29. ročník odborného semináře doktorandů
29th annual Doctoral Students‘ Seminar

ANORGANICKÉ NEKOVOVÉ MATERIÁLY

INORGANIC NON-METALLIC MATERIALS

7. – 8. února 2024
February 7-8, 2024

Ústav skla a keramiky, VŠCHT Praha
Department of Glass and Ceramics, UCT Prague

Úvod

Programem semináře jsou odborné přednášky doktorandů různých akademických pracovišť působících v oblasti chemie a technologie anorganických nekovových materiálů.

Organizátor semináře

Ústav skla a keramiky, VŠCHT Praha

Webové stránky

https://sil.vscht.cz/o-ustavu/ANM

Kontakt

Ing. Petra Šimonová, *simonovb@vscht.cz*

Poděkování

Ústav skla a keramiky děkuje všem zaměstnancům VŠCHT Praha a doktorandům, kteří se na přípravě semináře podíleli. Dále děkujeme firmě Glass Service, Silikátové společnosti České republiky z.s. a nadačnímu fondu prof. Rudolfa Bárty za poskytnuté ceny pro soutěžící.

Introduction

The program of the seminar consists of specialized lectures given by PhD students from various academic institutions working in the field of chemistry and technology of inorganic non-metallic materials.

Seminar Organizer

Department of Glass and Ceramics, University of Chemistry and Technology Prague (UCT Prague).

Website

https://sil.vscht.cz/about/INM

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Acknowledgments

The Department of Glass and Ceramics expresses gratitude to all employees of the University of Chemistry and Technology Prague and PhD students who contributed to the preparation of the seminar. Additionally, we would like to thank Glass Service, The Czech Ceramic Society and Prof. Rudolf Bárta's Foundation for providing prizes for the participants.

GLASS SERVICE

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Department of Glass and Ceramics
UCT PRAGUE

Program semináře 2024 / Seminar programme 2024

Středa 7. února 2024 / Wednesday 7th February 2024

Úvodní slovo / Opening remarks (9:30-9:40)

1. Blok / 1st session – 6 příspěvků / 6 lectures (9:40-12:00)

Ing. Tereza Přívracká (9:40-10:00)
Corrosion effect of organic buffers on bioactive glass

Ing. Jiří Jemelka (10:00-10:20)
Solution processed multi-layered thin films of $Ge_{20}Sb_5S_{75}$ and $Ge_{20}Sb_5Se_{75}$ chalcogenide glasses

Naser Hosseini MSc. (10:20-10:40)
Joining of SiC-based Ceramics to Themselves for Extreme Environments Applications

Přestávka / Coffee break (10:40-11:00)

Ing. Antonín Sojka (11:00-11:20)
Difficulties in preparation of truly pure polycrystalline Bi_2O_2Se

Ing. Aiman Albekova (11:20-11:40)
Preparation of luminescent nanofibers from bioactive glasses containing samarium

Ing. Tomáš Hostinský (11:40-12:00)
Structural analysis of lithium borophosphate glasses by 1/2D MAS NMR techniques

Pauza na oběd / Lunch break (12:00-13:00)

2. Blok / 2nd session – 4 příspěvky / 4 lectures (13:00-14:20)

Ing. Karolína Opavová (13:00-13:20)
Bioglass Particle Size Effect on Bioactive Behaviour in Dynamic Test Arrangement

Ing. Petra Šnoblová (13:20-13:40)

Adsorption and catalytic properties of precipitatively prepared cerium dioxide

Mohamed Abdelkader MSc. (13:40-14:00)
Directed Energy Deposition 3D Glass Printing for Realizing Customized Optical Sensors

Ing. Eva Bedrnová (14:00-14:20)
Využití odpadních stavebních kalů v adsorpčních technologiích

Exkurze do univerzitního pivovaru / Excursion to the university brewery (15:00)

Čtvrtek 8. února 2024 / Thursday 8th February 2024

3. Blok / 3st session – 6 příspěvků / 6 lectures (9:40-12:00)

Ing. Klára Pulcová (9:40-10:00)
Shrinkage and expansion of hydraulic binder from Metakaolin, $CaSO_4$ AII and $Ca(OH)_2$

Ing. Petra Šimonová (10:00-10:20)
Microwave sintered SnO_2-ZnO based ceramics

Ing. Jakub Suský (10:20-10:40)
Inter-lanthanide $LaYYbO_3$ perovskites doped with Er^{3+}

Přestávka / Coffee break (10:40-11:00)

Ing. Kateřina Křehlíková (11:00-11:20)
Ternary cesium lithium iodide crystals grown by vertical Bridgman method for scintillation applications

Guido de la Torre MSc. (11:20-11:40)
Heated and plasma treated silicon nitride with different sintering additives for biomedical application

Ing. Marek Rotter (11:40-12:00)
Optimalization of a debinding procedure for objects prepared by 3D printing of the low loaded photopolymeric ceramic suspensions

Pauza na oběd / Lunch break (12:00-13:00)

4. Blok / 4th session – 5 příspěvků / 5 lectures (13:00-14:50)

Naser Hosseini (13:00-13:10)
Young Ceramists Network presentation

Ing. Oleksii Lapenko (13:10-13:30)
Research of the sintering process of no cement refractory concretes with dead burned magnesia filler

Ing. Nikola Klusoňová (13:30-13:50)
Thermal Stability of Valuable Metals in Cathodes at Elevated Temperatures

Ing. Jan Zich (13:50-14:10)
Synthesis traps of polycrystalline Bi_2O_2Se

Ing. Erik Ščasnovič (14:10-14:30)
The activation energy of sintering of $(Mg_{0.2}Co_{0.2}Cu_{0.2}Ni_{0.2}Zn_{0.2})O$ and impact of sintering temperature and heating rates on microstructure and homogeneity.

Ing. Timur Babii (14:30-14:50)
Preparation of Ni-Co-Mn mixed oxide coatings on stainless-steel supports by sol-gel method and their activity in oxidation of volatile organic compounds

Vyhlášení výsledků a ukončení semináře / Winner announcement and end of the seminar (15:00)

Student Speech Contest

The presentation duration will be 15 minutes, followed by 5 minutes discussion from the evaluation committee and the audience. The committee will select the winner of the Student Speech Contest and a representative of The Czech Ceramic Society will select an independent winner who will receive the prize from The Czech Ceramic Society.

Members of the committee:

- prof. Dr. Dipl.-Min. Willi Pabst
- doc. Ing. Kateřina Rubešová, Ph.D.
- Ing. Richard Pokorný, Ph.D.
- Ing. Diana Horkavcová, Ph.D.

Representative of the Czech Ceramic Society:

- doc. Ing. Hana Ovčačíková

The list of participants:

- Ing. Tereza Přívracká
- Ing. Jiří Jemelka
- Naser Hosseini MSc.
- Ing. Antonín Sojka
- Ing. Aiman Albekova
- Ing. Tomáš Hostinský
- Ing. Karolína Opavová
- Ing. Timur Babii
- Mohamed Abdelkader MSc.
- Ing. Klára Pulcová
- Ing. Petra Šimonová
- Ing. Jakub Suský
- Ing. Kateřina Křehlíková
- Guido de la Torre Msc.
- Ing. Marek Rotter
- Ing. Oleksii Lapenko
- Ing. Nikola Klusoňová
- Ing. Jan Zich
- Ing. Erik Ščasnovič
- Ing. Petra Šnoblová

ABSTRAKTY
ABSTRACTS

Corrosion effect of organic buffers on bioactive glass

Tereza Přívracká

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The Simulated Body Fluid (SBF) is used for *in vitro* testing and its preparation is described in the ISO standard 23317:2014. Previous studies have showed that the TRIS buffer, which maintains the pH in SBF, accelerates the dissolution of bioactive glass/glass-ceramics and helps precipitate a hydroxyapatite (HAp) layer.

This work aimed to study the corrosion behavior of the TRIS and HEPES buffers and compare the rates of the bioactive glass dissolution, as well as their effect on the HAp formation.

Bioactive glass samples, in the form of bulk, were tested under the static-dynamic conditions by the solutions: i) SBF without TRIS buffer (**SBF**), ii) **SBF+TRIS** (ISO standard) and iii) **SBF+HEPES**. The concentrations of the elements (Ca, P and Si) in eluates were analysed using ICP-OES. The pH was measured by glassy electrode at the physiological temperature (36.5ºC). Glasses and newly formed layers were observed by SEM/EDS and XRD methods.

This work confirmed that TRIS and HEPES buffers used in SBF produced false positive results of *in vitro* tests. Moreover, the chemical composition, morphology and crystallinity of newly created apatite layer formatted in buffered SBF are different.

Acknowledgement: This work was supported from the grant of Specific university research – grant No A1_FCHT_2023_005.

Solution processed multi-layered thin films of $Ge_{20}Sb_5S_{75}$ and $Ge_{20}Sb_5Se_{75}$ chalcogenide glasses

Jiri Jemelka[1], Karel Palka[1,2], Petr Janicek[2,3], Stanislav Slang[2], Jiri Jancalek[2], Michal Kurka[2], Miroslav Vlcek[1,2]

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Chalcogenide glasses semiconducting materials widely studied due to their desirable optical properties, such as a wide IR transparency window, high refractive index, and high optical nonlinearity.[1] Chalcogenide glasses can be used in the form of bulk glass (lenses, windows), optical fibers, or a thin film an appropriate substrate. There are several ways to prepare the thin films. One option is to use the vacuum deposition techniques such as thermal evaporation, sputtering, or laser ablation.[1] Alternatively, the thin films can be prepared from the solution of chalcogenide glass in amine or amine-based solvent using spin-coating.[1,2] The solution path enables the modification of the solution by adding a new component to modify the properties of the resulting thin film.[2]

The thin films of non-toxic glasses of $Ge_{20}Sb_5S_{75}$ and $Ge_{20}Sb_5Se_{75}$ composition were deposited in specular optical quality from n-propylamine-methanol mixture based glass solution using a spin-coating method. The structure, composition, organic residue content, refractive index, optical band gap, surface roughness, and chemical resistance were studied in dependence on the annealing temperature. A significant increase in refractive index and chemical resistance caused by thermo-induced structural polymerization and release of organic residua were observed. The high chemical resistance of the hard-baked thin films allowed repeated direct depositions by spin-coating, increasing total thickness. Multi-layered thin films of alternating $Ge_{20}Sb_5Se_{75}$ and $Ge_{20}Sb_5S_{75}$ composition were successfully prepared for the first time by direct deposition.[3] Solution-based deposition of non-toxic $Ge_{20}Sb_5Se_{75}$ thin films in specular optical quality significantly widens the applicability of solution-processed chalcogenide glass thin films.

Acknowledgement: The authors appreciate the financial support from grant LM2023037 from the Ministry of Education, Youth and Sports of the Czech Republic.

References

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[2] S. Novak, L. Scarpantonio, J. Novak, M. Dai Pre, A. Martucci, J.D. Musgraves, N.D. McClenaghan, K. Richardson, *Opt. Mater. Express*, **3 (6)**, 729-738 (2013).
[3] J. Jemelka, K. Palka, P. Janicek, S. Slang, J. Jancalek, M. Kurka, M. Vlcek, *Sci. Rep.*, **13**, 16609 (2023).

Joining of SiC-based Ceramics to Themselves for Extreme Environments Applications

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In this work, CVD β-SiC and SiC_f/SiC ceramics were joined to themselves with in-situ formed SiC-ZrC composite interlayer and the mechanical properties of the joints were investigated. The $ZrSi_2$ alloy and $ZrSi_2$-C powders mixture were applied as interlayer raw materials on the joining surfaces of the materials in the form of slurry. Afterward, the pressure-assisted joining was performed via field-assisted sintering technology at different temperatures (1400 – 1650°C). The joints with pure $ZrSi_2$ alloy showed a non-homologous interlayer, consisting of both ZrC and remaining $ZrSi_2$. In addition, a significant reaction with SiC substrate was observed, leading to the dissolution of SiC and infiltration of the filler into the base materials. When $ZrSi_2$-C interlayer was used, the uniform SiC-ZrC composite interlayer was formed by in-situ reactions between $ZrSi_2$, C, and SiC-based ceramics. The strength of the CVD β-SiC joints was investigated using 4-point flexural test, while the mechanical performance of SiC_f/SiC joints was determined using shear strength measurements. The strength of the joints was significantly improved with the increasing joining temperature, and reached the initial strength of the reference, un-joined CVD β-SiC and/or SiC_f/SiC materials.

Acknowledgement: This work was supported by the Slovak Research and Development Agency under the contract no. APVV-SK-CZ-RD-21-0089 and APVV-19-0497. The support of the project VEGA 2/0116/22 is also acknowledged.

Difficulties in preparation of truly pure polycrystalline Bi_2O_2Se

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Bi_2O_2Se is a 2D layered material with potential applications in thermoelectrics. In this work, a series of polycrystalline Bi_2O_2Se samples with altered stoichiometry were prepared. They were characterized by X-ray diffraction, electrical conductivity, Seebeck coefficient and thermal conductivity. Thermoelectric (TE) data were measured as a function of temperature in the range of 300 K to 773 K. We came across a specific issue where significant variations in TE properties compared to the recently published data on undoped Bi_2O_2Se are apparent. Furthermore, even the published data from various sources are inconsistent. We also compared TE properties of published data on doped samples with our samples with altered stoichiometry and found similarities. Based on a thorough investigation, we have reached several conclusions that are relevant to this problem. We argue that the solubility of the doped elements is very limited. Instead, the doping process mainly induces changes in the stoichiometry of the parent Bi_2O_2Se. This is associated with formation of native point defects and foreign phases. It is worth noting that both native defects and foreign phases are mostly not observable by X-ray methods.

Acknowledgement: This research was supported by the GAČR via project 22-05-919S.

Preparation of luminescent nanofibers from bioactive glasses containing samarium

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The use of nanoparticles has expanded in many areas of human activity. The ability of nanoparticles to penetrate deep into tissues, cells and nuclei can be used in medicine. Significantly expanded possibilities of molecular diagnostics and identification of biomarkers unique to each patient create prerequisites for improved therapy and targeted drug administration. In connection with the use of nanotechnology, it is becoming relevant to study the safety issues of the use of nanoparticles and to evaluate their toxic effects on the environment and human health.

The goal of this project is the preparation of glass powders and nanofibers from them for personalized anti-tumor systems used for highly effective drug delivery. First, glass samples will be prepared by melting, then glass powders will be obtained from these samples by crushing. Nanofibers from glass powders will be produced using the electrospinning method. Due to their large surface area, controlled surface conformation, good surface treatment, complex pore structure, and high biocompatibility, these nanofibers have proven to be a promising material for the creation of therapeutic platforms and biosensors.

Acknowledgement: This work was supported from the grant of Specific university research – grant № A2_FCHT_2023_048_Albekova.

Structural analysis of lithium borophosphate glasses by 1/2D MAS NMR techniques

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Borophosphate glasses of the series (100-x)[0.4Li_2O-0.2Nb_2O_5-0.4P_2O_5]-xB_2O_3 were studied in a composition range of x = 0-48 mol% B_2O_3. Basic physical properties were determined, and thermal properties studied by differential thermal analysis, thermomechanical analysis. The glass structure was investigated using Raman spectroscopy and ^{11}B and ^{31}P MAS NMR. The electrical properties of the glasses were obtained by using impedance spectroscopy.

Focus of this contribution will be given on MAS NMR analysis of studied glasses. Structure of the glasses was probed with multiple MAS NMR experiments. To get first information about the glass structure, standard 1D experiments of ^{11}B and ^{31}P nuclei were performed. To probe interconnections between phosphate and borate species was further studied by 2D correlation techniques. Namely $^{11}B(^{31}P)$ D-HMQC sequence to investigate P–O–B connections. By combining all these methods, we were able to prepare an approximate structural model of these glasses, which could be used to explain the macroscopic properties of the studied glasses.

Bioglass Particle Size Effect on Bioactive Behaviour in Dynamic Test Arrangement

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The prediction of bioactive materials' reactivity is crucial in tissue engineering and clinical practice. It is essential to realize that the bioactivity of the implant is influenced not only by the material's reactivity but also by the test solution and its composition. Experimental conditions play a significant role in *in vitro* testing. Most of these tests are conducted under static conditions and provide valuable information, but they do not reflect the processes taking place in the human body.

This work aimed to observe bioactive behaviour and evaluate the effect of glass particle size on their reactivity. Crushed bioactive glass (Bioglass 45S5) was immersed in two solutions: TRIS-buffered SBF (ISO 23317:2014) and non-buffered SBF. Before the 24-hour test, performed under dynamic conditions more reflective of the physiological environment, original particles were characterized through particle size measurement and SEM/EDS analysis. Kinetics of the interaction processes were monitored through eluate analyses (pH, ICP-OES) and material analyses (weight changes, SEM/EDS, FTIR).

Results indicate that the smaller fraction of bioactive glass exhibited stronger interaction than the larger one, forming silica-rich and calcium-phosphate layers on the samples' surface. The findings also confirmed that the TRIS is not an inert buffer; it accelerates glass dissolution and promotes the precipitation of new Ca-P phases. This knowledge will aid in establishing a new arrangement for *in vitro* bioactivity tests of glass materials. This experiment showed that a smaller fraction interacts rapidly, and new layers are visible after just one day of exposure. This finding may lead to future use of smaller glass fractions and a reduction in testing time.

Acknowledgement: This work was supported from the grant of Specific university research – grant No. A2_FCHT_2023_042.

Preparation of Ni-Co-Mn mixed oxide coatings on stainless-steel supports by sol-gel method and their activity in oxidation of volatile organic compounds

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Transition metals-containing oxides are widely used in heterogeneous catalysis as catalysts for various reactions such as dehydrogenation, selective oxidation, selective reduction, removal of volatile organic compounds (VOC) and electrocatalysis [1]. The performance of metal oxide-based catalysts depends on the methods and conditions of preparation. The sol-gel reaction is advantageous for the preparation of mixed oxide coatings combining various catalytically active components. The prepared sol can be deposited on substrates to obtain supported catalysts. The aim of this work was preparation of supported catalysts with catalytically active oxide coatings combining Ni, Co, and Mn components deposited on stainless steel supports.

Ni-Mn, Ni-Co and Ni-Co-Mn mixed oxides coatings were prepared by sol-gel method. The salts of metals mentioned above were dissolved in distilled water together with complexing agent (tartaric acid) and water-soluble polymer (gelatin). The reaction mixture was stirred for several hours at 85-90 °C to evaporate water and obtain a desired volume of the mixture. The formed sol was deposited on the stianless steel substrates by dip-coating method.

The combination of Ni, Co, and Mn chlorides, tartaric acid and gelatin in the reaction mixture for the sol-gel reaction led to the formation of a sol precursor suitable for deposition on the supports and obtaining of homogeneous oxide coatings after calcination. The adhesion of catalytically active mixed oxides to stainless steel sheets and meshes was improved by pre-coating them with SiO_2 interlayer and repeated dip-drying cycles before calcination. The deposited Ni-Mn coatings contained a mixture of Ni-Mn mixed oxides (Ni_6MnO_8 and $NiMnO_3$), whereas the spinel-type mixed oxides were found as a major component in the Co-containing samples. The supported mixed oxide catalysts with Ni:Co, Ni:Mn, and Ni:Co:Mn molar ratios of 1:4, 1:1, and 1:1:1, respectively, showed good catalytic activity in the ethanol deep oxidation.

Acknowledgement: The authors thank the Czech Science Foundation (project No. 21-04477S) for the financial support.

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Directed Energy Deposition 3D Glass Printing for Realizing Customized Optical Sensors

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This research focuses on the innovative application of Directed Energy Deposition (DED) in 3D glass printing, specifically for fabricating customized optical sensors. The project aims to overcome the limitations of traditional glass manufacturing methods by leveraging additive manufacturing's flexibility and precision.

During the first year, significant progress was made in establishing the basic setup for the DED 3D glass printing machine, including developing a GUI MATLAB program to control the printing process. Utilizing a CO2 laser and glass optical fibers, the printer showed promising results in printing both 1D and 2D structures. Optimum printing parameters were identified, with a speed of 10 mm/s being the most effective.

FEM simulations were conducted to determine the ideal printing temperature for minimizing mechanical stress, indicating around 400 °C as optimal. This finding will guide modifying the printing setup to include temperature control.

Despite these advancements, challenges remain in achieving the desired optical quality in printed parts. Further tests, including FTIR and optical response analyses, are planned for the upcoming year to assess and improve the optical properties of the printed structures.

In conclusion, this study opens new avenues in the field of optical sensor manufacturing, promising more efficient and versatile production methods crucial for the rapidly evolving demands of modern technology.

Acknowledgment: This work was supported by the Student Grant Competition (SGS) of the Technical University of Liberec, under project no. SGS-2023-5321.

Využití odpadních stavebních kalů v adsorpčních technologiích

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Ve vyspělých zemích vede stavební průmysl k vyčerpávání přírodních zdrojů a produkci několika miliónů tun odpadu. Jedním z těchto odpadů jsou i práškové stavební kaly (WBS) [1,2]. V současnosti jsou využívány různé separační a recyklační procesy, které jsou účinné, ale pro udržitelný rozvoj sami o sobě nedostatečné, proto je důležité pro tyto materiály najít další způsoby použití [2,3]. Odpadní stavební kaly mají slibné povrchové vlastnosti a chemické složení (obsah Si, Ca, Al a Fe), toto z nich dělá vhodné kandidáty pro jejich uplatnění v environmentálně přátelských technologiích [3].

Pro práci byly použity dva vybrané odpadní stavební kaly: i) kal z výroby a opracování předpojatých betonových sloupů (B); ii) kal z výroby technického kamene (TS). Dále byl použit klinoptilolit jako zástupce zeolitů, které jsou známé pro své výborné adsorpční vlastnosti. Experimenty byly provedeny na modelových roztocích obsahující vybrané toxické ionty (Pb^{2+}, Cs^{+}, AsO_4^{3-}, CrO_4^{2-}) ve dvou různých koncentracích 0.1 a 0.5 $mmol \cdot L^{-1}$. Sorpční experimenty byly prováděny vsádkově po dobu 24 hodin. Experimentální data byla proložena Langmuirovou adsorpční izotermou pro stanovení sorpčních parametrů (Q_t —teoretická sorpční kapacita, K_L —Langmuirova adsorpční konstanta, R^2 — korelační faktor).

Adsorpce kationtů probíhala s vysokými sorpčními účinnostmi (> 90 %) pro všechny systémy kromě sorpce Cs^{+} na B, který má vysokou hodnotu pH nulového náboje (pH_{ZPC}) a je selektivním sorbentem aniontů. B byl schopen s vysokou účinnosti adsorbovat Pb^{2+}, protože tento iont v systémech s vysokým pH podléhá hydrolýze a vytváří vnitřní sférické komplexy s povrchem sorbentu. Adsorpce kationtů mohly být téměř všechny proloženy Langmuirovým adsorpčním modelem. Pro adsorpci aniontů AsO_4^{3-} a CrO_4^{2-} byl nejúčinnějším sorbentem B a sorbenty klinoptilolit a TS se ukázaly jako neúčinné. Pouze adsorpce aniontů na B mohla být popsána Langmuirovým adsorpčním modelem.

Poděkování: Tento výstup vznikl v rámci projektu Specifického vysokoškolského výzkumu – projekt č. A1_FCHT_2023_005.

Reference:

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Shrinkage and expansion of hydraulic binder from Metakaolin, $CaSO_4$ AII and $Ca(OH)_2$

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Along with the tendency to reduce CO_2 emissions, the replacement of Portland cement with new hydraulic binders is becoming a topical issue. One of the suitable materials for the preparation of such binders turns out to be low-quality clays, which are stored in large quantities as coal mine overburden. For clays calcination, the temperature range of 500-900 °C is used in order to obtain pozzolanic properties. Thanks to that the total energy balance and carbon footprint of the binder are significantly reduced comparing to Portland cement.

This research deals with the long-term shrinkage and expansion of a new hydraulic binder prepared from metakaolin, anhydrite II and hydrated lime. At the beginning, a series of mortars containing a variable ratio of raw materials, the addition of a plasticizer and constant water to binder ratio was designed. The 40x40x160 mm beam-shaped bodies containing measuring contacts were prepared and stored in three different environments: laboratory conditions, humidity environment (95% RH) and tap water. Subsequently, the longitudinal shrinkage and expansion were measured according to EN 12617-4 standard in a long-term period of one year. Furthermore, the phase composition of the selected mixtures was determined using X-ray diffraction, and the amount of ettringite, as a possible expansion phase of the hydrated binder, was monitored in particular. In addition, SEM images were taken on selected samples.

In conclusion, the results of measurements in different environments were compared and the best composition of the binder was determined in view of the expansion behaviour. The obtained results were also compared with Portland cement and the potential use in industry was assessed.

Acknowledgement: This work was supported from the grant of Specific university research – grant No A1: FCHT_2023_005.

Microwave sintered SnO_2-ZnO based ceramics

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Ceramics based on tin oxide (SnO_2) and zinc oxide (ZnO) are important for various types of applications, including varistors, electrodes, gas sensors or catalysts. SnO_2 ceramics is a very specific material because a non-densifying sintering mechanism is usually dominating during sintering. On the other hand, in ZnO ceramics the dominant sintering mechanism is densifying, therfore it is much easier to obtain a highly dense material by conventional sintering compared to SnO_2 ceramics. Combining ZnO and SnO_2 it is possible to create a composite containing the spinel phase Zn_2SnO_4, which could significantly improve the properties of the material.

In this contribution we present results obtained for SnO_2 and ZnO ceramics and its composite prepared by microwave (MW-) sintering and compared them with results obtained on conventionally (CV-) sintered samples [1, 2]. Samples were prepared from commercial powders of SnO_2 and ZnO which were used in pure form but also mixed in different weight ratios (8:2, 2:8, 1:1). These powders were uniaxially pressed, and the sintering process was done in a multi-mode MW-cavity with and without the SiC susceptor. Different thermal cycles were used by varying the sintering temperature, heating rate, dwell time and the number of the heating cycles. After sintering the samples were characterized by measuring linear shrinkage, bulk density, porosity, phase composition and elastic properties were measured on chosen samples.

Concerning and investigation of the MW process it was possible to sinter all the samples with and also without the SiC susceptor, which proves that investigated materials have sufficient dielectric properties for the MW- sintering. Apart from that during the sintering certain areas were observed where more incident power was needed to initiate the sample heating, this fact is connected with the specific material property. When comparing the CV- and MW- sintered samples, in case of pure oxides (SnO_2 and ZnO) no changes between CV- and MW- sintered samples were observed. However, in case of composites significant differences were observed especially for the sample batch which was mixed to obtain mostly the spinel phase Zn_2SnO_4. Therefore, it was found that MW-sintering have a positive influence on densification and therefore the creation of the Zn_2SnO_4.

Acknowledgement: This work was supported from the grant of Specific university research – grant No A1_FCHT_2023_005. The stay od P.Š. in France was founded by Barrande Fellowship Programme.

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Inter-lanthanide $LaYYbO_3$ perovskites doped with Er^{3+}

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Perovskites are compounds with a general formula ABX_3, capable of accommodating the crystallographic structures of most elements in the periodic table [1]. Inter-lanthanide perovskites exhibit interesting magnetic and optical properties and are also good proton conductors. The intensity of photoluminescence (PL) emission can be influenced by the composition of the host matrix and is characterized by sharp spectral peaks from their 4f electron transitions [2, 3]. The aim of this work was to prepare selected perovskites of the $LaY_{1-x}Yb_xO_3$ system (where x = 0; 0.05; 0.15; 0.25) doped with ≈0.5 at.% Er^{3+} ions by combustion synthesis and study of their physico-chemical properties, with a focus on optical properties. In addition to the impact of the chemical composition of perovskites on the resulting properties, the attention was paid to revealing the dependence of Yb^{3+} ion concentration on the mechanism of upconversion (PL) emission.

The studied samples were characterized in terms of their chemical composition (EDX microanalysis), crystallites size (SEM), their crystallographic structure (XRD analysis), diffuse reflectance spectra (UV-Vis-NIR spectroscopy), and steady-state and time-resolved PL emission spectra measured at the pumping wavelength of $\lambda \approx 977$ nm.

Substitution of Yb^{3+} ions for Y^{3+} in $LaY_{1-x}Yb_xO_3$ perovskites leads to the formation of an orthorhombic perovskite structure $LaYYbO_3$, at lower annealing temperatures with the same time of annealing the samples. In all perovskites, was observed the near-infrared Er^{3+}: $^4I_{13/2} \rightarrow {}^4I_{15/2}$ ($\lambda \approx 1.56$ μm) PL Stokes and the visible Er^{3+}: $^2H_{11/2}/{}^4S_{3/2} \rightarrow {}^4I_{15/2}$ ($\lambda \approx$ 520–550 nm) and Er^{3+}: $^4F_{9/2} \rightarrow {}^4I_{15/2}$ ($\lambda \approx 670$ nm) PL anti-Stokes emission. Based on the PL emission spectra and the decay times of Er^{3+} electronic transitions, the mechanism of upconversion PL emission was determined. The optimal concentration of Yb^{3+} ions in studied perovskites was found to be ≈3 at.% in order to prevent concentration quenching of PL emission. Studied inter-lanthanide perovskites seem to be promising materials with potential in optical applications.

Acknowledgments: This work was supported by the Ministry of Education, Youth and Sports CR (LM2018103). I also thank projects SG2023 and VA390011.

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Ternary cesium lithium iodide crystals grown by vertical Bridgman method for scintillation applications

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In general, a scintillator is an energy converter of ionizing radiation (based on particles or high energy photons) into photons in the UV-VIS-IR region. Initially, the ionization leads to excitation of materials' atoms and molecules into their excited states followed by the recombination of an electron-hole pair accompanied with photon emission and de-excitation back to their ground states. These emitted photons are detected by photodetectors, e.g., photomultiplier tube (PMT), and transformed into photoelectrons by multiplying the weak initial signal, which can be recorded with a software [1,2]. The scintillator together with the photodetector form the scintillation detector. Among solid scintillators, organic as well as inorganic ones, nowadays, the most with practical applications, were developed [2]. One of the most widely used material in radiation detection is thallium doped sodium iodide (NaI:Tl) [1] discovered by Hofstadter in 1948 [3]. Even though it has very good spectrometric response to γ-ray and a light yield of 45 000 photons/MeV, the main disadvantages of a NaI:Tl detector are poor energy resolution (ca. 6-8 %) and slow scintillation response (ca. 300 ns) [4–6]. Thus, a new scintillation material meeting present and future challenges is sought-after. In application areas, such as nuclear power generation, nuclear decommissioning and decontamination, border security, nuclear proliferation and nuclear medicine, neutrons and their detectors play crucial role [1,7]. Since 2008, the world has been facing the shortage of ^{3}He, which has become extremely expensive, as the supply became limited [8]. As ^{3}He proportional counters represent the industry standard for neutron detection [9], there is high demand for a new scintillation material.

For both neutron and X- and γ-ray detection, ternary alkali halides, such as $Cs_2Li_3I_5$ (CLI) could represent an alternative material. CLI exhibits a light yield of ca. 40 000 to 55 000 photons/neutron, a primary decay time of just 250 ns for thermal neutron interactions and 500 ns for γ-rays, and, when doped with Eu, emission maxima peaking at ca. 450 nm [10]. According to the CsI-LiI phase diagram reported by Sangster and Pelton [11], no ternary phase is present; however, the formation of a ternary CLI phase was already confirmed by Meyer and Gaebell in 1983 [12]. Nevertheless, the preparation of large CLI single crystals has not been published as of now. The goal of this work is to prepare single crystals of ternary CLI, confirm its phase purity and perform basic structural, physical, thermal, optical, luminescence, and scintillation characterizations. Further aim is to analyze the influence of doping of monovalent ions (e.g., Tl, In) in the matrix on its luminescence properties.

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Heated and plasma treated silicon nitride with different sintering additives for biomedical application

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Different compositions such as calcium silicate, cerium oxide, bioactive glass, and the combination of silicate compound with magnesium, silica, and calcium was studied as sintering additives of silicon nitride. The aim is to select sintering additives which form after sintering and/or post sintering treatment surfaces suitable for biomedical applications.

The processing parameters, such as initial composition, sintering temperature and time were determined, and the mechanical properties and microstructure of prepared silicon nitride-based materials were studied. The work was also focused on the modification of the surface using plasma etching and thermal technique. Parameters such as ratio of gases, exposure time, and nozzle distance were studied in order to find the optimal surface modification conditions.

Furthermore, the biological response of the surface was analyzed to determine its bioactivity using viability of murine cell and cytokine anti-inflammatory assays.

Acknowledgement: This work was supported by VEGA Project No. 2/0161/22, and JRP SAV TUBITAK project No. 546676.

Optimalization of a debinding procedure for objects prepared by 3D printing of the low loaded photopolymeric ceramic suspensions

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Digital Light Processing (DLP) is one of the additive manufacturing methods suitable for preparing ceramic objects. It allows the fabrication of geometrically difficult parts that can be modified immediately according to the customer's requirements. DLP consists of four main steps (design and preparation of a model for printing, printing from photosensitive ceramic suspension, debinding and sintering), while the debinding procedure is the most critical. The debinding process serves for carefully removing organic additives from printed parts and prevents them from the formation of various defects, such as cracks or delamination, connected with stresses originating from the decomposition of organic compounds, leading to decreased mechanical properties. Therefore, the proper and effective design of the debinding procedure was researched in this study.

Optimization was accomplished to obtain samples with maximal density and minimal defects. It was performed by nine different groups of samples with dimensions 14x14x15 mm. Each group was treated by a three-step cycle (dwell was performed at 200 °C, 350 °C and 450 °C for 1 hour, 3 hours and 1 hour, respectively) with different heating rates and atmosphere types (air, nitrogen and vacuum). The group consisted of six samples with wall thicknesses of 1.0 mm, 1.5 mm, 2.0 mm, 2.5 mm, 3.0 mm and 4.0 mm. Overall, fifty-four samples were prepared. Samples were fabricated from the ceramic suspension with 33.6 vol.% loading of ZrO_2 particles and printed on Original Prusa SL1 printer. All debinding procedures were accomplished in the debinding furnace CLASIC CZ, type: 2011 DEB and each sample was sintered by the same cycle in the high-temperature furnace CLASIC CZ, type: 0518S. After the fabrication process, samples were evaluated by mass loss after debinding, after sintering and total mass loss, visual observation of defects, and calculation of relative density, porosity, and hardness.

Vacuum was found as the best surrounding atmosphere for the debinding procedure. The main reasons are the less reactive conditions helping to limitation of the exothermic reactions leading to CO_2 production, and the underpressure, which helps with the removal of the gases from the thermal degradation of additives. Moreover, the debinding procedure in a vacuum was insensitive to chosen heating rates. Therefore, the cycle with the highest heating rates: 2 °C/min, 1 °C/min and 1 °C/min, was selected as the most efficient; the whole procedure took 12.5 hours. It must be emphasized that further sintering incorporated an initial heating step, which took 5.8 hours due to the removal of the residual carbon. The resulting samples reached 98.4±0.1 % of theoretical density, and this value was independent of wall thickness. Also, all mass losses and hardness (HV1 was 1366 ± 17) were constant with this variable. The only parameter changing with wall thickness was type and number of defects. This observation concluded that the designed procedure described above can provide samples with 1 – 3 mm thick walls if stress concentrators are eliminated.

Acknowledgement: We acknowledge CzechNanoLab Research Infrastructure supported by MEYS CR (LM2023051).

Research of the sintering process of no cement refractory concretes with dead burned magnesia filler

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Sol-gel refractory concretes have many advantages compared to ordinary cement concretes. One of the main factors limiting the use of these concretes is their low resistance to basic slags. This is mostly due to the impossibility of using magnesite raw materials resistant to basic slags, due to the fact that MgO causes almost instantaneous coagulation of the gel. A possible solution to this problem could be the reproduction of a recipe that does not contain finely ground magnesium oxide, but only large grains. To continue research in this direction, it is important to understand how the interaction between concrete components occurs during its sintering. This paper presents the results of the first part of the research in which the interaction of the components of such concrete during sintering was studied under idealized conditions. The work also provides a thermodynamic calculation of possible reactions of the interaction of the components of the mixture.

Acknowledgement: This study was financially supported by Slovak Grant Agency for Science – project VEGA 1/0060/22 and the Slovak Research and Development Agency – project APVV-17-0483.

Thermal Stability of Valuable Metals in Cathodes at Elevated Temperatures

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The development of electric vehicles (EVs) is one of the fastest-growing segmenst in the automotive industry. With the increasing expansion of EVs, demand is growing for recycling and reusing lithium-ion batteries (LIBs) materials, particularly valuable metals like lithium, nickel, manganese, and cobalt. The demand for these metals is rising, and spent LIBs can potentially serve as a secondary source.

This paper focuses on the material analysis and stability during temperatures from 100-800 °C of the cathode in lithium nickel manganese cobalt oxide ($LiNi_xMn_yCo_zO_2$, NMC) batteries, which are currently the most commonly used type. Samples from the cathode were examined using a scanning electron microscope (SEM), X-ray fluorescence (XRF) and diffraction (XRD) analysis, thermal analysis, and, most importantly, the determination of the leached ion content after dissolution in nitric acid by inductively coumpled plasma optical emission spectrometry (ICP-OES). The experiment was also conducted using demineralized water as the use of an aqueous solvent can reduce costs and ehnance safety in the recycling process. However, this procedure could not be applied to all the elements determined, which include lithium, nickel, manganese, and cobalt.

The studied material was initially subjected to thermal conditions, which enabled to investigate the impact of the initial material firing temperature on the results. This procedure simulates the first step of the pyrometallurgical-hydrometallurgical process, which is one of the methods used for recycling spent LIBs. It was observed that the organic binder evaporated from the material between temperatures of 300 and 400 °C. This effect was evident, for example, the SEM/EDS analysis showed a sudden decrease in the F content and a corresponding increase in the content of all the other elements.

Acknowledgement:

This work was financially supported from the grant of Specific university research – grant No. A1_FCHT_2023_005, while the infrastructure has been utilized in the frame of project No. CZ.02.01.01/00/22_008/0004617 Energy conversion and storage within the Jan Amos Komensky Operational Program financed by the European Union and from the state budget of the Czech Republic.

Synthesis traps of polycrystalline Bi_2O_2Se

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Bismuth oxyselenide (Bi_2O_2Se) is being investigated as a promising thermoelectric (TE) material, IR photodetector and 2D FET substrate. Its main advantage as a TE material is the combination of good transport properties, absence of toxic elements and reasonable price. As the pure material has low electrical conductivity, researchers focus on doping [1]. In this context, it is crucial to distinguish the effect of doping from a simple variation of the base material.

It is noteworthy that even for the same synthesis method, the reported transport properties of bulk polycrystalline samples are very different. [2,3,4]. To identify multiple factors contributing to the variation in transport properties, we prepared a series of samples from different synthesis compounds and with different synthesis routes.

In order to identify foreign phases in the prepared samples, we carried out an analysis using powder X-ray diffraction (PXRD), scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS), Raman spectroscopy and ATR IR spectroscopy.

In this work we present how impurities can affect the transport properties of undoped material and provide a reproducible low-temperature solid-state synthesis process for pure Bi_2O_2Se

Acknowledgement: We would like to thank GAČR for supporting this research via project 22-05-919S.

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The activation energy of sintering of $(Mg_{0.2}Co_{0.2}Cu_{0.2}Ni_{0.2}Zn_{0.2})O$ and impact of sintering temperature and heating rates on microstructure and homogeneity

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The first application of entropy stabilisation of ceramic materials was demonstrated by Rost et al. in 2015. They successfully synthesised a high entropy oxide (HEO) with a rock salt structure consisting of MgO, CoO, CuO, NiO and ZnO mixed in an equimolar stoichiometric ratio. After appropriate thermal treatment, this mixture forms a high entropy phase $(Mg_{0.2}Co_{0.2}Cu_{0.2}Ni_{0.2}Zn_{0.2})O$. To form a single phase high entropy structure the configurational entropy (Sconf) of the material must be $\geq$ 1.5R. This value is influenced by the differences in magnetic moment, vibrations, and arrangement of atoms in the crystal structure, with the atomic arrangement usually having the greatest influence on the configurational entropy changes.

The homogeneous high entropy ceramic (HEC) bulk bodies of $(Mg_{0.2}Co_{0.2}Cu_{0.2}Ni_{0.2}Zn_{0.2})O$ were successfully sintered. HEO was synthesised using oxidic precursors in stoichiometric equimolar ratios, mixed in a planetary ball mill and sieved through a 100 μm sieve to eliminate particle agglomeration.

Bulk samples were sintered by spark plasma sintering (SPS) using three different heating regimes, 25, 50 and 100 °C/min. The final sintering temperatures range from 700 to 1000 °C. The aim was to investigate the effect of different heating regimes on microstructure and chemical homogeneity. It was shown that the final sintering temperature mainly influenced density and homogeneity and that the different heating rates resulted in little or no change in investigated parameters. Samples sintered at the highest temperature of 1000 °C were determined by relative density greater than 98 % of the theoretical density and by a high degree of chemical homogeneity. Furthermore, the activation energy of the sintering process was calculated using a master sintering curve method.

Acknowledgement: The authors acknowledge the support of the Grant agency of the Czech Republic under grant no. 23-06856S. CzechNanoLab project LM2023051 funded by MEYS CR is gratefully acknowledged for the financial support of the measurements/sample fabrication at CEITEC Nano Research Infrastructure.

Adsorption and catalytic properties of precipitatively prepared cerium dioxide

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This study is focused on the adsorption and catalytic properties of cerium dioxide. Nanoparticles were synthesized through the precipitation method, and subsequently characterized by using X-ray diffraction, Raman spectroscopy, and transmission electron microscopy. Adsorption properties were investigated for ten organic pollutants found in the water environment. Nanoparticles were added to solutions of individual substances, and the resulting concentration decrease was monitored using UV-Vis spectroscopy. For nano-particles, where adsorption occurred, Raman spectra were measured to determine the type of sorption and further identify the binding site.