual sided of the basis between temperature axes then represent the equilibrium phase diagrams of binary systems of the relevant components. Since the work with three-dimensional alloy systems is complicated, two-dimensional sections are used for simplification. Isothermal section represents a horizontal plane perpendicular to the temperature axis of the 3D phase diagram, while poly-thermal section is perpendicular to its basis. A very few complete ternary systems with projection of liquidus and solidus are available, that is why is developed a program for simulating and modelling of ternary systems from isothermal and poly-thermal sections and the related calculation of phase proportions in the course of solidification and the calculation of values of distribution coefficients in dependence on temperature.

Programming environment Matlab was used for description of ternary systems, as it enabled scientific-technical numerical calculations, graphical representations, simulations and modelling. Geometric representation of areas of liquidus, solidus and solvus was achieved by the so called B-spline areas. The software constructs complete 2D and 3D models of specific ternary systems, calculates parts of each phase in the equilibrium solidification or calculates equilibrium distribution coefficients depending on the temperature.

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#### HGMS using permanent magnets for separation submicron particles

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Magnetic separation is one of the methods that are used to separate magnetic and nonmagnetic components from the input mixture. Separation of the iron ferromagnetic compounds is usually not difficult. On the other hand, the separation of the paramagnetic particles, for example mineral particles contained in water, requires high gradient field [1]. This condition is hard to meet in classical separators.

This article presents a new laboratory magnetic separator with two NdFeB permanent magnets constructed at the Department of Physics. Its efficiency of paramagnetic particle separation was compared with the efficiency of the classical electromagnetic separator constructed at the Department of Physics too. The advantages of the separator design based on permanent magnets are following: they do not need the external electrical power and much higher values of the magnetic field can be achieved. High gradient magnetic field inside the separator is further increased by steel wool [2, 3]. The magnitude and distribution of the magnetic field in separator space was obtained using 2D computer models created in the framework of the ANSYS package.

The water dispersion of the paramagnetic  $Fe_2O_3$  powder was used for verification of separation efficiency. The samples of the three fractions were prepared by two steps: firstly the sedimentation separation was made and secondly the ultrasound cavitation disintegration was applied. Each of the fractions repeatedly passed the separation process in both separators under the same conditions. Experimental results of the separation efficiency were evaluated using the turbidimeter TN-100 and by the spectrometer Spekol Carlzeiss Jena 376581. It is evident that NdFeB permanent magnet separator shows higher separation efficiency than the classical one. This effect can be even increased by additional NdFeB permanent magnets in the separator sleeve.

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#### Whiskers growth on lead-free solder under dynamic stress

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The global trend of reducing the use of elements and substances which have an adverse effect on the human body has left the response in the EU legislation. The European Parliament issued a directive in 2002 [1], which prohibit, or at least restricts, the usage of certain toxic substances and elements. One of these substances is lead. Lead in combination with tin forms eutectic alloy which is widely used for soldering electrical components on the printed circuit board (PCB) for its excellent mechanical and electrical properties and long term stability. Restrictions of the use of lead means a gradual transition to lead-free solder. Although their properties are inferior to leaded solders is practically the only alternative.

With the new tin alloy without the presence of lead in time were a problem in terms of quality solder joints. For lead-free solders, however, a spontaneous growth of needle-shaped crystal structure which are called whiskers. It was shown that the presence of lead prevents their formation. They pose a risk in terms of reliability. These are outgrowths from the material surface with a diameter of 0,3-10  $\mu$ m reaching typically lenght around 500  $\mu$ m [2]. They can also have other shapes [3]. The growth of these conductive structure is undesirable for electrical circuits. Outgrows the whisker distance between two adjacent contacts can easily cause a short circuit and thus malfunction of PCB or adjacent connectors. The current in thin whiskers can reach values of the order of tens of mA [2]. Effective protection does not exist. Therefore, it is necessary to understand the causes of their origin. They haven't been already fully understood. It is shown that for the growth of whiskers may increase tension (stress) within the solder alloy. This has several causes such as the presence of intermetallic alloy, the crystalline structure of the fault, a change of state, etc.

The internal stress in the layer increased mechanical stress in both dynamic and static in industrial applications. Commonly used solder thickness are much greater than 10  $\mu$ m. The aim of the experiment was to simulate the dynamic loading of two types of unleaded solders deposited on three different surfaces. These combinations are present in electrical common. As the base material used coupons copper, brass and phosphor-bronze. The samples were periodically deflected. This occurred repeatedly increasing stress in the layer of solder.

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#### Scintillation properties of Pr-doped garnets

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During last years extended research of scintillation properties was carried out on fast  $Pr^{3+}$ doped Lu or Y aluminum garnet crystals (or even on epitaxial crystalline layers) [1-3].  $Pr^{3+}$ ion belongs to group of rare earth ions and its neighbour from this group is widely used Ce<sup>3+</sup> scintillating ion. Due to strong crystal field in garnets  $Pr^{3+}$  ion can also exhibit 5d—4f fast allowed transitions similarly as those observed on Ce<sup>3+</sup> ions in crystals. Now, high quality LuAG:Pr crystals were prepared with light yield up to 20000 ph/MeV [4].

The main goal of this talk is to present scintillating properties of  $Pr^{3+}$ - doped both pure LuAG or YAG crystals and those of mixed ones (Lu,Y)AG. A method of gamma spectroscopy of scintillators is used to characterize these scintillating crystals [1]. Experimental set-up using HPMT multiplier was used to measure properties as photoelectron and light yields, energy resolution, proportionality, etc. in the energy range 10 keV - 2 MeV. We will also shortly discuss other possible materials with  $Pr^{3+}$  as thin epitaxial layers prepared by LPE method and first works on  $Pr^{3+}$ -doped ceramics. Finally, we will shortly mention so called multigarnet crystals (Lu,Y,Gd)<sub>3</sub>Al<sub>2</sub>Ga<sub>3</sub>O<sub>12</sub>) [3].

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## Modelling of deformed grains in polycrystals with regular polyhedra

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Local strain in structure can be estimated by measurement of deformation of grains on metallographic cut. In general, the effect of grains boundaries self-orientation caused by grains deformation can be observed. Stereological evaluation of the grain boundary orientation is relatively simple in case if axes of orientation are defined [1]. But the orientation is not the same as deformation and so it is necessary to refer to a correlation between the grain deformation and grain orientation.

It is completely impossible to describe actual shape of the grain in material structure exactly [2]. Therefore deformation of various idealized grain shapes can be investigated. For instance deformation of spheres can be considered since spheres are not space filling and have no edges. Deformation of cubes can be also investigated and mathematical analysis can be simplified, but cubes are clearly poor of approximations to the shapes of real grains unfortunately. In addition, deformation of tetrakaidecahedron can be examined. The shape of tetrakaidecahedron can be regarded as approximately identical to the shape of the grains observed metallographically in undeformed state [3].

We quantified the degree of orientation of single tetrakaidecahedron shaped grain in two different deformation directions (perpendicular to the surface of a square face of tetrakaidecahedron and perpendicular to edge between hexagon faces of tetrakaidecahedron). Using assumption of constant grain volume during plastic deformation we have found the correlation between orientation parameter and deformation characteristic in cases of both deformations. The results are independent on initial dimensions of tetrakaidecahedron. Orientation of grain surface depends only on its deformation and vice versa. Therefore, if we are able to measure and calculate orientation parameter then the deformation can be determined by means of derived equations. Estimation of grain boundary orientation degree is relatively simple (stereology metallography method of oriented test line acording Saltykov). Consequential conversion of its values to strain leads to determination of deformations in three main axes in arbitrary places of plastically deformed polycrystalline body below recrystallization temperature.

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#### Application of thermoanalytical methods to the study of glasses

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This paper presents the application of Differential scanning calorimetry (DSC) and Thermodilatometry (TD) to understanding of the thermal behaviour of various phosphate and borophosphate glasses. From DSC curves the values of the glass transition temperature,  $T_g$ , and the values of crystallization temperature,  $T_c$ , were evaluated. From TD curves, the coefficient of thermal expansion,  $\alpha$ , the glass transition temperature,  $T_g$ , and the dilatometric softening temperature,  $T_d$ , was determined.

Beside basic thermal parameters, the thermal stability towards crystallization, the influence of glass composition on the nucleation mechanism, nucleation rate and critical cooling rate of the melt for glass formation was also studied. For the evaluation of thermal stability of glasses,  $\Delta T=T_c-T_g$ , criterion and the Hruby criterion [1] were applied.

The mechanism of crystallization (surface or internal) was evaluated from the changes in the shape and position of the crystallization peak on DSC curves in dependence on the particle size of the studied glasses [2]. The results obtained by the method of Ray et al. [2] indicated that the most of studied phosphate and borophosphate glasses nucleate predominantly by the surface crystallization mechanism.

The nucleation rate was studied by the method [3] consists of heating the glass at different nucleation temperatures and different nucleation times in DSC equipment, before being crystallized at a fixed heating rate. The study of nucleation rate led to the conclusions, that a lot of phosphate and borophosphate glasses are saturated with stable nuclei at the temperature closely above the glass transition temperature within a relatively short time.

The critical cooling rate for glass formation was estimated by the method proposed by Ray et al. [4] through analysis of crystallization peaks obtained by heating solidified melts produced at different cooling rates. The obtained results showed that this method is applicable only for the glasses with higher contribution of the volume crystallization mechanism.

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## Phase diagram of Zn-Mn-O system for diluted magnetic semiconductor study

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Diluted magnetic semiconductors (DMS) have recently gained a lot of attention due to their practical application in spintronics as non-volatile memories or spin-FETs. ZnO doped with transition metal (TM) is one of the most promising candidates in this area. Because it is a very wide band-gap semiconductor (3.36 eV), relatively non-expensive and eco-friendly material. Due to the limited solubility of TM in ZnO matrix the increasing content of dopants can be accompanied by the formation of secondary phases.

We have studied the high-temperature phase equilibria of Zn-Mn-O system at 1275 K. A series of five samples with Mn/(Mn+Zn) = 0.2, 0.4, 0.5, 0.6 and 0.8 was prepared by a standard ceramic route including two calcination steps for 24 h at 1075 K and one sintering step for 48 h at 1275 K in air. For the phase stabilization at the temperature of interest the samples were quenched in liquid nitrogen. The resulting phase composition of the prepared samples was identified by X-ray diffractometry using Cu-K $\alpha$  radiation. The obtained diffraction data were analyzed by Rietveld method in HighScore Plus program.

Two phases, the zinc oxide and spinel, were found in the samples with the manganese content Mn/(Zn+Mn) 0.2 - 0.5. According to the diffraction data the manganese content in ZnO was estimated to 5 %. We identified the  $(Zn_{0.95}Mn_{0.05})O$  and tetragonal spinel  $Zn_{1.3}Mn_{1.7}O_4$  only in respective phase ratio. In the sample with Mn/(Zn+Mn) = 0.6 we found two tetragonal spinels as observed elsewhere [1] from the same space group symmetry  $I4_1/amd$ , but having different stoichiometry. The last sample of the series was a single phase spinel with composition  $Zn_{0.6}Mn_{2.4}O_4$ , which is in agreement with the global manganese content of 0.8.

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#### Preparation and characterization of Er doped potassium ytterbium lanthanum phosphate glasses

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In recent years considerable research effort has been focused on the preparation and properties of alkali rare earth phosphate glasses due to their possible application as materials for solid state lasers and scintillators. Attention has been paid to both meta-phosphate derived of HPO<sub>3</sub> and those based on  $H_3PO_4$ . While meta-phosphates vitrifies very easy, alone orthophosphates do not form glasses. Ortho-phosphates are able to form glassy mixture with alkali meta-phosphate.

The tested glassy samples were prepared by rapid quenching of molten mixture of starting  $K_2CO_3$ , YbPO<sub>4</sub>, LaPO<sub>4</sub>, ErPO<sub>4</sub> and P<sub>2</sub>O<sub>5</sub>. The glassy samples were prepared in the form of ingots 10x10x25 mm and discs about 10 mm in diameter and 6 mm in height.

In this contribution we present information about preparation and properties of five Er doped potassium ytterbium lanthanum phosphate glass samples. Their cations molar concentration was as follows n(K) = 0.7, n(Yb) = 0.135, n(La) = 0.16 and n(Er) = 0.005 and it was the same in all tested samples. The additions of P<sub>2</sub>O<sub>5</sub> to the individual starting charges were batched so the following compositions of resulting glasses should be obtained: (i) pure metaphosphate, (ii) mixture of 80 mol% of meta-phosphate and 20 mol% of ortho-phosphate, (iii) mixture of 50 mol% of meta-phosphate and 50 mol% of ortho-phosphate, (iv) mixture of 25 mol% of meta-phosphate and 75 mol% of ortho-phosphate, and (v) pure ortho-phosphate.

The following spectroscopic properties of prepared glassy samples, significant for future laser operation, were investigated: absorption spectra, emission spectra, and fluorescence decay time. The absorption spectra were measured in broad range from 200 up to 2500 nm to identify possible impurities, mainly the residual OH-absorption. The detail absorption spectra in vicinity of wavelength 0.98  $\mu$ m (laser pumping region) and 1.54  $\mu$ m (laser emission region) were also measured to calculate absorption cross-section for pumping and laser transition <sup>4</sup>I<sub>15/2</sub>  $\rightarrow$ <sup>4</sup>I<sub>11/2</sub> and <sup>4</sup>I<sub>13/2</sub>  $\rightarrow$ <sup>4</sup>I<sub>15/2</sub>, respectively. For particular transitions fluorescence spectra and fluorescence decay time were recorded simultaneously. For Er<sup>3+</sup> fluorescence excitation pulsed laser diode, operating at wavelength 975 nm, was used. It was found that the fluorescence decay time, corresponding to upper laser level <sup>4</sup>I<sub>13/2</sub> depopulation, progressively increases with the content of ortho-phosphate in glass composition starting from 2 ms for sample (i) up to 8 ms for sample (v). Obtained results are promising for future laser operation of prepared materials.

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## Optical and luminescent properties of Ce<sup>3+</sup> doped GdLuAG and GdYAG garnet scintillators

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Yttrium and lutetium aluminium garnets doped by trivalent rare earth ions  $Y_3Al_5O_{12}$ :RE and Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:RE are prospective scintillators used for detection of high energy particles, gamma and X-ray radiation in the bulk and thin films. We focused here on the study of Ce<sup>3+</sup> doped garnets admixed by Gd<sup>3+</sup> and Ga<sup>3+</sup> ions. The single crystalline (GdYLu)<sub>3</sub>(AlGa)<sub>5</sub>O<sub>12</sub>: Ce films were grown by liquid phase epitaxy from the BaO-B<sub>2</sub>O<sub>3</sub>-BaF<sub>2</sub> flux at the temperatures 1000 - 1070 °C. The structure and chemical composition was measured by the X-ray diffraction and electron probe microanalysis techniques, respectively. We studied in detail the photoluminescence (PL) and radioluminescence properties, kinetics of PL decay, and the photoelectron yield excited by the alpha particles.

This scintillators show allowed 5d - 4f transitions in  $Ce^{3+}$  centers with intense emission in the green - yellow spectral range and fast decay time 50-60 ns. Forbidden 4f-4f transitions are also observed at 310 nm originates from  $Gd^{3+}$  ions. [1] Energy transfer from  $Gd^{3+}$ sensitizer to  $Ce^{3+}$  was observed and studied for different concentrations of both ions. Detailed discussion of emission and scintillation properties of co-doped epitaxial garnet films and comparison with bulk single crystals will be presented.

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#### Single-molecule magnets progress

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Research for new single-molecule magnets (SMMs) is of immense interest. SMM behavior is due to a combination of large ground state spin (S) and a negative (easy axis) magnetoanisotropy as ganded by a negative zero-field splitting parameter, D. The inorganic core of SMM cluster is constructed by several metal ions at various oxidation states (or oxidation state combinations) usually bridged by oxides. These metal oxide cores are enveloped is an organic shell, which can be varied at will using soft chemistry methods. Thus, the cores of SMMs can be considered as a small piece of a metal oxide network, protected by organic groups in the periphery. Several examples of SMMs have been reported, which contain manganese ( $Mn_{12}$  and  $Mn_4$ )and iron (Fe<sub>8</sub> and Fe<sub>4</sub>)ions[1].

The first identified single-molecule magnet was a manganese oxide cluster with acetate ligands  $Mn_{12}O_{12}(O_2CCH_3)_{16}(H_2O)_4$ , often abbreviated as  $Mn_{12}Ac$  [2]. It has eight Mn(III) and four Mn(IV) metal centers in a  $Mn_{12}O_{12}$  core that is surrounded by 16 acetate groups. The molecule has 20 unparied electrons. The latter family has included  $Mn_{12}$  complexes with bulky carboxylates, different terminal ligation as well as different electronic characteristics. The family of  $Mn_{12}$  SMMs still remains the most well-studied series, even though other SMM-types have surfaced over the years [3]. The largest SMM cluster made so far has  $Mn_{84}O_{72}$  core that was built up by the reaction of  $Mn_{12}$  acetate with a permanganate salt [4]. Cyanide-bridged complexes, such as  $Mn_4Re_4$  cluster with tripodal phosphorus ligands, provide different influences on the magnetic behavior of SMMs than traditional oxide-bridged compounds. Continuing the exploration of the lower part of the periodic table, a few researchers are beginning to make mixed transition-metal and lanthanide SMMs [5]. New rising stars in molecule-based magnetic materials are the framework-structured weak ferromagnets. The framework structures are powerful carries for long-range ordering of spins.

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## Strategy for Obtaining Quality Applicants to College Engineering Programmes

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The number of college applicants continued to grow during the last decade, as did the number of accepted applicants, continuing the trend that started after 1989.

Tertiary education applicants are relatively successful in the admissions procedure. The majority of applicants, in terms of the admissions success rate, is primarily accepted into engineering study programmes. The reason for this being that the majority of colleges accept applicants for engineering study programme without them having to go through the admissions procedure. Today, engineering fields of study are perceived as programmes that are relatively easy to get into, thus becoming a "sure bet" for some applicants with these applicants often being accepted as a temporary solution.[1]

The strategy for obtaining quality high school students in particular rests on the nature of the graduates from individual high school majors and their transition to tertiary education. The primary idea behind the proposed strategy, realized at the Faculty of Electrical Engineering - CTU, is to direct planned activities, that lead to obtaining new quality students for engineering colleges, on high school students in the following order:

- 1. lyceums (technical) and gymnasiums,
- 2. secondary technical schools,
- 3. secondary apprentice schools,
- 4. post-graduate studies.

Activities that will lead to obtaining new quality students for engineering colleges: innovating and updating the web page for Applicants, holding talks with selected high schools for the purpose of recruiting potentially quality applicants, continuing with established events for high school students and organizing new events, organizing various events for accepted applicants and introducing student events organized by the college to accepted students.

Each activity performed within the scope of realizing the strategy for obtaining quality students can be divided into four basic time periods:

- a) period before submitting college applications,
- b) period before admission tests,
- c) period before enrolment,
- d) period before the start of the academic year.

With respect to the short-term success rate, we can declare that based on the proposed strategy, students accepted to FEE-CTU bachelor study programmes for academic year 2012/13 demonstrate better study results during their first year of studies (indicated by their GPA), than students enrolled in academic year 2011/12.

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#### The growth of whiskers on thick layers of lead-free solders

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This research focuses on the possible emergence of tin crystalline formations on thick layers of lead-free solders. For this test were selected two lead-free solders, which are commonly used in the electronics industry, 99Sn1Cu and 96Sn3,8Ag0,2Cu. The created samples were exposed to any mechanical static stress and suitable climatic conditions under the defined climatic conditions. The aim was to verify whether under the conditions that occur in many electrical devices, there is the growth of whiskers.

By the introduction of lead-free solders into the production and dissemination in electrical production was not only the necessary changes in soldering technology, but also arose problem of the growth of thin and long crystalline formations on the surface of the solder. Until then, they were known mainly from tin surface finish. At the lead solders presence of lead considerably reduces the effect of crystal growth, but at the lead-free alloys are used ingredients that often do not have this ability and conversely some growth even support. The growth of whiskers is described primarily created for thinner layers deposited solder, usually within 10 micron, where their presence is very abundant. However, there are significant thicker layers.

Whisker is crystalline tin formation (this phenomenon is not only the domain of tin, but also they significantly show on the layers of zinc, cadmium and some other metals) growing from the surface layer of tin. The characteristics of these formations are very diverse. On the formation of whiskers has affect many factors and therefore determine their incubation period is almost impossible. On the grown whiskers are often monitored these parameters: the shape, the incubation time, the growth rate, the density growth of whiskers, the length, the average.

The whisker has due to its structure very good conductivity. If the whisker grow to sufficient length and if its length exceeds the insulation distance between two conductive pathways, may cause long-term short. or it may creating short-term electric short circuit, which is hardly traceable. Or it may cause evaporation of whisker and thereafter rise to highly conductive plasma, which can cross streams to hundreds of amperes.

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### Photoluminescence of Erbium Doped K<sub>1-x</sub>Li<sub>x</sub>TaO<sub>3</sub>:Er Crystals

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Perovskite-type ABO<sub>3</sub> ferroelectrics and related materials are widely applied in various devices due to their exceptional properties that can be tailored by adding suitable impurities. Favorite model material for study of impurity induced phenomena in these highly polarizable materials is potassium tantalate (KTaO<sub>3</sub>). Crystals of this incipient ferroelectric possess a cubic inversion symmetric structure down to the lowest temperatures but the frequency of TO<sub>1</sub> soft phonon mode strongly decreases on cooling and the crystal structure tends to impurity induced ferroelectric phase transitions. Thus phase transition in K<sub>1-x</sub>Li<sub>x</sub>TaO<sub>3</sub> crystals to low-temperature polar phase that changes its character from dipole glass to tetragonal ferroelectric phase with increasing Li content in the vicinity of x = 0.022 is well-known for x > 0.01 [1]. On the other hand, we recently revealed in Er-doped KTaO<sub>3</sub> crystals pronounced stiffening of the TO<sub>1</sub> phonon mode and the suppression of the low-temperature instability towards ferroelectric phase transition [2]. Therefore investigation of Er admixture effect on properties of K<sub>1-x</sub>Li<sub>x</sub>TaO<sub>3</sub> crystals seems to be very interesting and present Er<sup>3+</sup> impurity ions are at the same time a sensitive luminescence probe of crystal structure behavior.

We performed a study of photoluminescence emission spectra of  $K_{1-x}Li_xTaO_3$ :Er (~0.1%) crystals with Li content up to  $x \approx 0.1$  within the 350 - 860 nm spectral region in dependence on temperature between 4.2 and 300 K. A detailed analysis of the structure of zerophonon lines within the emission bands corresponding to  ${}^4S_{3/2} \rightarrow {}^4I_{15/2}$ ,  ${}^4F_{9/2} \rightarrow {}^4I_{15/2}$ , and  ${}^4S_{3/2} \rightarrow {}^4I_{13/2}$  transitions of  $Er^{3+}$  (4f<sup>11</sup>) ions proved that the "major" and less concentrated "minor"  $Er^{3+}$  centers previously found in KTaO\_3:Er crystals and connected with  $Er^{3+}$  ions substituting K<sup>+</sup> and Ta<sup>5+</sup> ions [2], respectively, are present in  $K_{1-x}Li_xTaO_3$ :Er crystals as well. Moreover, crystal field splitting of the ground state  ${}^4I_{15/2}$  and of the first excited state  ${}^4I_{13/2}$  for the "major"  $Er^{3+}$  centre with non-cubic symmetry at 4.2 K was determined in dependence on Li content in  $K_{1-x}Li_xTaO_3$ :Er crystal. Noticeable feature of  $Er^{3+}$  photoluminescence in  $K_{1-x}Li_xTaO_3$ :Er crystal field splitting of K<sup>+</sup> ions by Li<sup>+</sup> ions. Observed temperature behavior of zero-phonon lines in the emission spectra of  $Er^{3+}$  centers photoluminescence is discussed with respect to phase transitions in the  $K_{1-x}Li_xTaO_3$ :Er crystals. Attention is also paid to the effect of Li doping on relative content of "major" and "minor"  $Er^{3+}$  centers in the crystals.

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#### The comparison of the optical spectra of carbon coatings prepared by magnetron sputtering and microwave plasma enhanced chemical vapor deposition and measured by the absolute photothermal deflections

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Our absolute photothermal deflection spectroscopy (PDS) setup allows to measure simultaneously the absolute values of the optical transmittance T, reflectance R and absorptance A spectra of thin coatings deposited on glass substrates in the spectral range 250 – 2200 nm with the typical spectral resolution 5 nm in the ultraviolet, 10 nm in visible and 20 nm in the near infrared region. The PDS setup provides the dynamic detection range in the optical absorptance up to 4 orders of magnitude. Here we demonstrate the usability of this setup by comparing the optical absorbance on a series of the diamond-like carbon (DLC) and nanocrystalline diamond (NCD) thin layers deposited on glass substrates using the magnetron sputtering and the microwave based surface wave-discharge in linear antenna chemical vapor deposition (CVD) processes.

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#### Study on crystallization kinetics of the molten glassy Er:LiGd(PO<sub>3</sub>)<sub>4</sub>

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The transformation of supercooled melt to crystalline phase is characterized by the rate equation of transformation  $f(\alpha) = d\alpha/dt$ , where  $\alpha$  is the volume fraction crystallized and t is the time. Further parameters describing crystallization process are the activation energy for crystallization E<sub>C</sub>, the pre-exponential factor A, the Avrami kinetic exponent n and the overall reaction rate constant k.

This contribution deals with analysis of crystallization kinetics of  $Er^{3+}$  doped lithiumgadolinium meta-phosphate glass,  $Er:LiGd(PO_3)_4$ , especially with determination of the Avrami kinetic exponent n. Exponent n can reach the values of 4, 3, 2 and 1 and depends on nucleation procedure, crystal growth mechanism and morphology. For transformation starting with nucleation on the surface and the following one-dimensional (1D) growth from the surface to the inside n is equal to 2. For volume nucleation and 1D growth n = 1, for volume nucleation and 2D growth n = 3, while for volume nucleation and 3 D growth n = 4. If the n value is not integer, the crystallization proceeds by more than one mechanism [1].

The glass was prepared by rapid quenching of molten mixture of starting Li<sub>2</sub>CO<sub>3</sub>, GdPO<sub>4</sub> and P<sub>2</sub>O<sub>5</sub> in stoichiometric relations [2]. The experiments were performed on powdered samples of particles size smaller than 96  $\mu$ m and between 96 - 106  $\mu$ m. Non-isothermal DSC data were scanned at eight heating rates  $\beta$  between 2 and 48 K/min.

The Avrami kinetic exponent n was determined by methods as follows:

(1) Of the dependence of the double logarithms of the left part of the JMA and Matusita equations  $\ln[-\ln(1-\alpha)]$  vs.  $\ln\beta$ . The kinetic exponent n is equal to the slope of the plot.

(2) According to the relation  $n = 1.26 \text{ S}^{0.5}$  [3] calculated values of the kinetic exponent n are in the range between 1.0 and 1.5, regardless of crystallization temperature and particles size. (Symbol S denotes the shape factor defined as the absolute ratio of the slopes of the tangents to the derivative thermoanalytical peak at the inflection points on the rising and falling regions of the peak.)

(3) Using the relation  $\alpha_M = \exp(n^{-1} - 1)$ , where  $\alpha_M$  is the maximum of the  $y(\alpha)$  function given by  $y(\alpha) = \Phi \exp(E_C/RT)$  [4]. The kinetic exponent n was calculated and for individual heating rates and particle sizes its values are in the interval from 1.2 to 1.3.

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#### Viscosity and thermal behavior of zinc phosphate glasses

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Phosphate glasses have low melting and softening temperatures in comparison with silicate and borate glasses [1]. These low-melting glasses are receiving significant attention since they have great potential for use in optoelectronic devices or in sealing glass applicatons. Viscosity and surface tension are basic physical properties of molten glasses in the high temperature region.

The glasses of the binary ZnO-P<sub>2</sub>O<sub>5</sub> system with 33.3–65 mol% of P<sub>2</sub>O<sub>5</sub> were prepared and studied. The glasses were prepared from reagent grade ZnO and  $(NH_4)_2HPO_4$  in batches of 7 g. In the first stage, the reaction mixture was heated at the rate of 1 °C/minute in Pt-Au crucible up to 600 °C. After the calcination the reaction mixture was melted for 1.5 h at temperatures between 900–1150 °C and poured into a cylindrical graphite mould. Obtained glasses were annealed for 10 h at a temperature close to their T<sub>g</sub>.

The viscosity behavior of zinc phosphate glasses have been studied using parallel-plate viscometer Physica MCR 101 and the measurement were carried out in the softening range of the measured glasses. The continuous changes in the sample height with increasing temperature were recorded. Viscosity was calculated assuming no slip condition to the contacting substrates, as established by Varshneya [2]. Thermal behavior of the prepared glasses was studied by DTA, DSC and HMTA. Their structure was studied by <sup>31</sup>P MAS NMR spectroscopy.

According to the DTA results all glasses crystallized on heating at the rate of 10  $^{\circ}$ C/min. Zinc metaphosphate was characterized by the highest glass transition temperature (449  $^{\circ}$ C). On the other hand the sample with the highest P<sub>2</sub>O<sub>5</sub> content (66 mol%) revealed the lowest value of T<sub>g</sub> (241  $^{\circ}$ C). From the HMTA curves the sintering temperature, deformation temperature, sphere temperature, hemisphere temperature and flow temperature were determined. <sup>31</sup>P MAS NMR spectra were clearly resolved and their decomposition gave quantitative data on the gradual transformation of Q<sup>3</sup> units to Q<sup>0</sup> structural units. By fitting the NMR spectra characteristic parameters of the decomposed spectra were obtained.

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#### Innovation of general and inorganic chemistry education in ICT Prague - Operational Programme Prague - Adaptability

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General and inorganic chemistry belongs to the fundamental subjects in the bachelor study programme in the ICT Prague. Every year, more than 800 students have to complete basic chemistry courses (e.g. General and Inorganic Chemistry, Basic Chemical Calculations, Inorganic Chemistry Laboratories) that are essential for the following study of other fundamental chemistry disciplines, as well as, for the study in special fields of chemistry.

In order to make the courses more attractive to students, to incorporate modern teaching tools into courses and to create new study materials, the Department of Inorganic Chemistry applied for, and subsequently achieved, a subvention from the Operational Programme Prague - Adaptability funding by the European Social Fund in 2012. The project is carried out by the academic staff, as well as, by PhD students of our department.

The main aim of the project is the complex modernisation of inorganic chemistry education and the improvement of graduates quality to meet the demands of a labour market. Therefore, the curriculum of the General and Inorganic Chemistry course has been changed so that general principles will be more implemented to inorganic chemistry systematics. This effort will be enhanced by the other activity of the project - the translation of a modern textbook: C.E. Housecroft, A.G. Sharpe: Inorganic Chemistry (4th edition, Pearson, 2012). Moreover, a study guide connecting the book with the course will be written and published.

New database of possible examination questions (counting thousands of items) has been formed. This database will be used either as a source for a test generator or for the work with students in seminars. To enhance success in the exams, the collection of solved problems and on-line list of useful web links will be created for the students. Teachers will dispose of new applications for the work with the SmartBoard in seminars.

This presentation will also introduce other activities of our OPPA project such as the programme changes of laboratories and a new concept in Basic Chemical Calculations. Many of them are useful in the study of materials chemistry that could be interesting for DMSRE attendees.

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#### Preparation and transport properties of layered semiconductor -Bi<sub>2</sub>O<sub>2</sub>Se

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Bi<sub>2</sub>Se<sub>3</sub>, which adopts the tetradymite structure, is a component of n-type legs of thermoelectric (TE) couples used in solid state coolers or generators having the dimensionless figure of merit ZT i 1 in the range around room temperature. Recently, it has been shown that some of these materials are bulk topological insulators (TI). Bi<sub>2</sub>Se<sub>3</sub> is degenerate semiconductor due to a high concentration of native defects that produce large concentration of free carriers. Such a high concentration of free carriers affects the exploration and potential usage of this material as topological insulator. In search for Bi<sub>2</sub>Se<sub>3</sub> related materials we found a mention of compound Bi<sub>2</sub>O<sub>2</sub>Se. Though tetragonal, Bi<sub>2</sub>O<sub>2</sub>Se, like Bi<sub>2</sub>Se<sub>3</sub>, has a layered structure composed of heavy elements. Such a relationship between Bi<sub>2</sub>O<sub>2</sub>Se and Bi<sub>2</sub>Se<sub>3</sub> suggests that Bi<sub>2</sub>O<sub>2</sub>Se might have interesting physical properties regarding TE and TI. Bismuth oxide-selenide Bi<sub>2</sub>O<sub>2</sub>Se in polycrystalline and single crystalline forms was studied. An optimal way of preparation of homogeneous materials was the first step of research. The prepared materials were then characterized by measurement of the lattice parameters (X-ray diffraction) and by measurement of transport and optical properties.

Primary characterization of polycrystalline material was based on measurement of transport properties in the temperature range 5-800 K. The aim here was to verify the possibility of its potential in TE applications. The results of measurement of the Seebeck coefficient S, electrical conductivity  $\sigma$  and thermal conductivity  $\kappa$  of polycrystalline Bi<sub>2</sub>O<sub>2</sub>Se in the range of temperatures from 5 to 300 K revealed that the compound Bi<sub>2</sub>O<sub>2</sub>Se is n-type partially degenerate semiconductor like Bi<sub>2</sub>Se<sub>3</sub>. Based on these measurements, dimensionless figure of merit ZT was calculated. However its value is too small for applications in this temperature range 5-300 K. Samples for high temperature characterization were prepared by hot-pressing at a pressure of 50 MPa. Three different temperatures were used for hot-pressing in order to examine the influence of the temperature on the properties of the prepared samples. Dimensionless figure of merit ZT calculated from the measured data reaches a maximum value of 0.2 at 800 K. Although the value is five times lower it increases further with temperature.

 $Bi_2O_2Se$  single crystals were characterized by measurement of in-plane electrical conductivity  $\sigma$ , the Hall coefficient  $R_H$ , and the Seebeck coefficient S carried out between 80 K and 470 K. A detailed study of transport properties has been done with a focus on concentration, scattering mechanism and the effective mass of free carriers.

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### Spark Plasma Sintering of Dielectric Ceramics ZrSnTiO<sub>4</sub>

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 $(Zr,Sn)TiO_4$  belongs to the system  $A_{1-x}^{4+}B_x^{4+}Ti^{4+}O_4^{2-}$ , in which radii of cations A and B must differ markedly to avoid substitutions in the basic lattice  $A^{4+}Ti^{4+}O_4^{2-}$  and subsequent resulting lattice defects affecting dielectric properties. Combination of Zr and Sn fulfills this requirement. In this work x = 0.2 is used and the material is denoted as ZST. Commercially available product from ZST is Stabilit E37 ( $\varepsilon_r$ = 37). Sintering temperatures of dielectric ZST ceramics are as high as 1350°C. It is difficult to fully densify ZST ceramics without sintering additives by conventional solid-state reaction methods. The relative permittivity was not significantly affected by various additives while the loss factor was strongly dependent on the type and the amount of additives. Our paper describes an attempt to lowering of sintering temperature and significant shortening of sintering time of the ZST ceramics by using of Spark plasma sintering (SPS) technique. The material was produced by reactive sintering from micropowders. As-received tablets were milled into a powder of the size fine enough for SPS processing (size of about 2  $\mu$ m). SPS process was done in "vacuum regime" and with applied pressure 80 MPa, with dwell time on maximum temperature 4 min. Other parameters were varied for individual samples, namely the temperature between 1100°C and 1300°C. Sample A (1100°C) had the lowest relative permittivity, which is stable with varying frequency in the whole range, 80Hz - 1MHz. Its loss factor was also the lowest and relatively stable, with values between 0.1 for low frequency and 0.01 for high frequency. The relative permittivity is a bit below the nominal value of the ZST material. When the sintering temperature rose to 1150°C, both the permittivity and loss factor grew and became frequency dependent, however only in a small extent. When the sample B (1150°C) was annealed to 1000°C for 2 hours in air, permittivity as well as loss factor remained very similar to the non-annealed sample. The loss factor is more frequency-dependent for the annealed sample. Further increase of the sintering temperature to 1300°C led to dramatic increase of the permittivity and loss factor at low frequencies, below 300 kHz, sample C. Up to 1 kHz permittivity of the sample C was higher than 150 and loss factor as high as 0.7. This fact must be due to inherent conductivity. The resistivity of this sample was the lowest. We modified the procedure for manufacturing of sample B by a longer dwell time, 5 min, to obtain sample D. Its resistivity was higher than for sample B. But the permittivity was high and very unstable versus frequency below 40 kHz. The loss factor was below 40 kHz unacceptably high. The temperature for performing by SPS a ZST dielectric with similar characteristics to Stabilit E37 is probably below  $1150^{\circ}$ C and may be even below 1100°C. Above 1100°C, a kind of phase transformation took probably place. Annealing in air does not improve significantly the properties. Longer dwell time worsened the dielectric behavior. Further SPS investigation on ZST including the size of initial powder, different dwell time at temperatures between 1000°C and 1100°C and structural description are under way.

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#### Statistics of heterogeneous nucleation

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The standard approach to the description of nucleation is based on formulation of a master equation, which predicts a change of average number of clusters of different sizes with time. Under common conditions the master equation can be written in a form of Fokker-Planck equation, which governs the time evolution of the distribution function of clusters of different sizes. Thus, the usual approach to the nucleation process operates with average number density of clusters and does not take into account its possible fluctuations. Within the frames of the standard theory the fluctuations are taken into account only for calculation of the transition rates of clusters from one size to another. This approach works well, for example, for homogeneous nucleation in the open system. On the other hand, the homogeneous nucleation in the open system. On the other hand, the homogeneous nucleation in the outper process with a non-linear feedback, because clusters originated earlier consume the building material for clusters originated later. Even if we suppose the fluctuations of a nucleation rate to be symmetric (Gaussian) around the average value at any given instant, we cannot be sure beforehand that the influence of the non-linear feedback will not lead to a shift of the total average number of originated clusters.

In the last decade attention of several researchers has been attracted to this problem [1, 2]. It was shown, that in the case of homogeneous nucleation in the closed system the fluctuations do not significantly change the average number of originated clusters, but lead to a substantial increase of standard deviation [3]. The aim of this paper is to apply a similar method to the description of heterogeneous nucleation. In this case the feed-back in nucleation process is even stronger than in the case of homogeneous nucleation, since originated clusters consume both the building material and available active sites. Consequently, the fluctuations can be important even in the open system, when the concentration of building material is kept constant. A large fluctuation at the beginning of nucleation process can consume a lot of active sites and lead to an abrupt decrease of distribution function of clusters in the future. Another example of a system where fluctuations play an important role is provided by a heterogeneous nucleation on highly curved substrates. In such systems the nucleation process strongly depends on the number density of monomers in the vicinity of the substrate, i.e. on distances comparable with the radius of curvature. If the curvature of substrate is high enough, the important volume becomes very small and contains only a small number of monomers, which leads to relatively higher influence of fluctuations.

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## **Direct wafer bonding for SOI**

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For decades, polished silicon wafer has been the key substrate for the electronic industry. Electronic devices and integrated circuits are manufactured on such substrates using processes of silicon epitaxy, implantation, diffusion, growth of dielectric layers, etc. However, conductive connection of the electronic device to the silicon substrate has several drawbacks such as parasitic capacitance and latchup. These can be reduced by utilization of advanced substrates based on the Silicon on Insulator (SOI) technology.

The SOI wafer consists of a thin single crystal layer of silicon isolated from the supporting silicon wafer by electrically isolating silicon dioxide layer. Manufacturing the SOI wafer starts with pasting two silicon wafers whereas at least one of them contains the oxide layer. This process is called wafer bonding. In case no glue is used, one speaks of direct wafer bonding. One of the bonded wafers is then ground off and polished to create a layer where electronic devices are made.

The quality of the SOI wafer produced by direct wafer bonding relates to the chemical state of the bonded surfaces and the subsequent annealing process, which transforms temporary Van der Waals bonds into permanent covalent bonds. The overall bond strength between the bonded wafers is proportional to the area density of atomic bonds between the surfaces. Transformation of the bonds during post-bonding annealing is accompanied by release of gaseous molecules. These may not diffuse out of the interface and in that case form bubbles (voids) deteriorating the SOI wafer quality. The rule "the more, the better" shall therefore not be applied for the bond strength.

This work deals with estimation of the bond strength on the as-bonded interface by the razor blade test. The results are discussed with respect to the quality of the final SOI wafer. The effect of pre-bonding plasma activation of wafer surface in various gases as well as the effect of hydrophilicity or hydrophobicity of the surfaces is discussed.

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#### Structure and properties of GeO<sub>2</sub> modified borophosphate glasses.

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Lithium borophosphate glasses doped with  $GeO_2$  were prepared and studied in the compositional series  $(100-x)[0.4Li_2O-0.4B_2O_3-0.2P_2O_5]$ -xGeO<sub>2</sub> within the concentration range of x=0-30 mol% GeO<sub>2</sub>. The structure of the glasses was studied by Raman and <sup>11</sup>B MAS NMR spectroscopy. Differential thermal analysis and thermodilatometry have been used to study thermal behaviour of the glasses.

According to the <sup>11</sup>B MAS NMR and Raman spectra, the glass structure consists mostly of BO<sub>4</sub> tetrahedra and planar BO<sub>3</sub> triangles, whereas P<sub>2</sub>O<sub>5</sub> is incorporated in the structural network in the form of tetrahedral PO<sub>4</sub> metaphosphate (Q<sup>2</sup>) and diphosphate (Q<sup>1</sup>) units. The ratio of BO<sub>4</sub>/BO<sub>3</sub> and PO<sub>4</sub> (Q<sup>2</sup>/Q<sup>1</sup>) structural units change insignificantly with increasing GeO<sub>2</sub> content. According to the Raman spectra within the spectral region of 400-600 cm<sup>-1</sup>, GeO<sub>2</sub> is incorporated in the structural network in the form of 3-membered and 6-membered rings of GeO<sub>4</sub> tetrahedra. The ratio of 6-membered/3-membered GeO<sub>4</sub> units increases with increasing GeO<sub>2</sub> content in the glasses.

The incorporation of GeO<sub>2</sub> the glass structure results only in a small changes of their properties. Glass density slightly increases with increasing GeO<sub>2</sub> content, whereas molar volume, dissolution rate and thermal expansion coefficient reveal a minimum for the glass with x = 15 mol% GeO<sub>2</sub>. Glass transition temperature, on the other side, reveals for the glass containing 15 mol% GeO<sub>2</sub> a maximum. DTA studies showed that all of glasses crystallize on heating within the temperature region of 540-560°C, nevertheless most of glasses reveal high thermal stability. Compounds formed by crystallization were Li<sub>3</sub>PO<sub>4</sub> and B<sub>2</sub>O<sub>3</sub>.

This work was supported from the research projects No. 13-00355S (Grant Agency of Czech Republic) and SG330001 (University of Pardubice).

#### Study of activation energy of Er:LiY(PO<sub>3</sub>)<sub>4</sub> glass

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Activation energy for crystallization  $E_C$  is one of the parameters of the kinetic triplet, which characterizes process of crystallization in undercooled melts. Several methods were deduced from non-isothermal measurements for determination of the activation energy for crystallization. The first group of methods contains Kissinger, Augis - Bennet, or Matusita - Sakka method. These methods are based on the change of crystallization peak temperature depending on the heating rate. The activation energy is obtained by plotting  $\ln(\beta/T_p^2)$ ,  $\ln(\beta/T_p)$  or  $\ln(\beta)$  versus inverse peak temperature  $(1/T_p)$ . Integral methods as Kissinger-Akahira-Sunose, Flynn-Wall-Ozawa, Tang, or Starink method are another group of methods for calculation of the activation energy. In this case activation energy is evaluated from the slope of the plot of logarithmic function containing heating rate or heating rate and temperature at the constant conversion degrees  $(T_{\alpha})$  versus inverse temperature  $(1/T_{\alpha})$  [1-3].

This contribution deals with calculation of the activation energy of meta-phosphate glass Er:LiY(PO<sub>3</sub>)<sub>4</sub> using above-mentioned methods. Studied glasses were developed and tested when searching for new scintillating materials [4]. The glassy samples were prepared by a direct synthesis of Li<sub>2</sub>CO<sub>3</sub>, YPO<sub>4</sub>, ErPO<sub>4</sub>, and excess of P<sub>2</sub>O<sub>5</sub> necessary for transformation of starting materials to meta-phosphate at about 1200 °C [4]. Bulk (discs 3.6 mm in diameter and 1.6 mm in height) and powder samples (particle size between 96 - 106  $\mu$ m and 196 - 206  $\mu$ m) of the meta-phosphate LiY(PO<sub>3</sub>)<sub>4</sub> glass were used for DSC measurements. Samples were studied by non-isothermal DSC in alumina crucible at heating rates 2, 8, 10, 18, 25 and 40 °C/min under nitrogen atmosphere.

Activation energies calculated using the first group of methods reached for the Kissinger method  $173\pm1$  kJ/mol, for the Augis - Bennet method  $181\pm1$  kJ/mol, and for the Matusita - Sakka  $190\pm2$  kJ/mol. On the contrary, the values of the activation energy calculated using the methods of the other group were scattered. The values of activation energy calculated from the Flynn-Wall-Ozawa were between 176 and 190 kJ/mol and from others between 168 and 183 kJ/mol.

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ANALYTICAL & ME	ASURING & TESTING	ANAMET Slovakia s.r.o. 1.Mája 794/29, 900 01 Modra	
A) Materiálové analýzy (fyzikálne a chemické metódy)			
Kombinované technik Testovanie žiaruvzdor (RUL, CIC, HMOR).	Termická analýza TG, DTA, DSC, STA, TMA, DMA, DIL. y (TA-MS, TA-FTIR). rných materiálov	Objektívne hodnotenie farebnosti. Prenosné a stolné spektrofotometre, leskomery, receptovanie farieb. Analýza svetla a meranie obrazoviek.	
Quantachrome Instruments metria, He pyknometr	Meranie špec. povrchu a porozity, Hg porozi- ia , sorpcia vodnej pary .	Distribúcia veľkosti a tvaru častíc, Zeta potenciál, molekulová hmotnosť, Morfológia, rotačné viskozimetre a rheometre.	
B) Aplikácia pre farmáciu, potravinárstvo, biochémiu, kozmetiku			
Biotage	Mikrovlnné syntézy, flash chromatografia, otage syntetizátory peptidov		
PARTICLE measuring systems	Čítače častíc vo vzduchu (až 100 l / min) a vo vodách, monitorovanie čistých priestorov podľa ISO 14644-1 a USP 797, Biologické monitorovacie systémy v súlade s požiadavkami ISO 14698-1, cGMP, GAMPR a farmakológie.		
C) Materiálové skúšobníctvo			
a vibračné komory (r vrátane shakeru TIR/ testov. Pochozné ko	Klimatické, termostatické, šokové, vákuové, korózne nožnosť TURN KEY riešení A), ESS a HALT/HASS mory WAZZI E	Komory pre simuláciu poveternostných podmienok (UV a slnečné žiarenie, 100% vlhkosť, korózna atmosféra v soľnej hmle-Salt Spray,atď.)	
Skúšanie požiarnej odolnosti materiálov. Kalorimetre (Bomb&Cone), Spaľovacie komory (horľavosť, bod vzplanutia, toxicita atď.)		Univerzálne hydraulické a elektromechanické skúšobné stroje pre skúšky v ťahu, tlaku a ohybe, Charpyho kladivá, MFI-tavný index, dart drop impact testery, teplota priehybu pri zaťažení HDT, stanovenie odolnosti za tepla podľa Vicata, meranie tuhosti, torzné skúšobné stroje.	
	D) Analyt	ické techniky	
Spaľovacie elementárne analyzátory C/H/N/S/O, Rapid N, TOC/TN autosampler pre kvapaliny a pevné vzorky, Izotopová hmotnostná spektroskopia.			
E) Analýza palív a maziv			
SETA         Analyics         Analýza palív a mazív (bod vzplanutia, destilačná skúška, viskozita, cetanové číslo atď.)           IR stanovenie biozložky v palive, analýza biopalív HPLC.			
www.anamet.cz		email: igor.culak@anamet.sk Tel: +421 905 249 664	





Váš výhradný dodávateľ laboratórnych prístrojov

Laboratórne prístroje a zariadenia Drieňová 34, 821 02 Bratislava 2 a zariadení ThermoFisher Scientific

Atómová absorpčná spektrometria (FAAS, GF-AAS) Indukčne viazaná plazma ICP-OES, ICP-MS Röntgenová fluorescenčná spektrometria XRF UV-VIS, VIS spektrofotometre IR, FTIR, IRa, FT-Ira, NIR Plynová chromatografia Kvapalinová chromatografia Hmotnostná spektrometria Elementárna analýza CHNS/O Spaľovacia analýza CI, S, N, TOX, AOX





Laboratórne informačné systémy LIMS Archívne laboratórne SW Chromatografické SW Viskozimetre, reometre Reologické vlastnosti plastov Plastometre, extrúdre Termostaty, kryostaty, obehové chladiace zariadenia Porozimetre, BET sorpčné systémy He pyknometre Zariadenie pre analýzu distribúcie veľkosti a tvaru častíc IR mikroskopy Zariadenia pre termickú analýzu (TGA, DTA, DSC, TMA ...)









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# Energodisperzné XRF analyzátory

-NEX CG – rozsah od <sub>11</sub>Na po <sub>92</sub>U, polarizované žiarenie, sekundárne terčíky, vynikajúci pomer signál/šum i pri nízkych koncentráciach, nevyžaduje externé chladenie, zabudovaný autosampler, zabudovaný vákuový a He systém

-UltraCarry – patentovaná metodika stanovenia prvkov vo vodách v ppb koncentráciach na vzorkou ovlhčenom filtri

### Vlnovodisperzné XRF analyzátory

-Mini Z séria – malé jednoúčelové zariadenie, na stanovenie S v nafte, Si povlaku na papieri, Zr a Ni v pokovovacej vrstve, zabudovaný autosampler

-**Primini** – rozsah od <sub>9</sub>F po <sub>92</sub>U, malý stolný analyzátor s výborným pomerom cena/úžitkové vlastnosti, nevyžaduje externé chladenie, zabudovaný autosampler

-Simultix 14 – rozsah od <sub>4</sub>Be po <sub>92</sub>U, až 40 fixných kanálov, možnosť kombinácie fixných kanálov a skenovacieho goniometra, zabudovaný autosampler, výkonný softvér

-ZSX Primus – rozsah od <sub>4</sub>Be po <sub>92</sub>U, možnosť kombinácie fixných kanálov a skenovacieho goniometra doplnená o mapovanie , zabudovaný autosampler, výkonný softvér,

## XRF analyzátor s celkovou reflexiou

-Nanohunter – nová technológia založená na meraní reflexie XRF žiarenia posúvajúca aplikáciu u kvapalných vzoriek do ppb oblastí (limity zrovnateľné s ICP, AAS)

## XRD difrakčný analyzátor

 -Rôzne typy zariadení podľa aplikačného zamerania na analýzu práškových materiálov, vnútromolekulových štruktúr, fázových rozhraní, mikropnutí a iných parametrov



Elementárna analýza

Biologická analýza

Materiálová analýza

RTG komponenty

### Elementárna analýza -nedeštruktívna analýza

-stanovenie prvkov od Be po U v pevných, kvapalných vzorkách, práškoch, filmoch - v koncentráciach ppm až 100%









