Book of abstracts of the 1st outdoor seminar of the Department of Optical Materials: **Advances in the new materials engineering and study**

***„We are only as strong as we are united, as weak as we are divided*“**
– J. K. Rowling

Editors: Maksym Buryi and Robert Král

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Foreword

The seminar is dedicated to the main activities of the department of Optical Materials, FZU – Institute of Physics of the Czech Academy of Sciences in the course of national and international collaborations. In particular, the advanced methods of the (nano)materials synthesis, fabrication and characterization will be presented and discussed in detail. The aim of the seminar is to establish more of the effective paths of communication and data exchange by creating and maintaining links among the teams of national and international experts in various fields of physical and chemical knowledge, both experimental and theoretical. Moreover, the connection to the medical, biological, archeological, forensic and historical science is provided. This will in turn create the basis for the synergy of sciences and pave the way for the wider collaboration with the world-wide experts. In this respect, it is appropriate to mention that the expected outcome is new hybrid materials design by applying the set of experimental and theoretical techniques. The most specific ones presented at the seminar are electron paramagnetic resonance, X-ray photoelectron spectroscopy, and density functional theory. Their combination complemented with the other experimental methods is the strongest tool giving feedback to any technology, thus leading to the desired material synthesis and fabrication. The expected practical outcomes finding implementation in industry are various sensors, light emitting devices, scintillators, dating methods, etc. Therefore, the importance of the meeting can hardly be underestimated. We believe that this first outdoor seminar of the department will continue becoming annual stretching out the ideas of national and international collaboration for the improved R&D.

The organizing committee consisting of Dr. Maksym Buryi, Dr. Robert Král and Ing. Alena Beitlerová (further mentioned in the text as “committee”) would like to express deepest gratitude to the chair of the Optical Materials department, Prof. Martin Nikl for financial and moral support. Further, the committee is thankful for the participation of all the attendees including: (i) employees of the department; (ii) Dr. Alice Hospodková, the head of the Department of semiconductors, FZU – Institute of Physics of the Czech Academy of Sciences; (iii) Dr. Irene Villa, the international expert in luminescence and materials for radiotherapy and medicine from the Milano-Bicocca University; (iv) Prof. Michał Piasecki, the head of the department of Theoretical Physics from the Jan Dlugosz University, the international expert in materials engineering and theoretical physics; (v) MSc. Tomáš Hostinský, the expert on glasses synthesis from the University of Pardubice; (vi) Dr. Kristýna Králová from the Faculty of Arts, Charles University in Prague. The committee also acknowledges the help of Dr. Zdeněk Kožíšek from the FZU – Institute of Physics of the Czech Republic with the seminar preparation.

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Luminescent and scintillating nanomaterials for imaging, diagnostic, and therapy

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Luminescent and scintillating nanomaterials offers the great advantage of the tunability of their physico-chemical properties and of their emission features through the control of their structure, morphology, and doping. The scientific community is continuously drawing up novel surface engineering/doping and materials design approaches to obtain the best nanomaterial to satisfy the more and more demanding requirements of most advanced applications in photonics, in detection of ionizing radiation, and in theranostics. In this context, I propose an excursus on the results obtained from diverse luminescent and scintillating nanomaterials. Starting from rare earth ions doped and undoped inorganic scintillating nanoparticles (HfO_2) [1] to the development of brand-new hybrid and composite nanoscintillators [2], I will show a systematic approach to adapt the nanomaterials qualities according to the targeted application. Especially, I will present the exploitation of luminescent and scintillating nanomaterials with ad-hoc features for in vitro/in vivo imaging and for radiotherapeutic protocols [3]. Lastly, I will show how the use of hybrid and composite nanoscintillators with fast timing qualities represents a potential breakthrough in medical diagnostics for the requirements for early-stage cancer diagnostic [4-5].

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How to effectively improve the desired properties of materials?

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Is well known that the chemical composition, structure, temperature, stress or strain caused by external pressure or temperature changes influence on structural, optical, magnetic or super-conducting properties of solids. Band gap engineering through the modification of the composition is an effective method to adjust electronic and optical properties of such a mixed compound to meet special requirements for its successful operation in a particular spectral range. Superlattices, thin films as well as multilayer structures have attracted great attention because they provide new opportunities in device manufacture by new material develop. The properties of low dimension structures differ dramatically from their bulk materials, such as the polarization, dielectric constant and tunability etc. Ultra-thin films are subject of current interest due to possible probing of deep inter-band transitions. Miniaturization and nano scaling will lead to effects that modify the electronic behavior; the latter still requires improved fundamental understanding in the reduced dimensions. The stress or strain from temperature changes of lattice mismatch between thin films and substrate gives an extraordinary opportunity for materials engineering. Thanks to these phenomena, constructed (transformed) materials, demonstrating totally different properties, than are desired in nature. Recently has been spectacularly demonstrated that lattice mismatch between thin films and/or substrate gives an extraordinary opportunity for materials engineering. Rapid advances in reliable computational DFT-based methods have paved a broad way towards increasing importance of so-called "theoretical experiments", when thoroughly performed calculations replace or forego experiments and even predict unknown materials and their properties. In present talk we also discuss the opportunities (and disadvantages) of quantum chemical calculations towards search for new efficient materials.

Nanomaterials synthesis and characterization. Synergy of different types of (nano)materials

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Downscaling to nanomaterials (wires (1D), ultrathin films (2D) and quantum dots (0D)) results in the rise of the radiation detector efficiency. The practical examples are zinc oxide (ZnO), gallium nitride-based structures like InGaN/GaN multiple quantum wells (MQW) and complex lead bromides, $XPbBr_3$ (X = Cs (or other alkali metal), MA (methylammonium) or FA (formamidinion) etc.), XPB. ZnO has excellent physical properties and its production at the nanoscale, e.g., in the form of nanorods using hydrothermal growth method is cheap. Moreover, it can be combined with other inorganic and organic (nano)materials in the form of a composite. As the complementary part, the GaN-based structures or XPB can be used. It is noteworthy, that GaN has structural and scintillating properties (especially, timing) very similar to those of the ZnO. There are ZnO/GaN composites where ZnO is grown in the form of the vertically aligned nanorods on the GaN substrate. The opposite orientation of the composite growth is also known where GaN nanorods are grown on the ZnO substrate. As another part of the composite, XPB grown as larger nanoparticles or quantum dots can be used. Note, that XPB materials, in general, have prominent timing characteristics, large light and quantum yields. In combination with the piezoelectric properties discovered in all the mentioned materials, the charge transfer occurs due to the field effect, thus supporting e.g., photovoltaic effect, energy transfer, luminescent, and scintillating properties.

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High temperature thermal analyses DTA-TGA-MS: review and installation

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This contribution deals with introduction to thermal analyses and mainly focuses on installation of new high temperature thermal analyzer in our department. The installed basic setup is highly versatile containing three different methods such as differential thermal analysis (DTA)/differential scanning calorimetry (DSC), thermogravimetry (TGA), and mass spectroscopy (MS). All techniques are coupled together allowing to perform experiments independently or simultaneously. In this way one can record changes in sample regarding: (i) heat flow e.g. exo- and endo-thermic effects (e.g. phase transitions, reactions, etc.), (ii) mass change (decomposition, reactions, etc.), and (iii) detection of evolved gases and their fragments. Therefore, in simultaneous regime the setup enables to obtain complex picture about processes occurring in sample under thermal treatment and their easier identification. This basic setup allows measurements under both isothermal and non-isothermal conditions in wide temperature range 25-1300 °C with heating and cooling rates 1-100 K/min and various atmospheres e.g. inert (Ar, N_2, CO_2), oxidative (O_2), or reductive (H_2, CO). The apparatus is equipped with easy-fit system allowing quick exchange of DSC/DTA sensors in matter of minutes to enhance its sensitivity in specific temperature region (up to 1700 °C). On the contrary, the high temperature setup requires much larger modifications exchanging all necessary parts for those that can sustain extreme temperatures (e.g. tungsten, carbon). In this way, it is possible to conduct experiments in 25-2000 °C, heating rates 1-100 K/min, however, with limitations on used atmosphere, only inert (high purity Ar, N_2) and reductive (e.g. H_2, CO) can be used.

Surface changes induced by inductively coupled plasma

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High temperature annealing, and plasma treatment are the universal techniques to process surface. Plasma contains energetic electrons, ions, excited radicals, and molecules that allows surface states adjustment while keeping the sample at a relatively low temperature. Silicon dioxide and zinc oxide micro powder (average size about 1 μm) has been treated in the inductively coupled plasma (ICP). Plasma treated SiO_2 and ZnO micro powder has been characterized by the attenuated total reflectance Fourier transform infrared spectroscopy (ATR FTIR), FTIR Raman spectroscopy, low temperature photoluminescence spectroscopy (PL) and scanning electron microscopy (SEM), The phase sensitive method is applied for time resolved PL measurements. Using 20 kHz excitation, it is possible to measure mean value of the PL decay with 500 ns time resolution. The measured data are discussed, and their interpretation is supported by the literature.

The work was supported by the CAS-SAS Mobility Plus project SAV-23-13.

EPR Dosimetry: unique and sharp tool for radiation emergency and archaeological-forensic applications

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Electron paramagnetic resonance (EPR) allows quantitative and qualitative study of electron traps in materials. Due to its sensitivity, accuracy and non-destructiveness, EPR is considered the gold standard in retrospective dosimetry, i.e. radiation exposures where no man-made detector is present. The same principle can be applied in many branches. This presentation introduces the basic principle and its (dis)advantages, shows cases we are investigating and indicates the direction in which we are planning to improve the method further.

Peculiarities of charge trapping in crystalline and amorphous complex metal oxides

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The properties of optical materials are affected not only by the composition of the material matrix and the optically active dopants but also by defects in the structure. Such defects can reduce optical performance. Applications where it is important not to reduce the performance of optical components by defect losses include laser technology and photonics. In these areas, e.g. europium-doped yttrium aluminium garnet (YAG) and tellurite glasses are used. Doped YAG is an attractive material in areas where luminescence is used. YAG:Eu^{3+} is known for red light emission, but recently efficient and stable photoluminescence of Eu^{2+} has also been observed. Tellurite glasses are characterized by good thermal and mechanical stability and high chemical resistance. A great advantage is the possibility to optimize their properties due to the wide range of glass formation between binary and ternary glasses. By combining these materials, the production of low-cost glass-ceramics with high light yields can potentially be achieved. The aim of this work was to understand the factors affecting the optical performance of these materials. Electron paramagnetic resonance was used in correlation with thermally stimulated luminescence and radioluminescence. Defect formation in YAG:Eu and in glass of TeO_2-BaO-ZnO composition was studied. Charged oxygen vacancies and O-type defects were revealed and characterized.

Niels Steensen: last polymath, his life and research

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Niels Steensens contribution to natural sciences is of a great importance as he was one of the first to suggest a modern approach to the scientific research in general. 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. He 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 and crystallography as well as crystal growth. Steensens observed that crystals do not grow from inside like flowers, but from outside as a result of material deposition on the surface of the existing crystal. Furthermore, Steensen divided crystals into different groups according to parent material and thus disproved the common belief that all crystals were formed from ice [1]. It was also Steensen, who 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]. In 1675 Steensen became priest and theologist and did not continue the scientific research.

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Application of photoelectron spectroscopy for analysis of surfaces and buried interfaces

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In this lecture, we will give an overview of the usability of the AXIS Supra X-ray photoelectron spectrometer for the analysis of various types of materials from the field of energy applications, catalysis, sensors, and microelectronics. We use X-ray photoelectron spectroscopy (XPS) to measure chemical composition, bonding configurations and electronic properties of surfaces, epitaxial thin films, nanostructures, nanoparticles, and heterostructures. We will show how combination of multiple approaches such as angle-resolved XPS, UPS, Ar ion cluster beam sputtering, inelastic ion spectroscopy, and exposure of surfaces by laser offers further information about the topmost surface layers and the deeply buried heterointerfaces. In particular, we will present our recent achievements in the analysis of B-doped nanocrystalline diamonds, Er-doped ZnO nanowires, Au@CeO_x core@shell nanoparticles, electrodes for dual batteries, 2D materials, epitaxial AlN(0001) layers, and GaP(As)/Si(100) heterointerfaces. Limitations of the methods, especially in information depth, doping sensitivity, lateral and in-depth resolution, will be discussed. Finally, we compare the results obtained in the laboratory with the results obtained using soft and hard X-ray photoelectron spectroscopy at synchrotrons and discuss possible expansions of the measurement capabilities of the Kratos AXIS Supra spectrometer.

Growth of selected scintillation crystals by micro-pulling-down method

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Inorganic scintillation single crystals have been utilized in many fields of technology and research, such as high-energy physics, environmental monitoring, geological survey and oil well logging or astronomy. The Medical imaging and especially security scanning are nowadays the applications mostly stimulating the development of new scintillation materials due to increasing demands on their performance. To accelerate the single-crystal material development, fast material composition screening is desirable. The micro-pulling-down method is one of the most suitable for such a task due to its low material consumption and high growth rate which allows growing a crystal within several hours. It has been developed to its present form in the 90's for oxide crystals [1, 2]. Aluminum perovskites and garnets represent important groups of promising scintillation materials [3]. While the former has not been much studied due to difficulties of their crystal growth, the latter have been studied quite deeply due to relatively easier preparation. The crystal growth of selected perovskite and garnet crystals by the micro-pulling-down method will be presented and discussed together with the growth peculiarities and brief overview of their structure and luminescence characteristics.

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Crystal growth of sulfide scintillators by micro-pulling-down method

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Sulfides were one of the first substances used for detection of ionizing radiation. The ZnS:Ag powder was used already at the beginning of 20th century for detection of alpha particles in Geiger-Mardsen experiments which led to the creation of Rutherford's model of atom. After that the use of sulfides as scintillators subsided, mostly due to the difficulty of crystal growth. However, sulfides were commonly used in powder for as phosphors and luminophores. Research of ternary alkali-rare earth sulfides ($ALnS_2$) in powder/microcrystalline for have shown a very promising results for use as scintillators and laser active media. However, bulk single crystals are necessary for such applications. This motivated the development of apparatus for crystal growth of high melting sulfides. In our group we have modified halide micro-pulling-down apparatus for growth of binary and complex sulfides. In this contribution I will show and explain the modifications as well as first grown crystals and preliminary results.

High-frequency EPR spectroscopy

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Electron paramagnetic resonance (EPR) spectroscopy is a very powerful tool for investigation of a variety of materials that contain unpaired electrons. It is thus important in branches of physics, chemistry and biology or medicine. It is sensitive to the static electronic properties of paramagnetic system as well as to ionic or molecular dynamics. EPR spectroscopy is also widely used in study of lattice defects created by light and ionizing irradiation. Commercial EPR spectrometers are typically operated at fixed microwave (m.w.) frequencies of 10 and 34 GHz and a magnetic field of 0 – 1.4 T. However, higher m.w. frequencies and fields (HFEPR) lead to drastic increases of the absolute sensitivity as a result of the larger m.w. quantum energy. Furthermore, the advantages of HFEPR also lie in the delivery of much higher spectral resolution, which allows precise determination of the electronic and geometric structures of lattice defects and local properties of materials. Today, HFEPR spectrometers become widespread in scientific research due to commercial availability of m.w. sources up to THz region and cryomagnets with the field up to 25 T allowing quite simple construction of laboratory setups.

In this our report, we review our results recently obtained in investigation of optically active impurity ions in wide bandgap oxide materials by applying the HFEPR technique at frequencies 100-1000 GHz with the special attention to Gd-contained crystals. The advantages of HFEPR will also be demonstrated in studies of other functional materials, which are perspective as a media for spin qubit hosting manipulated by electric field.

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What is happening in the group of Luminescence and scintillation materials?

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In this presentation, I will give a brief introduction of the research conducted in the group of Luminescence and scintillation materials. Main research branches will be addressed, and novel experimental techniques will be presented and potentially offered for novel collaborations. Namely, time-resolved cathodoluminescence spectroscopy and ultra-fast scintillation decay measurement under the pulsed X-ray excitation will be mentioned.

In the first part, long-lasting research on ternary sulfide single crystals will be introduced. These materials possess several application potentials in fast-timing scintillators [1] (Pr^{3+}-doped $KLuS_2$), in solid-state lasers [2] (Tm^{3+}, Yb^{3+}-doped $KLuS_2$), in solid-state white LED technology [3] (Eu^{2+}-doped $KNaLuS_2$) and potentially as well in persistent phosphors [4] (Ce^{3+}, Sm^{3+} co-doped $KLuS_2$). Novel approaches will be described as ways to synthesize these materials as bigger single crystals (halide micro-pulling down or vertical Bridgman) or as high-quality crystalline layers (plasmatic reactive sputtering deposition technique). Next, another direction includes other fast emitters which could be used in time-of-flight applications, such as InGaN/GaN heterostructures or inorganic perovskites embedded in various matrices. As a last part, novel garnet-based materials are introduced as phosphors giving strong persistent luminescence. These could overcome the efficiency of widely used $SrAl_2O_4:Eu^{2+}$, Dy^{3+}.

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InGaN/GaN QWs on semipolar facets and their impact on PL properties

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InGaN/GaN QW heterostructures can be used as very efficient fast scintillators for particle radiation detection. However, presence of unintentional impurities and defect bands in their luminescence spectra significantly slow down their luminescence response and decay time. Possible way to suppress incorporation of some defects into the structure is growth on structured surfaces with semipolar facets. We will compare photoluminescence of structures with 10xQW prepared on buffers with 5 different morphologies. In the presentation we will describe different ways to prepare self-organized 3D structured surfaces, we will show and discuss the impact of buffer morphology on InGaN/GaN QW luminescence. The discussion will be supported by results of PL, cathodoluminescence, positron annihilation spectroscopy and SIMS. Finally, we will suggest new design of structures promising improved luminescence properties.

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Phosphate and borophosphate glasses modified with transition metal oxides

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In the group of Prof. Koudelka and Mošner, phosphate and borophosphate glasses have been studied at the Department of General and Inorganic Chemistry since 1997. During this time, countless glass systems have been prepared and studied in terms of their basic physicochemical properties, thermal and electrical properties as well as their structure using IR, Raman and NMR spectroscopy methods.

Phosphorus oxide, P_2O_5, is a well-known glass-forming oxide and allows the preparation of glassy materials known as phosphate glasses. Phosphate glasses have properties different from conventional silicate glasses, including a higher coefficient of thermal expansion, lower values of thermal properties, the ability to dissociate other oxides better, and poorer chemical and thermal stability, which are based on differences between P_2O_5 and SiO_2. In order to improve their chemical and thermal stability, but also to enhance other properties (optical, electrical, biocompatibility, solubility, etc.), these glasses are modified with additional oxides. These include mainly transition metal oxides but also oxides of alkali metals and alkaline earths and rare earth oxides. Another method of modification is by adding another glass-forming oxide, such as boron trioxide, B_2O_3, to form borophosphate glasses. In most cases, adding other oxides does not change the properties of the glass linearly. This phenomenon can be described as a kind of synergistic effect defined as the Mixed Glass Former Effect (MGFE).

While production of phosphate and borophosphate glasses are nowhere near the scale of silicate glasses, the vast number of combinations with other oxides make them suitable materials that can be tailor-made for a range of applications. These glasses are commonly used in optoelectronics, as laser glasses, biomaterials, low-temperature sealing glasses, matrices for immobilization of radioactive waste, solid-state electrolytes with high ionic conductivity or precursors for the preparation of glass ceramics.