30. ročník odborného semináře doktorandů
30th annual Doctoral Students‘ Seminar

ANORGANICKÉ NEKOVOVÉ MATERIÁLY

INORGANIC NON-METALLIC MATERIALS

5. – 6. února 2025
February 5-6, 2025

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

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 nadačnímu fondu prof. Rudolfa Bárty, firmě Glass Service, Silikátové společnosti České republiky z.s. a České sklářské společnosti 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

Contact

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

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 Prof. Rudolf Bárta's Foundation, company Glass Service, The Czech Ceramic Society and The Czech Glass Society for providing prizes for the participants.

Silikátová společnost České republiky z.s

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Program semináře 2025 / Seminar programme 2025

Středa 5. února 2025 / Wednesday 5th February 2025

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

1. Blok / 1st session (9:40-12:00)

Soutěž České Silikátové společnosti / Czech Ceramic Society Competition

6 příspěvků / 6 lectures

*Ing. Herbert Kindl (9:40-10:00)**

Development and characterization of nanoparticle-infused phosphite glass rods via the micro-pulling-down method

Ing. Lucie Kotrbová (10:00-10:20)

Temperature dependence of thermal conductivity of silica-based materials as materials for high temperature thermal energy storages (HT-TES)

Ing. Marek Rotter (10:20-10:40)

SPS as an effective pre-sintering technique for transparent/translucent HEC

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

*Ing. Klára Betáková (11:00-11:20)**

Physico-chemical properties of clinker-free hydraulic binder from metakaolin, anhydrite II and lime

*Ing. Vojtěch Ševčík (11:20-11:40)**

Preparation of carbonate-free $BaCeO_3$ powder for REBCO superconductors

Ing. Petra Šimonová (11:40-12:00)

Energy-efficient ceramic processing: Insights into microwave sintering of SnO_2-ZnO-based ceramics

* Příspěvky označeny * jsou také součástí Soutěže České Sklářské společnosti / Contributions marked with * are also part of the Czech Glass Society Competition.

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

2. Blok / 2nd session (13:00-14:10)

Soutěž České Silikátové společnosti / Czech Ceramic Society Competition

3 příspěvky / 3 lectures

*Ing. Kateřina Křehlíková (13:00-13:20)**
Ternary manganese(II) chloride as a new red-emitting neutron scintillator

Ing. Daniel Valášek (13:20-13:40)

Mechanical and Structural Insights into High Entropy Carbides Produced from High Entropy Alloys

Lekshmi Jegan (13:40-14:00)
A Sustainable Approach to Develop Silicon-Graphite Anode for Advanced Lithium-Ion Batteries

Ing. Petra Šimonová (14:00-14:10)
YCN by European Ceramic Society and PCSA by American Ceramic Society

Přestávka / Coffee break (14:10-14:30)

3. Blok / 3nd session (14:30-15:30)

3 příspěvky / 3 lectures

Ing. Jiří Stibral (14:30-14:50)
Synthesis and characterization of substituted $CaSnO_3$ pigments

Ing. Patrik Sokola (14:50-15:10)

Optimization of photocurable zirconia suspensions for 3D printing

Ing. Lukáš Mauermann (15:10-15:30)

Optimization of the preparation of a new three-component clinker-free binder

Společenský program Pubkvíz / Social programme Pubquiz (17:30, Carbon)

* Příspěvky označeny * jsou také součástí Soutěže České Sklářské společnosti / Contributions marked with * are also part of the Czech Glass Society Competition.

Čtvrtek 6. února 2024 / Thursday 6th February 2025

4. Blok / 4th session – (9:40-11:40)

Soutěž České Sklářské společnosti / Czech Glass Society Competition[*]

5 příspěvků / 5 lectures

Ing. David Vaculík (9:40-10:00)
Viscosity and crystal growth in amorphous samples of $Ge_{25}Se_{75}$ chalcogenide glasses

Ing. Jan Zich (10:00-10:20)
Anomalous properties of Bi_2O_2Se monocrystals

Ing. Mansi Dua (10:20-10:40)
Additive manufacturing of advanced and porous glass ceramic photocatalytic 3D structures for wastewater treatment

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

Ing. Tomáš Hostinský (11:00-11:20)
Silver borophosphate glasses modified with tungsten and molybdenum oxide

Ing. Tomáš Pipota (11:20-11:40)
Preparation and dissolution of binary phosphate glasses

Pauza na oběd / Lunch break (11:40-12:40)

[*] Příspěvky z 5. 2. 2025 označeny * jsou také součástí Soutěže České Sklářské společnosti / Contributions marked with * are also part of the Czech Glass Society Competition.

5. Blok / 5th session (12:40-14:20)

5 příspěvků / 5 lectures

Ing. Timur Babi (12:40-13:00)
Supported catalysts with an active layer of transition metal oxides

Ing. Karolína Opavová (13:00-13:20)
Investigating the impact of solution composition on bioactive glass behaviour: Effect of MEM, SBF15, and TRIS-Buffered SBF

Ing. Tereza Přívracká (13:20-13:40)
Monitoring the immediate interaction of glass materials in demineralized water

Vyhlášení výsledků a ukončení semináře / Winner announcement and end of the seminar (14:00)

Czech Ceramic Society Competition

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 Czech Ceramic Society Competition. Participation in this competition is open to members of the Czech Ceramic Society and the winner will represent the Czech Republic at the Student Speech Contest at the XIXth Conference of the European Ceramic Society 2025.

Members of the committee for Czech Silicate Society Competition:

- doc. Ing. Hana Ovčačíková – Technical University of Ostrava
- Ing. Eva Bartoníčková Ph.D. – Brno University of Technology
- doc. Ing. Kateřina Rubešová, Ph.D.– University of Chemistry and Technology Prague

The list of participants:

- Ing. Klára Betáková
- Ing. Lekshmi Jegan
- Ing. Herbert Kindl
- Ing. Lucie Kotrbová
- Ing. Kateřina Křehlíková
- Ing. Marek Rotter
- Ing. Vojtěch Ševčík
- Ing. Petra Šimonová
- Ing. Daniel Valášek

Czech Glass Society Competition

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 Czech Glass Society Competition. The winner will receive a financial prize and an opportunity to contribute an article to the journal Sklář a Keramik.

Members of the committee for Czech Glass Society Competition:

- doc. Ing. Jaroslav Kloužek, CSc. – University of Chemistry and Technology Prague
- Ing. Richard Pokorný, Ph.D. – University of Chemistry and Technology Prague

The list of participants:

- Ing. Klára Betáková
- Ing. Mansi Dua
- Ing. Tomáš Hostinský
- Ing. Herbert Kindl
- Ing. Kateřina Křehlíková
- Ing. Tomáš Pipota
- Ing. Vojtěch Ševčík
- Ing. David Vaculík
- Ing. Jan Zich

Award of Foundation prof. Rudolf Bárta

Selected participants will be recognized with the Award of Foundation prof. Rudolf Bárta. All presentations in English are automatically considered for this award.

Members of the committee:

- doc. Ing. Jaroslav Kloužek, CSc. – University of Chemistry and Technology Prague
- Ing. Richard Pokorný, Ph.D. – University of Chemistry and Technology Prague
- Ing. Jan Hostaša, PhD. - CNR-ISSMC Italy
- Ing. Anna Prnová, PhD. – Institute of Inorganic Chemistry SAS

The list of participants:

- Ing. Timur Babi
- Ing. Klára Betáková
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- Ing. Lucie Kotrbová
- Ing. Kateřina Křehlíková
- Ing. Lukáš Mauermann
- Ing. Karolína Opavová
- Ing. Tomáš Pipota
- Ing. Tereza Přívracká
- Ing. Marek Rotter
- Ing. Patrik Sokola
- Ing. Jiří Stibral
- Ing. Vojtěch Ševčík
- Ing. Petra Šimonová
- Ing. David Vaculík
- Ing. Daniel Valášek
- Ing. Jan Zich

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ABSTRAKTY
ABSTRACTS

Development and characterization of nanoparticle-infused phosphite glass rods via the micro-pulling-down method

Herbert Kindl[1,2], Karol Bartosiewicz[2], František Zajíc[2]

[1]*Department of Inorganic Chemistry, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*

[2]*Institue of Physics of the Czech Academy of Sciences, Prague, Cukrovarnická 10/112, 162 00, Czech Republic*

kindlh@vscht.cz

Luminescent materials are extensively used in a wide range of applications, including white light-emitting diodes (LEDs), solid-state lasers, scientific instrumentation, sensor technologies, and medical imaging devices. The continuous advancement of these technologies necessitates improvements in luminescent properties alongside efforts to minimize production costs, pushing ongoing research and innovation in this field. In bulk luminescent materials one of the obstacles to achieving good luminescent properties, such as high quantum yield, is a luminescence quenching problem. The quenching of emitted radiation can occur due to several factors, primarily including energy transfer between closely spaced luminescent centers at high concentrations, reabsorption of light by the host lattice, or the trapping of charge carriers by intrinsic defects within the lattice.

This work focuses on the development of phosphite glass rods embedded with YAG:Ce nanoparticles. The motivation for preparing such composites was to mitigate quenching effects caused by volume phenomena typically observed in bulk luminescent materials. This can be achieved by embedding crystalline nanoparticles of a luminescent material into a low-melting-point matrix. This strategy effectively reduces the impact of total internal reflection, which traps emitted photons within the crystal lattice and restricts their extraction. As light emission occurs independently from each nanoparticle within this system, this approach can enhance luminescence efficiency. A method chosen for the preparation of these nanocomposites was the micro-pulling-down method (μ-PD). During the μ-PD process, the melt is pulled through a capillary in which microscopic cavities occur due to high pulling rates. As these cavities collapse, they release shock waves, effectively separating agglomerated nanoparticles. The main goal of this work was to optimize the growth process by customizing the hot zone and to determine the effects of different temperature and pulling rate programs on rod shape and nanoparticle distribution. Furthermore, samples will be characterized using XRD, SEM/EDS and optical, luminescent and scintillation properties will be estimated.

Temperature dependence of thermal conductivity of silica-based materials as materials for high temperature thermal energy storages (HT-TES)

Lucie Kotrbová, Tereza Uhlířová, Willi Pabst

[1]*Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*

lucie.kotrbova @vscht.cz

Materials for thermal energy storage (TES) is a hot topic in the world right now. One of the solutions is to use silica-based materials. Silica sand is already being used for quite some time as a TES material up to 600 °C. Significantly higher temperatures could be achieved by using silica refractories as TES materials. Of course, for TES applications, a detailed knowledge of the thermal and thermophysical properties of the materials is indispensable. Therefore, this work deals with the thermal conductivity of silica refractories and, for setting this into broader context, a comparison with other silica-based materials (sandstone) is made.

Based on the knowledge of phase composition (XRD), porosity (Achimedes method) and microstructure (SEM), theoretical predictions of thermal conductivity values at room temperature are calculated by theoretical bounds (upper Wiener bound, upper Hashin-Shtrikman bound), analytical predictions (power-law and exponential relation) and numerical simulations (for polycrystalline multiphase model materials with spherical, polyhedral and concave pores generated by the GeoDict® software package) and then compared with the experimental results obtained from two different transient methods, one of the contact type (modified transient plane source / MTPS technique), the other of the non-contact-type (laser-flash analysis / LFA technique). Special attention is given to the fact that the results of contact-techniques like MTPS are expected to be to a certain degree affected by the influence of the thermal contact resistance at the probe-material interface, which might introduce significant bias into the resulting values. An attempt is made in this work to assess the magnitude of this effect by modifying the contact surface and verifying the results by independent contact-free measurements via the LFA technique. Moreover, it is shown how the temperature dependence of the thermal conductivity can be obtained from that of the thermal diffusivity, measured via the LFA technique in a recently proposed continuous mode.

Acknowledgement: This work was supported from the grant of Specific university research – grant No. A1_FCHT_2024_008 and No. A2_FCHT_2024_018.

SPS as an effective pre-sintering technique for transparent/translucent HEC

Marek Rotter[1,2], Katarína Drdlíková[1], Daniel Drdlík[1,4], Monika Michálková[3], Karel Maca[1,4]

[1]*CEITEC - Central European Institute of Technology, Brno University of Technology, Brno, Czech Republic*

[2]*FunGlass - Centre for Functional and Surface Functionalized Glass, Alexander Dubček University of Trenčín, Trenčín, Slovakia*

[3]*Joint Glass Centre of the IIC SAS, TnUAD, FChPT STU, FunGlass, Trenčín, Slovakia*

[4]*Faculty of Mechanical Engineering, Brno University of Technology, Brno, Czech Republic*

Marek.Rotter@ceitec.vutbr.cz

High Entropy (Transparent) Ceramics (HE(T)Cs) represent a novel category of advanced materials which promise enhanced properties relative to conventional ceramics. To achieve transparency, the microstructure without pores, impurities and secondary phases is required.

Achieving this condition can be challenging to realise especially for HEC due to multicomponent composition. Therefore, proper design and selection of each component are crucial. In this work $(La_{0.1}Y_{0.5}Gd_{0.6}Yb_{0.7}Sm_{0.1})Zr_2O_7$ was used. The amount of each element was levelled to achieve a stable defect fluorite phase in a wide temperature range without phase transformation.

Spark Plasma Sintering (SPS) is usually used for fast densification. However, fast sintering may also inhibit the grain growth, leading to the retention of pores at the grain boundaries. This phenomenon can be effectively utilised as a pre-sintering treatment before a Hot Isostatic Press (HIP), facilitating the removal of residual porosity at the grain boundaries. Hence, this combined process effectively reduces the number of pores, which are the primary light-scattering centers and enables the production of translucent/transparent ceramic.

Physico-chemical properties of clinker-free hydraulic binder from metakaolin, anhydrite II and lime

Klára Betáková, Martina Šídlová

Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic

klara.betakova@vscht.cz

This study investigates the physico-chemical properties of hydraulic binder composed of metakaolin, anhydrite II, and lime. Calcining kaolinitic clay at temperatures between 500-900 °C imparts the desired pozzolanic properties, which induce hydraulic behaviour in the presence of activators. Lower calcination temperatures significantly reduce the carbon footprint and energy consumption of binder production compared to high-emission Portland cement. Additionally, there is potential for future binder production using waste inorganic clay-based materials and by-products from energy processes (CCPs). To utilize the binder in construction, its properties and long-term behaviour must be characterized. In this research, binder pastes with different compositions were designed and prepared using an appropriate water to binder ratio. The samples, cured in a controlled humidity environment, were analysed over time using mechanical testing and phase composition analyses (XRD, DTA-TG, SEM). The results were compared to Portland cement to evaluate potential industrial applications.

Acknowledgement: This work was supported from the grant of Specific university research – grant No A1: FCHT_2024_008.

Preparation of carbonate-free $BaCeO_3$ powder for REBCO superconductors

Vojtěch Ševčík[1]

[1]*Department of Inorganic Chemistry, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*

sevcikj@vscht.cz

$BaCeO_3$ is a mixed oxide with the potential to play an important role in the manufacture of REBCO superconductors. As an additive to the powder precursor at the melt-growth process stage of production of bulk superconducting components, it can positively influence the melt viscosity and phase equilibrium. Additionally, $BaCeO_3$ can serve as an alternative flux-pinning phase with improved properties in comparison to currently employed CeO_2 oxide. To be applicable in this role, however, the additive must not contain carbonates, which can have a significant negative impact on the growth of superconducting phases and their final performance. Finally, the small grain size of the additive is beneficial both for both melt-growth process and superconductor application parameters, which further limits the preparation options for such additives. The synthesis of fine-grain carbon-free precursor powder is thus a challenging task, especially when the phase contains an element with high affinity towards CO_2, as is the case with barium.

In this work, a $BaCeO_3/CeO_2$ powder mixture was prepared using reverse co-precipitation from a solution of barium and cerium cations. Several starting compounds and precipitating agents were tested to prepare a powder with high $BaCeO_3$ content while minimizing carbon contamination and limiting grin growth. The composition was determined using XRD and Raman spectroscopy, and the crystallite and grain size were obtained using XRD refinement and laser diffraction, respectively.

Energy-Efficient Ceramic Processing: Insights into Microwave Sintering of SnO_2-ZnO-Based Ceramics

Petra Šimonová[1], Clémence Petit[2], Willi Pabst[1], Christophe Meunier[2], François Valdivieso[2]

[1]*Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague, Technická 5, 166 28, Czech Republic*

[2] *Mines Saint-Etienne, Université Lyon, CNRS, UMR 5307 LGF, F-42023 Saint-Etienne, France*

simonovb@vscht.cz

Microwave (MW) sintering is an advanced, energy-efficient method for ceramic processing. It uses electromagnetic radiation to achieve rapid and uniform heating throughout the material. Compared to conventional sintering, MW sintering offers significant benefits, including lower energy consumption and shorter processing times, making it an attractive option for sustainable manufacturing. However, the material's dielectric properties are crucial for efficient MW sintering, often requiring susceptors to initiate and stabilize the heating process.

Ceramics based on SnO_2 and ZnO are widely used in applications including gas sensors, varistors, electrodes, and photoelectric devices due to their similar functional properties. Despite this, they exhibit fundamentally different sintering mechanisms: SnO_2 follows a non-densifying sintering mechanism, whereas ZnO exhibits classical densifying behavior. Investigating and comparing these materials under identical conditions is therefore crucial for understanding their processing and performance during sintering.

In this study, we demonstrate successful direct MW sintering of SnO_2-ZnO-based ceramics without using SiC susceptors. Samples were prepared by uniaxial pressing from pure SnO_2 and ZnO powders, as well as their mixtures, and sintered at 1200 °C (SnO_2 and mixtures) or 1000 °C (ZnO) with a heating rate of 50 °C/min and a dwell time of 5 minutes in MW- multimode cavity. Our results reveal that direct MW heating was achievable for all samples, with SnO_2 exhibiting faster heating initiation compared to ZnO, albeit with greater difficulty in maintaining a stable heating rate. Composite materials exhibited intermediate behavior, combining characteristics of both oxides.

A comparative analysis of power versus temperature curves for direct and indirect MW heating revealed distinct behaviors, with the presence of SiC in indirect heating enhancing densification and grain growth. These findings highlight the potential of MW sintering as an energy-efficient alternative for ceramic processing, particularly for SnO_2-ZnO systems, and provide valuable insights into optimizing this innovative technology for advanced material applications.

Acknowledgement: This work was supported from the grant of Specific university research – grant no A1_FCHT_2024_008. The stay of P.Š. in France was founded by Barrande Fellowship Programme.

References:

[1] Šimonová P. et al.: Elastic properties and microstructure evolution of Zn_2SnO_4-spinel-containing composite ceramics based on tin oxide and zinc oxide. *J. Eur. Ceram. Soc.* 2024.
[2] Šimonová P. et al.: Microwave sintering of tin oxide and zinc oxide mixtures – Formation of Zn_2SnO_4 spinel phase, densification and evolution of microstructure. *Ceram. Int.*, 2024. (under review)

Ternary manganese(II) chloride as a new red-emitting neutron scintillator

Kateřina Křehlíková[1,2], Vojtěch Vaněček[2], Robert Král[2], Petr Průša[2,3], Romana Kučerková[2], Vladimir Babin[2], Petra Zemenová[2], Jan Rohlíček[2], Zuzana Málková[4], Terézia Jurkovičová[4], Kateřina Rubešová[1], Martin Nikl[2]

[1]*Department of Inorganic Chemistry, Faculty of Chemical Technology, University of Chemistry and Technology, Prague, Technická 5, 166 28, Prague, Czechia*

[2]*Department of Optical Materials, Division of Solid State Physics, Institute of Physics of the Czech Academy of Sciences, Cukrovarnická 10/112, 162 00, Prague, Czechia*

[3]*Department of Dosimetry and Application of Ionizing Radiation, Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague, Břehová 78/7, 115 19, Prague, Czechia*

[4]*Department of Chemistry, Laboratory of Analytical Chemistry, Institute of Physics of the Czech Academy of Sciences, Na Slovance 1999/2, 182 00, Prague, Czechia*

Katerina.Krehlikova@vscht.cz

Neutron detectors play an important role in many applications related to nuclear power plants, radioactive waste depositories, spallation neutron sources, homeland security, medicine and materials analysis [1,2]. ^{3}He proportional counters represent the industry standard for neutron detection [3,4], but since 2008, the world has been facing ^{3}He shortage [2,5]. The current world supply of ^{3}He comes mostly from the decay of tritium originating from nuclear weapons programs, mostly in the U.S. and Russia. The depletion of ^{3}He reserves resulted in an abrupt increase of its price and became nearly unaffordable [6]. Therefore, there is still a high demand for alternative scintillation material, which would have potential to be mass-produced at low-cost from promptly available materials and technology [5]. High neutron detection efficiency, gamma-neutron separation and robustness for deployment are the most important properties sought after [2].

The ternary lithium manganese chloride (Li_2MnCl_4) could represent a suitable alternative for neutron detection, due to its high Li content (ca. 28.6 at%) and low density, which improves *n/γ* detection efficiency ratio in favor of neutron radiation, which is desirable in mixed radiation fields. Moreover, the red-infrared emission is favorable for the use of modern semiconductor photodetectors [6].

In this work, undoped, and Ce^{3+}, Eu^{2+} and Sm^{2+} doped Li_2MnCl_4 single crystals were grown by the vertical Bridgman method and studied from the structural, compositional, thermal and optical point of view.

Acknowledgements: The work is supported by the Operational Programme Johannes Amos Comenius financed by the European Structural and Investment Funds and the Czech Ministry of Education, Youth and Sports Project No. SENDISO - CZ.02.01.01/00/22_008/0004596. This work was supported by JSPS KAKENHI Grant Number K24KF00040. This research was conducted in the scope of the Japanese Society for Promotion of Science standard fellowship.

References:

[1] Kouzes et al., Nucl. Instrum. Methods Phys. Res., Sect. A, 784 (2015) 172–175
[2] Carturan et al., Eur. Phys. J. Plus, 129 (2014) 212
[3] Dujardin et al., IEEE Trans. Nucl. Sci., 65 (2018) 1977–1997
[4] Maslin et al., Nucl. Instrum. Methods Phys. Res., Sect. A, 1058 (2024) 168898

[5] Nagarkar et al., 2013 NSS/MIC (2013) 1–4
[6] Vaněček et al., Mater. Adv., 5 (2024) 8199–8207

Mechanical and Structural Insights into High Entropy Carbides Produced from High Entropy Alloys

Daniel Valášek[1], Dawid Kozien[2], Abdullah Jabr[3], David Salamon[1,3]

[1]*Department of Advanced Multifunctional Ceramics, CEITEC Brno University of Technology, Purkyňova 123, Brno, Czech Republic*

[2]*AGH University of Science and Technology, Faculty of Materials Science and Technology, Av. Mickiewicza 30, Krakow, Poland*

[3]*Department of Materials Science, Montanuniversitat Leoben, Franz Josef-Strasse 18, A-8700, Leoben, Austria*

daniel.valasek1@ceitec.vutbr.cz

High entropy alloys (HEA), first discovered in 2004, represent a class of advanced materials characterized by their multicomponent composition, containing at least five different elements in equimolar or near-equimolar ratios. With a mixing entropy exceeding 1.5R, these alloys exhibit exceptional properties, including enhanced strength and corrosion resistance, superior to those of traditional alloys.

Building on the discovery of HEA, high entropy ceramics (HECer) were introduced, which incorporate an additional non-metallic element into their crystal structure. The properties of HECer vary depending on the specific non-metallic element, such as oxygen, boron, nitrogen, or carbon. This provides a variety of HECer materials with distinct and enhanced properties, offering significant potential for advanced applications. For example, High Entropy Carbides (HEC) are a promising candidate for cutting tool applications thanks to their high hardness and oxidation resistance.

Preparation of HEC is a complicated process, which involves milling and sintering. Both processes have to be optimized, and different starting materials can be used to reach the desired HEC composition. As with conventional carbides, the HEC also require high temperatures and pressures for sintering and densification, which limits the methods available. The most common sintering technique for these materials is the Spark Plasma Sintering (SPS). In the presented work, vanadium, niobium, molybdenum, tantalum, and tungsten carbide synthesis was explored.

The starting materials used to produce the selected carbide were HEA and graphite. The amount of carbon was varied to explore its influence on the structure. This is a novel production route discovered only recently. Two milling methods were used for the preparation of the powder before sintering. These methods were high-energy ball milling and ball milling. The dominant sintering technique for these materials was SPS. The temperatures used for the SPS were from 1700 °C to 2000 °C and the applied pressure was 50 MPa. In addition, for selected compositions, pressure-less sintering was also tested to see if the desired materials could be prepared without the application of pressure and the presence of a vacuum.

HEA, HEC, and carbide composites were successfully prepared. Sintered samples were further characterized using scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray diffraction. The mechanical properties of the sintered materials were examined using Vickers hardness measurement and nanoindentation. Measurement of fracture toughness using the Single Edge Notched Beam (SNEB) method was also conducted.

Acknowledgements: The stay at AGH University in Krakow was supported by JECS Trust (2024 394).

A Sustainable Approach to Develop Silicon-Graphite Anode for Advanced Lithium-Ion Batteries

Lekshmi Jegan[1], Katerina Rubesova[2], Jiri Cervenka[3]

[1]*Department of Inorganic Chemistry, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*

[2]*Department of Thin Film and Nanostructures Department, Nanoscale Materials and Systems, FZU-Institute of Physics of the Czech Academy of Sciences, Na Slovance 1999/2, 182 00, Czech Republic*

jeganl@vscht.cz

The emerging era of mankind needs ample energy resources to fulfil its day-to-day requirements. However, the widespread use of fossil fuels for energy consumption has led to a looming threat of global warming and climate change. This alarming rise in environmental pollution has encouraged extensive research in developing clean energy alternatives to fossil fuels that use renewable energy technology, thereby lowering greenhouse gas emissions.

Among several clean and sustainable energy storage technologies, batteries particularly lithium-ion (Li-ion) batteries have gained limelight as a foremost source of power for application in recent years. The performance of batteries is determined mainly by its long-term performance along with high power and energy densities. Only the high-capacity battery constituents (the cathode and anode) can majorly contribute to the high energy density batteries. Silicon (Si), possessing a very high theoretical capacity (3580 mAh/g for $Li_{15}Si_4$), is considered as an outstanding electrode material for advanced LIBs [1]. However, the limited practical application of Si is accounted to its severe volume variations (>300%) during cycling and the formation of an unstable Solid Electrolyte Interface (SEI) layer on the anode surface, thereby disrupting the stability of the electrode.

In my future work, I will be focussing mainly on the SEI layer, its composition, selection of the electrolyte and additive for more defined and stable SEI layer. In addition, to obtain a stable and high-capacity anode, the addition of graphite to silicon will be also studied as potentially beneficial. Graphite, the most popular anode material in the market, having good stability can control the volume changes of silicon by acting as a buffer matrix [2]. In my previous work, the composites of silicon-graphite were studied as a suitable anode material for Li-ion battery exhibiting satisfactory performance in terms of capacity and stability. The composite complements the drawbacks of both materials, thereby delivering the targeted capacity for application in practical uses, particularly in electric vehicles.

References:

[1] Je M, Han DY, Ryu J, Park S. Constructing pure Si anodes for advanced lithium batteries. Accounts of Chemical Research. 2023 Aug 1;56(16):2213-24.
[2] Gautam M, Mishra GK, Bhawana K, Kalwar CS, Dwivedi D, Yadav A, Mitra S. Relationship between Silicon Percentage in Graphite Anode to Achieve High-Energy-Density Lithium-Ion Batteries. ACS Applied Materials & Interfaces. 2024 Aug 22;16(35):45809-20.

Synthesis and characterization of substituted $CaSnO_3$ pigments

Jiří Stibral, Žaneta Dohnalová

Department of Inorganic Technology, Faculty of Chemical Technology, University of Pardubice, Pardubice, Doubravice 41, 533 53, Czech Republic

St60060@upce.com

Inorganic mixed oxides with a perovskite structure are modern materials with interesting properties promising applications in a wide spectrum of manufacturing fields. The lattice of their ABX_3 structure is capable of hosting various substituents allowing the development of highly specific materials. They are cost-effective and easy to produce. Due to their unique internal structure, some perovskites are used as photocatalysts [1] or materials for advanced solar cells [2]. Inorganic perovskite pigments also exhibit anticorrosion properties and high reflectance effects making them ideal for use in so-called "cool pigments," which help reduce building heating caused by solar radiation [3].

In this study, $CaSn_{1-x}M_xO_3$ (x=0,1; 0,2; M=Mn, Fe, Co, Ni) perovskites were synthesized via three different methods: dry mechanoactivation, wet mechanoactivation, and precipitation method, followed by calcination at 950-1300 °C for 2 hours. The prepared pigments were then cooled and ground in an agate mortar. The resulting pigments were characterized using XRD analysis, laser diffraction, thermogravimetry, and spectrophotometric methods.

All the prepared perovskite pigments had colors ranging from grey to brown. The lowest calcination temperatures were not sufficient for complete reaction of the mixture, while higher temperatures led to the formation of a spinel phase in the samples. Pure pigments, $CaSn_{0,9}Mn_{0,1}O_3$ and $CaSn_{0,9}Fe_{0,1}O_3$, were successfully prepared by the dry milling and precipitation methods. Reflectance measurements indicated that these pigments can be classified as “cool pigments”. The precipitation method produced pigments with higher color saturation, and the narrowest particles size distribution compared to the other methods, making it the most suitable approach for future preparations.

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References:

[1] Zhang, W. F., Tang, J., & Ye, J. (2006). *Photoluminescence and photocatalytic properties of $SrSnO_3$ perovskite. Chemical Physics Letters, 418(1-3), 174–178.* doi:10.1016/j.cplett.2005.10.122.

[2] Kim, J. Y., Lee, J.-W., Jung, H. S., Shin, H., & Park, N.-G. (2020). *High-Efficiency Perovskite Solar Cells. Chemical Reviews.* doi:10.1021/acs.chemrev.0c00107.

[3] Dohnalová, Ž., Šulcová, P., & Bělina, P. (2019). *Pink NIR pigment based on Cr-doped $SrSnO_3$. Journal of Thermal Analysis and Calorimetry.* doi:10.1007/s10973-019-08522-z.

Optimization of photocurable zirconia suspensions for 3D printing

Patrik Sokola[1], Petr Ptáček[1]

[1] *Faculty of Chemistry, Brno University of Technology, Purkyňova 464/118, 612 00, Brno, Czech Republic*

xcsokola@vutbr.cz

DLP (Digital Light Processing) is a rapidly developing technology for 3D printing of ceramic materials. Traditionally, the suspension consists of a ceramic powder, a photocurable resin and a dispersant (which prevents particle agglomeration because it acts as a surfactant). A wide range of uses of 3D printed zirconia includes, for example, fuel cells or molar dental replacements [1,2].

The properties of the prepared ceramic suspension condition the quality of 3D prints and their subsequent mechanical properties. One of the most important factors describing a suspension is dynamic viscosity. Over the years, many rheological models describing the dependence of viscosity on volume filling with ceramics have been created, for example the models by Chong, Quemada, Liu or Horri [3]. In the paper presented Sokola et al. [3], they introduced a new semi-empirical model to relative viscosity ($\eta_{rel.}$) prediction in the form:

$$\eta_{rel.} = 1 + 2.5\varphi - \frac{C\varphi}{e} + C\varphi \, exp\left(\left(\frac{\varphi^2}{(\varphi - \xi\varphi_{MAX})^2}\right) - 1\right) \quad (1)$$

where φ represents volume filling of ceramics; φ_{MAX} represents maximum volume filling of ceramics, C is a fitting constant; e represents Euler number and ξ is a multiplication factor.

For two types of zirconia nanopowders (doped with 3 molar and 8 molar yttrium) and two types of photocurable medium (resin), a high correlation was found in the dependence of the relative viscosity on volume filling (R^2 > 97%) at both laboratory and elevated temperature for presented Equation (1).

In addition to determining the viscosity value, the kinetic stability of the prepared suspension is an equally important factor. All prepared suspensions from the research [3] were highly stable, which was confirmed by rheological measurements after 120 days, when the dynamic viscosity changed only within the value of the standard deviation. In addition to rheological measurements, a modern method of analysis in a centrifugal field using a LUMiSizer centrifuge was used to measure the stability of suspensions, which can not only assess the long-term stability of suspensions, but also help to set the optimal weight composition of the dispersant.

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References:

[1] SOKOLA, P. et al. Comprehensive Study of Stereolithography and Digital Light Processing Printing of Zirconia Photosensitive Suspensions. *Ceramics*. 2024, **7**(4), 1616-1638. DOI:10.3390/ceramics7040104
[2] LIAN, Q. et al. Accurate printing of a zirconia molar crown bridge using three-part auxiliary supports and ceramic mask projection stereolithography. *Ceramics International*. 2019, **45**(15), 18814-18822. DOI:10.1016/j.ceramint.2019.06.111
[3] SOKOLA, P. et al. The new rheological model for zirconia suspensions with long-term kinetic stability. *Ceramics International*. 2024, **50**(22), 46611-46620 DOI:10.1016/j.ceramint.2024.09.013

Optimization of the preparation of a new three-component clinker-free binder

Lukáš Mauermann

Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic

Lukas.Mauermann@vscht.cz

This work deals with the optimization of the preparation of a three-component (3C) binder based on calcinated clay, lime and anhydrite II. The optimization was based on the way of burning of raw components in the static kiln. The burning was performed in three ways, i.e.: (1) separately; (2) together by combining two components; (3) together by combining all three components. From the burned components and water in suitable ration were prepared mixtures (M1-M5). The compressive strength of the mixtures was assessed after 2, 7 and 28 days. Furthermore, on every selected samples of hardened binders the phase composition were determined. Based on these results was found out, that co-burning of the raw components leads to the formation of calcium aluminates and calcium silicates, which are less reactive in subsequent hydration reactions. These conclusions are supported by strength test results, which clearly show that mixtures prepared by separate burning exhibit the highest strength.

Viscosity and crystal growth in amorphous samples of $Ge_{25}Se_{75}$ chalcogenide glasses

David Vaculík[1], Jaroslav Barták[1], Petr Košťál[2], Michaela Včeláková[2]

[1]*Faculty of Chemical Technology, Department of Physical Chemistry, University of Pardubice, Studentská 573, 532 10 Pardubice, Czech Republic*

[2]*Faculty of Chemical Technology, Department of Inorganic Technology, University of Pardubice, Doubravice 41, 532 10 Pardubice, Czech Republic*

david.vaculik@student.upce.cz

Chalcogenide glasses have unique physical properties, making them a point of interest in many fields of study in recent years. They are widely used in the optical and electrotechnical industry due to their high refraction index and infrared spectrum transmittance, where the perfectly stable amorphous phase is required. Crystallization is undesirable in optical applications, but it is helpful in preparing phase-change materials for data storage. In both cases, the knowledge of crystallization kinetic is, therefore, vital.

Crystals are formed when the structural units have enough mobility. Higher mobility of structural units can be observed at higher temperatures, specifically in an undercooled melt area, which is a temperature interval limited by the temperature of glass transformation and melting temperature. To fully understand the kinetic properties of $Ge_{25}Se_{75}$ material, a series of viscosity and crystal growth rate measurements were performed. [1]

The crystal growth was observed using infrared microscopy, where the dual character of crystal shapes was observed. X-ray diffraction identified the crystals growing at lower temperatures as Ge_4Se_9 composition, similar with its structure to $GeSe_2$ [2]. At the temperature of around 380 °C, both Ge_4Se_9 and $GeSe_2$ crystals were observed. Above 380 °C, only $GeSe_2$ crystals were present, which showed that Ge_4Se_9 started to melt and reorganize into a $GeSe_2$ structure, which stayed present up to the melting point.

Measured kinetic growth data and their temperature dependence were described by an appropriate kinetic model, further enhanced using a decoupling parameter showing the fundamental relation between viscosity and kinetic growth rate. The chosen crystal growth model describes the measured data well and can be used to predict crystal growth behaviour in broad temperature intervals.

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References:

[1] J. Barták *et al.*, “Beyond the Surface: Interconnection of Viscosity, Crystal Growth, and Diffusion in Ge25Se75 Glass-Former,” *J Phys Chem B*, Oct. 2024.

[2] H. Fjellvåg, K. Kongshaug, and S. Stølen, “Crystal structure of Ge4Se9: A new germanium selenide with Se2 pairs breaking the edge-sharing GeSe4 tetrahedra in GeSe2,” *Journal of The Chemical Society-dalton Transactions - J CHEM SOC DALTON TRANS*, pp. 1043–1045, 2001.

Anomalous properties of Bi_2O_2Se monocrystals

Jan Zich[1], Antonín Sojka[1], Jiří Navrátil[1], Karel Knížek[2], Václav Holý[3], Čestmír Drašar[1]

[1]*University of Pardubice, Faculty of Chemical Technology, Studentská 573, 53210 Pardubice, Czech Republic*

[2]*Institute of Physics of the Czech Academy of Sciences, Cukrovarnická 10/112, 162 00 Prague 6, Czech Republic*

[3]*Masaryk University, Department of Condensed Matter Physics, Kotlarska 2, 61137 Brno, Czech Republic*

jan.zich@student.upce.cz

Bismuth oxyselenide (Bi_2O_2Se) is a quasi-2D semiconductor with exceptional properties, including ultra-high charge carrier mobility, making it a promising material for next-generation high-speed, low-power electronic devices. Despite its potential, several issues remain unresolved: its metallic behavior at low carrier concentrations, unconventional mobility increase with carrier density, T^2 dependent resistivity, and an unusually high out-of-plane permittivity (~150). These indicate a complex interplay of crystal structure, electronic properties, and phonon dynamics that has yet to be fully understood.

To address these gaps, we grew multiple sets of Bi_2O_2Se monocrystals and performed measurements of transport properties, IR reflectivity, Raman scattering, High-resolution X-Ray Diffraction and supported our findings with DFT calculation using WIEN2k. All phonon modes predicted by factor-group theory were observed and assigned.

In our results, we have observed two very low frequency phonons: one acoustic at ≈14 cm^{-1} (≈1.8 meV) and one optical near ≈24 cm^{-1} (≈3 meV) with both modes contributing to the anomalous low-temperature dependence of the thermal capacity and conductivity.

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Additive manufacturing of advanced and porous glass ceramic photocatalytic 3D structures for wastewater treatment

Mansi Dua[1], Akansha Mehta[1], Enrico Bernardo[2], Jozef Kraxner[1]

[1]*FunGlass, Alexander Dubcek University of Trencin, Studentska 2, 911 50, Slovakia*

[2]*Department of Industrial Engineering (DII), University of Padova, Via Marzolo 9, 35131 Padova, Italy*

mansi.dua@tnuni.sk

Additive Manufacturing (AM) is transforming the field of advanced porous materials, enabling precise control over structural design and functionality. In wastewater treatment [1], AM provides an innovative platform for fabricating tailored photocatalytic membranes with enhanced porosity and tunable surface properties. Previous studies demonstrated that recycled pharmaceutical vial glass-ceramic matrices boosted porosity, surface area, and mechanical stiffness while promoting catalytic activity [2]. The current research explores the fabrication of advanced glass-ceramic-supported photocatalytic membranes incorporating selected photocatalysts (TiO_2, Fe_2O_3, and $BiVO_4$). The waste pharmaceutical glass (60 wt%) was alkali-activated with 3M NaOH (30 wt%) and combined with 10 wt% photocatalyst material through mechanical mixing (500 rpm, 3 hours) followed by spray drying. The resulting spray-dried particles exhibited uniform spherical morphology, enhancing their processability for additive manufacturing. Stereolithography (SLA) 3D printing was employed to fabricate gyroid-structured scaffolds with well-defined porosity (70-95%), followed by sintering. The obtained 3D structures exhibited tailored bandgap energies (2.0-2.5eV), allowing efficient photocatalytic activity for organic pollutant degradation under visible light. This work highlights the synergistic potential of additive manufacturing and glass-ceramic matrices for developing next-generation photocatalytic 3D structures offering a sustainable and efficient solution for advanced wastewater treatment.

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References:

[1] M. M. Osiemo, G. M. Ogendi, and C. M'Erimba, Environmental Health Insights, 2019, 13, 117863021983698.

[2] A. Mehta et al., Journal of the European Ceramic Society, 2024, 44, 9, 5480–5489.

Silver borophosphate glasses modified with tungsten and molybdenum oxide

Tomáš Hostinský[1], Petr Mošner[1], and Ladislav Koudelka[1]

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

tomas.hostinsky@student.upce.cz

This contribution deals with study of mixed glass former effect (MGFE) and its effects in two series of borophosphate glasses of $40Ag_2O$-$20MO_3$-$(40-x)P_2O_5$-xB_2O_3 (where M = W, Mo) in a composition range of x = 0-30 mol% B_2O_3. Basic physico-chemical properties were determined, and thermal properties studied by differential thermal analysis and thermomechanical analysis. The glass structure was investigated using Raman spectroscopy and both 1D and 2D ^{11}B and ^{31}P MAS NMR spectroscopy. Behavior and oxidation states of tungsten and molybdenum throughout the series was investigated using EPR spectroscopy. The electrical properties of the glasses were obtained by using impedance spectroscopy. In both series, glass transition temperature, T_g, increases in range of 0-10 mol% B_2O_3, further additions results in slight decline of T_g values. Results from impedance spectroscopy reveal non-linear increase in DC conductivity which can be attributed to MGFE and therefore changes in the structure of the glass network. The second possible explanation of the development in DC conductivity can be attributed to the presence of reduced forms of W and Mo which can affect the resulting conductivity by the polaron hopping phenomenon. This phenomenon has already been studied and confirmed on glasses of similar systems [1]. Structure of the glasses was probed with multiple MAS NMR experiments. To get first information about the glass network, standard 1D experiments of ^{11}B and ^{31}P nuclei were performed. From obtained ^{31}P MAS NMR spectra is visible the transformation of the dominant phosphate structural units in the direction of Q^2 to Q^0 with B_2O_3 additions, and thus the shortening of the phosphate chains. Development in ^{11}B MAS NMR spectra reveals presence of both BO_4 and BO_3 structural units through the series. Interconnections between phosphate and borate species was further studied by multiple 2D correlation techniques. Namely 2D ^{31}P INADEQUATE sequence was used to probe direct trough bond connections between phosphate structure units, which confirmed shortening of phosphate chains. As next was done investigation of the P-O-B linkages using the $^{11}B(^{31}P)$ D-HMQC sequence. This experiment confirmed presence of both $P\text{-}O\text{-}B_{[4]}$ and $P\text{-}O\text{-}B_{[3]}$ structure units connection inside the glass network. And lastly 2D ^{11}B DQ-SQ sequence was used to investigation of presence and nature of B-O-B bonds. This experiment reveals presence and evolution of all three BO_3-BO_3, BO_3-BO_4 and BO_4-BO_4 pairs inside the glass network. 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.

References:

[1] A. Šantić, J. Nikolić, S. Renka, et al., *Solid State Ionics*, 2022, **375**, 115849

Preparation and dissolution of binary phosphate glasses

Tomáš Pipota[1], Delia S. Brauer[2] & Aleš Helebrant[1]
[1]*Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*
[2]*Otto Schott Institute for Materials Research, Friedrich Schiller University, Jena, 07737, Germany*

tomas.pipota@vscht.cz

The extraordinary properties of phosphate glasses have been the subject of interest over the past decades, and their solubility makes them particularly interesting for biomaterials applications. Their chemical durability, along with other properties, can be tailored to the desired application. This makes phosphate glass a truly versatile material. On the other hand, preparation of phosphate glasses can be a problematic task. Phosphate melts tend to be corrosive for most of the conventional crucible materials, which introduce impurities into the glass. Some phosphate glasses are also notoriously prone to crystallize during the preparation. Those aspects represent prominent reasons why the phosphate glasses (specifically simple, high phosphate-containing glasses) are not widely researched nowadays.

For phosphate glass-based (bio)materials application are typically multicomponent glass compositions chosen.[1] To understand the influence of the composition on the dissolution, it is important to be able to differentiate between the effects contributed by each component. This can be studied on simple, binary phosphate glasses. Additionally, using simple glass systems allow us to reveal the effect of impurities on the dissolution process of selected glasses.

This work aims to investigate the effects of modifier cations in binary phosphate glass compositions on dissolution behavior and to link those effects with structural characteristics. For this purpose, 3 series of binary phosphate glasses were prepared. Each series contains an alkaline earth cation (Ca^{2+}, Mg^{2+}, Sr^{2+}) as modifier, with the content of the respective oxide ranging from 45 to 60 mol%. Glasses were prepared by a two-step melt-quenching technique. Each series was prepared in different crucible material (silica, alumina, platinum). Compositions of prepared glasses were analysed by XRF, glassy state of all samples was tested by XRD. Dissolution studies were performed as a static test, utilizing deionized water as dissolution medium. The concentration of leached components in leachates was evaluated using ICP-OES. Dissolution kinetics parameters were computed using obtained concentration data. Results were compared with dissolution models.[2] The structure of the prepared glasses was analyzed using ^{31}P solid-state NMR and Raman spectroscopy.

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References:

1. ABOU NEEL, Ensanya A., David M. PICKUP, Sabeel P. VALAPPIL, Robert J. NEWPORT and Jonathan C. KNOWLES. Bioactive functional materials: a perspective on phosphate-based glasses. *J. Mater. Chem.* [online]. 2009, **19**(6), 690–701. ISSN 0959-9428, 1364-5501.
2. HELEBRANT, Aleš. Kinetics of corrosion of silicate glasses in aqueous solutions. *Ceramics – Silikáty*. 1997, **41**(4), 147–151

Supported catalysts with an active layer of transition metal oxides

T. Babii[1], K. Jirátová[2], J. Balabánová[2], F. Kovanda[1]

[1]*Department of Solid State Chemistry, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic*

[2] *Institute of Chemical Process Fundamentals of the CAS, Rozvojová 135, 165 02 Prague, Czech Republic*

babiit@vscht.cz

Transition metal-containing oxides are widely utilized in heterogeneous catalysis for applications such as dehydrogenation, selective oxidation, selective reduction, removal of volatile organic compounds (VOC), and electrocatalysis [1]. The performance of these catalysts depends on the preparation methods and conditions. Metal-organic frameworks (MOFs), particularly zeolitic imidazolate frameworks (ZIFs), provide a novel approach for synthesizing transition metal oxides with controlled compositions and structures. These frameworks can be deposited on stainless steel substrates to create supported catalysts with enhanced catalytic properties.

The aim of this work was the preparation of supported catalysts with catalytically active oxide coatings derived from ZIF-67, a cobalt-based MOF, deposited on stainless steel meshes and sheets. ZIF-67 was synthesized by dissolving cobalt nitrate and 2-methylimidazole in methanol. Stainless steel substrates were pre-treated through ultrasonic cleaning and acid etching to improve surface roughness and adhesion. In some cases, a SiO_2 interlayer was deposited onto the substrates via the sol-gel method to further enhance the stability of the coatings. The treated substrates were then dipped into the ZIF precursor solution for in-situ growth, followed by calcination to convert the MOF into cobalt oxide-based coatings.

The ZIF-derived coatings exhibited uniform deposition on the stainless-steel substrates, forming spinel-type cobalt oxides after calcination. Adhesion and stability were improved by surface pre-treatment and the inclusion of a SiO_2 interlayer. While cobalt was used as the primary metal in this study, future work will explore the incorporation of additional transition metals, such as nickel and manganese, into the ZIF structure to develop mixed oxide catalysts with tailored compositions and optimized catalytic performance.

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References:

[1] Bion, N.; Can, F.; Courtois, X.; Duprez D. Transition metal oxides for combustion and depollution processes. In: Védrine, J.C. (Editor), Metal Oxides in Heterogeneous Catalysis, Elsevier, **2018**, 287-353.

Investigating the Impact of Solution Composition on Bioactive Glass Behaviour: Effect of MEM, SBF15, and TRIS-Buffered SBF

Karolína Opavová, Karolína Pánová, Dana Rohanová

Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic

Karolina.Opavova@vscht.cz

Advancing the field of tissue engineering demands a deeper understanding of biomaterials' reactivity under conditions that closely mimic the human body. Bioactive glasses, known for their ability to form a strong bond with bone through the development of a hydroxyapatite layer, present particular challenges in *in vitro* testing. The reactivity of these materials is profoundly influenced by the composition of the test solution, making it critical to refine *in vitro* testing protocols to achieve more accurate and reliable results.

This study aimed to compare the bioactive behaviour of bioactive glass based on Bioglass 45S5 in Minimal Essential Medium (MEM) with data obtained using modified simulated body fluid SBF15 (with adjusted HCO_3^- ion concentration) and standardized TRIS-buffer SBF (ISO 23317:2014) for comparison. By investigating the interaction kinetics and precipitation on sample surfaces in these solutions, we seek to provide a more comprehensive understanding of how different environments affect glass reactivity. The experiments involved immersing crushed bioactive glass particles in the solutions under static-dynamic conditions over four days to better mimic physiological environments. Key analyses included weight change measurements, pH monitoring, ICP-OES, and SEM/EDS to assess changes in the material's surface and the eluate composition over time.

Results indicate that MEM exhibits distinct interaction kinetics compared to SBF15 and TRIS-buffered SBF, influencing the formation and composition precipitated layers. These findings underscore the importance of selecting appropriate test solutions for in vitro bioactivity assessments, particularly for highly reactive glass materials.

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Monitoring the Immediate Interaction of Glass Materials in Demineralized Water

Tereza Přívracká

Department of Glass and Ceramics, Faculty of Chemical Technology, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28, Czech Republic

Tereza.Privracka@vscht.cz

Glass corrosion, influenced by factors such as pH, temperature, and solution composition, plays a critical role in the performance of both specialty and utility glasses. Evidence of glass corrosion can be seen even in everyday contexts, such as surface degradation on dishwasher-cleaned glassware. The extent of corrosion ranges from slight surface matting to complete degradation. In all applications, it is essential to understand how glass interacts with a solution during the initial stages of exposure. This knowledge makes it possible to precisely modify the chemical composition of glass to meet specific requirements. A key focus is the analysis of the chemical composition of eluates from the very beginning of the interaction, which can be effectively carried out using ICP-OES.

This work aims to study the immediate dissolution of four types of glass (bioactive, sodium, potassium, and phosphate glass) in grit form under static conditions in demineralized water. The concentrations of the elements in eluates were analysed using ICP-OES, and pH measurements were performed with a glass electrode at laboratory temperature.

This study confirmed that glass reacts in an aqueous solution within the first minutes of interaction, demonstrating the rapid initiation of the glass/water reaction. To fully understand this interaction, it is essential to analyse the eluates, which will later be complemented by SEM/EDS and XRD material analyses.

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