

12<sup>TH</sup> CONFERENCE FOR YOUNG SCIENTISTS IN CERAMICS

**CYSC**

2017



12<sup>TH</sup> CONFERENCE  
FOR YOUNG SCIENTISTS IN CERAMICS

**BOOK OF ABSTRACTS**

October 18-21, 2017  
Faculty of Technology Novi Sad  
Novi Sad, Serbia



**12<sup>th</sup> CONFERENCE for  
YOUNG SCIENTISTS in CERAMICS**

**PROGRAMME  
and  
BOOK OF ABSTRACTS**

**October 18-21, 2017  
Novi Sad, Serbia**

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## **Preface**

*The 12<sup>th</sup> Conference for Young Scientists in Ceramics is organized by the Department of Materials Engineering, Faculty of Technology Novi Sad, University of Novi Sad, in cooperation with Young Ceramists Network (an initiative of the European Ceramic Society).*

*This Conference first started as the Students' Meeting back in 1998 when it was just a national meeting for Serbian PhD students. After three national, this year is going to be the ninth consecutive international conference held every second year. For several years now, the Conference has a well-earned reputation as an excellent opportunity for the promotion of the work in the field of ceramics done by early stage researchers, being MSc and PhD students or young doctors. Additionally, the young scientists will be in the position to attend sessions covering major general topics of broad interest which will be presented by experienced scientists through the invited lectures. In that way, young researchers will have a chance to participate in the active discussions with their senior colleagues who are all well-known scientists in their area of expertise. We strongly hope that the overall activities during this event will create for the young researchers a fruitful platform for finding new topics, ideas and approaches for their scientific research and an excellent opportunity for establishing connections and finding proposals for collaborations.*

*General idea behind the Conference was and will continue to be the building of the closely intertwined European scientific network by offering the platform for young scientists to meet, discuss and exchange ideas in the ever growing field of ceramics. It is our deepest belief that this approach will be beneficial for both young researchers and the European science as a whole. Therefore, we strongly appreciate that the European Ceramic Society identified the efforts and the enthusiasm we have put into this idea of creating the bridge between young researchers and we truly hope that the European Ceramic Society will support this initiative in the future. Special thanks to the JECS Trust Fund for strong financial support of the Meeting. The Conference was also recognized by the Serbian Ministry of education, science and technological development as well as by the Provincial Secretariat for Higher Education and Scientific Research and we would like to thank them for their endorsements, too. A total number of 115 presentations given by young researchers, 1 plenary talk, and 13 invited talks coming from 25 countries with multidisciplinary profiles will be presented during the conference. It should be emphasized that presented topics cover research subjects of the highest scientific interest: experimental, theoretical and applicative aspects of synthesis, processing, advanced nano/microscale and functional characterization of various types of structures and ceramic materials.*

*We wish to express our thanks to the members of the local organizing committee in Novi Sad for their effort and time during preparation of the Conference, and especially to thank our endorsers and sponsors for making this event possible.*

*Editor*

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**PROGRAMME**

**WEDNESDAY, OCTOBER 18, 2017.**

**09.00 – 11.00 h – Registration** (Conference desk)

**11.00 – 11.30 h – Oppening** (Rectorate-Amphitheater)

**11.30 – 12.15 h – PL-1 Invited lecture** (Rectorate-Amphitheater)

**Jon Binner, *United Kingdom***, Progress in manufacturing high and ultra-high temperature ceramic matrix composites

**12.15 – 13.15 h – Welcome Party** (Rectorate-Ceremonial Hall)

**13.15 – 14.00 h – IL-1 Invited lecture** (Rectorate-Amphitheater)

**German Salazar-Alvarez, *Sweden***, From synthesis to large, ordered self-assembled arrays of nanoparticles

**14.00 – 14.45 h – IL-2 Invited lecture** (Rectorate-Amphitheater)

**Markus Winterer, *Germany***, ZnO – a diva among nanoparticles

**14.45 – 15.15 h – Coffe Break** (Blue Hall & Class Room, R10)

**15.15 – 16.45 h – Section 1**

***Synthesis of ceramic powders - 1*** (Blue Hall)

- 15.15 – 15.30 h    **OA-1 A. Levis, *et al.*, Germany**  
Comparison of nanocrystalline iron oxides with different valences made by a combination of a plasma and a hot wall CVS reactors
- 15.30 – 15.45 h    **OC-2 C. Ianasi, *et al.*, Romania**  
Superparamagnetic nanocomposites obtained by using different concentration of iron III acetylacetonate in SiO<sub>2</sub>-PVA inorganic - organic hybrid system
- 15.45 – 16.00 h    **OA-3 A. Sarkar, *et al.*, Germany**  
Multicomponent equiatomic rare earth oxides
- 16.00 – 16.15 h    **OA-4 O.A. Kovalenko, *et al.*, Ukraine**  
Synthesis of Ca, Zr-doped BaTiO<sub>3</sub> nanopowder with co-precipitation method
- 16.15 – 16.30 h    **OA-5 S. Ognjanovic, *et al.*, Germany**  
Gas phase synthesis of nanocrystalline cobalt-iron oxides and their structural characterization
- 16.30 – 16.45 h    **OA-6 A. Bjelajac, *et al.*, Serbia**  
XPS study of N-doped TiO<sub>2</sub> nanotubes obtained by different heat treatment

***Ceramic processing - 1*** (Class Room, R10)

- 15.15 – 15.30 h    **OA-7 V. Nečina, *et al.*, Czech Republic**  
Preparation of spinel ceramics by electric-current assisted sintering (ECAS) with special regard to carbon contamination and its characterization
- 15.30 – 15.45 h    **OA-8 N. Omerović, *et al.*, Serbia**  
Preparation of ZnO dispersions suitable for sensors fabrication using inkjet printing
- 15.45 – 16.00 h    **OC-9 N. Busatlic, *et al.*, Bosni & Herzegovina**  
The testing of influence of metakaolin on the mechanical properties of phosphate bonded refractory materials
- 16.00 – 16.15 h    **OA-10 S. Ilic, *et al.*, Serbia**  
Sintering behavior of nanocrystalline mullite powder
- 16.15 – 16.30 h    **OA-11 D. Baranovskyi, *et al.*, Ukraine**  
Evolution of the porous structure of ceramic based on BaTiO<sub>3</sub> at different sintering rates
- 16.30 – 16.45 h    **OA-12 J.F. Gomez Perez, *et al.*, Hungary**  
Solvent exchange assessment for liquid phase exfoliated Black Phosphorus

**16.45 – 17.00 h – Coffe Break** (Blue Hall & Class Room, R10)

**17.00 – 18.15 h – Section 2**

***Ceramic processing - 2*** (Blue Hall)

- 17.00 – 17.15 h    **OC-13 H Curto, *et al.*, France**  
Processing of ceramics by additive manufacturing (stereolithography) coupled with microwave sintering
- 17.15 – 17.30 h    **OA-14 J.S. Gebauer, *et al.*, Germany**  
Printing, structure and reactive laser sintering of advanced ceramics
- 17.30 – 17.45 h    **OC-15 V. Marak, *et al.*, Czech Republic**  
Preparation of laminated ceramics via slip-casting method followed by SPS
- 17.45 – 18.00 h    **OA-16 R. Pinho, *et al.*, Portugal**  
Spark plasma texturing - an alternative sintering process for lead free piezoceramics
- 18.00 – 18.15 h    **OA-17 M. Pilch, Poland**  
Influence of nitrogen flow during sintering of bismuth manganite ceramics on grain morphology and their surface disorder
- 18.15 – 18.30 h    **OC-18 S. Umerova, *et al.*, Ukraine**  
Rheological aspects of formation thin (1 μm) smooth (Ra=20–25 nm) films of nanocrystalline BaTiO<sub>3</sub> by screen-printing method

***Electroceramics - I*** (Class Room, R10)

- 17.00 – 17.15 h **OA-19 I. Lewandowska, et al., Poland**  
Influence of cationic ordering on (Ln,Ba)CoO<sub>3</sub> perovskite properties
- 17.15 – 17.30 h **OA-20 M. Čebela, et al., Serbia**  
Multidisciplinary approach to BiFeO<sub>3</sub>: Hydrothermal synthesis and theoretical investigation of crystal structure and multiferroic properties
- 17.30 – 17.45 h **OA-21 K. Dzierzowski, et al., Poland**  
Structure and conductivity of rare earth doped lanthanum niobate
- 17.45 – 18.00 h **OC-22 A. Dzunuzovic, et al., Serbia**  
Properties of PbZr<sub>0.52</sub>Ti<sub>0.48</sub>O<sub>3</sub> - NiZnFe<sub>2</sub>O<sub>4</sub>, CoFe<sub>2</sub>O<sub>4</sub> multiferroic composites obtained by auto-combustion synthesis
- 18.00 – 18.15 h **OA-23 T. Boldizar, et al., Hungary**  
Electrical and stability investigations of layered HfTe<sub>2</sub> semimetal
- 18.15 – 18.30 h **OC-24 B. Bajac, et al., Serbia**  
Synthesis and characterization of nanostructured multiferroic thin film memristors

**THURSDAY, OCTOBER 20, 2017.**

**09.15 – 11.15 h – Section 3**

***Bioceramics – I*** (Blue Hall)

- 09.15 – 09.30 h    **OC-25 E. Jidrova, *et al.*, Czech Republic**  
Preparation of biocompatible and bioresorbable fibrous structures
- 09.30 – 09.45 h    **OA-26 E. Cichoń, *et al.*, Poland**  
New calcium phosphate bone cements
- 09.45 – 10.00 h    **OC-27 D. Don Lopez, *et al.*, Spain**  
Freeze casting and 3D printing of ceramic and polymer composites for bone tissue regeneration: bicalcium phosphates, bioactive glass and silk
- 10.00 – 10.15 h    **OA-28 M. Lasgorceix, *et al.*, Belgium**  
Micro-patterning of calcium phosphate bioceramics with femtosecond laser
- 10.15 – 10.30 h    **OA-29 P. Stastny, *et al.*, Czech Republic**  
Customized porous calcium phosphate scaffolds prepared by epoxy-based gel-casting method
- 10.30 – 10.45 h    **OC-30 M. Vilotić, *et al.*, Serbia**  
Analysis of the multifractal intrinsic nature of dental nanocomposites' surface
- 10.45 – 11.00 h    **OA-31 L. Gil Flores, *et al.*, Spain**  
TZP based-ceramic composites sintered by microwave technology for biomedical applications
- 11.00 – 11.15 h    **OA-32 M. Lukic, Serbia**  
Evolved gas analysis during sintering of HAp ceramics: influence of heating rate

***Composites*** (Class Room, R10)

- 09.15 – 09.30 h    **OC-33 E. Pietrzak, *et al.*, Poland**  
Acrylates as monomers in fabrication of oxide powders by gelcasting
- 09.30 – 09.45 h    **OC-34 A. Miletić, *et al.*, Serbia**  
Reinforcement of polylactide using silicon(IV)-oxide nanoparticles
- 09.45 – 10.00 h    **OA-35 P. Rożek, *et al.*, Poland**  
New porous geopolymer-based materials for potential sorption application
- 10.00 – 10.15 h    **OC-36 P.M. Lada, *et al.*, Poland**  
Characterization of zirconia grains in ZrO<sub>2</sub> - Ti composites

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- 10.15 – 10.30 h **OA-37 J. Zygmuntowicz, et al., Poland**  
Characterization of ceramic-metal composites obtained by gel-centrifugal slip casting methods
- 10.30 – 10.45 h **OC-38 S. Dmitrovic, et al., Serbia**  
Synthesis and characterization of spider silk-maghemite composite
- 10.45 – 11.00 h **OC-39 K.A. Nagy, et al., Hungary**  
Comparison of Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> containing elastic nanocomposite hydrogels
- 11.00 – 11.15 h **OC-40 M. Ivanovic., Serbia**  
Effect of sodium hydroxide concentration on the structure of metakaolin based geopolymers

**11.15 - 11.45 h – Coffe Break** (Faculty of Technology - Amphitheater)

**11.45 – 12.30 h – IL-3 Invited lecture** (Faculty of Technology - Amphitheater)  
**Andraz Kocjan, Slovenia**, Complexity of the relationships between microstructure, ageing and mechanical properties of 3Y-TZP ceramics

**12.30 – 13.15 h – IL-4 Invited lecture** (Faculty of Technology - Amphitheater)  
**Ákos Kukovecz, University of Szeged, Hungary**, Stories told by ceramics - authentication, forgeries and fraud analysis

**13.15 – 14.30 h – Lunch** (Faculty of Technology -Ceremonial Hall)

**14.30 – 16.45 h – Section 4**

*Engineering and refractory ceramics - 1* (Blue Hall)

- 14.30 – 15.15 h **IL-5 Invited lecture - Claus Rebholz, Cyprus**, Thermal stability of hexagonal boron nitride (h-BN) nanostructures
- 15.15 – 15.30 h **OA-41 C. Moorehead, et al., USA**  
Meso-scale microstructural flaw quantification in boron carbide using microCT
- 15.30 – 15.45 h **OA-42 D. Koziń, et al., Poland**  
Synthesis of B<sub>4</sub>C nanopowders from different precursors
- 15.45 – 16.00 h **OC-43 M. Lesniak, et al., Poland**  
Characterization of zinc-containing raw glazes: correlations between composition, structure and properties
- 16.00 – 16.15 h **OC-44 A. Glukharev, et al., Russia**  
Influence of the reduced graphene oxide (rGO) on the microstructure and properties of the ceramics ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub>



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- 16.15 – 16.30 h     **OC-45 K. Janik, et al., Poland**  
Structure and properties of the graphene and diamond rich – copper composites fabricated by the high pressure high temperature method
- 16.30 – 16.45 h     **OA-46 P. Tazbierski, et al., Poland**  
Study of the mullite synthesis by ceramic method

### ***Bioceramics - 2*** (Class Room, R10)

- 14.30 – 15.15 h     **IL-6 Invited lecture - Chantal Damia, France,**  
Processing of calcium phosphate bioceramics: how to improve osteointegration?

### ***Electroceramics - 2*** (Class Room, R10)

- 15.15 – 15.30 h     **OA-47 P. Andricevic, et al., Switzerland**  
Three-dimensionally enlarged photoelectrodes by a protogenetic inclusion of vertically aligned carbon nanotubes into CH<sub>3</sub>NH<sub>3</sub>PbBr<sub>3</sub> single crystals
- 15.30 – 15.45 h     **OA-48 A Simoes, et al., Brazil**  
Study of triboelectric devices based on ZnO nanorods and PDMS:GO composites for energy harvesting application
- 15.45 – 16.00 h     **OA-49 C. Constantinescu, et al., France**  
Laser processing of para-/ferro-electric ceramics as thin films for highly-tunable microcapacitors
- 16.00 – 16.15 h     **OA-50 L. Rossi, et al., Switzerland**  
Thermal coarsening of metal-on-oxide nanowire thin films
- 16.15 – 16.30 h     **OC-51 T Boteva, et al., Bulgaria**  
Diamond electrodes for electrochemical applications
- 16.30 – 16.45 h     **OA-52 P. Winiarz, et al., Poland**  
Selected properties of titanium doped yttrium niobate

### **16.45 – 17.00 h – Coffe Break** (Blue Hall & Class Room, R10)

### **17.00 – 18.30 h – Section 5**

#### ***Optics - 2*** (Blue Hall)

- 17.00 – 17.15 h     **OA-53 I. Vorona, et al., Ukraine**  
Solid-state reactive sintering of Yb<sup>3+</sup>:YAG and Er<sup>3+</sup>, Yb<sup>3+</sup>:YAG laser ceramics
- 17.15 – 17.30 h     **OA-54 D. Milojkov, Serbia**  
Luminescent Pr<sup>3+</sup> doped fluorapatite nanoparticles with different excitation-emission profiles
- 17.30 – 17.45 h     **OA-55 M. Stan, et al., Poland**  
Sintering and preparation of yttrium iron garnet powders

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- 17.45 – 18.00 h **OC-56 V. Radmilovic, et al., Serbia**  
Ag nanowire/Al-doped ZnO composite for transparent electrode applications
- 18.00 – 18.15 h **OC-57 O.A. Kornienko, et al., Ukraine**  
Phase relations in the La<sub>2</sub>O<sub>3</sub>-Er<sub>2</sub>O<sub>3</sub> system at 1500 °C
- 18.15 – 18.30 **OA-58 D. Kosyanov, et al., Russia**  
Micro-Raman study of 5-15 at.% Yb<sup>3+</sup>:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> optical ceramics

### *Catalysts* (Class Room, R10)

- 17.00 – 17.15 h **OA-59 P. Svera, et al., Romania**  
Precursor influence on photocatalytic performance of Cd<sub>x</sub>E<sub>1-x</sub>S compounds (E=Zn, Co)
- 17.15 – 17.30 h **OA-60 J. Zaffran, et al., Israel**  
Designing efficient catalysts for solar water splitting: Metallic doping of NiOOH material
- 17.30 – 17.45 h **OA-61 D Nicheva, et al., Bulgaria**  
Effect of the synthesis method on the catalytic activity of NiCo<sub>2</sub>O<sub>4</sub>
- 17.45 – 18.00 h **OA-62 V. Mackert, et al., Germany**  
Comparing the microstructure of dry powders and colloidal dispersions
- 18.00 – 18.15 h **OA-63 A.-M. Putz, et al., Romania**  
Ketoprofen loading capacity and the release profile in mesoporous silica prepared from mixed precursors
- 18.15 – 18.30 h **OA-64 E. Djurdjic, et al., Serbia**  
Peak assignment – short ordering at B-site in spinel ferrite

### 20.00 h – Meeting of Young Ceramicst Network

Restaurant “Fontana” – *Meeting of Young Ceramist Network of the European Ceramic Society*, Invited speakers: A. Kocijan, M. Lasgorceix and I. Stijepović

**FRIDAY, OCTOBER 21, 2017.**

**09.15 – 11.15 h – Section 6**

***Electroceramics* – 3 (Blue Hall)**

- 09.15 – 09.30 h **OA-65 D. Szalbot, *et al.*, Poland**  
Influence of calcium dopant on dielectric and electric properties of BaBi<sub>2</sub>Nb<sub>2</sub>O<sub>9</sub> ceramics
- 09.30 – 09.45 h **OA-66 N. Ilic, *et al.*, Serbia**  
BiFeO<sub>3</sub> thin films: influence of doping on structure and properties
- 09.45 – 10.00 h **OA-67 L.M. Riemer, *et al.*, Switzerland**  
Stress-induced phase transition in lead free relaxor ferroelectric composites
- 10.00 – 10.15 h **OC-68 G. Ferreira Teixeira, *et al.*, Brazil**  
Structural and photoluminescence properties of the (Ni,Zn) Fe<sub>2</sub>O<sub>4</sub>-BaTiO<sub>3</sub> multiferroic composite nanopowders
- 10.15 – 10.30 h **OC-69 I. Turcan, *et al.*, Romania**  
Novel functional properties of BaTiO<sub>3</sub>-ferrite magnetoelectric composites
- 10.30 – 10.45 h **OA-70 V.K. Veerapandiyan, *et al.*, Austria**  
Compositional tuning of B-site barium titanate solid solutions
- 10.45 – 11.00 h **OA-71 D.A. Lagunas *et al.*, Mexico/Spain**  
KNN-based materials sintered via microwave heating technology: From synthesis to sintering
- 11.00 – 11.15 h **OA-72 M. Fernandes, *et al.*, Portugal**  
Effect of sintering atmosphere in the microstructural development and dielectric properties of BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub>

***Bioceramics* - 2 (Class Room, R10)**

- 09.15 – 09.30 h **OT-73 A. Abdelghany, *et al.*, Egypt**  
Effect of gamma irradiation doses on the bioactivity of borate analogous to Hench bioglass and their glass ceramic
- 09.30 – 09.45 h **OC-74 D. Pugar, *et al.*, Croatia**  
Preparation and characterization of poly(vinyl-alcohol)/chitosan blends with ZnO nanoparticles
- 09.45 – 10.00 h **OA-75 L. Pejchalova, *et al.*, Czech Republic**  
Shaping of bioceramic hydroxyapatite scaffolds on micro level
- 10.00 – 10.15 h **OC-76 M. Stevanović, *et al.*, Serbia**  
Electrophoretically deposited bioactive hydroxyapatite/chitosan coatings loaded with gentamicin
- 10.15 – 10.30 h **OA-77 K. Krol-Morkisz, *et al.*, Poland**  
Hydroxyapatite-based thermal stabilizers for acetal resins

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- 10.30 – 10.45 h    **OA-78 I. Dinic, et al., Serbia**  
Synthesis of biocompatible upconverting nanoparticles for non-specific cell labeling
- 10.45 – 11.00 h    **OA-79 D. Haidu, et al., Romania**  
NAA, a valuable analytic method for elemental characterization for medicinal crop plants
- 11.00 – 11.15 h    **OC-80 M. Gluszek, et al., Poland**  
Rheological response of non-Newtonian composites based on silica powder and poly(propylene glycol)

**11.15 - 11.45 h – Coffe Break** (Rectorate-Amphitheater)

**11.45 – 12.30 h – IL-7 Invited lecture** (Faculty of Technology -Amphitheater)  
**Dragan Damjanovic, Switzerland,** Interfaces in ferroelectrics: Domain walls and grain boundaries

**12.30 – 13.15 h – IL-8 Invited lecture** (Faculty of Technology -Amphitheater)  
**Jean-Pierre Erauw, Belgium,** Achieving homogeneity in spark plasma sintering

**13.15 – 14.30 h – Lunch** (Faculty of Technology -Ceremonial Hall)

**14.30 – 17.00 h – Section 7**

*Engineering and refractory ceramics - 2* (Blue Hall)

- 14.30 – 15.15 h    **IL-9 Invited lecture - Michael Schneider, Austria,** High performance piezoelectric AlN MEMS resonators for precise sensing of liquid properties
- 15.15 – 16.30 h    **IL-10 Invited lecture - Goran Štefanić, Croatia,** Synthesis, characterization and applications of metastable ZrO<sub>2</sub> solid solutions

*Non-crystalline materials* (Blue Hall)

- 16.00 – 16.15 h    **OA-81 A. Alzahrani, et al., United Kingdom**  
Effect of TiO<sub>2</sub> on crystallizations of alkali alumina-silicate glasses
- 16.15 – 16.30 h    **OA-82 R. Jadach, et al., Poland**  
Structural and optical characterization of modified germanate glass system
- 16.30 – 16.45 h    **OA-83 J. Nieroda, et al., Poland**  
Preliminary investigation of amorphous metal surface for application in the power industry
- 16.45 – 17.00 h    **OA-84 P. Goj, et al., Poland**  
Molecular dynamics simulations of borosilicate glasses

**Equipment** (Class Room, R10)

14.30 – 15.15 h **Slavko Žižek, Slovenia**  
Demonstration of JEOL microscopes

**Electroceramics - 4** (Class Room, R10)

15.15 – 16.00 h **IL-11 Invited lecture - Lucjan Kozielski, Poland**, Green energy and piezoelectric

16.00 – 16.15 h **OA-85 O.A. Condurache, et al., Romania**  
Dielectric and structural characterization of ferroelectric-relaxor crossover in  $\text{Eu}_{0.01}\text{Ba}_{0.99}\text{Ti}_{1-0.0025-y}\text{Zr}_y\text{O}_3$  ceramics

16.15 – 16.30 h **OC-86 J. Guzmán Minguez, et al., Spain**  
Improving synthesis and processing of dense Sr-ferrite magnets

16.30 – 16.45 h **OC-87 P. Zachariasz, et al., Poland**  
Characterization of the superconductor-multiferroic type materials based on  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  /  $\text{YMnO}_3$

16.45 – 17.00 h **OA-88 V.A. Lukacs, et al., Romania**  
Grain size effect on dielectric properties of dense submicron  $\text{BaTiO}_3$  ceramics

**17.00 – 17.15 h – Coffe Break** (Blue Hall & Class Room, R10)

**17.15 – 18.30 h – Section 8**

**Ceramics processing - 3** (Blue Hall)

17.15 – 17.30 h **OA-89 J.U. Nandhini, et al., India**  
Thermodynamic analysis and experimental investigation of multicomponent transition metal oxide ceramics

17.30 – 17.45 h **OA-90 I. Sever, et al., Croatia**  
Stabilization of high concentrated alumina suspensions by different dispersants

17.45 – 18.00 h **OA-91 S. Ivanchenko, et al., Ukraine**  
Obtaining  $\text{BaTiO}_3$  ceramic tapes with thickness less then 500 nm by tape casting method from previously cooled suspension

18.00 – 18.15 h **OA-92 I. Primorac, et al., Croatia**  
Wear mechanisms of monolithic ceramics

18.15 – 18.30 h **OA-93 A. Gerle, et al., Poland**  
Kinetics of topochemical reaction of magnesia spinels:  $\text{Mg}(\text{Cr}_{0.5}\text{Fe}_{0.5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0.5}\text{Cr}_{0.5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0.5}\text{Fe}_{0.5})_2\text{O}_4$  solid solutions with sulphur oxides

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### *Coating & films* (Class Room, R10)

- 17.15 – 17.30 h    **OC-94 A. Miletic, *et al.*, Serbia**  
Nanocomposite Ti-Si-N coatings prepared with different rotations
- 17.30 – 17.45 h    **OA-95 L. Navarro López, *et al.*, Spain**  
Failure behaviour for thermal barrier coatings by Acoustic Emission
- 17.45 – 18.00 h    **OC-96 M. Storozhenko, *et al.*, Ukraine**  
Composite powder materials of FeNiCrB<sub>0</sub>SiC-CrB<sub>2</sub> system for plasma sprayed coatings
- 18.00 – 18.15 h    **OC-97 M. Gawęda, *et al.*, Poland**  
Cerium and phosphorus modified silicon oxycarbide layers on metallic substrates
- 18.15 – 18.30 h    **OA-98 A. Rakoczy, *et al.*, Poland**  
Investigation of yttria-stabilized zirconia coating

### **19.00 h – Social Event**

**SATURDAY, OCTOBER 21, 2017.**

**09.15 – 10.30 h – Section 9**

***Ceramic processing - 4*** (Blue Hall)

- 09.15 – 09.30 h    **OC-99 M. Nikolic, *et al.*, Serbia**  
Synthesis of SiO<sub>2</sub>/CoFe<sub>2</sub>O<sub>4</sub> multifunctional three-layered core-shell particles for invertase immobilization
- 09.30 – 09.45 h    **OT-100 R. Kusiorowski, *et al.*, Poland**  
MgO-ZrO<sub>2</sub> ceramics based on recycled magnesia-carbon bricks
- 09.45 – 10.00 h    **OA-101 M. Parfenova, *et al.*, Russia**  
3D computer model and crystallization paths for system FeO-SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub>
- 10.00 – 10.15 h    **OA-102 S. Lukic *et al.*, Germany**  
From gallium oxide to zinc modified gallium oxynitride nanoparticles as promising material for photocatalytic water splitting
- 10.15 – 10.30 h    **OC-103 A. Boros, *et al.*, Hungary**  
Synthesis of inorganic polymers from various raw materials

***Traditional ceramics - 1*** (Class Room, R10)

- 09.15 – 09.30 h    **OT-104 I.N.G. Simsek, *et al.*, Turkey**  
Effect of chamotte amount and particle size on thermal expansion behavior of ceramic sanitaryware products
- 09.30 – 09.45 h    **OT-105 A. Kovacs, *et al.*, Hungary**  
Synthesis of kaolinite nanoscrolls using solution/stirring and solvothermal methods
- 09.45 – 10.00 h    **OT-106 S. Conte *et al.*, Italy**  
High temperature viscosity of porcelain stoneware bodies
- 10.00 – 10.15 h    **OC-107 N. Slijepčević, *et al.*, Serbia**  
Characterization of solidification/ stabilization sediment treatment with native clay – pilot plant scale
- 10.15 – 10.30 h    **OT-108 K. Pasiut, *et al.*, Poland**  
The influence of addition of the zirconium oxide on some technological parameters of the raw porcelain glaze

**10.30 – 11.15 h – IL-12 Invited lecture** (Faculty of Technology- Amphitheater)  
**Kristóf Kovács, Hungary**, From structure-property relations to forensic investigations

**11.15 – 11.30 h – Coffe Break** ((Faculty of Technology - Amphitheater))

**11.30 – 12.00 h – IL-13 Invited lecture** (Faculty of Technology -Amphitheater)  
**Offer Schwartzglass, Israel**, Modeling heat dissipation in ceramic packages of integrated circuits

**12.00 – 13.00 h – Section 10**

***Traditional ceramics - 2*** (Blue Hall)

- 12.00 – 12.15 h    **OT-109 I. Balczar, et al., Hungary**  
Manufacture of alkali activated cements using air-cooled slag
- 12.15 – 12.30 h    **OT-110 H. Hullar, et al., Hungary**  
Production of slag based alkali-activated cement foams
- 12.30 – 12.45 h    **OT-111 R. Soldati, et al., Italy**  
Powder rheology of spray dried bodies for porcelain stoneware slabs
- 12.45 – 13.00 h    **OT-112 K. Zwiazek, et al., Poland**  
Investigation into influence of binding regulators on thermomechanical properties of cement free castables

***Ceramics processing - 5*** (Class Room, R10)

- 12.00 – 12.15 h    **OA-113 M. Piciorus, et al., Romania**  
Sonocatalyzed sol-gel derived nanoporous silica evaluated mainly by morpho-textural properties
- 12.15 – 12.30 h    **OA-114 T. Uhlířová, et al., Czech Republic**  
Numerical calculation of effective elastic and thermal properties of open-cell model foams based on computer-generated digital microstructures
- 12.30 – 12.45 h    **OA-115 A. Nesterovic, et al., Serbia**  
Challenges in fabrication of ferroelectric thin films suitable for use in tunable microwave devices

**13.00 – 13.15 h – Closing** (Blue Hall)



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**INVITED TALKS**

Plenary Lecture, PL-1

**PROGRESS IN MANUFACTURING HIGH AND ULTRA-HIGH  
TEMPERATURE CERAMIC MATRIX COMPOSITES**

Jon Binner, Virtudes Rubio, Andrea D'Angio, Matt Porter, William Hillman  
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There is an increasing demand for advanced materials, for aerospace and other applications, with temperature capability ranging from 1500 °C to well over 2000 °C and able to survive highly corrosive environments whilst subject to intense heat fluxes and mechanical stresses. The interaction of environmental conditions together with the requirement that dimensional stability is maintained makes the selection of suitable materials extremely challenging. This paper discusses the design, development, manufacture and testing of a new class of ceramic matrix composites based on C fibre and SiC preforms enriched with ultra-high temperature ceramic (UHTC) powders and with a matrix infiltrated by either RF- or microwave-heated chemical vapour infiltration (CVI). These composites will form of suite of materials suitable for application in severe aerospace environments.

**Keywords:** UHTCMC

Invited Lecture, IL-1

**FROM SYNTHESIS TO LARGE, ORDERED SELF-ASSEMBLED  
ARRAYS OF NANOPARTICLES**

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The assembly of semiconducting and magnetic nanoobjects into ordered superstructures, e.g., mesocrystals [1], is attractive from the scientific and technological viewpoints due to their symmetry and potential applications. Specifically, the synthesis and self-assembly of non-spherical nanoparticles has been identified as one of the major challenges, and opportunities, for tomorrow's materials.

In this talk I will present: (i) how monodisperse iron oxide nanocubes and nanospheres with average sizes between 5 and 27 nm can be synthesized by thermal decomposition and the synthesis conditions that generate nanocubes suitable for producing large ordered arrays [2], (ii) how the mesostructure and mesocrystal habit can be tuned through nanoparticle interactions and external magnetic field [3], and (iii) the self-assembly IONs followed by time-resolved SAXS experiments on levitating droplets where the acoustic levitator enabled substrate-free evaluation of reaction kinetics within

a droplet and on the liquid-air interface [4]. Several stages during droplet drying can be identified from transitions in the scattering behaviour and correlated with existing nucleation theories.

### References

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- [2] E. Wetterskog, M. Agthe, A. Mayence, J. Grins, D. Wang, S. Rana, A. Ahniyaz, G. Salazar-Alvarez, L. Bergström, *Sci. Technol. Adv. Mater.*, **15** (2014) 55010.
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Invited Lecture, IL-2

## ZINC OXIDE – A DIVA AMONG NANOPARTICLES

Markus Winterer

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Although zinc oxide (ZnO) is a simple oxide, its chemical and physical properties are quite complex. Therefore, ZnO may be perceived as a multifunctional material with applications as TCO, in LEDs, spintronics, or thermoelectrics. However, ZnO also shows some rather surprising anomalies, especially in the form of nanoparticles.

My talk will review our previous work on zinc oxide over the last twenty years. It covers gas phase synthesis, dry powder and colloidal processing and detailed structural characterization. It will describe the role of defects and doping, surface chemistry, and aging of zinc oxide nanoparticles and highlights unexpected results which can be explained by the physicochemical properties of zinc oxide.

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Invited Lecture, IL-3

**COMPLEXITY OF THE RELATIONSHIP BETWEEN  
MICROSTRUCTURE, AGEING AND MECHANICAL  
PROPERTIES OF 3Y-TZP CERAMICS**

Andraž Kocjan

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Yttria-stabilized tetragonal zirconia (3Y-TZP) is today's ceramic material of choice in dentistry owing to its improved aesthetic appearance compared to metals, excellent biocompatibility and mechanical properties. The mechanical performance and the susceptibility to controversial low-temperature degradation (LTD) or ageing process are ultimately governed by stress- and moisture-induced tetragonal-to-monoclinic ( $t-m$ ) transformation, respectively. The ageing resistance of 3Y-TZP ceramics can be improved by increasing the amount of yttria and reducing the grain size of the tetragonal grains. However, both approaches result in a trade-off, that is, the reduced transformability of the tetragonal phase ( $t-m$ ) also negatively affects the mechanical properties of the 3Y-TZP.

In the present paper, the complexity of the relationships between microstructure, ageing and mechanical properties of 3Y-TZP ceramics will be presented and discussed. It will be shown that  $t-m$  transformation not only depends on factors such as grain size and yttrium concentration but also on the internal residual stresses that evolve during sintering-related phase-partitioning and during mechanical (airborne-particle abrasion) and thermal (annealing) handling procedures, as encountered in everyday dental laboratory processing of 3Y-TZP. It will also be shown how unique microstructures, addressing the conflicting demand for fabricating ageing resistant, but still, mechanically robust 3Y-TZP ceramics can be obtained by colloidal processing and (rapid) sintering of mesoporous 3Y-TZP powder or by co-doping of commercially available, CAD/CAM machinable 3Y-TZP pre-sintered blanks.

Invited Lecture, IL-4

**STORIES TOLD BY CERAMICS – AUTHENTICATION, FORGERIES  
AND FRAUD ANALYSIS**

Ákos Kukovecz

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This talk will introduce some of the analytical instruments relevant for the investigation of ceramics in general, and historical pottery and glass in particular. Elemental analysis, electron microscopy and Raman spectroscopy are all useful methods in this field and we will review their ceramic-specific capabilities and limitations. The second part of the talk will discuss case studies related to authentication, forgery and fraud analysis in the field. The latter will include art pieces currently exhibited in top museums worldwide.

**Acknowledgement:** This research effort was partially supported by the OTKA NKFIH K 112531 grant and the “Széchenyi2020” program in the framework of GINOP-2.3.2-15-2016-00013 “Intelligent materials based on functional surfaces—from syntheses to applications” project.

Invited Lecture, IL-5

**THERMAL STABILITY OF HEXAGONAL BORON NITRIDE (h-BN)  
NANOSTRUCTURES**

Nikolaos Kostoglou, Claus Rebholz

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Boron nitride (BN) is a quite versatile inorganic compound used for a wide range of industrial applications due to a combination of unique characteristics, including high oxidation resistance, large thermal conductivity, good electrical insulation, chemical inertness, excellent lubricity, non-toxicity and environmental friendliness. Even though BN is well-known for a variety of crystalline structures, its hexagonal polymorph (i.e. h-BN) is the most stable one exhibiting a structure similar to graphite. In recent years, much attention has been drawn towards nanoscale h-BN, since improved properties (e.g. mechanical strength) are expected. In this study, an extensive series of hexagonal BN nanostructures with different structural properties were investigated for their high-temperature resistance and oxidation behavior up to 1500 °C under air-flow conditions. Both commercially available and laboratory synthesized h-BN materials were

systematically evaluated for their high-temperature properties through thermal gravimetric analysis (TGA) in combination with differential scanning calorimetry (DSC). Additional characterization techniques were employed, including nitrogen adsorption/desorption at 77 K, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy in order to investigate thoroughly their texture/porosity, structure, surface chemistry, morphology and elemental composition, respectively. The experimental results revealed that purity and specific surface area seem to have a crucial role on the thermal stability and oxidation resistance of BN materials.

Invited Lecture, IL-6

### **PROCESSING OF CALCIUM PHOSPHATE BIOCERAMICS: HOW TO IMPROVE OSTEOINTEGRATION?**

Chantal Damia, Isabelle Julien, Patricia Pascaud-Mathieu,  
Karine Beaubrun-Giry, Amandine Magnaudeix, Eric Champion

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The reconstruction of bone defects resulting from trauma, malformations, cancer or pathologies, *e.g.* osteomyelitis, remains a significant clinical issue. The current approaches include autografts, allografts or synthetic biomaterials but none of them are completely satisfactory. If bone autograft, the gold standard, contains the necessary components for the bone induction and growing (*i.e.* extracellular matrix, growth factors and osteocompetent cells), the harvesting of autologous bone tissue induces medical complications such as pain, infection, hemorrhage and increased morbidity. Furthermore, the limited amounts of autograft bone available as well as an excessive graft resorption may make their use impossible. Alternatively, allografts can be used but they can transmit infectious diseases.

First (bioinert) and second (bioactive) generation of synthetic biomaterials have been developed for the filling of bone defects for many years. Among them, calcium phosphate ceramics (CPCs), such as hydroxyapatite (HA)  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , have been used since the early 1980's. Due to a chemical composition close to the bone mineral, they are highly biocompatible and osteoconductive. When implanted, the new bone tissue grows on their surface from the host bone. However, this bone ingrowth only occurs up to about 1 cm from the bone apposition and therefore full bone repair is restricted to defects of small dimensions.

In order to stimulate osteogenesis and thus extend the applications of porous HA ceramics to the repair of large osseous defects, researchers aim to develop a new generation (the third one) of synthetic bioceramics able to induce both bone formation (*i.e.* osteoinductive materials) and angiogenesis.

We propose here to expose three ways to improve osseointegration of calcium phosphate ceramics:

- (i) the modification of the chemical composition of the material using the ability of the apatite structure to accept ion substitutions. For example, silicon is involved in bone calcification and the carbonate group leads to be closer to natural bone;
- (ii) the elaboration of ceramics with controlled macro-micro-porous architecture in order to improve the cell colonisation of scaffolds;
- (iii) the functionalisation of the ceramics surface by covalent bonding of active molecules which are able to promote osteogenesis, *e.g.* bone morphogenetic protein (BMP), and angiogenesis, *e.g.* vascular endothelial growth factor (VEGF).

Invited Lecture, IL-7

## **INTERFACES IN FERROELECTRICS: DOMAIN WALLS AND GRAIN BOUNDARIES**

Dragan Damjanovic

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Ferroelectric materials exhibit a wide range of functionalities, including piezoelectricity (used in sensors, actuators, transducers), pyroelectricity (used for infrared sensors), large dielectric permittivity (capacitors, dielectric resonators), and switchable polarization (memory applications). These functionalities can be controlled by manipulating ferroelectric domain walls. The ferroelectric domain walls are interfaces that separate regions of homogeneous polarization. Typical width of a domain wall is considered to be about 1 nm, although this depends on definition. The properties of domain walls can be and usually are qualitatively different than in the material on each side of the wall: they can be conductors (or even superconductors) in an insulating matrix, have crystal structure that is different than the bulk material, can be electrically charged, attract defects and exhibit other exotic properties. Since polarization can be switched by an electric field, the domain walls can be created and moved by the field. This offers interesting possibilities, for example to create and move a quasi-2D conducting interface in an insulating ferroelectric material, opening the possibility for a new type of ferroelectric memory devices. In this talk, I will first give an introduction to properties of ferroelectric domain walls, and then review the recent progress in the field, with an emphasis on results obtained in  $\text{BiFeO}_3$ ,  $\text{BaTiO}_3$ , and  $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$ . The grain boundaries in ferroelectric polycrystalline materials will also be addressed, in particular their interaction with the domain walls.

Invited Lecture, IL-8

### **ACHIEVING HOMOGENEITY IN SPARK PLASMA SINTERING**

Jean-Pierre Erauw, L. Boilet, V. Dupont, M. Cambier, V. Lardot, F. Cambier

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Spark Plasma Sintering has attracted a large interest in recent years owing to its ability to enable full densification of a broad range of materials in a very short processing time. Heating rates of several hundreds of Kelvin per minute are regularly reported in the literature, combined with very short holding periods at the maximum temperature. If the very fast direct electrical heating has been shown very effective to achieve high densities of both conductive and non-conductive powder compacts, the high heating rates used do not allow reaching a steady state temperature field in the experimental setup and often result in a non-uniform temperature spatial distribution which in turn impacts both the transient densification behavior as the homogeneity of the final microstructure of the sintered specimen.

In the case of non-conductive powders, it has been theoretically shown that the optimization of the tool dimensions enables to reduce the temperature gradients within the compact; in the case of conductive powders, one way to better control the temperature distribution field throughout the sintering cycle and ultimately suppress the occurrence of temperature gradients in the parts is to rely on hybrid heating, i.e. combining the direct electrical heating characteristic of the FAST process with an additional external heating source.

In the present presentation, the effectiveness of both solutions for the homogeneous densification of representative powders will be presented and discussed. Other sources of temperature heterogeneities related either to the tool setup or to peculiar material properties, will be briefly addressed as well.



Invited Lecture, IL-9

## HIGH PERFORMANCE PIEZOELECTRIC AlN MEMS RESONATORS FOR PRECISE SENSING OF LIQUIDE PROPERTIES

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The group III-V material aluminium nitride (AlN) is frequently used in micro electromechanical systems (MEMS) based sensors due to its compatibility with CMOS technology [1]. These MEMS sensors have found numerous application scenarios in modern technical systems and serve due to their miniaturized design to increase as almost invisible assistants our daily quality of living. Despite current advances, the field of continuous monitoring of liquid properties like density and viscosity has not yet been permeated by MEMS based solutions and is still reliant on sampling and expensive macroscopic measurement equipment. MEMS resonators with high quality factors generally offer excellent measurement sensitivity when operated in resonance, since variations in resonance frequency and quality factor due to changes of the surrounding medium properties can be detected very accurately. Resonators immersed in liquids on the other hand will exhibit increased mechanical damping with increased liquid viscosity and density, which in return leads to drastically reduced quality factors compared to operation in air and thus reduced sensitivity. A new class of resonance modes called the roof-tile shaped modes (see inset in Fig. 1) offer high quality factors of 10 even in highly viscous calibration standards S200 or D500 and a quality factor of up to 366 in water [2].

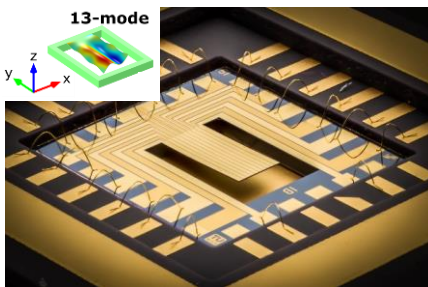


Figure 1. AlN MEMS resonator for liquid property sensing (inset shows a FEM simulation of a 13-mode)

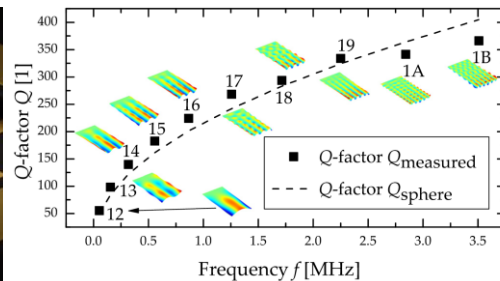


Figure 2. Q-factor of roof-tile-shaped modes in deionized water

The developed MEMS resonator platform offers high quality factors in highly viscous liquid media and is therefore a promising approach for future inexpensive liquid monitoring solutions. One exemplary device is depicted in Fig. 1. Figure 2 shows the quality factors of MEMS resonators based on pure AlN thin films in deionized water for

different orders of root tile-shaped modes [2]. The resonators can be further improved by optimized electrode designs, tailored to each mode shape individually [3]. One such optimized device is depicted in Fig. 1.

This excellent performance makes this new type of resonance mode an ideal candidate for liquid monitoring applications in portable systems or “on board” technical systems, such as automobiles or aircrafts.

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Invited Lecture, IL-10

## **SYNTHESIS, CHARACTERIZATION AND APPLICATIONS OF METASTABLE ZrO<sub>2</sub> SOLID SOLUTIONS**

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Very good physical and chemical properties (strength, toughness, chemical resistance, high melting point) makes zirconia (ZrO<sub>2</sub>) an attractive material in the production of cutting tools, engine parts, dies for wire drawing or hot extrusion, pumps, impellers, refractories, etc. ZrO<sub>2</sub> is inert in the human body and used as a biomaterial especially in dentistry.

In dependence on temperature ZrO<sub>2</sub> appears in three different polymorphs: monoclinic (*m*-ZrO<sub>2</sub>), tetragonal (*t*-ZrO<sub>2</sub>) and cubic (*c*-ZrO<sub>2</sub>). Only *m*-ZrO<sub>2</sub> polymorph is thermodynamically stable at room temperature (RT), *t*-ZrO<sub>2</sub> polymorph become stable above 1170 °C and *c*-ZrO<sub>2</sub> above 2370 °C. The *t*-ZrO<sub>2</sub>→*m*-ZrO<sub>2</sub> transition on cooling exhibits many of the characteristics of martensitic transformation in steel and could not be suppressed by rapid cooling. However, in the products of ZrO<sub>2</sub> synthesis at RT often, besides *m*-ZrO<sub>2</sub>, often appear metastable *t*-ZrO<sub>2</sub> and in some cases even metastable *c*-ZrO<sub>2</sub>. Various factors which influence the appearance of *t*-ZrO<sub>2</sub> polymorph at RT have been extensively investigated. Several proposed models emphasize the role of anionic impurities, crystallite size (surface energy), structural similarities between the precursor phase and the obtained *t*-ZrO<sub>2</sub> product, lattice strains, oxygen vacancies, etc.

In this presentation I will give a brief overview of our research related to the influence of different synthetic routes (thermal decomposition, sol-gel method, hydrothermal crystallization, mechanosynthesis) on the formation and the stability of low temperature *t*-ZrO<sub>2</sub>. A special emphasis will be given to the researches associated

with ZrO<sub>2</sub>-type solid solutions with aliovalent undersized cations and their impact on the stabilization of high-temperature ZrO<sub>2</sub> polymorphs at RT. The obtained results will be used to discuss the proposed models of stabilization of low temperature *t*-ZrO<sub>2</sub>.

Invited Lecture, IL-11

**GREEN ENERGY AND PIEZOELECTRIC ENERGY HARVESTING DEVICES**

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Self-powered and batteryless sensor network, with piezoelectric vibration energy harvesters is an idea whose time has come. Victor Hugo once remarked: “You can resist an invading army; but you cannot resist an idea whose time has come.”

All anthropogenic vibration energy is generated directly from petrol burning or from electrical energy that is generated by fossil fuels that in turn produces CO<sub>2</sub> resulting in global warming. Today’s buildings use a large portion of the grid’s energy – and then waste 30% of that energy. Creating a more intelligent building using sensor nodes geared toward reducing high-power-consuming equipment could have a huge impact on conserving energy, reducing waste, and (arguably most important) saving some money on our bills.

In modern building automation, wireless sensor networks (WSNs) and the Internet of Things (IoT) are becoming more and more prevalent in new and old buildings alike. Piezoelectric Energy Harvesters (EH) enable us to add “smarts” to existing building infrastructures without worrying about battery replacement, additional wiring or installations especially in hard-to-reach areas.

Invited Lecture, IL-12

**FROM STRUCTURE-PROPERTY RELATIONS TO FORENSIC INVESTIGATIONS**

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After a brief introduction of the Institute of Materials Engineering of the University of Pannonia the lecture will focus onto the imaging methods applied for education and research. The Institute and its predecessors provide a good example of the trinity of the role of universities: research, education, and dissemination of knowledge at

a broader scale of the society. These three roles are mutually helping and strengthening each other. Besides of research of the Institute (traditional and high-tech ceramics, bioceramics, recycling of waste as secondary raw materials, inorganic polymers, clay based nanocomposites, etc) a significant part of activities is the consulting for the industry. Failure analysis, quality control, and development of new products are mostly requested by the various automotive companies of the region. The institute is heavily involved in national and EU-level projects such as development of glass foams and foamy insulating materials, morphology development of drugs, development of water treatment technologies, processing of biowaste and polymer debris, development of self-cleaning surfaces, recovering valuable elements of red mud, development of perovskite solar cells, just to mention a few. R/D and education are supported by sophisticated instrumentation, out of which examples of the application of imaging methods (light and electron microscopy, X-ray tomography) will be presented. Examples will cover structure-property relations of high-tech ceramics, analysis of glass defects, composite failure analysis, CT-testing of electronic circuits, non-destroying analysis of ball bearings in assembled motors, testing of archaeological samples, and various forensic investigations. The institute is the winner of a huge grant leading to the construction of a new electron microscopy centre with high resolution analytical transmission and scanning electron microscopes. The instrumentation and expertise provides a good background for future co-operations at an international level.

Invited Lecture, IL-13

### **MODELING HEAT DISSIPATION IN CERAMIC PACKAGES OF INTEGRATED CIRCUITS**

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Advanced electroceramics are industrial materials that owing to their insulating qualities are useful in the production of electronic components, substrates and packages for the intergated circuit (IC's) industry and market. For IC's the advanced ceramic package should satisfy 3 main demands: minimum BOM (build of materials, cost), efficient heat transfer (functionality) and hermetic sealing (reliability). Higher digital gate densities (millions per sqmm, moore law), increasing operating frequencies (system clock > GHz) and increasing number of IC's IO's (>1000) result in increasing power consumption and heat dissipation. The excess heat is dissipated through the silicon die, ceramic package and PCB substrate. These technology trends are forcing a growing need to use special methodology and CAD (Computer Aided Design) tools to analyse the resulting heat transfer and its effect on the IC functionality.

In this presentation we will cover: IC's ceramic packages trends in respect to power penalty, methodology and modelling tool (Comsol Multiphysics) for IC's power thermal analysis

**ORAL PRESENTATIONS**

OA-1

**COMPARISON OF NANOCRYSTALLINE IRON OXIDES WITH  
DIFFERENT VALENCES MADE BY A COMBINATION OF A PLASMA  
AND A HOT WALL CVS REACTORS**

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Controlling the oxidation state of iron and the crystal structure of iron containing compounds is the key to improved materials such as iron oxide nanoparticles for cancer treatment or heterogeneous catalysis [1]. Iron oxides contain iron in different oxidation states and form different phases for one valence state ( $\alpha$ -Fe<sup>3+</sup><sub>2</sub>O<sup>2-</sup><sub>3</sub>,  $\beta$ -Fe<sup>3+</sup><sub>2</sub>O<sup>2-</sup><sub>3</sub>, etc.).

Chemical vapor synthesis (CVS) allows the reproducible production of pure nanocrystals with narrow size distribution where particle formation and growth take place in the gas phase. Through the controlled variation of synthesis parameters CVS enables the synthesis of diverse iron oxide phases. Synthesis of different phases is accomplished by varying partial pressures of oxygen, residence time in the furnace and furnace power. In this study the energy for the CVS process is supplied by a hot wall furnace and a microwave plasma. The advantage of an plasma reactor as the first CVS stage is the fast and complete precursor decomposition at low temperatures. This results in a larger process window for the hot wall reactor in the second stage [2].

We study the comparison and combination of these two methods for the synthesis of crystalline iron and iron oxide nanoparticles with desired composition using Fe(acac)<sub>3</sub> as precursor. Iron nanoparticles synthesized from Fe(acac)<sub>3</sub> form a protective carbon matrix at the particle surface [3]. The nanoparticles are examined regarding their structure, surface and valence by XRD, BET, TEM and XPS.

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OA-2

**SUPERPARAMAGNETIC NANOCOMPOSITES OBTAINED BY USING  
DIFFERENT CONCENTRATION OF IRON III ACETYLACETONATE  
IN SiO<sub>2</sub>-PVA INORGANIC – ORGANIC HYBRID SYSTEM**

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A series of three hybrid xerogel samples, with different iron oxide content (10%, 15%, 20% Fe<sub>2</sub>O<sub>3</sub>) were prepared by using one pot acid catalysed sol-gel method. SiO<sub>2</sub>-PVA hybrid matrix was obtained by mixing tetraethoxyortosilicate, TEOS, polyvinyl alcohol, PVA with molecular weight of 72,000. As iron source was used iron (III) acetylacetonate. The hybrid nanocomposite xerogel samples were heat treated at 220 °C, 260 °C and 300 °C. The fired magnetic nanocomposites samples (containing iron oxide/silica/carbon) were characterized by using, thermal analysis, IR spectroscopy, X-ray diffraction, nitrogen adsorption-desorption isotherms, Mössbauer spectroscopy and magnetic measurements. From XRD data it was observed that at lowest calcination temperature, of 220 °C, the obtained nanocomposites showed an increasing the structure ordering with iron concentration. The spinel (maghemite) phase starts to crystallize. At 260 °C XRD features of maghemite phase became more evident. At 300 °C, the  $\gamma \rightarrow \alpha$  Fe<sub>2</sub>O<sub>3</sub> temperature was reached, so, beside maghemite, hexagonal hematite phase was detected. The general crystallite average size, at 300 °C, was evaluated at ~15 nm, not showing a significant dependence of iron oxide content. The Mossbauer measurements confirm the XRD data, supplementing them with the information concerning the identity of spinel phase and data helping to distinguishing between the sources of hematite formation: the iron oxide amorphous phase or already crystallised maghemite. Based on *hysteresis loop rectangularity coefficient*, *Mr/Ms*, the room temperature magnetic measurements revealed that, without doubt, the obtained nanocomposite samples have a superparamagnetic behaviour. The nanocomposite sample, with 20% Fe<sub>2</sub>O<sub>3</sub>, fired at 300 °C, was presenting the maximum value of saturation magnetization, *Ms* = 54 emu/g, having the coercive field of 0.093 KOe, and the surface area of about 400m<sup>2</sup>g<sup>-1</sup>. The sample prepared with the lowest iron oxide concentration, of 10% Fe<sub>2</sub>O<sub>3</sub>, fired at 260 °C, presents superparamagnetic behaviour and the highest surface area of 763 m<sup>2</sup>/g, which actually is a significant result, mainly if this morpho-textural property is regarded in tandem with the saturation magnetization of ~30 emu/g.

OA-3

## MULTICOMPONENT EQUIATOMIC RARE EARTH OXIDES

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Since the last decade an increasing number of researches have focused on high entropy alloys (HEAs)[1] which crystallize into single phase structures, in spite of the presence of multiple elements (at least five) in equiatomic ratios due to the high configurational entropy of these systems. It was recently shown that the concept of high configuration entropy can be extended to cubic rocksalt type multicomponent equiatomic oxide (MEOs) systems as well, by populating a single sublattice by five transition metal (TM) cations in equiatomic amount.[2,3]

This discovery was the motivation for our work to widen the dimensionality of MEOs. In this study [4], we have demonstrated that MEOs comprising of 3–7 different rare earth cations can be successfully synthesized by nebulized spray pyrolysis. These new systems crystallized into a phase pure fluorite-type structure independent of the number of rare earth cations present. The study showed that the formation of single phase in these systems is not governed by high configurational entropy like in the above mentioned transition metal based system [2,3] but rather by the presence of Ce<sup>4+</sup> ions. Functional properties like optical band gap were investigated to perceive the potential of these materials for practical applications. The results revealed unexpected lowering of the band gap compared to the known (undoped or doped) rare earth oxides. The band gap lowering in these oxides could be associated with the high amount of oxygen vacancies present in these systems. Hence, these materials can be of interest in applications where oxygen vacancies are of importance (like catalysis, oxygen storage capacity, etc.) or lower band gap is desirable (like in photocatalysis).

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OA-4

**SYNTHESIS OF Ca, Zr-DOPED BaTiO<sub>3</sub> NANOPOWDER WITH CO-PRECIPIATION METHOD**

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Many works devoted to obtaining nanodispersed BaTiO<sub>3</sub> powder modified with different dopants for suitable properties providing. In particular, recently considerable attention has been given to obtaining modified nanopowders BaTiO<sub>3</sub> possessing relaxor behavior order to ensure reliable work of dielectrics. Generally, Ca,Zr,Mn, ,Pb and rare earth elements such as Nb,Y adds order to provide stress, inhibit grain growth and provide Pinching effect, and hence to increase dielectrics relaxor behavior.

However, there is still an issue associated with obtaining satisfactory stoichiometry of the obtained powder. From this viewpoint Ca,Zr-doped BaTiO<sub>3</sub> were prepared with co-precipitation method via multiligand complexes formation and influence of the precursor type on Ca,Zr-doped BaTiO<sub>3</sub> stoichiometry were investigated. Their stoichiometry, crystal structure was examined in order to determine preferential solubility site of Ca,Zr ions in perovskite structure. Stoichiometry Ca,Zr-modified BaTiO<sub>3</sub> will be evaluated considering different precursor type. X-ray, IR spectroscopy and X-ray fluorescence analysis were carried out to obtain the knowledge on the occupation site in the Ba<sub>1-x</sub>Ca<sub>x</sub>Ti<sub>1-y</sub>Zr<sub>y</sub>O<sub>3</sub> perovskite structure. These results proved influence complex formation on Ca,Zr-modified BaTiO<sub>3</sub> stoichiometry.

OA-5

**GAS PHASE SYNTHESIS OF NANOCRYSTALLINE COBALT-IRON OXIDES AND THEIR STRUCTURAL CHARACTERIZATION**

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Magnetic nanoparticles, and cobalt-ferrites in particular, have been studied extensively in the past decades due to their potential applications in data storage, magnetic fluids, medicine, solar cells, sensors and catalysis. The magnetic and catalytic properties of these materials depend strongly on their size, shape and the cation distribution across the tetrahedral and octahedral crystallographic sites. As a first step in studying the all-important link between structure and properties, a series of

nanocrystalline  $\text{Co}_{3-x}\text{Fe}_x\text{O}_4$  ( $x = 0, 0.4, 0.8, 1.2, 1.6, 2, 2.5$  and  $3$ ) samples have been synthesized in the gas phase by chemical vapor synthesis (CVS) and characterized by X-ray diffraction (XRD), extended X-ray absorption fine structure (EXAFS), dynamic light scattering (DLS), transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDX). The EXAFS spectra were analyzed by reverse Monte Carlo (RMC) method to solve the local structure of the synthesized nanoparticles. The influence of the chemical composition of the nanomaterials is discussed with respect to their micro-, crystal- and local- structure.

OA-6

**XPS STUDY OF N-DOPED  $\text{TiO}_2$  NANOTUBES OBTAINED BY DIFFERENT HEAT TREATMENT**

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The ~60 nm wide and ~2.5  $\mu\text{m}$  long  $\text{TiO}_2$  nanotubes were obtained by anodization of Ti films sputtered on FTO glass. For N-doping the samples were annealed in ammonia atmosphere. The effect of pre and post-annealing in air on the nature and amount of incorporated N was studied by XPS technique. It was shown that the sample annealed just in ammonia had the biggest amount of N (3.8 %) among the studied samples. Pre-annealing in air led to smaller amount of N (1.4 %). Moreover the sample that sustained post-annealing in air contained even smaller amount of N (0.7 %). The sample that was annealed in air both before and after annealing in ammonia contained bigger amount of N (0.8 %) compared to the one that was just post-annealed in air. The fitting results of the N 1s line showed that in all cases N was incorporated interstitially in  $\text{TiO}_2$  structure, whereas substitutional N and N in the form of  $\text{NO}_x$  species were removed during post-annealing in air and/or ionic sputtering.

OA-7

**PREPARATION OF SPINEL CERAMICS BY ELECTRIC-CURRENT ASSISTED SINTERING (ECAS) WITH SPECIAL REGARD TO CARBON CONTAMINATION AND ITS CHARACTERIZATION**

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This work deals with the preparation of transparent spinel ceramics by electric-current assisted sintering (ECAS), also known as spark plasma sintering (SPS), with special regard to attempts towards reducing the carbon contamination without using sinter additives (e.g. LiF). The main advantage of this technique, compared to conventional sintering, is the possibility to achieve fast densification. However, the disadvantage in the case of high heating rates is the high degree of carbon contamination, which is especially undesired when preparing transparent ceramics. In this work the samples were prepared from a commercial spinel powder (S25CRX, Baikowski, France) with a declared median crystallite size of 200 nm using commercial ECAS equipment (KCE<sup>®</sup>-FCT HP D 10-SD, FCT Systeme, Germany). Various sintering schedules were chosen that differ in maximum temperature (1300–1700 °C), heating rate (4–100 °C/min), dwell times (0–30 min), loading schedule (up to maximally 50 MPa) and sintering atmosphere (vacuum or 5 kPa argon). Also procedures using hexagonal boron nitride as a barrier against carbon penetration into the samples were tested. The samples were characterized from the viewpoint of bulk density, grain size (determined from scanning electron micrographs using stereology-based image analysis) and elastic properties (measured by the impulse excitation technique using commercial equipment RDF A23, IMCE, Belgium). The influence of sintering parameters on the resulting transparency of the samples is determined via the in-line transmittance measured by spectrophotometry (UV-VIS-NIR Spectrophotometer UV-2450, Shimadzu, Japan) in the wave length range 190–900 nm.

**Acknowledgement:** This work is part of the project “Preparation and characterization of oxide and silicate ceramics with controlled microstructure and modeling of microstructure-property relations” (GA15-18513S), supported by the Czech Science Foundation (GAČR).

OA-8

**PREPARATION OF ZnO DISPERSIONS SUITABLE FOR SENSORS  
FABRICATION USING INKJET PRINTING**

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Inkjet printing enables a digitally controlled ejection of picoliter-sized droplets and their precise positioning onto a variety of substrates. Therefore, it shows great promise to be the right deposition technology used in applications such as electronic devices and displays, sensors or solar cells. For successful implementation of the technology the key element is precise chemical formulation of inkjet ink, which should be stable, printable and functional. Basically, functional inkjet inks are dispersions of materials with semiconducting, conducting, luminescent or magnetic functionalities. In this work, stable dispersions of ZnO nanoparticles were prepared using gum arabic (GA; natural polyanionic polysaccharide with carboxylic groups as anchors) and Solsperse<sup>®</sup> 40 000 (anionic phosphated alkoxyated polymer of Lubrizol) as dispersants. After adding an appropriate amount of ZnO in an aqueous solution of dispersant (GA or Solsperse), ball milling was performed in a Retch PM 100 planetary mill at a constant milling speed of 250 rpm using a YSZ (Yttrium Stabilized Zirconia) jar and balls. The effect of different milling times (15, 30, 60, 90, 120, 150 and 180 min) on average particle sizes and on particle size distribution was investigated. Inks were prepared by diluting ZnO dispersions with water and adding 1, 2-propylene glycol and n-propanol in appropriate ratios. An ageing test of 3 weeks for both dispersions and inks was performed. Prepared inks were printed as a sensitive sensor layer using a Fuji Dimatix DMP-3000 inkjet printer. After optimization of the basic printing parameters such as jetting voltage, frequency and waveform, stable droplets of prepared inks were obtained. Sensor fabrication was conducted in two steps. In the first step, interdigitated electrodes of commercial silver were printed on PET substrate, and in the second step 10 layers of functional ZnO inks were printed on top of the electrodes. Electrical characterization and humidity response measurements revealed that sensors with Solsperse dispersant possessed better electrical properties in comparison to GA and a very good response to high humidity levels with fast response times.

OC-9

**THE TESTING OF INFLUENCE OF METAKAOLIN ON THE  
MECHANICAL PROPERTIES OF PHOSPHATE BONDED  
REFRACTORY MATERIALS**

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During the last decades it is clearly expressed the manufacturing of chemically bonded refractory materials. Studies have shown that the bond between the filler and the binder is not possible at normal temperatures when it is mixed without added binding accelerator. In this work the influence of metakaolin as an accelerator of binding of phosphate bonded refractory materials based on silica sand is examined. As binder in the refractory material was used 50% solution of mono aluminum phosphate (MAP). The prepared samples were cylindrical with diameter of 50 mm and height of 50 mm. The prepared samples were dried 24 hours at room temperature, and then annealed at temperatures of 120 °C, 400 °C, 800 °C and 1200 °C for a period of 3 hours. In thermally treated test bodies main physical properties based on which one can evaluate the achieved quality are determined.

OA-10

**SINTERING BEHAVIOR OF NANOCRYSTALLINE MULLITE  
POWDER**

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The nanocrystalline mullite powder synthesized by sol-gel method was compacted and sintered using two different sintering methods. The starting mullite powder calcined at 1200 °C was sintered at temperature ranging from 1100 to 1500 °C for 1 minute under mechanical pressure of 4 GPa. The same powder was also cold-isostatically pressed under 200 MPa to form powder compacts which were pressureless sintered at 1550 and 1600 °C for 4 h. Relative densities of pressureless sintered samples were about 90 %TD, while the maximum density of high pressure sintered samples was achieved at 1400 °C and found to be 99.7 %TD. Although application of high pressure results in very high density it also leads to phase transformation. As confirmed by XRD analysis, a part of mullite had decomposed into kyanite, high pressure polymorph of sillimanite, and

corundum. The largest amount of kyanite phase was found in samples sintered at 1400 °C. Regardless of sintering method, the microstructure of sintered samples consisted of intertwined needle-like grains. The highest Vickers microhardness of 15.4 GPa was measured in the sample with the highest density.

OA-11

**EVOLUTION OF THE POROUS STRUCTURE OF CERAMIC BASED  
ON BaTiO<sub>3</sub> AT DIFFERENT SINTERING RATES**

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The article describes the experimental investigation of the porous structure of samples based on the BaTiO<sub>3</sub> nanopowder during sintering. BaTiO<sub>3</sub> nanopowder for this research was obtained in IPM NASU by decomposition of crystalline barium titanate oxalate with a specific surface area of 1m<sup>2</sup>/g at non-isothermal conditions at a rate controlled heating in a rotating reactor. Sintering of barium titanate was carried out at heating rate from 100 to 3000 °C/hour to 800–1300 °C. The main methods of investigation of sintered samples were method of static volumetric adsorption structures of nitrogen adsorption at low temperatures, XRD, and other microscopy.

In this work nanopowder of barium titanate with a surface area of 25,6 m<sup>2</sup>/g was used. There is has been a great deal of interest in the study of the porous structure of samples based on the BaTiO<sub>3</sub> nanopowder during sintering since the important properties of this material depend on grain size and porous structure.

To optimize the sintering conditions, the powder consolidation was carried out by non-isothermal sintering with heating rate from 100 to 3000 °C/hour. Adsorption structural research of sintered samples showed that the sintering process is dependent on the heating rate.

OA-12

**SOLVENT EXCHANGE ASSESSMENT FOR LIQUID PHASE  
EXFOLIATED BLACK PHOSPHORUS**J. Gómez-Pérez<sup>1</sup>, Á. Kukovecz<sup>1,2</sup><sup>1</sup>*Department of Applied and Environmental Chemistry, University of Szeged,  
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Liquid phase exfoliation (LPE) is a common technique for the production of 2D materials. The scalability of the process is among its benefits but it also has some disadvantages as the sorption of the solvent onto the surface of the material or its oxidation during storage. In literature N-methyl-2-pyrrolidone (NMP) is the most common and effective solvent for LPE of Black Phosphorus (BP) due to the minor difference in surface energy between BP and NMP; BP has a surface energy of c.a. 60 mJ·m<sup>-2</sup> [1] and therefore NMP with a surface tension of 40.79 mNm<sup>-1</sup> has a surface energy at 20°C comparable to the one of the BP allowing an efficient exfoliation process. In a more detailed explanation polar and dispersive contributions to surface tension must be taken into account [2].

Although NMP has good characteristics that allow high concentrations of exfoliated BP (c.a. 0.4 mg·mL<sup>-1</sup>), it also has a poor volatility and wide electromagnetic absorption and therefore this phosphorene cannot be used directly either for optical investigation or the fabrication of electronic devices [3]. As expected this inconvenient is also persistent for another common solvent with good LPE efficiency (e.g. N-Cyclohexyl-2-pyrrolidone) and it is the reason why efforts have been made to exchange the exfoliated BP into other solvents. For example the authors in [4] using saturated NaOH NMP solution for the exfoliation were able to stabilize BP in water and authors in [1] were able to exchange exfoliated BP in CHP to chloroform using several centrifugation steps.

Acetone, ethanol and hexane were chosen to evaluate as receptors of the exfoliated BP in NMP due to their higher volatility. First was assessed the LPE performance of the solvents for a sonication time of 1 hour at 25°C in argon atmosphere and a sonication bath power of 120W. The exchange process was evaluated by AFM, TEM and light scattering. AFM shows that the histograms of height and diameter are comparable after the solvent exchange and the TEM confirms the presence of the few layer Black Phosphorus in the suspension.

**Keywords:** Phosphorene, exfoliated black phosphorus, solvent exchange.

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OC-13

**PROCESSING OF CERAMICS BY ADDITIVE MANUFACTURING  
(STEREOLITHOGRAPHY) COUPLED WITH MICROWAVE  
SINTERING**

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To develop a quick manufacturing of alumina and/or zirconia complex-shaped parts, stereolithography (additive manufacturing) and microwave sintering are coupled.

The stereolithography is based on layer by layer photo-polymerisation of ceramic powder slurry according to a CAD file and permits to obtain pieces with complex shape. The first part of this study focusses on the development of adapted slurries for the stereolithography (material, dry matter content, resin viscosity, ...) and on the determination of the proper printing parameters (diving time, exposure time of light, diving depth...).

The green pieces manufactured by stereolithography are sintered by using microwave/matter interactions. This process allows a quick *in-situ* debinding and sintering of the green samples. The appropriate parameters (heating rate, dwell time, sintering temperature...) are investigated for the different kinds of green samples (material, dry matter content, shape...).

Finally, the microstructural (density, grain sizes) and mechanical properties (Vickers hardness, Young's modulus, fracture toughness...) are studied to establish the relationship between the shaping and sintering parameters, and the final product properties.



OA-14

## PRINTING, STRUCTURING AND REACTIVE LASER SINTERING OF ADVANCED CERAMICS

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In recent years, inkjet printing of electronic devices is getting increased attention, due to the possibility of high throughput production of devices like transistors, biomaterials, sensors, photo catalysts, solar cells and sensors at low price [1]. Even though organic semiconductors are already widely used for printed components, partially inorganic materials are advantageous due to their high durability and their superior device performance. However, it is still necessary to investigate ceramic semiconductors as printing materials, because they are difficult to print and normally need high process temperatures excluding the use of flexible polymeric substrates.

In order to overcome these challenges we study the complete process chain creating semiconducting metal oxide structures, from producing stable inks out of CVS synthesized nanoparticles, printing and UV-laser sintering. Using an UV-laser ( $\lambda = 325$  nm,  $E_g = 3.8$  eV) most common metal oxides like  $\text{TiO}_2$  ( $E_g = 3.3$  eV),  $\text{In}_2\text{O}_3$  ( $E_g = 3.75$  eV) and  $\text{SnO}_2$  ( $E_g = 3.6$  eV) are sintered resonantly above the band gap ( $E_g$ ) [2]. This method allows to produce high temperature locally and temporally with low laser power and to create advanced ceramics out of printed nanoparticle films without damaging the substrate. Thus UV-laser sintering can be used for flexible substrates and the electrical performance of the printed device can be precisely controlled [3]. Furthermore, the stabilization of different metal oxide materials in the same dispersion media allows mixing of different nanoparticle species in the same ink. After creating mixed material structures through inkjet printing, we show with XRD and EDS that UV-laser sintering enables the creation of complex ternary metal oxide ceramics through reactive laser sintering.

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OC-15

**PREPARATION OF LAMINATED CERAMICS VIA SLIP-CASTING  
METHOD FOLLOWED BY SPS**

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In this study the technology of ceramic laminates preparation was developed. The laminates consisted from layers of pure alumina and mixture of alumina and 5 vol% nano silicon carbide. Bi- and tri-layered ceramic samples as well as monolithic standards were prepared via slip-casting route followed by SPS. Laminates and standards in form of cylinders with 48.5 mm in diameter and 15 mm in height were sintered at 1450 °C and 2 min dwell with a heating rate of 50 °C/min in vacuum under pressure of 40 MPa. This fabrication route extended durability of graphitic dies in SPS and affected physical and mechanical properties of prepared ceramics.

OA-16

**SPARK PLASMA TEXTURING – AN ALTERNATIVE SINTERING  
PROCESS FOR LEAD FREE PIEZOELECTRICS**

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Spark plasma texturing is an alternative method to densify potassium sodium niobate ((K<sub>1-x</sub>Na<sub>x</sub>)NbO<sub>3</sub>, KNN) ceramics. KNN is a lead-free piezoelectric with good electromechanical properties and a relatively high Curie temperature ( $T_C = 420$  °C). Due to these features, KNN is the most studied lead-free piezoelectric and one of the most promising systems to substitute some of the lead-based ceramics currently available in the market. However, the densification of KNN ceramics is very challenging. The high temperatures needed for sintering with the high vapour pressure of the alkalines results in secondary phases that compromise the full densification and the final stoichiometry, hence the electromechanical properties.

In order to solve KNN densification, we have used one of the most popular techniques among the field assisted sintering techniques – the spark plasma sintering (SPS). Previous reports of KNN ceramics sintered by SPS state densifications of 96 % and ameliorated dielectric properties [1,2].

In this study, we sintered KNN ceramics by an alternative SPS method called spark plasma texturing (SPT), described as an edge-free SPS [3]. Our KNN SPT ceramics present very high density (99.8 %) with an average grain size of 1.4  $\mu\text{m}$ ,  $\epsilon_r = 328$ ,  $\tan \delta = 0.05$ ,  $d_{33} = 125 \text{ pC}\cdot\text{N}^{-1}$ ,  $g_{33} = 43.1 \times 10^{-3} \text{ Vm}\cdot\text{N}^{-1}$ ,  $P_r = 46.9 \text{ }\mu\text{C}\cdot\text{cm}^{-2}$  and  $E_C = 9.4 \text{ kV}\cdot\text{cm}^{-1}$ . The comparison between SPT and SPS is established and an explanation is proposed to enlighten the phenomenon behind the densification enhancement.

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OA-17

## **INFLUENCE OF NITROGEN FLOW DURING SINTERING OF BISMUTH MANGANITE CERAMICS ON GRAIN MORPHOLOGY AND THEIR SURFACE DISORDER**

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Bismuth manganite ceramics, which consists of two phases, cubic *I23* and orthorhombic *Pbam*, were obtained previously by high temperature sintering in air. The powdered ceramics were sintered first time in gaseous nitrogen flowing around the pellets at stabilized temperature  $T_s = 870\text{--}1120 \text{ K}$  for 4 h. Grain structure and chemical composition was observed by scanning electron microscopy. We observed change in the grain shapes, *i.e.* the number and size of the hexagon-based parallelepipeds increased. The orthorhombic  $\text{BiMn}_2\text{O}_5$  phase formation was favored. The local disorder was tested by X-ray photoemission spectroscopy. Multi-component lines assigned to O 1s, Mn 2p, Bi 4f were detected. The ratio of intensities of particular components was influenced by the sintering conditions. The shape of valence band was not influenced by the sintering conditions. Electric resistivity was measured in 200–750 K range. The activation energy increased slightly when the sintering temperature increased. The ceramics shown stabile semiconductor features.

OC-18

**RHEOLOGICAL ASPECTS OF FORMATION THIN (1  $\mu\text{m}$ ) SMOOTH  
( $R_a = 25 \text{ nm}$ ) FILMS OF NANOCRYSTALLINE  $\text{BaTiO}_3$  BY  
SCREEN PRINTING METHOD**

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The development of next generation electronic devices is closely linked to cross-coupling between different functional properties. Currently multifunctional materials research is based on designing of meta-nanostructures or laminated nanocomposites for a particular set of properties and applications. Particularly, modern technologies of printed electronics for MLCC, SOFC, DSSC, electroanalytical sensors require developing of appropriate structures in form of thin layers with smooth interface between functional layers using nanosized particles. Among many methods of colloidal forming the screen-printing is more promising one due to low cost, rapidity and the possibility of obtaining smooth uniform layers with high density and different structures in one-step. The process of screen-printing is a paste falling through the mesh and forming the film on the substrate. The films quality depends on printing setting and rheological properties of the pastes. However, the pastes rheology plays a crucial role for production of high quality films because screen-printing involves tangential stress on the paste, the paste passage through the aperture of screen mesh and its recovery on a substrate. It is known that traditional screen printing pastes are generally required to be shear thinned, thixotropic over definite time scale and shear rate, stable for screen/shelf-life and low solvent evaporation. Therefore, terpineol and its derivatives are used as a solvent, ethyl cellulose as an organic binder because of their ability to form thixotropic systems with suspensions of particles of various origination. However, miniaturization trend assumes using of nanosized solid particles and therefore developing of new compositions of suspensions based on them. High chemical activity and big specific surface area of nanoparticles cause the complex rheology of the appropriate pastes. Moreover, a few times bigger polymer molecules flocculate several nanoparticles, leading to formation of rough prints. According to this fact, the polymer was plasticized with plasticizer dibutyl phthalate and the 19 pastes with three types of flow were obtained. Pronounced viscoelastic properties and the prevalence of structural bonds polymer-polymer characterized the «thixotropic» pastes because of presence of unbounded nanoparticles. “Rheopexic – pseudoplastic – thixotropic” flow of the pastes has indicated the almost full flocculation and a slight viscoelasticity due to pseudoplastic region. “Rheopexic – thixotropic” flowing type of the pastes indicated the full flocculation of  $\text{BaTiO}_3$  nanoparticles with EthCell molecules. According to the conducted analyzes and obtained results, we make three important conclusions: to obtain smooth films the polymer molecules must flocculate whole  $\text{BaTiO}_3$  nanopowder at the stage of paste preparation; the smoother surface is peculiar for films of plasticized pastes exhibiting rheopexic

properties at low shear stresses; for obtaining thin smooth layers by screen printing method the plasticized paste with “rheopexic – thixotropic” character of flow should be used.

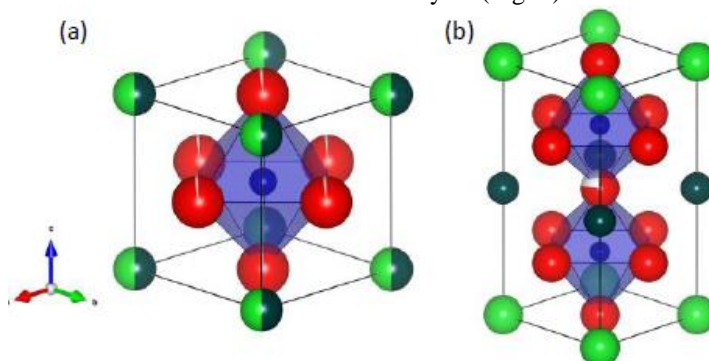
OA-19

## INFLUENCE OF CATIONIC ORDERING ON (Ln,Ba)CoO<sub>3</sub> PEROVSKITES PROPERTIES

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The cobaltites with perovskite structure are intensively studied due to their electric and magnetic properties [1–3]. One of the most promising compounds in this group is lanthanum barium cobaltite. It exhibits interesting phenomena of ordering A-site cations depending on the synthesis conditions. La<sup>3+</sup> and Ba<sup>2+</sup> ions can be both randomly distributed in A-cation sublattice as well as form layers (Fig. 1).



*Figure 1. Disordered cubic (a) and ordered tetragonal (b) perovskite structure of lanthanum barium cobaltite (figure produced by Vesta software [4] on the basis of the crystallographic data published in [5])*

Such ordering can affect transport properties of these compounds [6]. This work focuses on synthesis and characterization of materials with specific crystal structure and A-site cations ordering.

Samples will be synthesised by solid state route starting from lanthanides and cobalt oxides and barium carbonate. Different ordering of lanthanum and barium cations will be obtained by modifying sintering conditions. Structural, transport and electrical properties depending on ordering level will be characterized by X-ray diffraction method and Electrochemical Impedance Spectroscopy.

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OA-20

**MULTIDISCIPLINARY APPROACH TO BiFeO<sub>3</sub>: HYDROTHERMAL SYNTHESIS AND THEORETICAL INVESTIGATION OF CRYSTAL STRUCTURE AND MULTIFERROIC PROPERTIES**

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Bismuth ferrite (BiFeO<sub>3</sub>) has recently drawn attention due to its outstanding multi-functional properties, as well as the lead-free material. BiFeO<sub>3</sub> nanoparticles synthesized by strictly controlled hydrothermal process, with high geometric molded particles of a small size and with high degree of purity (99.74 %) were obtained. In addition, electronic and magnetic properties of BiFeO<sub>3</sub> were investigated using combination of experimental and theoretical methods. Structural analysis showed that non-annealed powder can be perfectly fitted to rhombohedral phase of *R3c* space group, where final product represents the single-phase with high crystallinity. EPR measurements suggested existence of electrons trapped by vacancies or defects, while magnetic behavior of synthesized material was investigated by SQUID. Theoretical studies were performed using a full potential linearized augmented plane-waves plus local orbital (FP(L)APW+lo) method, based on density functional theory (DFT). In addition, a structure prediction has been performed and 11 additional BiFeO<sub>3</sub> modifications have been proposed. In the next phase, an *ab initio* optimization of predicted structures has been performed and the structure of the  $\gamma$ -phase has been elucidated.

OA-21

## STRUCTURE AND CONDUCTIVITY OF RARE EARTH DOPED LANTHANUM NIOBATES

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Rare earth orthoniobates are ceramic materials belonging to ABO<sub>4</sub> group. Since 1961 these materials have been widely studied due to their interesting ferroelectric and optical properties. In the last decade, their ionic conductivity was investigated. One of the main potential applications of these materials are solid oxide fuel cells. At room temperature RENbO<sub>4</sub> has monoclinic structure while at high temperature (e.g. in LaNbO<sub>4</sub> above 500 °C) phase transition to tetragonal structure occurs [1,2]. Figure 1 shows monoclinic and tetragonal unit cells of RENbO<sub>4</sub>.

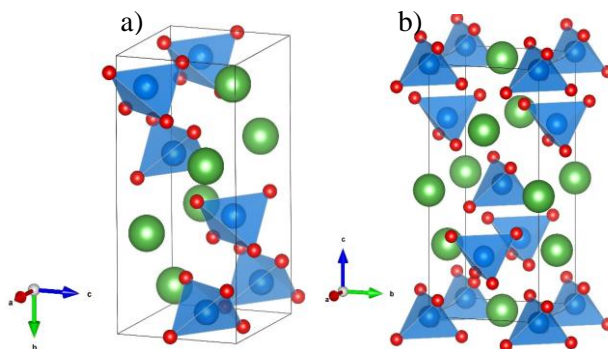


Figure 1. Unit cell of RENbO<sub>4</sub>: a) monoclinic, b) tetragonal (Nb - blue, RE - green, O - red) [3]

Properties of La<sub>1-x</sub>Pr<sub>x</sub>NbO<sub>4</sub> and 2% Ca doped La<sub>1-x</sub>Pr<sub>x</sub>NbO<sub>4</sub> samples are presented in this work. The samples were prepared by solid state reaction method. Structural and electrical properties were characterized by X-ray diffraction method and electrochemical impedance spectroscopy, respectively.

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OC-22

**PROPERTIES OF  $\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3$  -  $\text{NiZnFe}_2\text{O}_4$ ,  $\text{CoFe}_2\text{O}_4$   
MULTIFERROIC COMPOSITES OBTAINED BY AUTO-  
COMBUSTION SYNTHESIS**

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Nickel zinc ferrite (NZF), cobalt ferrite (CF) and barium titanate (BT) nanosized powders were synthesised by auto-combustion method. Multiferroic composites with the formula  $x\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3+(1-x)\text{CoFe}_2\text{O}_4$  and  $x\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3+(1-x)\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$  ( $x=0.8$  and  $0.9$ ) were prepared from nickel zinc ferrite, cobalt ferrite and barium titanate powders by mixing in planetary ball mill for 24 h. Pellets were sintered at different temperatures in order to obtain dense, two phased composites. XRD data indicated the formation of well crystallized structure of PZT and NZF/CF phase in the composite ceramics. SEM micrographs revealed a uniform grain distribution of both phases without any secondary phases.

Magnetic measurements of all sintered composites were carried out and presented in Figure 1. All the composite samples exhibit typical ferromagnetic hysteresis loop, indicating the presence of the order magnetic structure. Saturation magnetization decreases with increasing PZT content, because of non-magnetic PZT phase.

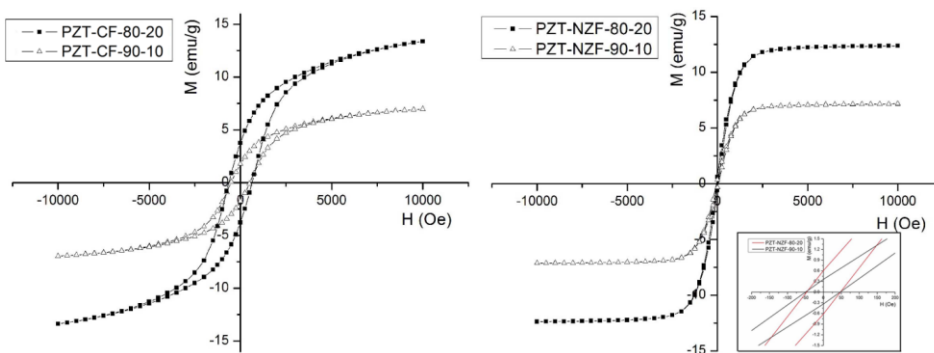


Figure 1: Magnetic measurements of the PZT-CF and PZT-NZF composite ceramics



OA-23

## ELECTRICAL AND STABILITY INVESTIGATIONS OF LAYERED HfTe<sub>2</sub> SEMIMETAL

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Hafnium-ditelluride is a semimetal that belongs to the group-IV transition metal dichalcogenides (TMDC). It features a layered structure held together by van der Waals interactions, and it can be exfoliated into thin two-dimensional sheets.

Much of the interest in the layer-structured transition-metal dichalcogenides has been in the series of structural phase transitions that are usually observed in the metallic members of this class of materials. Most of the group IV dichalcogenides are semiconductors, with a band gap between the top of the full chalcogen p band and the bottom of the transition-metal d band [1,2].

Despite the vast amount of reports on various TMDC structures, information about HfTe<sub>2</sub> nanomaterials are almost entirely missing. In this study, we investigate the changing of the electronic properties of HfTe<sub>2</sub> during oxidation by time-dependent I-V measurements and diffuse reflection spectroscopy (DRS) [3,4].

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OC-24

**SYNTHESIS AND CHARACTERIZATION OF NANOSTRUCTURED  
MULTIFERROIC THIN FILM MEMRISTORS**

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In recent years, discovery of new materials has led to development of modern technologies. In the beginning of this century multiferroic materials caught the attention of researchers due to their unique property to exhibit more than one ferroic property, and achieve coupling between them. Beside, magnetoelectric coupling, the possibility of application of multiferroic materials in field of memristive devices has shown some promising results.

This research explores the process of deposition, structural and electrical characterization of multiferroic multilayer thin films memristor devices, based on BaTiO<sub>3</sub>/NiFe<sub>2</sub>O<sub>4</sub>/BaTiO<sub>3</sub> composition. The films were obtained by spin coating of precursor sols on platinum coated silicon substrates. The multilayer thin films have well-defined layer structure composed of desired phases, with microstructure on nanometer scale and layer thickness of 40–60 nm. Electrical characterization shows weak memristive response, which is influenced by sintering temperature of the sample.

OC-25

**PREPARATION OF BIOCOMPATIBLE AND BIORESORBABLE  
FIBROUS STRUCTURES**

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Treatment of large wounds is often connected with replacement of the damaged tissue. The tissue can be substituted by a living tissue of the same kind or by an artificial materials. Nowadays, for example bones and joints are still replaced by metal implants. Beside the risk of an autoimmune reaction, there is also a problem with different mechanical properties of the implant and the natural body tissue. Therefore, new materials and technologies, which could enable production of bioresorbable implants, are searched. Because of the natural structure of the tissues, fiber materials are very

promising. A very suitable process for production of fiber materials is electrospinning. Electrospinning enables preparation of nano- or microfibers of different materials and various morphologies using electrostatic voltage. Natural (e.g. collagen) or artificial (e.g. polycaprolactone, chitosan) polymers are suitable materials for soft tissues replacements. As hard tissues implants can be used Ca-P materials such as hydroxyapatite or  $\beta$ -tricalcium phosphate.

Polymer-calcium phosphate composites were prepared in our laboratory using the electrospinning method. The aim was the preparation of a structure suitable for healing of smaller bone defects. The role of the polymer component (fibers) is to anchor the ceramic particles in the structure and to support the proliferation of the implant. The ceramic particles should enhance the mechanical properties of the implant and support bone tissue growth (ossification) of the structure.

Structures composing only from ceramic (calcium phosphate) fibers were prepared, too. Preparation of these structures requires more precise preparation of the precursors, control of the spinning parameters and following thermal treatment. These fibers should have better mechanical toughness than the composite structure, the disadvantage is their brittleness which makes worse the manipulation with the samples. Better ossification and in-growing into the living tissue of this structure is expected.

Cytotoxicity tests were provided on both kinds of samples. The influence of the composition and morphology of the fiber structure on the cell growth was assessed

OA-26

## **NEW CALCIUM PHOSPHATE BONE CEMENTS**

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Calcium phosphate bone cements (CPCs) are commonly used in medicine due to their self-setting ability, biocompatibility and bioactivity. Unfortunately, CPCs are characterized by poor injectability what makes them more difficult to use in minimally invasive internal surgical procedures. Other drawback of CPCs is their insufficient mechanical properties, especially low compressive strength and brittleness. In order to obtain better physicochemical and biological properties CPCs can be modified with different additives.

In this study, new hybrid cements based on  $\alpha$ -tricalcium phosphate ( $\alpha$ -TCP) and HAp/Chitosan granules were obtained. To improve injectability and obtain antibacterial properties sodium alginate and silver nanoparticles were applied. In the experimental part setting times, microstructure and injectability of CaP cement pastes were evaluated. The study confirmed that the 1.0 wt.% addition of sodium alginate and 0.1 wt.% silver

nanoparticles can significantly improve/enhance the surgical handiness and injectability of bone cements.

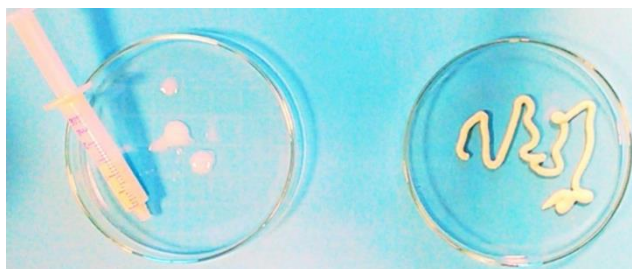


Figure 1. Prepared bone cements non-injectable without and injectable with sodium alginate

OC-27

**FREEZE CASTING AND 3D PRINTING OF CERAMIC AND POLYMER COMPOSITES FOR BONE TISSUE REGENERATION: BICALCIUM PHOSPHATES, BIOACTIVE GLASS AND SILK**

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Different kind of ceramic scaffolds were created in order to promote and enhance the bone cell growing inside of them to regenerate bone tissue. The calcium phosphates have very similar chemical composition to human bone inorganic tissue and bioactive glasses can get dissolve and later precipitate as layers of hydroxyapatite, bone's main inorganic component. All these ceramic compounds have demonstrated in previous studies their biocompatibility and their good properties as materials used for bone tissue regeneration.

In the other hand, silk is a natural biopolymer that also has already been used to enhance the growth of different human tissues and it has shown great results.

Our studies focused in getting all this materials together through the novel *freeze casting* technique. This technique is based in the controlled freezing process of an aqueous ceramic suspension that, after freezing and sublimating the water of the suspension, creates a characteristic lamellar structure that confers the scaffolds an interconnected high porosity with reasonable mechanical properties. This are needed characteristics for a scaffold to be used as a base for bone tissue regeneration inside of them. The created scaffolds were chemically, physically and biologically characterized.

It is important to highlight the very good results obtained in the *in vivo* tests. The scaffolds were implanted in a rabbit skull and the bone tissue was regenerated in only ten weeks, with a high cell regeneration rate and an obtained high vascularization of the bone tissue.

Right now, our studies are focused in trying to recreate the same scaffolds materials with a 3D printer instead with the *freeze casting* technique. After obtaining these scaffolds in both processing techniques, they will be chemically, physically and biologically characterized and compared.

OA-28

### **MICRO-PATTERNING OF CALCIUM PHOSPHATE BIOCERAMICS WITH FEMTOSECOND LASER**

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The bioactivity of synthetic bone implants is highly impacted by their surface topography, especially by the presence of micro-patterns likely to influence cell behaviour.

In this study, laser machining technology was employed in order to produce controlled regular micro-patterns on dense calcium phosphate surfaces, without any contamination. The choice of the source was directed towards a femtosecond pulsed laser, with 1030 nm wavelength and 270 fs pulse width, in order to limit the thermal impact of such a process and thus to avoid the unwanted phase transformations potentially induced by the temperature elevation. Beta tricalcium phosphate substrates with perfectly controlled micro-patterning and without any secondary phase were obtained by controlling the process parameters (laser power, scanning speed, pulse frequency). An accurate optimization of the process parameters allowed to obtain micropatterns with several complex designs. The microstructural characteristics were investigated by microscopy (optical, confocal, scanning electron) and the phase identification was performed by X-Ray Diffraction and Raman spectroscopy. This work allowed to highlight the effects of the process parameters on the patterning.

The influence of surface micro-patterning on osseous cells behaviour was highlighted *in vitro*. In particular, an elongation of the cells shape was observed along linear grooves made by laser machining, whereas cells appeared more spread on smooth surfaces with same chemical composition. These results show that linear patterning should promote cell migration. The effect of the micro-patterning design, and particularly the presence of angular shapes, on cell adhesion, is currently being evaluated *in vitro* with stem cells.

To conclude, femtosecond laser machining technique seems to provide an interesting alternative to conventional ceramic surface treatments of calcium phosphates. This technology, which allows a minimization of the thermal impact, appears promising and can now be envisaged for the surface treatment of calcium phosphate ceramics or even calcium phosphate coatings used for bone tissue engineering.

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**Keywords:** CaP ceramics; laser patterning; physico-chemical surface characterization; biological assessment

OA-29

## **CUSTOMIZES POROUS CALCIUM PHOSPHATE SCAFFOLDS PREPARED BY EPOXY-BASED GEL-CASTING METHOD**

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Porous calcium phosphate (CaP) scaffolds are used in medicine for bone regeneration in a case of a bone tissue loss for decades. Owing to specific shapes of defects there is a need for implants' individualization. We investigate a possibility of employing CAD/CAM technology and a multi axis milling of porous ceramics in the field of a preparation of customized bioceramic scaffolds.

Calcium phosphate foams with a proper structure were prepared by combination of a direct foaming method and an epoxy gel-casting. Before a heat treatment, foams had compositions of pure hydroxyapatite (HA), pure tricalcium phosphate ( $\beta$ -TCP) and mixture of both, so called biphasic calcium phosphates structure. RTG measurements after sintering were conducted in order to describe changes in phase composition during heat treatment. Rheology of prepared suspensions as well as mechanical and structure characterizations of CaP foams were investigated. A machinability of CaP foams in a different stage of the heat treatment and approaches improving machinability were

evaluated as well. Practical usage of the multi axis milling has been demonstrated at the example of mandibular defect.

OC-30

**ANALYSIS OF THE MULTIFRACTAL INTRINSIC NATURE OF  
DENTAL NANOCOMPOSITES' SURFACE**

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In this study, a multifractal approach was used for describing the complexity of three-dimensional (3-D) surface characteristics of polished dental nanocomposites. Six representative particle-reinforced polymer composites, used as restorative materials in dentistry, were investigated. The cured and polished surfaces were scanned by AFM after which an approach, based on computational algorithms for multifractal analysis was applied on AFM raw data. The generalized dimension  $D_q$  and the singularity spectrum  $f(\alpha)$  characterized the local scale properties of polished nanocomposites' surface geometry at nanometer scale. The composites had the multifractal spectrum  $f(\alpha)$  width from  $\Delta\alpha = 1.7672$  to  $\Delta\alpha = 2.5401$ , indicating their differently emphasized multifractality. It turned out that the multifractal nature is an existing dental composites' property of a real surface, which cannot be taken into account by classical surface geometrical analysis.

OA-31

**TZP BASED-CERAMIC COMPOSITES SINTERED BY MICROWAVE TECHNOLOGY FOR BIOMEDICAL APPLICATIONS**

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Statistics provided by the American Association of Oral and Maxillofacial Surgeons show that 69% of adults ages 35 to 44 have lost at least one permanent tooth to an accident, gum disease, a failed root canal or tooth decay. Most of these patients benefit greatly from dental implants and the use of biomaterials is becoming increasingly necessary.

Zirconia-based composites are commonly used for developing metal-free restorations and dental implants because of its superb mechanical properties, biocompatibility and esthetics. Therefore, the aim of the present study is to sinter zirconia doped with ceria and toughened with alumina (Ce-TZP/Al<sub>2</sub>O<sub>3</sub>) by non-conventional method; microwave sintering technology.

The resulting samples of microwave sintering are compared with the composites sintered by conventional method. Its mechanical properties, final density and microstructure have also been studied. It has been shown that the properties of the samples are the same or even better for microwave sintering, reducing production costs and processing times.

Furthermore, the studied material is compared with zirconia doped yttria that have already been sintered under the same conditions.

OA-32

**EVOLVED GAS ANALYSIS DURING SINTERING OF HAp CERAMICS: INFLUENCE OF HEATING RATE**

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With development of new synthesis methods and continual decrease of particle size of synthesized ceramic powders down to nanometer level, the question of an adequate processing step becomes more important. Even slight changes in materials chemistry, stoichiometry, evolution of structural ionic species, and defect formation will influence sintering behavior of nanoceramics. Hydroxyapatite (HAp) ceramics



synthesized by a conventional chemical precipitation method usually contain traces of carbonates incorporated in crystal lattice of HAp at the position of hydroxyl groups (A-type carbonated HAp) and/or at the position of phosphate groups (B-type carbonated HAp). It was reported that such structural species have very important practical consequences on HAp sintering behavior; however, there are no data on influence of heating rate parameter. This study focuses on analysis of release of structural carbonate and water molecules during non-isothermal sintering of HAp with different heating rates (2, 10, and 50 °C/min). Evolution of structural species was *in situ* monitored during sintering experiments. A detailed physicochemical characterization of synthesized nanopowder and sintered ceramics will be presented.

OC-33

### ACRYLATES AS MONOMERS IN FABRICATION OF OXIDE POWDERS BY GELCASTING

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One of the extensively studied forming method is gelcasting. This method allows to obtain high-quality, complex-shaped ceramic parts by means of an *in situ* polymerization, through which a macromolecular network is created to hold the ceramic particles together. The samples obtained by this method show both homogenous microstructure and high densification. The key role in this process is played by the suitable selection of organic additives such as monomers, dispersants, activators and initiators of polymerization. These substances influence on the rheological properties of ceramic suspensions and mechanical strength of green bodies.

Research on new, low toxic organic additives lead to the improvement of the properties of green bodies. Moreover, the use of environmentally friendly monomers for gelcasting opens wider application possibilities. Another advantage of application of selected additives is their ability to act as dispersing agents and monomers at the same time for selected nanopowders.

The aim of the research was the optimization and preparation of ceramic slurries based on alumina and zirconia powders. The slurries were prepared by using gelcasting process. Subsequently, the influence of organic additives (containing hydroxyl, carboxyl and acryloyl groups in a molecule) on the rheological and mechanical properties (apparent viscosity, mechanical strength) and stability (zeta potential, pH) of ceramic slurries has been investigated. Then the properties of both green and sintered elements have been measured. The microstructure of the obtained samples and additive distribution in green bodies were observed on SEM.

**Keywords:** gelcasting, alumina, acrylates, low-toxic monomers

OC-34

**REINFORCEMENT OF POLYLACTIDE USING SILICON(IV)-OXIDE NANOPARTICLES**

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Nanocomposites as advanced hybrid materials are under huge interest recent years. Nanoparticles are very promising additives for polymers because of their specific properties like high volume to specific surface area ratio, and when added in small amounts to the polymer they cause significant improvements regarding mechanical and thermal properties. One of the challenges is preparing homogenous nanocomposites because forces between particles are very strong and they often make aggregates. In this work, two different techniques for nanocomposites preparation are used. In first one, films with different amounts of silicon(IV)-oxide nanoparticles were prepared by solution casting method. Second series of nanocomposites was prepared using new technique, electrospinning, which enables production of nanofibers and with thermal treatment nanofiber-based films were obtained. Polymer matrix for all samples was polylactide, which is biobased and biocompatible polymer, with pure mechanical and thermal properties. The influence of presence of silicon(IV)-oxide nanoparticles was examined through mechanical and thermal testing of samples, and it is shown that small amount of nanoparticles cause improvement of mechanical and thermal stability of polylactide.

OA-35

**NEW POROUS GEOPOLYMER-BASED MATERIALS FOR POTENTIAL SORPTION APPLICATION**

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Geopolymers are inorganic materials consisting three-dimensional aluminosilicate framework formed by Si and Al tetrahedrons linked by oxygen bridges. Geopolymers are synthesized by alkali-activation of such raw materials as fly ash, metakaolin, red mud or blast-furnace slag. They exhibit thermal, acidic and alkaline resistance as well as relatively high compressive strength [1]. Apparent density of geopolymers is higher than 1 g/cm<sup>3</sup>. In recent years several approaches to apply geopolymers as sorption materials have been made. Lightweight geopolymers, e.g. geopolymer foams, are also relatively

new field of research and may be used as thermal insulators, catalyzer carriers, sensors or adsorbents [2]. The aim of this paper is to connect these two areas of research, i.e. to develop porous, lightweight geopolymeric material for sorption application.

Metakaolin was used as a source of silica and alumina and sodium hydroxide as an alkali-activator. Additionally expanded glass aggregates were utilized to act as porous filler. The synthesis was carried out by mixing those components with water and different ratios of starting materials and various activator contents were assessed. Then such mixtures were molded to obtain cubic samples. Molds were sealed and kept in 80 °C for 24 hours in order to enhance geopolymerization. Subsequently geopolymer samples were cured in room temperature and tested after 28 days.

Apparent density and compressive strength of obtained geopolymers were measured. Their structural characterization was conducted by X-ray diffraction and infrared spectroscopy. Microstructure of the samples was observed in a scanning electron microscope. Sorption properties were also determined.

It is possible to obtain durable lightweight (with apparent density lower than 1 g/cm<sup>3</sup>) geopolymers on the basis of expanded glass aggregates.

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OC-36

## CHARACTERIZATION OF ZIRCONIA GRAINS IN ZrO<sub>2</sub>-Ti COMPOSITES

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Zirconium oxide stabilized by 3 mol% yttrium is characterized by properties like high flexural strength, high fracture toughness, high hardness, wear resistance, low thermal conductivity and corrosion resistance in acid and alkalis. High strengths coincided with high tetragonal phase content and critical average grain size (<3 μm). One of the effects of the metallic phase addition could be a change in the matrix grain size.

In this study a pure ceramic and composite samples were prepared from the nano-size ZrO<sub>2</sub> powder stabilized by 3 mol% Y<sub>2</sub>O<sub>3</sub> and 3 vol.% Ti powder with particle size about 15 μm. Materials were formed by uniaxial pressing with the pressure 50-150 MPa.

Sintering process was conducted in an air and in an argon atmosphere at temperatures 1300°C - 1550°C with the retention time of 2 h.

The selected physical properties of the green body and sintered ZrO<sub>2</sub>-Ti composites were determined by Archimedes method. The microstructural characterization was carried out using the scanning electron microscope (SEM). Stereological analysis was made by using computer program. The stereological analysis showed the differences between the zirconia grain sizes after sintering in air and argon inert atmosphere. Additionally, the various sizes of zirconia grains in the composite samples were higher than ZrO<sub>2</sub> grains in the monolith zirconia samples. The SEM observation of the composite cross-section confirmed that the Ti particles are homogeneously distributed in analyzed areas.

OA-37

### **CHARACTERIZATION OF CERAMIC-METAL COMPOSITES OBTAINED BY GEL-CENTRIFUGAL SLIP CASTING METHOD**

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The Al<sub>2</sub>O<sub>3</sub>-Ni composites were fabricated by a novel gel-centrifugal casting method. This technique combines the consolidation of powders by the centrifugal force with the reaction of radical polymerization of an organic monomer which simultaneously proceeds within the Al<sub>2</sub>O<sub>3</sub>-Ni slurry. Composites made by gel-centrifugal casting have the shape of hollow cylinders.

Alumina matrix graded composites with nickel particles were investigated. Slurries with a 50 vol.% solid content were consolidated using centrifugation at 3000 rpm. The composite properties were characterized using X-ray diffraction and scanning electron microscopy. Microstructural characteristics were investigated from outer surface towards the inner side of the hollow cylinder. The selected physical and mechanical properties such as hardness, fracture toughness of the composites were investigated.

The microstructural observations revealed that the outer surface of all composites samples consisted of the maximum volume fraction of nickel particles in contrast to the inner part of the tube which was devoid of the metal particles.

OC-38

### **SYNTHESIS AND CHARACTERIZATION OF SPIDER SILK-MAGHEMITE COMPOSITE**

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Spider silk (SS) is a biopolymer that outperforms some of the strongest natural or man-made materials known. Fibers of *Pholcus phalangioides* spider were used as a template for obtaining SS coated with superparamagnetic maghemite nanoparticles ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> NPs) – strong composite fiber responsible in a magnetic field. The material was synthesized by one-pot precipitation technique. X-ray diffraction analysis of the sample confirmed the presence of maghemite nanocrystalline phase, and EDS measurements confirmed the presence of iron oxide in the composite. From SEM and TEM images it could be seen that the SS fibers were completely and homogeneously covered with  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> NPs and SQUID analysis were used to describe magnetic properties of the obtained composite.

OC-39

### **COMPARISON OF Al<sub>2</sub>O<sub>3</sub> AND TiO<sub>2</sub> CONTAINING ELASTIC NANOCOMPOSITE HYDRGELS**

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Hydrogels have good water-holding capacity between the cross-linked polymeric networks. Here we present two different nanocomposite hydrogels produced by *in situ* free-radical co-polymerization. The Al<sub>2</sub>O<sub>3</sub> nanoparticle containing hydrogel is expected to behave as an optical sensor with high mechanical strength according to the literature [1]. The aim of this study is to investigate the effects of replacing Al<sub>2</sub>O<sub>3</sub> nanoparticles with TiO<sub>2</sub> on the morphological properties and elastic behaviour of the hydrogel. The starting materials are N,N-dimethylacrylamide (DMAA), acrylic acid (AA), Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> nanoparticles. The nanoparticles are the cross-linking agent between the polymer chains. The hydrogel is formed on poly-methyl methacrylate (PMMA) colloidal arrays.

The produced  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> (Degussa P25) nanoparticles are characterized by Transmission Electron Microscopy and the morphology of obtained hydrogels is observed by Scanning Electron Microscopy. The colour changing ability can have several potential applications in e.g. complex, multi-stimulus responsive sensors.

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OC-40

### **EFFECT OF SODIUM HYDROXIDE CONCENTRATION ON THE STRUCTURE OF METAKAOLIN BASED GEOPOLYMERS**

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One of the potential raw materials for preparation of geopolymers is kaolin. Thermally treated kaolin-metakaolin powder was mixed with alkali activator solutions with solid to liquid ratio of 0.85. The activation of metakaolin in the alkaline solution occurred at the surface of metakaolin particles, resulting in their dissolution and release of Si<sup>4+</sup> and Al<sup>3+</sup> ions. These ions are involved in geopolymerization reaction, yielding in the formation and growth of the geopolymeric gel. This study aim is to analyse the effect of different concentration of sodium hydroxide (NaOH) (10M–16M) on geopolymerisation process. The changes were followed during four weeks every seven days. The structure and the composition of raw and obtained materials were studied by X-ray diffraction (XRD) and Fourier transforms infrared spectroscopy (FTIR). The results showed variable degree of dissolution of metakaolin and formation of solid geopolymer matrix, which is a consequences of the different concentration of NaOH. FTIR analysis revealed alumino-silicate precursor that develops via meta-phase. Vibrational line shifts were observed for Al–O, Si–O and O–H groups during the polymerization for all samples. That confirmed that the inorganic polymer was formed by cross-linking of Al and Si. The microstructures of the geopolymer samples after 28 days reveal a homogeneous matrix with dispersed platy particles associated with residual metakolin phase.

OA-41

**MESO-SCALE MICROSTRUCTURAL FLAW QUANTIFICATION IN  
BORON CARBIDE USING microCT**

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Failure in brittle materials is governed, in part, by the specific microstructure of that material, with heterogeneous microstructures generally leading to reduced properties and performance. In ceramics used for armor, an increase in compressive strength can be an indicator of improved ballistic performance. In hot-pressed boron carbide (B4C) ceramic plates, failure has been attributed to carbonaceous flakes observed on the fracture surfaces of failed specimens. However this current work shows agglomerates, in addition to carbonaceous flakes, present in boron carbide. The density and distribution of these agglomerates is believed to influence the ballistic performance. Therefore, it is of interest to improve the quantification of meso-scale microstructural agglomerates so that strategies can be developed to mitigate them. This work is focused on the characterization of dumbbell-shaped compression specimens that were machined from a single tile of pressure-aided densification (PAD) B4C, using high-resolution micro-computed tomography (microCT). Large agglomerates of non-B4C material, up to 125  $\mu\text{m}$  in diameter, were observed in addition to the typical smaller carbonaceous flakes and aluminum-based phases. Agglomerate size distribution, orientation, morphology, and spacing was quantified both parallel and perpendicular to the pressing direction. Furthermore, the role of such agglomerates during compression was further investigated using partially loaded specimens in order to inform future processing refinement and improve ballistic performance.

OA-42

**SYNTHESIS OF B<sub>4</sub>C NANOPOWDERS FROM DIFFERENT  
PRECURSORS**

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In this study, nanopowders of boron carbide (B<sub>4</sub>C) were produced using two different methods. In the first method ammonium borate prepared by dissolving of boric acid in ammonia solution at 50°C was used as a precursor. Sources of carbon were suspensions of graphene, soot and active carbon powders in isopropyl alcohol. Both solutions were mixed together and then liquid was evaporated in a drier.

In the second method water solutions of some organic substances as triton X-100, glucose, glycerin and polyvinyl alcohol were used as starting materials. In such solutions boric acid was dissolved and then they were dried overnight at 90 °C to form powders. To decompose organic phases the dried powders were calcined in air at 700 °C for 2 h.

All powders were placed into graphite crucibles and then heat treated at temperature from 1300 to 1500 °C for 1h under argon flow. The final powders were characterized by the X-ray diffraction (XRD) and scanning electron microscopy (SEM) with X-ray energy dispersive spectroscopy (EDS). The results show that morphology of the B<sub>4</sub>C powders slightly depends on temperature of the synthesis and strongly depends on carbon source.

OC-43

**CHARACTERIZATION OF ZINC-CONTAINING RAW GLAZES:  
CORRELATIONS BETWEEN COMPOSITION, STRUCTURE  
AND PROPERTIES**

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The amorphous phase in ceramic glazes is usually aluminosilicate glass, which is formed with various oxides [1]. The amphoteric properties of Zn<sup>2+</sup> ions allow them to be glass-forming ions (Zn<sup>2+</sup> ions exist in a tetrahedral coordination) or be modifying ions (Zn<sup>2+</sup> ions exist in an octahedral coordination) [2,3]. In view of these facts, one might expect that the introduction of zinc ions (Zn<sup>2+</sup>) would result in significant changes in the chemical and physical properties of aluminosilicate ceramic glazes.



In this work, we examined a number of glazes from  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaO-MgO-K}_2\text{O-Na}_2\text{O-ZnO}$  system, in which the variable is zinc oxide and the share of the silicon oxide to alumina, at a constant content of other components ( $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ). Glazes were applied to the ceramic body and fired under identical conditions, i.e. at 1230 °C in a total time of 14 hours. For all glazes, structural studies (XRD, FTIR, MAS-NMR) were performed. The scanning electron microscope (SEM-EDS) was done in order to determine the state of the microstructure of glazes. The obtained data on the structure and microstructure of glazes were correlated with results of thermal tests (DTA, DL, HSM). It allowed to define the properties of zinc-aluminosilicate ceramic glazes at high temperature. In this way, the research allowed to determine the impact of molar ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3$  and variable content of ZnO on the structure and phase composition of glazes and the nature, type of formed crystalline phases.

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OC-44

## **INFLUENCE OF THE REDUCED GRAPHENE OXIDE (rGO) ON THE MICROSTRUCTURE AND PROPERTIES OF $\text{ZrO}_2\text{-Y}_2\text{O}_3$ CERAMICS**

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Nowadays, the development of the novel nanoceramic materials with unique characteristics is one of the major directions in the material science. The use of nanosized precursors for such materials manufacturing makes it possible to design their chemical, physical and mechanical properties. Powder particle properties (i.e. particle size distribution, mean size, agglomeration degree etc) strongly affect on the final microstructure and properties of the ceramics. Here the control of grain growth during ceramics sintering is regarded critical to achieve nanoceramics. In some studies it has been shown that graphene (Gr) and reduced graphene oxide (rGO) addition can act as effective grain growth retarding agent in metal-matrix composites, aluminum, nickel and copper, in particular. Thus the aim of the present work was the manufacturing of rGO-zirconia based ceramics nanocomposites and the investigation of the effect graphene addition on microstructure, phase composition and conductivity of the ceramic-matrix composites.  $9\text{Y}_2\text{O}_3\text{-91ZrO}_2$  (mol%) was chosen for the investigation (YSZ). Reduced

graphene oxide was synthesized via Hammers method with following thermal exfoliation. Precursor powder YSZ was synthesized by the sol-gel synthesis in a variation of reverse co-precipitation of inorganic salts. Based on rGO characteristics and particle size distribution the amount of rGO addition was calculated. Nanocomposite ceramic material YSZ+rGO was isostatically compacted and annealed at 1550 °C for 3 hours on air. Via SEM, XRD, Raman spectroscopy and impedance spectroscopy it was shown that the small addition of rGO (0.25 wt.%) to ceramics significantly improves the microstructure of the final composite with no change in electrical conductivity. The increase of rGO content up to 2.5 wt.% induces cracks formation and porosity increase.

OC-45

**STRUCTURE AND PROPERTIES OF THE GRAPHENE AND  
DIAMOND RICH - COPPER COMPOSITES FABRICATED BY THE  
HIGH PRESSURE HIGH TEMPERATURE METHOD****K. Janik<sup>1</sup>, T. Czeppe<sup>1</sup>, L. Jaworska<sup>2</sup>, P. Figiel<sup>2</sup>**<sup>1</sup>*Institute of Metallurgy and Materials Sciences Polish Academy of Sciences,  
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Composites were fabricated by high-pressure, high-temperature (HP-HT) sintering method. The fabricated compositions of composite samples contained 20 vol.% of copper powder and 80 vol.% of graphene or artificial diamond. The Bridgman type of equipment was used. In the process the similar parameters of sintering were applied: 7.7 GPa pressure and 1050 °C temperature. The powders were preliminary carefully mixed by using ultrasonic vibration and were homogenised with the agate mortar. The process of sintering consisted of the following steps: 90 s of heating, 60 s of sintering and 5 s of cooling. As a result samples 13.8–15.4 mm in diameter and 4.0–4.7 mm in thickness were achieved. In case of consolidations of the powders copper-diamond composites was excellent, but the graphene-copper composites content was dependent on the powder fraction. The larger fraction (smaller powders dimensions) caused better consolidation of the samples. To determine the influence of the sintering process on the chemical character of the graphene addition the Raman spectroscopy was used. The microstructure of the samples was investigated with light, scanning (SEM) and transmission (TEM) electron microscopes. Using Archimedes method the density of the samples was determined and compared to the theoretical density of the powders. Thermal properties of the composites like heat capacity and high temperature thermal stability were determined by the DSC method. Also thermo-mechanical analysis (TMA) was used to determine coefficients of thermal expansion as well as the evolution of the elastic and damping properties.

OA-46

## **STUDY OF THE MULLITE SYNTHESIS BY CERAMIC METHOD**

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Synthetic mullite is an important product used in various branches of engineering and technology due to the unique combination of properties such as good mechanical strength, excellent thermal shock resistance, high creep resistance, low coefficient of thermal expansion, and good chemical stability at elevated temperature. An important feature of mullite is very good thermal shock resistance as a consequence of low thermal expansion coefficient. From the quantitative participation of mullite in the material such as ceramic depends e.g. porcelain whiteness, chemical resistance, mechanical strength over long periods of use as well as the deformation resistance of refractory materials. The above characteristics of mullite depend on the properties of the raw materials used including: chemical form, purity, grain size, production methods and synthesis temperature.

The results of ceramic preparation of synthetic mullite with stoichiometric starting material ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) obtained from  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  oxides were reported. The products prepared under varying conditions were tested with regard to thermal quantitative phase composition (XRD) and microstructure (SEM). An interpretation of physical characteristics depending on the properties of the raw materials and the temperature of the synthesis has been presented.

OA-47

**THREE-Dimensionally ENLARGED PHOTOELECTRODES BY A PROTOGENETIC INCLUSION OF VERTICALLY ALIGNED CARBON NANOTUBES INTO CH<sub>3</sub>NH<sub>3</sub>PbBr<sub>3</sub> SINGLE CRYSTALS**

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We demonstrate that single crystals of methylammonium lead bromide (MAPbBr<sub>3</sub>) could be grown directly on vertically aligned carbon nanotube (VACNT) forests. The fast-growing MAPbBr<sub>3</sub> single crystals engulfed the protogenetic inclusions in the form of individual CNTs, thus resulting in a three-dimensionally enlarged photosensitive interface. Photodetector devices were obtained, detecting low light intensities (~20 nW) from UV range to 550 nm. Moreover, a photocurrent was recorded at zero external bias voltage which points to the plausible formation of a p-n junction resulting from interpenetration of MAPbBr<sub>3</sub> single crystals into the VACNT forest. This reveals that vertically aligned CNTs can be used as electrodes in operationally stable perovskite-based optoelectronic devices and can serve as a versatile platform for future selective electrode development.

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OA-48

**STUDY OF TRIBOELECTRIC DEVICES BASED ON ZnO NANORODS AND PDMS:GO COMPOSITES FOR ENERGY HARVESTING APPLICATION**

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With the increase needing of saving natural resources, green technologies are getting attention of scientific community. Devices that can harvest energy from the

environment are a great choice to reduce the use of natural resources. Triboelectric devices can be used for harvest mechanical energy from the environment and convert to electrical energy and among different materials, zinc oxide is one promising material used to build those devices; its semiconducting and triboelectric properties presents in its nanostructures have been extensively studied for application in nanogenerator devices.

Aligned with these perspectives, we have graphene, known for its high electrical conductivity and transparency, ideal to be used in flexible devices. Besides those characteristics, it can be used as a modifier of morphology agent or as a support for the growth of nanoparticles in a substrate.

Zinc oxide nanorods were synthesized by chemical bath deposition at lower temperature in a flexible copper substrate without the seed layer step and characterized by FEG/SEM images; polydimethylsiloxane (PDMS) and a PDMS:graphene oxide (PDMS:GO) composite was used as upper triboelectric material with zinc oxide nanorods as bottom triboelectric material; together they were tested as a triboelectric device aiming to increase the output power[1]. The addition of graphene oxide to PDMS indeed influence the results, with this configuration, we obtained output power density value around 942 nW/cm<sup>2</sup>, making it possible to be used as a nanogenerator device.

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OA-49

## LASER PROCESSING OF PARA-/FERRO-ELECTRIC CERAMICS AS THIN FILMS FOR HIGHLY-TUNABLE MICROCAPACITORS

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In paraelectric / ferroelectric materials, the ions can segregate to create internal electric fields. The prevailing polarization in any domain can be modified by applying an external electric field, a feature that makes such materials highly desirable in tunable elements for developing reconfigurable digital or analog thin film radiofrequency

devices. Among such materials, barium strontium titanate solid solutions ( $\text{Ba}_{1-x}\text{Sr}_x\text{TiO}_3$ , or BST) are extensively studied in various compositions, structures, and configurations. Due to their complex stoichiometry, the choice processing technique is pulsed laser deposition (PLD) for attaining high quality, low-cost, eco-friendly thin films. This comprehensive parametric study, based on high-resolution transmission electron microscopy (HR-TEM) and X-ray diffraction (XRD) investigations, reveals and then correlates the influence of the deposition parameters and substrate types to thin films structure, morphology, and interface phenomena, versus their electrical properties in the millimetre waves region up to 60 GHz. We found that electrostrictive resonant effects in loss tangent can be reliably controlled by efficiently designing thin film composition, architecture, and device shape, leading to optimal control of the Q-factor in tuneable electronic components. By further integrating the BST thin films in metal-insulator-metal (MIM) and interdigitated (IDC) microcapacitors, high dielectric tunability (40 to 95%), with low losses ( $\text{tg}\delta: 10^{-3}-10^{-4}$ ), and at low applied electrical fields (10–50 V), are demonstrated.

OA-50

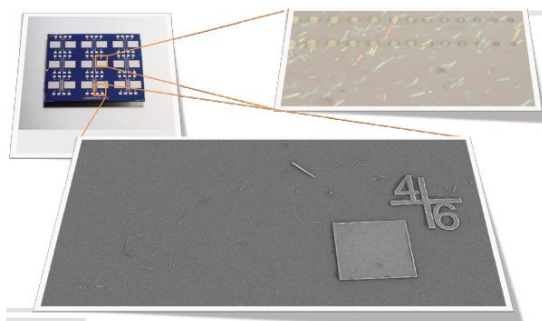
**THERMAL COARSENING OF METAL-ON-OXIDE NANOWIRE  
THIN FILMS**

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From painting, biocompatible coatings to solar cells, titanium dioxide ( $\text{TiO}_2$ ) has a very wide range of applications and is one of the most studied oxide material in the last 30 years [1,2,3]. In most of these applications, titanium dioxide is used in the form of nanoparticles as a starting material. Much has learned on the coarsening behaviour of nano  $\text{TiO}_2$ , mainly because of recent studies almost exclusively addressed to the coarsening of these, nearly spherical, isotropic nanoparticles. Indeed, the degree of the particle anisotropy might induce a strong shape-dependent variation in properties of matter, which raises the question how the coarsening of anisotropic particles proceeds at various interfaces.



In this study coarsening behavior of 2D thin films of titanate nanowires coated with few nanometer thick noble metal (Ag, Pt, Rh, Pd, Au) thin film were studied by using various techniques: atomic force microscope (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and resistivity measurements. The aim of this study was to shed light on the temperature dependent structural and geometrical evolution of ceramic/noble metal interfaces down to the single particle, the individual titanate nanowire and precious metal nanoparticle level. Thin films of titanate nanowires were prepared by wetchemical method. From room temperature up to 900 °C, different surface diffusion regimes were identified which correspond to the different titanate phases, from titanate  $\text{H}_2\text{Ti}_3\text{O}_7$  to anatase and to rutile phase. Understanding the sintering behavior of 1D oxide nanowires and metal nanoparticles will open up new routes to design efficient heterogeneous catalyst and photocatalyst systems.

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OC-51

## DIAMOND ELECTRODES FOR ELECTROCHEMICAL APPLICATIONS

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Diamond is a very attractive material for many applications due to its unique property. Highly boron-doped conductive diamond films are a subject of considerable interest as an electrode material because of their electrochemical properties, such as wide potential window, very low background current, chemical and physical stability.

One of the most efficient and promising applications of boron-doped diamond (BDD) electrodes is their use for the electro reduction of compounds that are difficult to reduce. This is the case of nitrates, which in the environment represent a health risk. The electrochemical reduction of nitrate is a multielectronic charge transfer process and the reaction mechanism is very complex. A large variety of nitrogen compounds can be formed such as NO<sub>2</sub>, NO, N<sub>2</sub>O, N<sub>2</sub>, NH<sub>2</sub>OH, N<sub>2</sub>H<sub>4</sub> and NH<sub>4</sub><sup>+</sup> (NH<sub>3</sub>) which are dissolved in the solution or are volatile compounds. The reduction potential and the nature of the products of nitrate reduction strongly depend on experimental conditions such as pH, applied potential, and coexisting ions.

The subject of our research work is the investigation of boron doped nanocrystalline diamond layers functionalized with photosensitive molecules. The diamond thin films were deposited on silicon substrate by Hot Filament Chemical Vapour Deposition (HFCVD) technique from a mixture of methane and hydrogen. As photosensitive species were chosen manganese phthalocyanine molecules. To optimize the process of the attachment of the photosensitive molecules, the diamond layers were preliminary modified with UV/O<sub>3</sub> - processing, O<sub>2</sub>-plasma and NH<sub>3</sub>/N<sub>2</sub>-plasma.

The modified and functionalized with phthalocyanine samples were used as working electrodes in three-electrode cell. Potassium nitrate was used as electrolyte. The electrochemical properties of the obtained layers studied by Cyclic Voltametry (CV) show the wide potential range (almost twice larger in comparison to the classical Pt electrode) and photosensitivity of the diamond films functionalized with phthalocyanine.

In the present work we have studied the influence of the chemical nature of the cation, (K<sup>+</sup>) on the reactional mechanism at BDD electrodes and analyze the dissolved and volatile species using spectrophotometry and chromatography.



OA-52

## SELECTED PROPERTIES OF TITANIUM DOPED YTTRIUM NIOBATE

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Rare earth niobates have recently been gaining attention as materials with considerably high ionic conductivity [1-3]. These compounds crystallize in defect fluorite structure yttrium niobate -  $Y_3NbO_7$  is an oxygen ion conductor in a wide range of oxygen partial pressures [1]. Acceptor doping of the material, due to an increase of the oxygen vacancies concentration, should rise the total conductivity and effectively improve ionic conductivity. In this work, titanium was chosen as aliovalent dopant because of the small difference in ionic radius of  $Ti^{4+}$  and  $Nb^{5+}$  [4]. Therefore series of titanium doped yttrium niobates were synthesized through solid state reaction method and thoroughly investigated.

The X-Ray diffraction showed that the obtained compounds contain only defect fluorite phase and no impurities were detected. The SEM images of samples showed dense granular structure on the surface with polyhedral pores. The highest values of total conductivity were obtained in humid atmosphere at 750 °C for the sample with 15% concentration of Ti. In humid conditions total conductivities were higher than in dry in the whole temperature range. The activation energies of conductivity were between 0.55 eV and 1.65 eV.

Acceptor doped yttrium niobate is a good candidate as ionic conductor for potential applications in fuel cells as well as other electrochemical devices. Its electrical and structural properties and chemical stability was shown in this study.

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OA-53

## SOLID-STATE REACTIVE SINTERING OF Yb<sup>3+</sup>:YAG AND Er<sup>3+</sup>,Yb<sup>3+</sup>:YAG LASER CERAMICS

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Polycrystalline Yb<sup>3+</sup>:YAG and co-doped Er<sup>3+</sup>,Yb<sup>3+</sup>:YAG are promising materials for laser and photonics applications, primarily for active media of diode-pumped solid-state near-infrared lasers due to excellent solubility of dopants into garnet matrix, high optical and structural quality, which are beneficial properties for obtaining of efficient laser devices. Nowadays there is an increasing interest in realizing YAG-based materials via advanced ceramic technology.

In the present work we report on realization by reactive sintering method [1] of Yb<sup>3+</sup>:YAG ceramics doped with 5 to 15-at.% Yb<sup>3+</sup> ions and of Er<sup>3+</sup>,Yb<sup>3+</sup>:YAG ceramics doped with 5-at.% Yb<sup>3+</sup> and 0.5 to 1.5-at.% Er<sup>3+</sup> ions. A special attention was paid to the realization method. Ball-milling conditions of starting oxide powders were refined to achieve aggregate-free homogeneous powder mixture. Optimal sintering temperature range suitable for the formation of highly-dense transparent ceramics under normal recrystallization conditions was found to be 1750–1800 °C. The effect of Yb<sup>3+</sup> and Er<sup>3+</sup> ions on phase composition evolution, microstructure, optical and luminescent properties of sintered samples was experimentally investigated. Furthermore, quasi continuous-wave lasing of Yb<sup>3+</sup>:YAG was studied under the pump at 970 nm with a fiber-coupled diode laser. A highest slope efficiency of 47% was achieved with a 15-at.% Yb<sup>3+</sup>:YAG ceramics.

The results are of interest for realization of advanced ceramic diode-pumped lasers for near-IR range, at 1.0-mm but also for eye-safe wavelength of 1.6 μm, to be used in various applications, such as range-finders, optical and communication devices or for light converters of semiconductor detectors and solar cells.

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OA-54

## LUMINESCENT Pr<sup>3+</sup> DOPED FLUORAPATITE NANOPARTICLES WITH DIFFERENT EXCITATION-EMISSION PROFILES

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The fluorapatite (FAP) nanoparticles doped with rare earth elements have been extensively studied as a potential luminescence inorganic material for *in-vitro* and *in-vivo* cells and tissue bioimaging [1-3]. Luminescent nanoparticles of undoped and Pr<sup>3+</sup> doped fluorapatite (Pr-FAP) have been successfully synthesized by co-precipitation method followed by calcination. Obtained materials are characterized by XRD, FTIR-FAR, PLE-PL and SEM-EDAX methods, and theoretical prediction of structural changes was also done.

Emission of FAP nanoparticles occurs in violet-blue region of visible part of spectrum under excitation in UV region. Introducing the Pr<sup>3+</sup> into the lattice causes the red or blue shift depending of Pr<sup>3+</sup> concentration and synthesis conditions. MCR-ALS analyses of fluorescence spectra and *ab initio* calculation indicated that Pr<sup>3+</sup> ions are located in Pr-FAP lattice at Ca2 (*6h*) sites. Assumed mechanism of substitution of Ca<sup>2+</sup> with Pr<sup>3+</sup> ion is accompanied with charge compensation by incorporation of CO<sub>3</sub><sup>2-</sup> ions into the structure or by vacancies formation on metal ions position. Synthesized nanoparticles can be potentially used as a luminescent nanomaterials in biomedicine for targeting, bioimaging, drug delivery and therapy.

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OA-55

## SINTERING AND PREPARATION OF YTTRIUM-IRON GARNET POWDERS

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The yttrium-iron garnet -YIG- ( $Y_3Fe_5O_{12}$ ) is one of the most important material with the ferromagnetic properties. It can be used as a basic material in microwave, acoustic, optical and magneto-optical applications. Multiple applications of YIG are a consequent of its excellent electromagnetic properties, including low dielectric losses.

The aim of this study was to obtain yttrium-iron-garnet YIG in the solid state reaction between the yttrium-iron perovskite ( $YFeO_3$ ) and  $\alpha$ - $Fe_2O_3$ . The first step was to obtain perovskite from iron and yttrium nitrate solutions with citric acid, where the ratio of total yttrium and iron to citric acid was 1:2. The next step was to introduce a 4% PVA solution which prevents accidental pyrolysis. The mixture was dried: at 100 °C for 24 hours, at 200 °C for 3 hours, and calcined at 750 °C. Obtained powder with stoichiometric addition of iron oxide was homogenized in an alkaline medium. Then a mixture was dried for 24 hours at 100 °C. The uniaxially cylindrical pellets and next isostatically at 250 MPa were pressed. Received in this way pellets were sintered at 1450 °C for 2 hours to obtain a material without open porosity. Elimination of residual porosity was achieved by hot isostatic pressing (HIP) at 1350 °C under argon. Based on SEM images, the microstructure analysis of obtained material was carried out.

OC-56

**Ag NANOWIRE/Al-DOPED ZnO COMPOSITE FOR TRANSPARENT ELECTRODE APPLICATIONS**

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For their ease and cost-effectiveness of synthesis and processing, silver nanowires (AgNWs) have been extensively studied as a transparent electrode alternative to conventional solutions for the application in solar cells. The main focus of this research was elucidating the mechanisms which occur during annealing of AgNWs before a layer of aluminum doped zinc oxide (AZO) is deposited on them, for the enhancement of properties essential for a transparent electrode in a solar cell. Microstructural characterization using scanning electron microscopy (SEM) and scanning transmission electron microscopy (STEM) revealed that solid-state wetting and subsequent welding occurred only between AgNWs whose contact geometry is characterized by an enormous difference in radii of curvature. The AgNW in contact through a smaller radius of curvature partially dissolves, Ag atoms diffuse and are incorporated in a welded zone between the AgNWs whose crystallographic orientation is inherent from the AgNW in contact through a large radius of curvature. Wetting angle between two welded AgNWs was measured to be below 4.8°, indicating almost complete wetting. It has been demonstrated that welding of AgNWs significantly reduces electrical resistivity while preserving high optical transparency, properties essential for transparent electrodes utilized in solar cells.

OC-57

### PHASE RELATIONS IN THE $\text{La}_2\text{O}_3\text{-Er}_2\text{O}_3$ SYSTEM AT 1500 °C

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System with lanthana and erbia are perspective for the development of transparent ceramics, laser and other optoelectronic components, ceramics for the intermediate temperature solid oxide fuel cells with high ionic conductivity.

The objective of this work is to investigate the phase relations in the binary  $\text{La}_2\text{O}_3\text{-Er}_2\text{O}_3$  system at 1500 °C in air in the whole concentration range. Powders of  $\text{La}_2\text{O}_3$ ,  $\text{Er}_2\text{O}_3$  (99.99 %) were used as raw materials. The samples were prepared from nitrate solutions with their subsequent evaporation and decomposition at 1000 °C for 2 h. Thermal treatments at 1100 °C for 744 h and at 1500 °C for 225 h in air were carried out. The phase compositions were investigated by X-ray diffraction, petrography, microstructural phase and electron microprobe X-ray analyses.

The study of solid state reaction of  $\text{La}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  at temperature 1500 °C showed that three types of solid solutions based on hexagonal modification of A- $\text{La}_2\text{O}_3$ , cubic modification of C- $\text{Er}_2\text{O}_3$  and intermediate phase with perovskite-type structure of  $\text{LaErO}_3$  (R) with rhombic distortions in the  $\text{La}_2\text{O}_3\text{-Er}_2\text{O}_3$  system are formed. These solid solutions were separated with the two-phase fields: (A + R) and (C + R). The boundaries of the homogeneity fields for the solid solutions based on A- $\text{La}_2\text{O}_3$ , C- $\text{Er}_2\text{O}_3$  and  $\text{LaErO}_3$ , as well as lattice parameters for solid solutions were determined.

The refined solubility of  $\text{Er}_2\text{O}_3$  in A- modification of  $\text{La}_2\text{O}_3$  is about 13 mol % at 1500 °C (225 h). The lattice parameter of the unit cell varies from  $a = 0.6523$  nm,  $c = 0.3855$  nm in pure  $\text{La}(\text{OH})_3$  to  $a = 0.6477$  nm,  $c = 0.3815$  nm in the sample containing 85 mol%  $\text{La}_2\text{O}_3\text{-15 mol% Er}_2\text{O}_3$ . It has been found that the boundaries of the R- phase homogeneity field in the concentration range of 45-51 mol%  $\text{Er}_2\text{O}_3$ . The lattice parameters of the unit cell R phase varies from  $a = 0.6056$ ,  $b = 0.5843$ ,  $c = 0.8442$  nm in single-phase sample, containing 50 mol%  $\text{La}_2\text{O}_3\text{-50 mol% Er}_2\text{O}_3$  to  $a = 0.6060$ ,  $b = 0.5856$ ,  $c = 0.8416$  nm and to  $a = 0.6068$ ,  $b = 0.5862$ ,  $c = 0.8442$  in two-phase sample (A + R), containing 55 mol%  $\text{La}_2\text{O}_3\text{-45 mol% Er}_2\text{O}_3$ .

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OA-58

## MICRO-RAMAN STUDY OF 5-15 at.% Yb<sup>3+</sup>:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> OPTICAL CERAMICS

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Ytterbium-doped yttrium aluminum garnet Yb<sup>3+</sup>:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> is an active laser medium of exceptional interest for creation of high-power and passively Q-switched solid-state lasers for different applications in optical communications, pollution monitoring, nonlinear optics, material processing, medical surgery and so on. The present work is targeted at chemical analysis, composition analysis, and identification of crystal structure peculiarities of synthesized transparent SiO<sub>2</sub>-doped 5-15 at.% Yb<sup>3+</sup>:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> ceramics of optical quality using Raman spectroscopy [1].

Raman spectra were obtained on microscopes WiTecAlpha 500 A/R and Morphology D3SE-ID. The Raman spectrometers were used with a laser wavelength  $\lambda$  of 532 nm and 785 nm, respectively.

The chemical composition homogeneity was confirmed for different-sized garnet crystallites along the grain boundaries and in their volume at doping with Yb<sup>3+</sup> ions to 15 at.%. The presence of all peaks characteristic for yttrium aluminum garnet structure when comparing the Raman spectra of synthesized 5-15 at.% Yb<sup>3+</sup>:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> ceramics with data for commercial Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> single crystals (Database The RRUFF™ Project) indicates the absence of lattice deformation within the entire studied concentration range of the active additive Yb<sup>3+</sup>. The possibility was shown for estimation of an influence of Si<sup>4+</sup> ions from the sintering additive SiO<sub>2</sub>, replacing Al<sup>3+</sup> ions, on the symmetry of garnet aluminum sublattices, using the shift values for peaks of modes of the elementary unit (AlO<sub>4</sub>).

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OA-59

**PRECURSOR INFLUENCE ON PHOTOCATALYTIC PERFORMANCE  
OF Cd<sub>x</sub>E<sub>1-x</sub>S COMPOUNDS (E=Zn, Co)**

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Results have shown that chalcogenic compounds are most suitable for photocatalytic water splitting reaction using solar radiation due to their activity in the visible range. In this work Cd<sub>x</sub>E<sub>1-x</sub>S (E=Zn, Co) doped with Pd<sup>2+</sup> photocatalysts were obtained in hydrothermal environment at 150 °C and different E concentrations. The final products were characterized by powder X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Brunauer–Emmett–Teller (BET) and UV–visible spectroscopy. All photocatalysis experiments for hydrogen evolution from water splitting process were conducted in the presence of sulfide ions under visible light and determined by gas chromatography.

OA-60

**DESIGNING EFFICIENT CATALYSTS FOR SOLAR WATER  
SPLITTING: METALLIC DOPING OF NiOOH MATERIAL**

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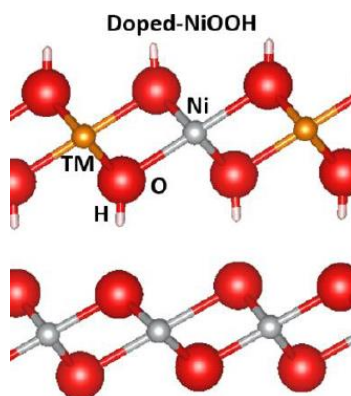
Solar energy is one important challenge of the current century. However, energy storage remains a considerable issue. Photoelectrochemical cells are devices aiming to convert solar energy into hydrogen fuel by water splitting. The process requires solid catalysts, often based on noble metals. We propose here to study an earth-abundant element based material already known for its outstanding catalytic activity, nickel oxyhydroxide (NiOOH).

NiOOH properties are strongly dependent on doping, generally involving metallic elements. To this date, only Fe presents a significant interest, attested by several experimental studies. Using computational methods, and especially the density



functional theory (DFT), we performed a complete scan of the first row of the transition metal (TM) block of the periodic table. We concluded that only middle and late TM may be relevant for NiOOH doping, whereas early TM have globally an inhibiting effect on the reaction. Moreover, we were able to correlate the reactivity of such elements to specific bulk properties of NiOOH material. We demonstrate in particular that high metal-oxygen bond ionicity is not suitable for efficient catalysts in water splitting process.

This theoretical work provides new insights in surface reactivity and hydrogen production. It also expects to give some guidelines to experimentalists and industrialists to elaborate new materials for photoelectrochemical cells.



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OA-61

## EFFECT OF THE SYNTHESIS METHOD ON THE CATALYTIC ACTIVITY OF NiCo<sub>2</sub>O<sub>4</sub>

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With the increasing worldwide demand for electrical energy storage, development of rechargeable batteries with high storage capacity and cycling stability are urgently needed. Rechargeable Zn-air batteries have received increasing attention due to their

prominent advantages such as drastically high energy density, cost efficiency, safety as well as environmental compatibility. The power density of zinc–air battery depends mainly on the performance of the air electrode. In fact, it is extremely difficult to exploit one electrocatalyst with satisfactory activities for both ORR and the reversible OER.

Recently, ternary spinel nickel cobaltite ( $\text{NiCo}_2\text{O}_4$ ) has caught much attention owing to the number of intriguing advantages, such as cost-effective, naturally abundant and environmentally benign. Moreover, it was reported that  $\text{NiCo}_2\text{O}_4$  possesses a much better electrical conductivity and electrochemical activity than other binary transition metal oxides.

For this reason, the aims of the present work were synthesis and investigation of  $\text{NiCo}_2\text{O}_4$  as potential bifunctional catalysts. The materials are prepared by means of different methods, namely precipitation reaction and hydrothermal method. They were characterized by physical-chemical and electrochemical methods. The obtained X-ray diffractograms are typical for  $\text{NiCo}_2\text{O}_4$ . The position and intensity of the peaks correspond to the spinel  $\text{NiCo}_2\text{O}_4$  phase. The IR spectra are typical for cubic spinels with two bands of high intensity. The electrochemical demeanor of the samples determined from cyclic voltammetry and steady state polarization study exhibit a good electrochemical activity.

OA-62

## **COMPARING THE MICROSTRUCTURE OF DRY POWDERS AND COLLOIDAL DISPERSIONS**

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Solution based processing of nanoparticles (NPs) is suitable for economic and material-efficient production of granular films and green bodies on large surface areas. Stable dispersions with low degree of agglomeration are required to produce pore- and crack-free high-quality films with optimal mechanical and electrical properties. Uncontrolled agglomeration of dispersed particles during processing can lead to microstructural inhomogeneities like density gradients or voids. Control over particle-particle interaction during the preparation and processing of colloidal dispersions is necessary to prevent such structural defects. As the structure of the colloidal dispersion has a direct, profound influence on the microstructure of the produced green body or film, characterizing it is of great importance.

Therefore, we study deagglomeration and stabilization process of chemical vapor synthesized (CVS)  $\text{SnO}_2$  NPs with small-angle X-ray scattering (SAXS), dynamic light scattering (DLS) and high-resolution scanning electron microscopy (HRSEM).  $\text{SnO}_2$  NPs were electrostatically stabilized in colloidal dispersions to investigate the

microstructural evolution varying dispersion pH, zeta potential and electrolyte concentration. Finally, size distribution, agglomerate formation, particle arrangement in the agglomerates and fractal dimension in the dispersed and dry powder states are compared.

OA-63

### **KETOPROFEN LOADING CAPACITY AND THE RELEASE PROFILE IN MESOPOROUS SILICA PREPARED FROM MIXED PRECURSORS**

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In order to be tested for their drug loading and release properties, mesoporous silica particles systems were synthesised. Mixtures of silica precursors, such as, tetraethoxysilane (TEOS) and methyltriethoxysilane (MTES), in the presence of different alkyltrimethyl-ammonium bromides pore templates was used, in acid (nitric acid) catalysed sol-gel process. The obtained products showed specific surface areas in the 883–1580 cm<sup>2</sup>g<sup>-1</sup> range. The maximum value of surface area corresponds to intermediate precursor content, specifically of 30 mol% MTES. The total pore volumes and the average pore sizes show a clear trend of decreasing with the increase of MTES content, from 1.5 to 0.45 cm<sup>3</sup>g<sup>-1</sup>, and from 5.7 to 2.5 nm, respectively. In parallel to the decrease of the pore size, the SAXS measurements evaluated that interpore spacing was also decreasing. TEM images show ordered arrays of very long tubules characteristic for MCM41 type materials. The best loading capacity for ketoprofen (the chosen drug) was found for the sample synthesized with low content of MTES. The highest MTES content (50% MTES) sample showed the release profile corresponding to the measured kinetic of approximately 1.5 times slower than in the case of the sample without MTES content. Resuming, at the time, the mentioned sample seems to be an appropriate candidate as controlled drug delivery system.

OA-64

**PEAK ASSIGNMENT – SHORT ORDERING AT B-SITE IN  
SPINEL FERRITE**

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Spinel ferrites have general formula  $AFe_2O_4$  ( $A=Mg, Mn, Fe, Co, Zn, Ni, Cu$ ). It is known that in a bulk form  $ZnFe_2O_4$  has normal spinel structure and  $NiFe_2O_4$  possess inverse spinel structure. Structural characterization of  $NiFe_2O_4$  nanoparticles has shown their mixed spinel structure [1]. In order to investigate the cation distribution among the tetrahedral and octahedral positions Raman spectroscopy has been performed on  $ZnFe_2O_4$  and  $NiFe_2O_4$  nanoparticles. This method is extremely sensitive to the atomic short-range ordering. Spinel structure is characterized by five Raman active internal modes (or phonons) ( $A_{1g}+E_g+3F_{2g}$ ). In small particle size hinders the phonon propagation and hence induces Brillouin zone following making all the phonons Raman active, which leads to band broadening of the Raman lines to the increase of cationic disorder. We consider the peak assignments and intensities of Ni-O, Fe-O and Zn-O vibrations on octahedral site by deconvolution of Raman spectra.

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OA-65

## INFLUENCE OF CALCIUM DOPANT ON DIELECTRIC AND ELECTRIC PROPERTIES OF BaBi<sub>2</sub>Nb<sub>2</sub>O<sub>9</sub> CERAMICS

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Bi-layered ferroelectric materials including SrBi<sub>2</sub>Ta<sub>2</sub>O<sub>9</sub> and SrBi<sub>2</sub>Nb<sub>2</sub>O<sub>9</sub> have been paid much attention because they show good long-term reliability properties such as fatigue and retention for the ferroelectric memory (FRAM) applications. The most useful aspect of these materials are their high fatigue resistance against polarization switching. When Sr is replaced with Ba<sup>2+</sup> ions (BBN), structural disorder is induced and material exhibits significant broadening of the phase transition. The causes of this diffuse phase transition were widely discussed in the previously published articles [1,2]. Moreover the dielectric properties of BBN ceramics show strong axial properties, so they seems to be very promising material for pressure sensor applications [3]. Such applications required materials with not only good dielectric properties, but also good mechanical quality (high density), which is connected with the improvement of the sinterability. Based on literature information calcium dopant seems to be a very promising modifier.

In the first part of the presentation the impact of calcium dopant on microstructure and crystal structure will be discussed. The phase purity was revised by X-ray diffraction analysis. The results indicate that small size Ca<sup>3+</sup> ions were successfully substituted into Ba-site with no secondary phase detection. The addition of calcium caused inhomogeneity of microstructure, namely the occurrence of bigger grains increased with the increase of admixture content. The second part of the presentation would be focused on dielectric and electric properties of this interesting material. The pure BBN ceramics, as well as the modified ones by calcium, show the frequency dependence of the dielectric response typical for relaxor ferroelectrics i.e. the significant reduction of  $\epsilon'_{\max}$  and shift of the corresponding temperature ( $T_m$ ) towards higher values with increasing frequency.

**Keywords:** lead-free ceramics, BBN, dielectric properties

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OA-66

## BiFeO<sub>3</sub> THIN FILMS: INFLUENCE OF DOPING ON STRUCTURE AND PROPERTIES

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Thin films of BiFeO<sub>3</sub> and doped BiFeO<sub>3</sub> were prepared by chemical solution deposition method. Because of low electrical resistivity of BiFeO<sub>3</sub> which often prevents it to exhibit ferroelectric properties, several elements were used as dopants, partially replacing Bi<sup>3+</sup> (Y<sup>3+</sup>, Sr<sup>2+</sup>) or Fe<sup>3+</sup> (Ti<sup>4+</sup>, Zr<sup>4+</sup>) ions. Dopants may also reduce the size of the grains improving the magnetic properties.

Pure BiFeO<sub>3</sub> phase was formed for all compositions without preferred orientation. All films are around 300 nm thick, with relatively densely packed round grains of the size well below 100 nm as it is presented with micrographies in Fig. 1. Some of the dopants have lowered the size of the grains and increased electrical resistivity enough for ferroelectric hysteresis to be measured at fields above 100 kV/cm

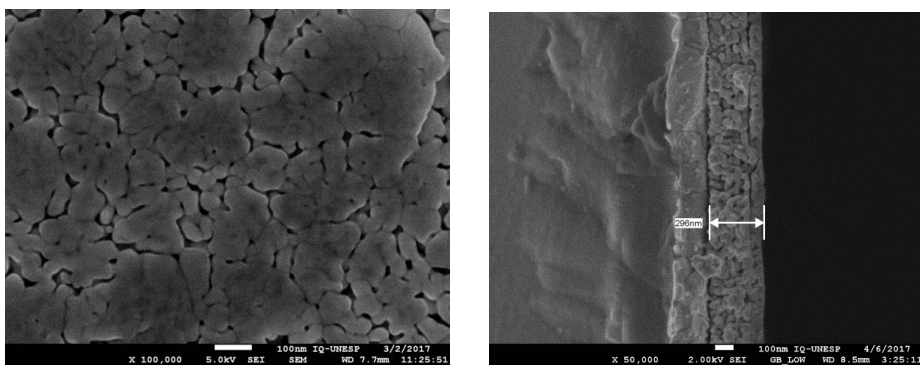


Figure 1. SEM micrographies of surface (a) and cross section (b) of BiFeO<sub>3</sub> thin film

OA-67

**STRESS-INDUCED PHASE TRANSITION IN LEAD-FREE RELAXOR FERROELECTRIC COMPOSITES**

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Piezoelectric materials are considered an enabling technology generating an annual turnover of about 20 billion dollars. At present, lead-based materials dominate the market with their known risk to health and environment. One of the three key competitors for their replacement is the class of sodium bismuth titanate (NBT)-based relaxor ferroelectrics, the use of which is limited by thermal depolarization. An increased thermal stability has recently been experimentally demonstrated for composites of  $\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-}0.06\text{BaTiO}_3$  with ZnO inclusions. However, the exact mechanism for this enhancement still remains to be clarified. In this study, piezoresponse force microscopy and  $^{23}\text{Na}$  NMR spectroscopy were used to demonstrate that the incorporation of ZnO leads to a stabilization of the induced ferroelectric state at room temperature. Temperature-dependent measurements of the relative dielectric permittivity  $\varepsilon'(T)$  and the piezoelectric coefficient  $d_{33}$  revealed an increase of the working temperature by 37 °C. A simple mechanics model suggests that thermal deviatoric stresses stabilize the ferroelectric phase and increase, as well as broaden, the temperature range of depolarization.

OC-68

**STRUCTURAL AND PHOTOLUMINESCENCE PROPERTIES OF THE  
(Ni,Zn) Fe<sub>2</sub>O<sub>4</sub>-BaTiO<sub>3</sub> MULTIFERROIC COMPOSITE NANOPOWDERS**

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Ferroics and multiferroics are among the most attractive multifunctional materials. In recent years, multiferroic composite consisted of two or more ferroic orders, such as ferroelectric, ferromagnetic, or ferroelastic, in which coupling between these phases can produce magnetoelectric effect, has been drawing significant attention. Besides others, (Ni, Zn)Fe<sub>2</sub>O<sub>4</sub>-BaTiO<sub>3</sub> composites have attracted considerable attention not only due to fundamental research of magneto-electric effect, but also for the potential applications in many electronic devices such as drug delivery, heterogeneous catalysis, levitated railway system, magnetic-refrigeration, microwave devices, antennas, memory devices, etc. In this composite, (Ni,Zn)Fe<sub>2</sub>O<sub>4</sub>(NZF) possesses magnetic behavior, and the ferroelectricity is due to the presence of BaTiO<sub>3</sub>(BT). In the present work, it was investigated the structural and photoluminescence properties of NZF-BT composite nanopowders prepared by auto-combustion method and the different ratio of NZF/BT was explored. XRD indicated well crystallized powders but with small presence of secondary phases respecting to BT. By FE-SEM analyses was noticed the agglomerates with wide particles size distribution, as well as the difference of the particles morphology between (Ni,Zn)Fe<sub>2</sub>O<sub>4</sub> and BaTiO<sub>3</sub>. FT-IR spectra display interactions between metal-oxygen around 500-600 cm<sup>-1</sup>. The different ratio of NZF/BT effects on the optical band gap values indicating the formation of intermediary levels modifying the Fermi level. Photoluminescence (PL) analysis provides important information about the materials structures, however the PL behavior of these composites is not very well known. The presence of PL emission suggests that the structures of the materials are medium range disordered. All samples showed a broad band emission centered around 450 nm, being a region characteristic of resultant emission from the presence of shallow defects in materials structures. This behavior is reinforced by deconvolution of PL bands, which shows the contribution of three sub bands to emission (blue, green and yellow emission), being that the largest area of spectra is occupied by a band correspondent to blue emission.



OC-69

## NOVEL FUNCTIONAL PROPERTIES OF BaTiO<sub>3</sub>-FERRITE MAGNETOELECTRIC COMPOSITES

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The magnetolectric (ME) multiferroics possess simultaneous magnetic and ferroelectric order, displaying a ME coupling that consists in variation of a dielectric property under magnetic field variation or *vice versa*. This facilitates the magnetic–electric energy conversion, thus being attractive for applications as magnetic field probes, transducers, actuators, sensors, passive filters for communications etc. [1,2].

In this study, the dielectric and magnetic properties of BaTiO<sub>3</sub>–ferrite multiphase magnetolectric composites are reported. The composite ceramics were prepared starting from di-phase mixtures of appropriate amounts of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and BaTiO<sub>3</sub> powders (10, 20, 30 and 40 vol.%  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), that were uniaxially pressed into pellets at 150 MPa and sintered at 1200 °C for 1 h.  $M(H)$  loops at room temperature show a “wasp-waisted” feature (Fig. 1), determined as result of the formation of magnetic phases with contrasting coercivities (hard BaFe<sub>12</sub>O<sub>19</sub> and soft Ba<sub>12</sub>Ti<sub>28</sub>Fe<sub>15</sub>O<sub>84</sub>) in different ratios. These extrinsic contributions play an important role, modifying the electrical properties of ceramics with large amount of magnetic phases, causing space charge effect and Maxwell-Wagner relaxations<sup>3</sup>. Thus, measurements in the MHz – GHz range (Fig. 1) were carried out for determining intrinsic contributions to electrical response of this kind of composites.

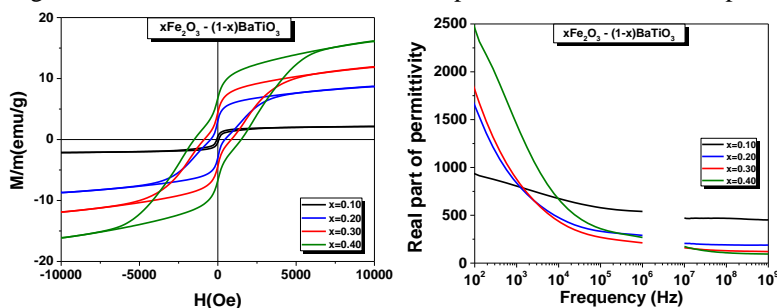


Figure 1.  $M(H)$  loops of Fe<sub>2</sub>O<sub>3</sub>-BaTiO<sub>3</sub> at room temperature – left; Real part of permittivity frequency dependencies of Fe<sub>2</sub>O<sub>3</sub>-BaTiO<sub>3</sub> at room temperature – right.

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OA-70

## COMPOSITIONAL TUNING OF B-SITE BARIUM TITANATE SOLID SOLUTIONS

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B-site substitution of barium titanate (BaTiO<sub>3</sub>) with homovalent or heterovalent dopants is the basis of solid solutions that are of recent interest due to their increasing importance for dielectric, piezoelectric, energy storage and microwave applications. High dielectric permittivity – stable over a large temperature range – and large high-field piezoelectric coefficients are typically sought-for figures of merit. Yet, compositional tuning to attain specific properties has been largely based on macroscopic observations and very little is known about the fine material structure on the short range that is necessary to induce those properties. B-site substitution in BaTiO<sub>3</sub> can produce, depending on the cation type and amount, different local structures such as chemically ordered regions (CORs) or chemical clusters (CCs). CORs and CCs can impact in many ways the macroscopic dielectric behaviour, since different atomic arrangements can produce different charge or strain on the local scale, inducing ferroelectric, paraelectric or relaxor behaviour.

In this work, we investigated several B-site doped barium titanate materials using a combination of high resolution X-ray diffraction, Raman spectroscopy and macroscopic dielectric property measurements. The aim of the work is to attempt to develop correlations between the macroscopic properties and the local structures/phases of BaTiO<sub>3</sub> solid solutions that specifically result in relaxors. Heterovalent (Nb<sup>5+</sup>), homovalent (Zr<sup>4+</sup>) and charge compensated (Ga<sup>3+</sup>, Ta<sup>5+</sup>) substituents have been selected. Charge imbalances necessary for relaxor behaviour are directly induced upon heterovalent doping and local strain fields induce charge imbalances indirectly upon homovalent substitution. The latter effect is expected to be weaker than the former, thus giving rise to relaxor behaviour at much higher substituent content. Further, charge-compensated substitutions introduce randomly distributed electric field interaction locally, resulting in diffuse phase transitions which have been also associated with relaxors.

Our work demonstrates that the combination of microscopic and macroscopic methods including techniques sensitive to short range interactions (i.e. Raman spectroscopy) is decisive for elucidating the influence of the local chemical arrangement on the macroscopic properties of the material.

OA-71

**KNN-BASED MATERIALS SINTERED VIA MICROWAVE HEATING TECHNOLOGY: FROM SYNTHESIS TO SINTERING**

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Piezoelectric materials based on  $\text{Pb}(\text{Zr,Ti})\text{O}_3$  has been widely used by their remarkable piezoelectric and ferroelectric properties, however, the high content of lead represent possible ecological and human hazards. The  $(\text{K,Na})\text{NbO}_3$  (KNN) based piezoelectrics are environmentally friendly and exhibits close properties, so its study represents an effort to eliminate the use of lead. The fabrication of KNN ceramics follows the conventional ceramic processing route, however it requires prolonged times for heating. An alternative method using microwave is described in this work. The synthesis of KNN with perovskite structure has been carried in a monomode microwave. Sintering the KNN by microwave technology reduces the time of processing in a 80% time in comparison with conventional method, obtaining similar structural and microstructural properties.

OA-72

**EFFECT OF SINTERING ATMOSPHERE IN THE MICROSTRUCTURAL DEVELOPMENT AND DIELECTRIC PROPERTIES OF  $\text{BaLa}_4\text{Ti}_4\text{O}_{15}$**

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$\text{BaLa}_4\text{Ti}_4\text{O}_{15}$  (BLT) is a material with excellent microwave properties due to its high permittivity ( $\epsilon_r$ ), high quality factor ( $Q$ ) and a near zero temperature coefficient of resonant frequency ( $\tau_f$ ) [1,2]. BLT is currently used in microwave electronics industry as a building block for resonators and microwave films such as capacitors, substrates, among others.

It is well known that the physical properties of ceramics are dictated by the microstructure of the material [3]. Therefore, by altering the microstructure, the electrical properties may be designed.

There are several ways to modify the microstructure, one of them is the sintering atmosphere, which can affect the grain boundary mobility, the structure, and the electrical response of the material. This is well known for functional oxides such as BaTiO<sub>3</sub> and SrTiO<sub>3</sub>, among others. However, for BLT this knowledge is still limited.

The aim of this study is to investigate the effect of the sintering atmosphere on the GB characteristics of high Q (low dielectric losses) BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub> (BLT) to engineer its electrical response.

BLT ceramics were sintered at 1530 °C in air, oxygen, and nitrogen. High dense and anisotropic microstructures, with elongated grains, were developed in the different atmospheres. However, the grain growth kinetics is different in nitrogen and oxygen: in nitrogen, a stagnant grain growth regimen was achieved, whereas in oxygen grains can grow along the sintering time. Consequently, in oxygen coarser and more anisotropic microstructures were obtained with large abnormal grains having a very high aspect ratio.

Samples sintered in oxygen (O<sub>2</sub>) atmosphere shows higher dielectric constant ( $\epsilon_r$ ) and, consequently higher dielectric losses ( $\tan \delta$ ), than both sintered in air or nitrogen (N<sub>2</sub>). Also, the electrical conductivity presents changes for the different atmospheres, showing that different species are responsible for the conduction mechanism.

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OT-73

## **EFFECT OF GAMMA IRRADIATION DOSES ON THE BIOACTIVITY OF BORATE ANALOGUS TO HENCH BIOGLASS AND THEIR GLASS CERAMICS**

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Presented work focusing on manifestation of gamma irradiation successive doses effect on the bioactivity or bone adhering efficiency of samples from patented Hench's and modified borate bioglass upon immersion for extended times in stimulated body fluid (SBF). Combined analytical techniques including FT infrared, X-ray diffraction and scanning electron microscopic measurements were carried out to follow and identify the change of these studies with both gamma irradiation doses and time of immersion. FTIR

spectra before and after both irradiation and immersion processes reveal vibrational bands due to main silicate groups and sharing of some phosphate units in mixed wavenumbers. Such processes show mainly growth of the vibrational bands due to water, OH or related groups and the vibrational modes due to main network forming groups remain unaffected by irradiation. The X-ray diffraction data indicate that irradiation with 10 Mrad causes no changes in the amorphicity of the Hench's bioglass as before irradiation while diffraction pattern of immersed sample reveal distinct peaks after four weeks of immersion. The irradiated sample (10Mrad) show only a peak after two weeks and more different peaks after four weeks of immersion. SEM micrographs are observed to be distinct revealing the appearance and growth of nodular shaped micro-crystallites with prolonged times of immersion.

OC-74

**PREPARATION AND CHARACTERIZATION OF POLY(VINYL-ALCOHOL)/CHITOSAN BLENDS WITH ZnO NANOPARTICLES****D. Pugar, T. Haramina**

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This research focuses on relating structure and mechanical properties of nanocomposites with antimicrobial activity. Organic nanoparticles of chitosan (CS) and inorganic nanoparticles of ZnO, both with cumulative antimicrobial activity, are added in a non-toxic poly(vinyl-alcohol) (PVA) matrix. CS reduces crystallinity of PVA inducing degradation of mechanical properties, but the properties of the polymer matrix can be improved by chemical crosslinking with glutaraldehyde (GA) and by adding ZnO nanoparticles.

A solution of low molecular chitosan in dilute acetic acid was mixed with solution of fully hydrolysed PVA at different weight ratios, by means of direct blend process. ZnO nanoparticles were synthesized by the precipitation method from  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  and added to PVA/CS blends. The resultant suspensions were casted and then dried in oven in order to obtain a thin flexible films. The structure of these mono-, bi- and ternary systems with different composition were characterized by means of dynamic mechanical analysis (DMA).

The DMA analysis showed that CS reduces the crystallinity degree of PVA leading to a higher amount of the amorphous phase contributing to the  $\alpha$  relaxation that corresponds to the glass to rubber transition. However, the mobility of the amorphous phase can be restricted with crosslinking. Moreover, ZnO NPs improve mechanical properties of the PVA/CS mixture and reduce the negative effect of CS on PVA properties.

OA-75

**SHAPING OF BIOCERAMIC HYDROXYAPATITE SCAFFOLDS ON  
MICRO LEVEL**

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Due to increasing average age of population around the world, there are more patients who need surgery of musculoskeletal system. In some cases, it is even necessary to implant new bone scaffold or replace joints. Hydroxyapatite is the most frequently used material for its biocompatibility and bioactivity.

On the other hand, mechanical properties constraints its use in porous scaffolds. The method providing both good mechanical stability and high porosity is freeze – casting. This method utilises controlled freezing to form lamellar structure. During freezing of slurry, ice crystals grow in direction of temperature gradient. In next step, ice crystals are removed by lyophilisation and lamellar structure is revealed. This structure is relatively unstable and needs to be reinforced by sintering. There are many aspects affecting resulting structure – freezing schedule, slurry concentration etc.

We used water/ceramic slurries in various concentrations of hydroxyapatite (7,5 % vol., 10 vol.%, 15 vol.% and 20 vol.%). These slurries were used for preparation of porous bone scaffolds. We tried to add 3D mash to form net of channels and we observed favourable changes in porosity. 3D mash was removed during sintering. Resulting bioceramics structure was highly porous (up to 90 %). These scaffolds had an acceptable ration between porosity and mechanical stability, too. We considered, adding of 3D mash, as good way to form channels of desired properties.

OC-76

**ELECTROPHORETICALLY DEPOSITED BIOACTIVE HYDROXY-  
APATITE/CHITOSAN COATINGS LOADED WITH GENTAMICIN**

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Hydroxyapatite (HAP) is a widely used biochemical material, because it promotes osteointegration, has outstanding biocompatibility and chemical composition similar to natural bone. In order to improve the mechanical and antimicrobial properties of HAP, biocompatible, natural polymer chitosan and antibiotic gentamicin were employed. The obtained composite hydroxyapatite/chitosan/gentamicin (HAP/CS/Gent) was assembled on pure Ti plates using the cathodic electrophoretic deposition process from an aqueous suspension.

The aim was to produce a uniform bioactive coating with improved mechanical strength and favorable antibacterial properties. HAP/CS/Gent coatings were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FE-SEM). Addition of the polymer significantly improved morphology, and bioactivity of thus formed composite. HAP/CS/Gent composite coatings exhibited strong antibacterial activity against *Staphylococcus aureus* and *Escherichia coli*.

OA-77

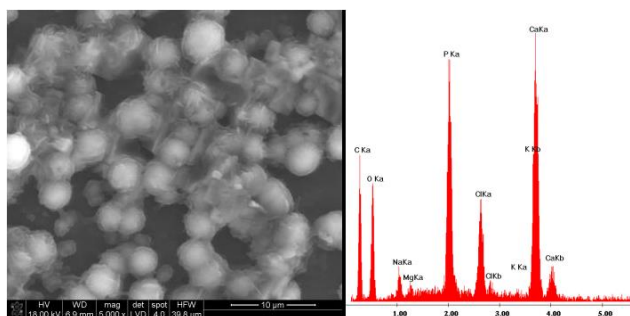
**HYDROXYAPATITE-BASED THERMAL STABILIZERS FOR  
ACETAL RESINS**

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Hydroxyapatite (HAp,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ) is a calcium phosphate ceramic widely-known for its bioactivity. HAp nanopowder can be easily used as a polymers modifier in order to create bioactive, inorganic-organic composites with high mechanical properties intended for bone implants. One of the thermoplastic polymer, that can be modified with HAp, is polyacetal (POM, polyoxymethylene). POM ( $[-\text{O}-\text{CH}_2-]_n$ ) is a highly crystalline engineering material that is characterized by its high mechanical strength and

stiffness, low friction coefficient, very good dimensional stability, toughness and excellent chemical resistance. The poor thermal stability is the biggest disadvantage of POM [1]. Pure HAp was incorporated into the POM matrix to obtain POM/HAp bioactive composites [2]. Unfortunately, the thermal stability of POM decreased significantly. To prevent this undesired effect the HAp surface was chemically modified with poly(ethylene glycol) (PEG) of different molar mass using 1,6-hexamethylene diisocyanate (HDI) as a coupling agent. Then, the new HAp-g-PEG hybrid compound was introduced to polyacetal. It was confirmed with thermogravimetric method (TG) that HAp-g-PEG acts as a thermal stabilizer for polyacetal resin. Thermal stability of POM was increased even by 30 °C. Furthermore, POM/HAp-g-PEG composites were still bioactive what was confirmed by incubation in simulated body fluid (SBF) (Fig 1).



*Figure 1. SEM images and EDX analysis after 3 weeks of incubation in SBF:  
POM/5% HAp-g-PEG*

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OA-78

**SYNTHESIS OF BIOCOMPATIBLE UPCONVERTING  
NANOPARTICLES FOR NON-SPECIFIC CELL LABELING**

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Unique optical properties, such as ability to convert NIR to visible light, high quantum yield and extraordinary photostability of the lanthanide-doped upconverting nanoparticles (Ln-UCNPs) have been proven already to be useful in biomolecule labeling and cell imaging. However, beside possessing favorable intrinsic characteristics, for such application UCNP should be also hydrophilic and biologically safe. Having this in mind, we developed a facile solvothermal processing of hydrophilic/biocompatible NaYF<sub>4</sub>:Yb,Er UCNPs with a help of poly(lactic-co-glycolic acid). Synthesized particles were analyzed by X-ray powder diffraction (XRPD), field emission scanning electron and transmission electron microscopy (FE-SEM/TEM), Fourier transform infrared (FTIR), Energy dispersive X-ray (EDX) and photoluminescence (PL) spectroscopy. To asses a biological safety of their use, viability of human gingival fibroblasts (HFG) and primary cell cultures of head and neck squamous carcinoma cells (HNSCC) was additionally evaluated by a colorimetric MTT assays

OA-79

**NAA, A VALUABLE ANALYTIC METHOD FOR ELEMENTAL CHARACTERIZATION FOR MEDICINAL CROP PLANTS**

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There are many analytical methods used for determination of crop plants elemental content, that generally implying the vegetal matrix disintegration. The Neutronic Activation Analysis, NAA, represents a specific and accurate analysis technique, classified as primary ratio method which does not necessarily requires the preliminary step of digestion, such way avoiding certain errors. By using NAA technique, the elemental analysis, for seven medicinal crop plants, such as, *coriander, dill, Echinacea, lavender, chamomile, mint and plantain*, that were cultivated in unpolluted areas, in Romania, in order to be used as raw material for phytopharmaceutical products, was accomplished. The essential nutrients (K, Ca, Mg, Na, Fe, K, Mn, Zn), micro- and trace elements (Co, Cr, Cu, Ni, Se, V), as well as the undesirable, potentially toxic elements (Al, As, Ba, Co, Cr, Ni, Sb) together with rare earth elements are monitored in this context. The compared results with those presented in the specialized literature, analyzed from point of view of their contributions to intake and toxicity of metals for a person reveal that medicinal plants can represent an useful contribution to the food provided intake of three essential macronutrients (K, Ca, Mg). The results remove any concern because raw material contents are very low in As, Ba, Co, Cr, Ni and the rare earths concentrations normal and acceptable from toxicological point of view. Seemingly the Al and Fe content is of some worry, the facts revealed that that metal bioaccumulation property is recognized and attributed to several plant species that are used for phytoremediation of contaminated soils. This dual implication, beneficial for soil depollution, but critical in transmitting the accumulated metals to humans, was considered. Based on the valuable obtained results, the tested plant materials can be considered as an internal secondary plant standard.

OC-80

**RHEOLOGICAL RESPONSE OF NON-NEWTONIAN COMPOSITES  
BASED ON SILICA POWDER AND POLY(PROPYLENE GLYCOL)**

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Shear thickening fluid (STF) is a good example of an innovative ceramic-polymer composite which has the ability to suppress and dissipate impact energy. There are colloidal systems consist ceramic powder, often SiO<sub>2</sub>, dispersed in an organic carrier fluid such as poly(ethylene glycol) or poly(propylene glycol). The typical feature of these liquids is an increase in viscosity with increasing shear stress.

The mechanism of shear thickening can be interpreted by a few theories. It can be explained by order–disorder transition, hydrodynamic clustering and particle flocculation. The main assumption is the increasing of the internal friction forces, with a growing shear rate, and consequently rising viscosity. This phenomenon still requires research in various fields to achieve entire knowledge of this process.

This work presents the studies based on poly(propylene glycol) and amorphous silica powder made by Stöber method. Studies show that the rheological response mainly is due to the breakout of hydrogen bonds (shear thinning) and reorganization of the ceramic powder particles in suspension, leading to the increase of internal friction and partially jam the fluid flow (shear thickening). The characterization of the material was obtained using rotational rheometer equipped with plate-plate system. This work includes comprehensive results of flow and viscosity curves, oscillation, thixotropy, creep and recovery measurements.

OA-81

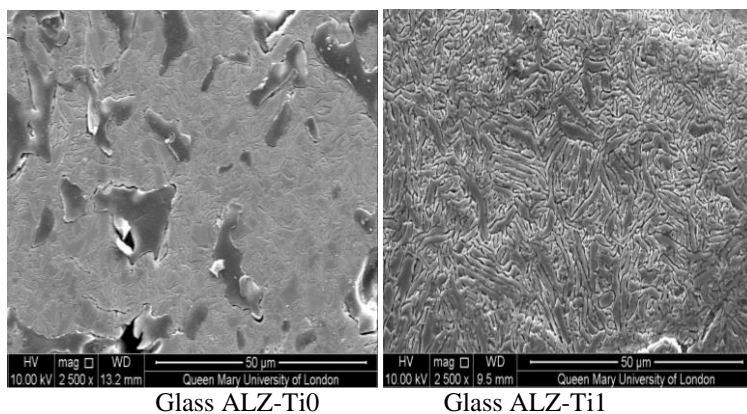
## EFFECT OF TiO<sub>2</sub> ON CRYSTALLIZATIONS OF ALKALI ALUMINA-SILICATE GLASSES

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The addition of TiO<sub>2</sub> in certain surface crystallised glasses known to act as nucleation agents to promote the development of a high density of internal nucleation sites and used as a key to the achievement of controlled crystallisation. Therefore, the aim of this study was to investigate the effect of TiO<sub>2</sub> on the crystallizations of a newly developed alkali rich alumina-silicate glasses. Wherein, two glass compositions based on multicomponent system containing sodium-magnesium-alumina-silicate were synthesized using melt-quench method. Glass ALZ-Ti1 contains 1.0 mol% TiO<sub>2</sub> and ALZ-Ti0 is a titanium free glass. Both experimental glasses were ground into fine powders (<125 μm) then heat-treated at the exact crystallization schedule. Glasses and glass-ceramics were characterized before and after crystallizations using differential scanning calorimetric (DSC), dilatometry (DIL), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The glass transition temperatures of the glass and the end glass-ceramics were extracted from DSC and DIL experiments on both glasses. This poster presentation will further discuss, the effect of TiO<sub>2</sub> on crystallisation of a newly developed alkali alumina-silicate glasses in term of thermal behaviours and the residual glassy matrix after crystallisation when TiO<sub>2</sub> is included in the parent glass composition.



*Figure: SEM photomicrographs showing the effect of on the residual glassy matrix content after crystallisation, (left) before and (right) after including TiO<sub>2</sub> in the parent glass composition*

OA-82

## STRUCTURAL AND OPTICAL CHARACTERIZATION OF MODIFIED GERMANATE GLASS SYSTEM

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Optical glass materials emitting at the MIR spectra region (Mid Infrared) and UV-VIS-NIR spectra region (Ultraviolet – Visible – Near Infrared) have been widely studied recently. Those belong to the group of optoelectronic materials. Their application can be in optical fiber as a source of optical radiation in mentioned regions.

The research is focused on the influence of type and amount of modifier on luminescence efficiency in the visible and infrared region of germanate glasses. Additionally an attempt to define the correlation between structure and luminescence properties of received glasses was taken.

The investigated glassy system  $\text{GeO}_2\text{-Ga}_2\text{O}_3\text{-(25-x)BaO-xM}$ , where M-modifier ( $x=5, 10, 15, 20$  and  $25$  mol%) was doped with rare earth ions. Following study of prepared glasses' samples were executed: X-ray Diffraction measurements (Panalytical Empyrean powder diffractometer using  $\text{Cu K}\alpha$  ( $\lambda_{\text{K}\alpha} = 1.54186 \text{ \AA}$ )), SEM/EDS (SEM-FEI Nova 200 NanoSEM), FTIR spectra (Bruker Company Vertex 70v spectrometer and Horriba Yvon Jobin LabRAM HR micro-Raman spectrometer), luminescence spectra (Acton Spectra Pro 2300i monochromator in the range of 1350–2300 nm and laser diode  $\lambda_{\text{exc}}=976$  nm).

Some metal oxides which will play role of modifier in this research can cause bond breaking, other polymerization of glass structure. Those and any other phenomenon will be investigated. Structural changes may cause changes in luminescence properties. Correlation between structural and optical properties will be studied.

**Keywords:** germanate glasses, luminescence, structure, spectroscopy

OA-83

**PRELIMINARY INVESTIGATION OF AMORPHOUS METAL  
SURFACE FOR APPLICATION IN THE POWER INDUSTRY**

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Metallic glass is a metallic material which exhibit long-range disorder in its structure. It is mostly three or more component alloy, where some of them are magnetic metals. The amorphous metal is technical glass, however it is much tougher and less brittle than regular oxide glasses and ceramics. Additionally unlike regular glass which is used in electrical insulators on power lines, amorphous metals are characterized by high electrical conductivity. At the same time, they exhibit very good magnetic properties (e.g. low-magnetization loss). The above mentioned properties are very useful in electrical engineering industry and this material is more and more popular as a substance for high-efficiency transformer core production. Transformers with cores made of amorphous metals present lower losses while they working in relation to the regular one. However, production technology of amorphous metal core is still evolving, and thus even higher efficiency of transformers is expected. A raw material must be carefully investigated and characterized before the main production process is started.

Presented work contains results of complementary examination of amorphous metal material. Scanning electron microscopy, x-ray photoelectron spectroscopy and Raman spectroscopy were used as methods for characterizing the raw material and to control an annealing process. Adequate parameters let to obtain surface with enhanced amount of useful oxides. Additionally, surface cleaning process was optimized with surface free energy measurement by means of Drop Shape Analyzer as a control method.

Used methods allow to fully investigate the amorphous metal samples. Information about surface character and appropriate cleaning pretreatment were received and let to apply the best parameters in further experiments.

OA-84

## MOLECULAR DYNAMICS SIMULATIONS OF BOROSILICATE GLASSES

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Vitrification is one of the most effective method of nuclear waste immobilisation due to the potential durability of the final waste form and possibility to the accommodation of a wide variety of contaminants. The most commonly used glasses for this purpose are borosilicate. They are characterized by a good glass forming ability, chemical durability, thermal stability and ability to incorporate many kinds of waste elements. One of the main fission product in nuclear waste is <sup>137</sup>Cs which influence on structure and properties of the glasses was studied using molecular dynamics simulations.

Classical molecular dynamics simulations were conducted to simulate influence of gradual substitution of Na<sub>2</sub>O by Cs<sub>2</sub>O on structure of glasses from the SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO-Na<sub>2</sub>O system.

According to the conducted simulations increasing Na<sup>+</sup> by Cs<sup>+</sup> substitution degree is accompanied by diminishing decrease of B-O distance and increase of O-B-O bond angle. The same parameters in the alumino-silicate part of the network persist not changed. The presence of Cs<sub>2</sub>O initiates the change of the coordination number of boron to oxide, from 4 to 3 and [BO<sub>3</sub>] triangle formation. The sodium by caesium substitution increases the glass density but on the other hand increases also molar volume by expanding the size of the interstitials. In the studied glasses Na<sup>+</sup> atoms are mainly compensators of negative charges of [AlO<sub>4</sub>]<sup>-</sup> and [BO<sub>4</sub>] tetrahedra. It was observed that Cs<sup>+</sup> preferentially substitutes sodium in the charge compensating positions.

The analysis revealed that the glass structure modifiers start aggregating together and form clusters. The caesium atoms increase clustering ability of the modifiers and thus it could increase crystallization ability of the glassy matrix.

OA-85

**DIELECTRIC AND STRUCTURAL CHARACTERIZATION OF FERRO-ELECTRIC-RELAXOR CROSSOVER IN  $\text{Eu}_{0.01}\text{Ba}_{0.99}\text{Ti}_{1-0.0025-y}\text{Zr}_y\text{O}_3$  CERAMICS**

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$\text{BaZr}_y\text{Ti}_{1-y}\text{O}_3$  (BZT) solid solutions have attracted considerable attention due to their spectacular dielectric properties. Although few studies revealed better dielectric properties of BZT ceramics if substituted with earth-rare ions [1], the subject has not been investigated in depth. In addition, the rare-earth ions doped perovskite oxide ceramics possess excellent photoluminescence (PL) performance.

In the present paper, preparation and properties of  $\text{Eu}_{0.01}\text{Ba}_{0.99}\text{Ti}_{1-0.0025-y}\text{Zr}_y\text{O}_3$  ( $y = 0; 0.05; 0.15$  and  $0.3$ ) ceramics prepared by solid state reaction are described. Electrical characterization by impedance spectroscopy in the temperature range of  $(-150$  to  $150)^\circ\text{C}$  shows a composition-induced ferroelectric-to-relaxor (Fe-Re) crossover when increasing  $y$ , with very high values of permittivity and low losses at room temperature. The nonlinear dielectric properties of ceramic samples, *i.e.* tunability and  $P(E)$  loops were discussed in terms of compositionally-induced Fe-Re crossover.

A correlation between microstructure and PL response will be further performed in order to develop a non-contact, quick-response and compositional-change-sensitive spectroscopic method to probe the phase transition region.

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OC-86

**IMPROVING SYNTHESIS AND PROCESSING OF DENSE  
Sr-FERRITE MAGNETS**

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Permanent magnets are ubiquitous materials in both industry and society, for their ability to create spontaneous magnetic fields, such as in motors, generators, biomedical applications (RMN, sensors) or for daily household applications. Rare-earth-based permanent magnets (Sm or Nd) have been dominating the market for decades. This is due to the fact that RE-magnets provide large remanence (spontaneous field) and coercivity (resistance to demagnetization) simultaneously, which is a rare occurrence in nature. However, RE are considered critical raw materials as China controls almost 97% of RE-magnets production and export quotas. As a consequence, research has emerged whose main goal is to find alternative magnets that can substitute RE in selected applications. One of the most promising routes consists on creating or producing permanent magnets based on hexaferrites.

In this work, we present an attempt to further understand the correlation between magnetic properties and microstructure in dense strontium ferrite  $\text{SrFe}_{12}\text{O}_{19}$  (SFO) ceramics. We have fabricated dense SFO ceramics by both conventional and advanced sintering techniques. We observe that massive grain growth is easily activated at temperatures above 1200 °C, required for achieving maximum densification. However, coercivity dramatically decreases with grain size. We will present and discuss strategies to circumvent this issue, such as using grain inhibitors ( $\text{SiO}_2$ ) and employing fast sintering techniques such as flash sintering or spark plasma sintering. In addition, we present strategies for maximizing both remanence and coercivity simultaneously in SFO by tuning the starting powders properties through the control of synthesis and processing parameters.

Our results bring forward novel routes for fabricating and processing hexaferrites magnets, which have the potential to reduce sintering times, environmental and manufacturing costs, that are crucial at the industrial level.

OC-87

**CHARACTERIZATION OF THE SUPERCONDUCTOR-MULTIFERROIC  
TYPE MATERIALS BASED ON  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  /  $\text{YMnO}_3$**

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Presentation deals with physico-chemical properties of three ceramic composites based on  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ / $\text{YMnO}_3$  with proportions 2:1, 1:1 and 1:2.  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) is a first discovered high-temperature superconductor with transition point near 93 K [1]. In turn,  $\text{YMnO}_3$  is class (I) multiferroic with a strong coupling of magnetism and ferroelectricity [2]. A multiferroism of  $\text{YMnO}_3$  is arising at 72 K by long range order of Mn spins and question is how the magnetism of  $\text{YMnO}_3$  is affecting on superconductivity state of YBCO and in fact on properties of composite structure.

Both ceramic components were synthesized by solid state reaction method in separately routines. Finally, the calcination products were combined and formed as pellets under the pressure of 800 MPa. Above pressure was deducted due to the largest contribution of superconducting phase in YBCO component as well as the highest critical current density. After the heat treatment in oxygen atmosphere at 920 °C [3] the specimens were monitored by X-ray diffraction (XRD) method to evaluate the proper stoichiometry of ceramics.

In order to understand a specific behavior of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ / $\text{YMnO}_3$  composites the different measurement methods as SQUID magnetometry, four point electrical resistivity and impedance spectroscopy (EIS) will be used.

In the present work the results of magnetic susceptibility and electrical resistivity of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ / $\text{YMnO}_3$  will be discussed. Both methods are convenient for characterizing the critical current  $J_C$  and critical temperature  $T_C$  of the superconducting state.

Also, complementary results of the ferroelectricity are to be presented. It is expected the modulation of temperature point for ferroelectric ordering in manganite  $\text{YMnO}_3$  phase.

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OA-88

## GRAIN SIZE EFFECT ON DIELECTRIC PROPERTIES OF DENSE SUBMICRON BaTiO<sub>3</sub> CERAMICS

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Current demands in microelectronics for miniaturization, integration and topological compatibility with Si structures, high performances and use of environmental friendly technologies recommend BaTiO<sub>3</sub> as one of the main multifunctional materials used for building passive and active components. In particular, a high value capacitance in multilayer ceramic capacitor structures and tunable components impose the execution of dense BaTiO<sub>3</sub> ceramic single layers with thickness about 1 μm, therefore grain sizes below 500 nm. Technological reasons give great concern to knowing if the functional characteristics are maintained at such small grain size levels.

Since ferroelectricity is a cooperative long-range-order phenomenon generated by collective interaction of dipolar units, with decreasing number of dipoles as result of size reduction, the ferroelectric state becomes unstable and important property changes are expected with respect to the bulk state. The problem of the existence of a critical size in ferroelectrics is still under discussion and producing novel downscale ferroelectric nanostructures with specific geometries, to probe their properties as a function of their size and to discriminate between intrinsic or extrinsic effects, is of interest [1]. Variations of the functional properties of BaTiO<sub>3</sub> ceramics with reducing GS start when approaching GS to about 1 μm, but major deviations from the bulk properties are expected in the sub-micron range [2].

Commercial BaTiO<sub>3</sub> fine powders (60 nm particle size) were sintered by spark plasma sintering (SPS) at different temperatures, in order to obtain dense ceramics with various submicrometer grain sizes. After sintering, the ceramics were annealed in air, in order to reduce the oxygen vacancies characteristic in the SPS process. Phase purity and morphology of final dense ceramic samples (density > 95%) were investigated using XRD and SEM. Permittivity reduction and flattening of permittivity versus temperature dependence was observed when reducing grain size. This effect is interpreted as being related to an increased contribution of non-ferroelectric grain boundaries when grain size is in nanoscale range.

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OA-89

**THERMODYNAMIC ANALYSIS AND EXPERIMENTAL  
INVESTIGATION OF MULTICOMPONENT TRANSITION METAL  
OXIDE CERAMIS**

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Transition metal oxide ceramics have great potential in the field of semiconductor electronics, sensors and energy. However, as the quest for new materials with improved, advanced or novel properties is ever increasing, the possibility of developing multicomponent equimolar oxide ceramics become important with the prospect of synergising the properties of the individual oxides and/or arriving at exceptional properties that are not easy to obtain otherwise. Recent research [1–3] has shown interesting results in terms of the structure and the properties. The factors stabilising these oxides are yet to be understood. It is, therefore, necessary to develop a theoretical basis in order to understand such behaviour. In a first attempt to decrypt the behaviour, a Gibbs energy database was developed using which equilibrium calculations can be performed. These calculations are expected to reduce the number of trials required and help in identifying the synthesis conditions to produce a multicomponent oxide of a chosen crystallochemical nature. Based on a critical review of the available experimental information and assessed Gibbs energy functions for the boundary subsystems of FeO-MgO-NiO-CuO system [4–6], a set of self-consistent Gibbs energy database was constructed using the CALPHAD approach. Experimental validation was carried out for the selected compositions synthesised through a reverse co-precipitation (RCP) method. The calculated phase fraction plots were in reasonable agreement with that obtained from the Rietveld refinement of the X-ray diffraction data of the synthesised samples. Further confirmation was made through elemental mapping by energy dispersive spectroscopy. It was concluded that the established Gibbs energy database could be used for phase equilibrium calculations at various temperatures and overall compositions for the candidate system to identify the single phase region desirable for synthesis of multicomponent equimolar nanocomposite oxide ceramics.

**Keywords:** Multicomponent equimolar ceramics, Thermodynamic database, CALPHAD

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OA-90

**STABILIZATION OF HIGH CONCENTRATED ALUMINA  
SUSPENSIONS BY DIFFERENT DISPERSANTS**

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Slip casting is a widely used colloidal technique for the consolidation of ceramic powders from a suspension. The colloidal approach ensures homogeneous green body microstructure, therefore reducing post-sintering machining and production costs. The process itself consists of several steps. Firstly, dispersion of powder with controlled characteristics in a liquid, which is usually water, followed by stabilization of the new obtained suspension using different dispersants. When an optimum dispersion of particles is achieved and thus low slip viscosity, ceramic slurry can be poured in a porous mold, usually a gypsum one.

The influence of different dispersants on rheological properties of highly concentrated alumina ( $\text{Al}_2\text{O}_3$ ) suspensions was investigated. Used dispersants were Darvan C-N (an ammonium polymethacrylate water solution), 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt and citric acid. The amount of dispersants was varied from 0.1-1.25 wt.% in order to determine the optimal one. Rheological properties of prepared suspensions were determined by measuring the apparent viscosity at different shear rates. The optimum amount for Darvan was 0.75 wt.%, 0.1 wt.% for 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt, and 0.3 wt.% for citric acid. The obtained results show that the most effective dispersant is 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt given that only 0.1 wt.% was needed to stabilize the 70 wt.%  $\text{Al}_2\text{O}_3$  suspension.

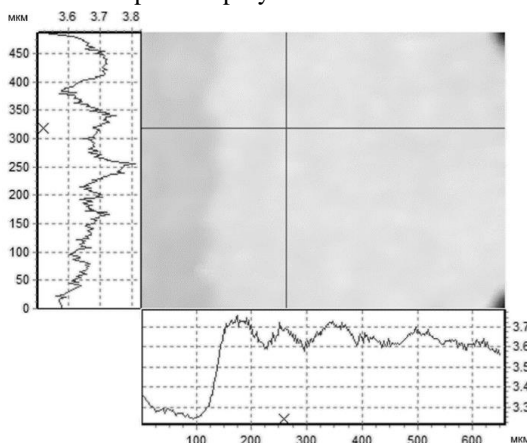
**Acknowledgements:** This work has been fully supported by Croatian Science Foundation under the project IP-2016-06-6000: Monolithic and composite advanced ceramics for wear and corrosion protection (WECOR).

OA-91

**OBTAINING BaTiO<sub>3</sub> CERAMIC TAPES WITH THICKNESS LESS THAN 500 nm BY TAPE CASTING METHOD FROM PREVIOUSLY COOLED SUSPENSION****S. Ivanchenko, S. Umerova, D. Baranovsky, A. Ragulya***Frantsevich Institute for Problems of Materials Science NASU, Krzhizhanovsky str., 3, 03680, Kyiv-142, Ukraine  
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The main problem that occurs during casting of thin tapes on lyophobic substrate is discontinuity of tape. Where a suspension is casted a thin layer, there is a fracture surface due to suspension collection into drops due to poor wetting of lyophobic surface of the carrier. There are several ways to solve this problem: change the chemical composition of suspension to improve the wetting or use of substrate without lyophobic coating. Of particular interest is the method of eliminating defects when both methods are unacceptable.

For suspension creation as solid phase was used BaTiO<sub>3</sub> nanopowder produced by “Nanotechcenter” (ua) with a particle size of 20–25 nm. A polymer solution of polyvinyl butyral (PVB) with molecular weights from 40 000 to 75 000 g/mol in organic solvents ethanol and butanol was used as the organic binder. Dibutyl phthalate was used as plasticizer. In this work was used the suspension of the following composition: the concentration of the polymer solution from 5 to 10 wt.%. The ratio of polymer-plasticizer was 2:1, and ratio of powder-polymer solution was 1:2.



*2-D profile of the film obtained from the pre-cooled to temperature of 5 °C suspension TCS-27*

Improvement of suspension wetting angle were acquired by its pre-cooling. Cooled to 5 °C suspension shows a reduction of wetting angle to the substrate by 7°. Roughness criteria of the tape obtained from pre-cooled suspension with a temperature of 5 °C were:  $R_z = 85$  nm,  $R_a = 24$  nm. The average thickness was 350 nm and tapes

shows good continuously. Roughness criteria of the film obtained from the suspension with temperature of 25 °C were:  $R_z = 110$  nm,  $R_a = 43$  nm. The average thickness was 900 nm and tape was strongly fragmented.

The obtained data shows that pre-cooling of suspension is not only improves continuous of the tape on lyophobic carrier without changing the chemical composition, but also reduce the thickness and roughness of the film.

OA-92

## **WEAR MECHANISMS OF MONOLITHIC CERAMICS**

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Advanced ceramics have been extensively used as anti-wear materials for various engineering applications, because of the wide range of their good properties, such as high temperature stability, hardness and strength, good wear and corrosion resistance, particularly at elevated temperatures where metal matrix composites have proven to be inadequate. Among the advanced oxide engineering ceramics, alumina ceramics have the unique combination of desirable properties for different applications: good wear and corrosion resistance, high hardness, excellent oxidation resistance and low production costs. Wear is one of the most commonly encountered industrial problems, leading to frequent replacement of components. Solid particle erosion can be defined as the degradation of material that results from repeated impacts of small solid particles. It occurs in a gaseous or a liquid medium containing solid particles. The present medium can change the velocity and direction of erodents (solid particles). If the erosion occurs at impact angles between 0° and 30°, it is regarded as abrasive erosion, while the one occurring between 60° and 90° is called impact erosion. Depending on the material and operating parameters, erosive wear can occur with plastic deformation and/or brittle fracture. Ductile materials will undergo wear by a plastic deformation process, where the material is removed by the displacing or cutting action of the eroding particle. On the other hand, eroded brittle material will be removed by the formation and intersection of cracks that cause grain ejection.

In this investigation, solid particle erosion tests were conducted to investigate the erosion wear behavior of the slip cast monolithic high purity alumina ( $Al_2O_3$ ) advanced ceramics at different particle impact angles at the room temperature. The erodent impact angles were 30°, 60° and 90°. Dry silicon carbide (SiC) was used as an erodent. Erosion mechanisms of all prepared ceramic samples were evaluated by measuring the weight loss and roughness parameters ( $R_a$ ,  $R_z$ ,  $R_m$ ).

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OA-93

**KINETICS OF TOPOCHEMICAL REACTION OF MAGNESIA  
SPINELS:  $\text{Mg}(\text{Cr}_{0,5}\text{Fe}_{0,5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0,5}\text{Cr}_{0,5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0,5}\text{Fe}_{0,5})_2\text{O}_4$  SOLID  
SOLUTIONS WITH SULPHUR OXIDES**

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Spinel-containing materials belong to an important group of refractories used as high-temperature unit linings. A crucial element of the conditions in which they are used is gaseous corrosion caused by sulphur oxides:  $\text{SO}_2$  and  $\text{SO}_3$ . In previous investigations into reactions of magnesia spinels with sulphur oxides it was found that spinels reactivity could be considerably influenced by a phase transition (order – disorder) in the structure of spinels, resulting in a change of their reactivity in relation to  $\text{SO}_2/\text{SO}_3$ .

The aim of the study was to investigate the kinetics of topochemical reactions between equimolar solid solutions of magnesia spinels and sulphur oxides before and after order – disorder phase transformation. The studies of the reaction of equimolar solid solutions  $\text{Mg}(\text{Cr}_{0,5}\text{Fe}_{0,5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0,5}\text{Cr}_{0,5})_2\text{O}_4$ ,  $\text{Mg}(\text{Al}_{0,5}\text{Fe}_{0,5})_2\text{O}_4$  with  $\text{SO}_3$  was undertaken due to the fact that in basic refractories spinels forms solid solution. Before kinetic investigations the spinels solid solutions were characterized by determining their: phase composition, density, porosity, specific surface area and degree of inversion vs. temperature. The investigations confirmed that the main product of reaction identified in all the cases was  $\text{MgSO}_4$  and  $\text{R}_2\text{O}_3$  (R = Al, Fe, Cr).

To take kinetic measurements, a semi-flow reactor was designed and constructed, which enabled a direct measurement, of changes in the mass of a sample during the reaction without having to stop the process. Investigations were carried out within the temperature 773 and 973 K and time range of: 0–7 h. Mixture of air and  $\text{SO}_2$  (13 %) was used.

Based on obtained results the kinetic curves were drawn. Obtained results have been compared with the kinetic results from previous work for one-phase spinels:  $\text{MgAl}_2\text{O}_4$ ,  $\text{MgFe}_2\text{O}_4$ ,  $\text{MgCr}_2\text{O}_4$ . The influence of degree of inversion in spinels structure on kinetics of the process were discussed.



OC-94

**NANOCOMPOSITE Ti-Si-N COATINGS PREPARED WITH  
DIFFERENT ROTATIONS**

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Thin ceramic coatings have been widely used for protecting the surfaces of various manufacturing tools and mechanical components. Different physical vapor deposition techniques are used for their preparation. In industrial physical vapor deposition systems substrates move in a planetary-like manner to ensure uniform coating deposition on all surfaces. In this study, influence of the type of substrate rotation on phase composition, crystal orientation, microstructure, and surface morphology of nanocomposite Ti-Si-N coatings was investigated. Coatings were prepared in an industrial magnetron sputtering unit and characterized by X-ray photoelectron spectroscopy, X-ray diffraction, scanning electron microscopy, transmission electron microscopy, nanoindentation technique, stylus profilometry and atomic force microscopy. It was found that the type of rotation has great impact on crystal orientation and microstructure, and consequently on mechanical properties and surface morphology. Coating prepared with single rotation was of columnar and porous microstructure with crystallites oriented in 111 direction. On the other hand, there were no signs of columnar growth for coatings prepared with double and triple rotations which were of compact and dense microstructure with crystallites oriented in 200 direction. Coatings prepared with higher number of rotations were smoother and of considerably higher hardness.

OA-95

## FAILURE BEHAVIOUR FOR THERMAL BARRIER COATINGS BY ACOUSTIC EMISSION

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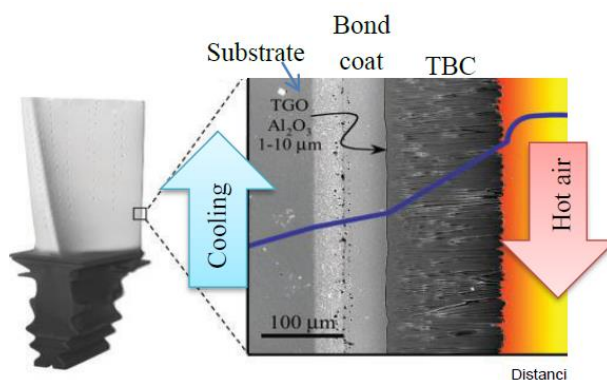
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Thermal barrier coatings (TBC) have great potential used in gas turbines due to that allows operate at high temperatures. These coatings are multi-layer systems consisting of: yttria-stabilized zirconia ceramic top layer (YSZ), an bond layer whose function is to reduce tensions between the ceramic layer and the substrate, both layers were deposited by plasma suspension (SPS) on a metal substrate.

This work presents the initial results obtained to analysis the formation and propagation of cracks into different layers of the TBC, using a 3-point bending test supported by non-destructive method such as acoustic emission (AE), which is a test widely used in different fields to detect the energy that is propagated through a material in the form of elastic waves, this technique will allows to differentiate in which layer the crack is generated or if it passes through different layers.

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**Keywords:** Thermal barriers, three point bending test, acoustic emission (EA)

OC-96

## COMPOSITE POWDER MATERIALS OF FeNiCrBSiC-CrB<sub>2</sub> SYSTEM FOR PLASMA-SPRAYED COATINGS

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Among the spraying materials self-fluxing alloys have been widely used in many industrial fields to protect the machine parts against wear and corrosion. The structure of FeNiCrBSiC plasma-sprayed coatings consists of Ni- or Fe-based metallic matrix and dispersed hard boride and carbide grains [1,2]. However, due to the short time of spraying process the size of hard phases is not enough to provide wear-resistance under high-demand conditions [3].

The wear resistance of FeNiCrBSiC plasma sprayed coatings can be substantially improved by reinforcement with hard borides such as CrB<sub>2</sub>, TiB<sub>2</sub>, ZrB<sub>2</sub>. The goal of this study is to develop the composite powder materials of the FeNiCrBSiC-CrB<sub>2</sub> system for the plasma-spraying wear-resistant coatings

In this study commercially available Fe-based self-fluxing alloy (Fe – base, Ni – 37 wt.%, Cr – 14 wt.%, Si – 2.5 wt.%, C – 1.4 wt.%, B – 2.2 wt.%, Mo – up to 2%) was mixed with 20 wt.% of CrB<sub>2</sub> powder (98%, 2–3 μm). The powder mixture was pressed in bulks and sintered then in vacuum at 1100 °C for 40 minutes.

The structure of the FeNiCrBSiC-CrB<sub>2</sub> composite material consists of Fe-based matrix and distributed in boride grains. It has been found that during sintering process chromium boride interacts with components of self-fluxing that leads to the dissolution of CrB<sub>2</sub> particles and formation of complex chromium-iron-molybdenum boride grains of 10–20 μm in size.

The FeNiCrBSiC-CrB<sub>2</sub> composite material was crushed and sieved within the range of 60–100 μm in order to achieve excellent fluidity of powder during plasma spraying. Each particle of composite powder material represent itself conglomerate comprising both the metal matrix and boride grains. The plasma spraying of developed composite powder materials results in the formation of high-quality composite coatings.

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OC-97

## CERIUM AND PHOSPHORUS MODIFIED SILICON OXYCARBIDE LAYERS ON METALLIC SUBSTRATES

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Materials based on silicon oxycarbide might be simply called black glasses. It is due to the fact that besides the carbon bonded into the structure of an amorphous material, the free carbon is present. It is possible to obtain black glasses in the form of thin layers with the use of the proper organosilicon precursors. Subsequent ceramization is required [1,2]. Such materials are characterised by high corrosion resistance, chemical and thermal stability. Their bioactivity was also proven [3]. In terms to improve e.g. mechanical and surface properties or tendency to crystallisation, it is possible to modify Si-O-C materials with aluminum, boron, cerium, phosphorus ions or even nanoparticles [e.g. 4].

In this work, modifications of silicon oxycarbide based materials by cerium and phosphorus ions were proposed to enhance corrosion resistance and bioactivity, respectively. Cerium ions enable to obtain self-healing, germicidal and fungicidal properties [5]. Oligo- and polysilsesquioxanes were obtained with the sol-gel method and afterwards deposited with dip-coating technique on metallic substrates. Apart from structural and morphological studies, phosphorus doped layers were examined to evaluate their bioactivity with the use of the so-called Kokubo test. Obtained coatings are continuous and hermetic. Both types of layers differ strongly in the value of surface properties. Obtained modified black glasses might be applied as protective layers on various metallic substrates as materials for medicine, catalyst or in fuel oxide cells.

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**Keywords:** silicon oxycarbide, black glasses, cerium, protective layers, spectroscopy

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OA-98

## INVESTIGATION OF YTTRIA-STABILIZED ZIRCONIA COATING

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Improving of the efficiency of gas turbines can be achieved by increasing the exhaust gas temperature. However, the temperature of gases influence on the microstructure of components located inside hot section. In order to protect base metal against high temperature and aggressive corrosion environment, different type of coating are performed including thermal barrier coatings (TBC). Thermal barrier coatings perform the important function of insulating components, like gas turbine and jet engine parts, operating at elevated temperature. The most commonly use material is yttria-stabilized zirconia (YSZ) which characterized by resistance to fatigue up to 1150 °C. In this paper investigation of YSZ coatings deposited on nickel alloy was investigated. Research includes microstructure analysis using light and scanning electron microscopy equipped with EDS spectrometer, X-ray diffraction and measure of surface profile.

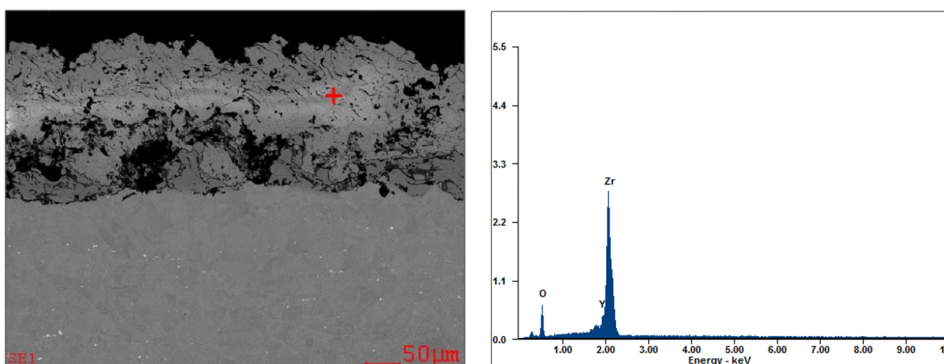


Figure 1. Thermal barrier coating: a) microstructure; b) EDS spectrum

OC-99

**SYNTHESIS OF SiO<sub>2</sub>/CoFe<sub>2</sub>O<sub>4</sub> MULTIFUNCTIONAL THREE-LAYERED CORE-SHELL PARTICLES FOR INVERTASE IMMOBILIZATION**

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Multilayered core-shell particles were synthesized by wet-chemical process. The monodispersed silica core particles (having average size of 0.4 μm) were prepared by hydrolysis and condensation of tetraethylorthosilicate and used as templates for assembly of three layers. The first internal layer was prepared by deposition of cobalt ferrite nanoparticles (obtained by co-precipitation) on the surface of functionalized silica core particles. The second layer on the previously obtained particles with silica core and ferrite (CoFe<sub>2</sub>O<sub>4</sub>) shell was formed by deposition of silica nanoparticles from a silicate solution. The obtained silica layer was mesoporous which enabled immobilization of invertase from *Saccharomyces cerevisiae* within silica layer. To prevent enzyme leaching from the bilayered core-shell particles, the latter were covered with the third external silica layer obtained by deposition of silica nanoparticles from a silicate solution. The obtained composite particles with three functional layers allowed the use of enzyme for many repeated cycles and magnetic separation from reaction mixture.

OT-100

**MgO-ZrO<sub>2</sub> CERAMICS BASED ON RECYCLED MAGNESIA-CARBON BRICKS**

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The increasing amounts of waste and the simultaneous decrease of waste disposal space pose a big problem for communities. It would be environmentally unthinkable and expensive for end users of refractories to store the spent refractories at landfill sites. One of the methods for solving this problem may be recycling of end-of-life refractories.

Magnesia-carbon (MgO-C) refractories are mainly applied in the steel industry. They consist of magnesia (dead-burned or fused) and carbon. Depending on the specific

application, these refractory materials have various life cycles after which they are replaced by new ones. Consequently, a large amount of spent MgO-C is produced. This waste may be used as a potential source of magnesia for modified magnesia refractories like MgO-ZrO<sub>2</sub>.

The mechano-physical properties of magnesia-zirconia refractory ceramics based on the recycled spent MgO-C materials have been investigated. The MgO-C waste was previously calcined to remove carbon and the obtained secondary magnesia clinker was used to prepare MgO-ZrO<sub>2</sub> refractories. It was applied as an individual component of raw materials mix or as a base of fused MgO-ZrO<sub>2</sub> co-clinker obtained by the melting process in a laboratory electric-arc furnace.

After firing at 1660 °C the ceramic properties of the MgO-ZrO<sub>2</sub> obtained were measured. Linear shrinkage, open porosity, apparent density, cold crushing strength, gas permeability, thermal shock resistance (water cycles) as well as resistance to corrosion by two different corrosive agents were determined. The research results show that recycling of spent MgO-C refractories is possible. Moreover, an addition of zirconia improved the refractories' properties.

OA-101

### **3D COMPUTER MODEL AND CRYSTALLIZATION PATHS FOR SYSTEM FeO-SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub>**

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System FeO-SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> is used in the description of pyrometallurgical processes, as well as in the study of petrological and ceramic objects. The system describes the processes of sintering of iron-containing ores and slags, which are used in the production of steels and the melting of non-ferrous sulfides. The complexity of this ternary system investigation lies in the fact that there is the fragmentary description of liquidus surfaces and immiscibility regions of two melts [1,2].

In addition, only the schematic description is given for the binary system SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub>, and the system FeO-Fe<sub>2</sub>O<sub>3</sub> is presented as a part of the system Fe-O, taking into account two compounds: magnetite FeO·Fe<sub>2</sub>O<sub>3</sub>=R<sub>1</sub> and wüstite Fe<sub>x</sub>O=R<sub>2</sub> (congruently and incongruently melting, respectively) [2]. The accepting of wüstite composition corresponding to the formula FeO will not be correct at the considering of ternary system. The binary system FeO-SiO<sub>2</sub> contains a congruently melting compound fayalite 2FeO·SiO<sub>2</sub>=R<sub>3</sub>. We have developed the assumed prototype of the system FeO-SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> (A-B-C) based on the available experimental data for the liquidus surfaces and binary systems, and taking into account the schematic phase diagram of V.A. Zharikov

[3]. It is assumed that there are four eutectic invariant reactions in the system:  $L_{E1} \rightarrow A + R_1 + R_2$ ,  $L_{E2} \rightarrow R_1 + R_2 + R_3$ ,  $L_{E3} \rightarrow B + R_1 + R_3$ ,  $L_{E4} \rightarrow B + C + R_3$ . The model of phase diagram includes two surfaces of liquids immiscibility: one surface is situated near the component FeO, second surface adjoins to SiO<sub>2</sub> and occupies the most part of phase diagram. There are 8 liquidus surfaces corresponding to three initial components (FeO, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>), two polymorphous modifications of SiO<sub>2</sub> (cristobalite and tridymite) and three compounds (R<sub>1</sub>-R<sub>3</sub>); 22 ruled surfaces on the boundary of phase regions with melt and 12 ones on the boundary of solid-phase regions. Obtained model of phase diagram for system FeO-SiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> can be modified with the appearance of new data for its topological structure, and it can be used for the calculation of crystallization paths and the analysis of microconstituents for any concentration fields.

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OA-102

**FROM GALLIUM OXIDE TO ZINC MODIFIED GALLIUM  
OXYNITRIDE NANOPARTICLES AS PROMISING MATERIAL FOR  
PHOTOCATALYTIC WATER SPLITTING**

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Solar irradiation can directly split water into its components, hydrogen and oxygen, by photocatalysts. The highest quantum yields for a single particle photocatalyst is reported for metal oxides. The most promising materials involve cationic species of a  $d^0$ - or  $d^{10}$ -electronic configuration, such as Ga in  $\text{Ga}_2\text{O}_3$ , which is able to evolve  $\text{H}_2/\text{O}_2$  simultaneously in a stoichiometric ratio when loaded with suitable co-catalyst. Because  $\text{Ga}_2\text{O}_3$  has a large band gap of about 4.6 eV it works just in the UV regime. Domen et. al. developed zinc doped gallium-oxynitride ( $\text{Ga}_{1-x}\text{Zn}_x$ )( $\text{N}_{1-x}\text{O}_x$ ) by nitridding mixture of  $\text{Ga}_2\text{O}_3$  and ZnO in a solid solution, which is efficient for absorbing visible light with a bandgap of 2,6 eV [1].

Chemical vapor synthesis (CVS) is a highly suitable method to prepare well-defined photocatalyst materials with a large specific surface area and a high degree of crystallinity.  $\text{Ga}_2\text{O}_3$  is doped with zinc by CVS and nitrided under  $\text{NH}_3$  flow (300 sccm) at 800°C for 30 min. showing 7x and 32x higher  $\text{O}_2$  and  $\text{H}_2$  evolution rate, respectively, when compared to pure  $\text{Ga}_2\text{O}_3$ . By adjusting the precursor entry positions in the CVS system and combining two reactors it is possible to get a physical mixture of  $\text{Ga}_2\text{O}_3$ -ZnO in the gas phase or even core-shell structures, depending on the process parameters. X-ray Diffraction (XRD) analyzed by Rietveld refinement reveals crystal phases, cell parameters, as well as crystallinity and amount of Zn. These results are supported by High-Resolution Semi-Electron Microscopy (HRSEM) in combination with Energy Dispersive Spectroscopy (EDS). The specific surface area of the samples is analyzed by using low temperature nitrogen adsorption. The bandgap is determined by using UV-VIS Spectroscopy. The samples are tested regarding their photocatalytic water splitting potential. A novel map correlates structural characterization results and process parameter fields with catalytic performance for  $\text{Ga}_2\text{O}_3$  nanoparticles.

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OC-103

**SYNTHESIS OF INORGANIC POLYMERS BASED ON DIFFERENT  
RAW MATERIALS**A. Boros, T. Korim, I. Balczár*Institute of Materials Engineering, University of Pannonia, Egyetem Street 10, H-8200,  
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Over the past decades, alkaline activated inorganic polymer (AASP) foams, as eco-friendly porous materials, have been great attention due to their favorable production conditions (e.g. low energy requirements) and their promising properties (low shrinkage after foaming, mechanical and chemical stability, high temperature resistance etc.). These materials can be used, inter alia, in photocatalytic degradation processes or as catalyst supports, membrane supports, heavy metal adsorbents. The primary process used for the manufacture of AASP foams are taken over from the gas-concrete production. The components added to the aqueous “geopolymer” slurry (e.g. Si or Al powder) are able to generate in situ H<sub>2</sub> gas due to the oxidation of Si or Al metal in a strong alkaline environment. The gas formation can also be achieved by the addition of H<sub>2</sub>O<sub>2</sub> because H<sub>2</sub>O<sub>2</sub> is decomposed into water at high pH. However, with these foaming processes generated pores are typically closed, which limits the application of these materials. AASP foams with a mesoporous matrix and one-way lamellar macroporosity can be produced mainly by combined processes (saponification/peroxide decomposition/gelcasting). With this technique, foams with up to 85% pore content (~70% open porosity) can be produced. Although their low compressive strength (~ 0.45 MPa) limits the application possibilities, by increasing the strength, these AASP foams may become applicable, such as catalyst supports in photocatalytic degradation processes.

In the study metakaolinite-based AASP foams were produced by saponification/peroxide decomposition/gelcasting combined method. As an activating component the mixture of NaOH and water glass was used, and different vegetable oils, sodium oleate and hydrogen peroxide were added in order for saponification reaction and foaming. The combined foaming process led to the formation of interconnected pores: macroporosity was created due to the in situ formation of the soap molecules in the saponification reaction. This combined route allowed the production of designed porosity AASP foams. During experiments were investigated the porosity, pore size distribution and compressive strength of the specimens. The results show that the size and volume of pores can be controlled by the percentage of oil and peroxide.

OT-104

**EFFECT OF CHAMOTTE AMOUNT AND PARTICLE SIZE ON  
THERMAL EXPANSION BEHAVIOUR OF CERAMIC  
SANITARYWARE PRODUCTS**

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Vitreous ceramics are the materials that consist of clay, kaolin, feldspar and quartz. They are sintered with a glazed surface at around 1250 °C. These ceramics are commonly called as sanitaryware and they are used in hygienic parts of everyday life such as closets, washbasins, shower stalls, urinals. Fine fire clay (FFC) and vitreous slip are the types of slips used in the production of ceramic sanitaryware. Large size products are usually produced with FFC sludge, which generally require less deformation behaviour. Chamotte material, a kind of sintered clay, is used in FFC bodies for providing low sintering deformation to the body. In this study, thermal expansion behaviour of FFC bodies was investigated depending on the chamotte amount and particle size. The developed recipes were tested with a dilatometer. It is observed that thermal expansion coefficient decreased with decreasing chamotte amount. Simultaneously, deformation of body has reduced depending on the chamotte amount. On the other hand, thermal expansion coefficient was observed to be proportional to chamotte particle size while it did not have effect on deformation of body.

OT-105

**SYNTHESIS OF KAOLINITE NANOSCROLLS USING  
SOLUTION/STIRRING AND SOLVOTHERMAL METHODS**

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Kaolinite nanoscrolls were produced using kaolinite-cetyltrimethylammonium chloride (kaolinite-CTAC) intercalation complex. These halloysite-like nanoscrolls can be applied as nanoscale reactors, as nanofillers for clay polymer nanocomposites and as carriers for the controlled release of reagents etc. Kaolinite-dimethyl-sulfoxide and kaolinite-urea precursors were synthesized using cost-efficient homogenization method. These precursors were found to be favourable to produce kaolinite-methanol complexes using the solution/stirring and the solvothermal methods, as well. The formed kaolinite-methanol complexes were suitable to form kaolinite-CTAC complex. Finally, the created

kaolinite-CTAC complex was extensively washed, and this resulted in the formation of kaolinite nanoscrolls. The influence of the solution/stirring and solvothermal method on the formation of the kaolinite complexes and nanoscrolls was investigated using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), thermogravimetry (TG), scanning and transmission electron microscopy (SEM, TEM).

OT-106

### **HIGH TEMPERATURE VISCOSITY OF PORCELAIN STONEWARE BODIES**

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The viscosity of porcelain stoneware at high temperatures is crucial to understand the vitrification path, the viscous flow sintering kinetics and the pyroplastic deformation of this material. For the determination of the porcelain stoneware final viscosity is necessary to consider both the viscosity of the liquid phase formed by the melting of feldspars – and other minerals – and the viscosity of the body made up of a suspension of crystals dispersed in the melt. Fundamental theoretical background exists with semi-empirical constitutive laws of viscous flow sintering and glass densification as well as high viscosity liquids and melts [1]. Since different approaches are needed to measure/estimate the two viscosities, the best model for ceramic tiles has been defined by contrasting experimental data with calculated viscosities within the IPERCER project [2]. It worth be noticed that the parameterization depends on the chemical composition of the liquid phase and the persistence of crystals in the melt (related to the solubility of solids into liquid). Further variables, such as porosity, particles size and shape, play a significant role. Existing models for high temperature viscosities of glasses and melts present some limits that will be overviewed. In particular, the well-known model proposed by Fluegel [3] takes into account a large number of oxides, allowing its application to melts characterized by wide ranges of composition. On the other hand, the maximum concentration of alumina expected by the model is too low for the systems here in study, generating a significant error between the calculated and the measured viscosities. The more recent model proposed by Giordano et al. [4] is based on a lower number of oxides, but the alumina levels are more compatible whit those of the systems of interest.

**Keywords:** porcelain stoneware; viscosity; liquid phase; crystals

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OC-107

**CHARACTERIZATION OF SOLIDIFICATION/STABILIZATION  
SEDIMENT TREATMENT WITH NATIVE CLAY-PILOT  
PLANE SCALE**

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Fast technological development and exploitation of surface waters degraded water resources, and at the same time the sediment as well, which represents habitat of numerous organisms. Remediation ex situ technic solidification/stabilization was the most applicable treatment of contaminated sediment. S/S treats the sediment by translating it into heavily soluble stable oxides or with the addition of silicates and the alkali, is converted to an insoluble glass mass, because of safe disposal. Clay and modified clays showed as convenient immobilization agent for the treatment of contaminated sediment. Clays play important part in environment because they represent natural "sponge" of pollutants, binding their anions or cations through ionic exchange or through adsorption. Large specific surface, chemical and mechanic stability, layered structure, high capacity of cation exchange and other characteristics make clays excellent adsorbent. Physical characteristics of such treated sediment are capable for compact disposal and maximum use of space at the landfill. The problematic of sediment from Great Backi Canal contaminated with heavy metals was showed in this work. The method of sediment treatment by solidification/stabilization applying immobilized agent such as clay was shown here. A research was performed on a pilot plant under atmospheric conditions, after which it was approached to the main goal of this work and that was characterization of disposed sediment after the treatment. The characterization of disposed sediment was done by using modified sequential extraction, bioavailability with HCl, as well as compressive strength of such stabilized mixtures of sediment and clay. The quality of sediment was determined by pseudo-total content of metals in the initial sample of sediment and the results showed that in accordance to the tested metals the contaminated sediment was of class 4 from the aspect of copper and zinc, i.e. of class 3 i.e. of class 2 from the aspect of nickel and cadmium. By analyzing of disposed mixtures after the treatment on the pilot landfill it was established that all treated samples showed decreasing of metal concentration in soluble phase. There was a transition of metals into the fourth residual phase and on this way their mobility was decreased as well as their impact on the environment. According to EPA S/S materials with the strength bigger than 0.35 MPa are considered too have satisfactory compressive strength. This minimal value was recommended to create stable foundation for disposal of such materials on landfills. Both mixtures of disposed sediment and clay as immobilized agent showed values far above 0.35 MPa.

OT-108

**THE INFLUENCE OF ADDITION OF THE ZIRCONIUM OXIDE ON  
SOME TECHNOLOGICAL PARAMETERS OF THE RAW  
PORCELAIN GLAZE**

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The research focused on the influence of addition of zirconium oxide on some technological parameters of porcelain ceramic glazes, which were created to fire at temperature 1230°C. The examined glazes had a constant molar ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3 = 4$ ,  $\text{CaO}/\text{MgO} = 1$  and  $\text{Na}_2\text{O}/\text{K}_2\text{O} = 1$ . To this glazes, the zirconium oxide was added in five different amounts (1,5; 3; 6; 12; 24 wt%). Glazes were ground in a planetary mill for 30 minutes and dried. Raw glazes were used to designate the characteristic temperature at High Stage Microscopy (HSM). Fired glazes were used to dilatometric measurements.

As the result of examination some parameters were obtained such as: characteristic temperatures obtained by HSM and dilatometric measurements by mechanical dilatometer, coefficient of thermal expansion by dilatometric method and the viscosity of glaze as a function of temperature, using Vogel–Fulcher–Tammann models. The obtained results indicate on a lot of important dependencies between the content of zirconium oxide in glaze and examined properties.

**Keywords:** glaze, porcelain, zirconium oxide, HSM, viscosity

OT-109

**MANUFACTURE OF ALKALI ACTIVATED CEMENTS USING AIR-  
COOLED SLAG**

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Alkali activated cements play an increasing role as a successful substitute of ordinary Portland cements due to their superior durability and environment friendliness. Based on the chemical composition of these binders, alkaline cements can be divided to two basic categories: high- and low-calcium cements. Most commonly used starting material for high-calcium system is blast furnace slag (BFS), which can be activated using alkali hydroxides, carbonates or silicates.

This study introduces a novel activation method to manufacture high-calcium alkali-activated cements (AACs) based on inactive air-cooled blast furnace slag (ACS), which is well-crystallized and hydraulically unreactive. To enhance its reactivity we developed a new procedure, called mechanochemical activation. Mechanochemical activation is technically an intensive grinding, where the formed amorphous phase reacts later with the alkaline solution.

During experiment grinding parameters were altered (grinding time, the mass ratio of sample to grinding body, and rotation speed). The structural and morphological changes of mechanochemically activated ACS were followed by X-ray diffraction (XRD), and Fourier transform infrared (FTIR) spectrometry, scanning electron microscopy (SEM), as well as determination of particle size distribution. The applicability of activated ACS in alkali activated cements was characterised using compressive strength at 28 days.

The ACS partially amorphised by mechanochemical activation proved to be a valuable component for AACs with its maximum strength (49.1 MPa) in mortar surpassing that of granulated slag (30.2 MPa).

OT-110

## **PRODUCTION OF SLAG BASED ALKALI-ACTIVATED CEMENT FOAMS**

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The development of sustainable construction and building materials with reduced environmental footprint is attracting increased interest in the housing and construction industry worldwide. Recent innovations have led to the development of alkali activated cement foam (AACF), which combines the performance benefits and operational energy savings. These new binders can be produced from industrial wastes, such as blast furnace slag with comparable properties to the commercial autoclaved aerated concrete.

Our aim was to produce alkali activated cement foams derived from industrial waste blast furnace slag with similar properties to the autoclaved aerated concrete. AACFs were manufactured using blast furnace slag, sodium silicate and sodium hydroxide (as activating solution), H<sub>2</sub>O<sub>2</sub> (as foaming reagent) and sodium oleate solution (as surfactant). The weight percent of the foaming reagent (1.0, 1.3, 1.5, 2.1 wt%) and surfactant solution (4.8, 6.6, 8.3 wt%) were altered and the effect on the nature, size, distribution of the voids was investigated. Physical properties (compressive strength, bulk density and thermal conductivity) of the foams were also measured at the age of 7 days.

According to results slag based AACFs proved to be valuable insulating material with comparable properties to the autoclaved aerated concrete.

OT-111

## POWDER RHEOLOGY OF SPRAY DRIED BODIES FOR PORCELAIN STONEWARE SLABS

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The technological behavior of porcelain stoneware bodies strongly depends on the rheological properties and response to compaction of spray-dried powders during the deposition and pressing stages, particularly in the case of large slabs. Although the literature offers some insights into the characteristics of spray dried powders for ceramic tiles (both red and porcelain stoneware), no data are available on bodies utilized in large slabs (>4 m<sup>2</sup>) by novel technologies [1,2]. In order to fill this gap, a systematic approach to properties and behavior of spray dried powders for porcelain stoneware large slabs was carried out within IperCER project [3]. For this purpose, nine industrially-manufactured spray dried bodies were characterized for particle size and agglomerate size distribution; shape, moisture and internal porosity in function of agglomerate size; rheological properties of powders (flowability, static and dynamic angles of repose, poured and tapped densities) with effect on the compaction and firing behavior. The aim was to gather the information necessary to model the behavior during deposition (in mould or on tape). Results reveal the occurrence of dissimilarities with significant effects on some rheological properties (flowability and tapped density, but not angles of repose), leading to some differences in the features of compacted bodies.

In conclusion, there is room to improve the performance of spray dried bodies by pointing out a compromise between powders flowability, compaction and densification during firing. The challenge is to transfer this progress to the management of industrial spray driers, at present focused on energy efficiency and constancy in the moisture content.

**Keywords:** Porcelain Stoneware, Powder Rheology, Spray Dried Bodies.

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OT-112

**INVESTIGATION INTO INFLUENCE OF BINDING REGULATORS ON  
THERMOMECHANICAL PROPERTIES OF CEMENT FREE  
CASTABLES**

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This paper presents an influence of selected chemical additions on thermomechanical properties of cement free castables. The plasticizers, superplasticizers and new generation additives used in production of refractory castables were described. Mechanism of chemical reaction in the system: additive - concrete has been presented. Experimental part of the project includes research on the optimized concrete mixes to achieve satisfactory properties: bulk density, apparent porosity, crushing strength, permanent linear change after firing, phase composition, refractoriness under load, linear expansion, TG/DTA. The influence of following additives were tested: Skorbet-N, Castament FS 20 and TPS. The research confirm the positive effect of the additives on rheological properties of concrete mixture, reduction of the mixing water and increasing of mechanical properties of materials after drying and firing at 1000 °C temperature. Additionally, the results of testing refractoriness under load and linear expansion showed, that the addition of Skorbet-N, Castament FS 20, TPS has not been deteriorate the thermomechanical properties of cement free castables.

OA-113

**SONOCATALYZED SOL-GEL DERIVED NANOPOROUS SILICA  
EVALUATED MAINLY BY MORPHO -TEXTURAL PROPERTIES**

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Two series of silica wet gels were synthesized via ultrasonic assisted sol-gel processing, at 35 °C and 45 °C, respectively. Silica precursor, tetraethoxysilan, TEOS, was hydrolysed and polymerized under different initially alkaline conditions. The used

base catalyst was the ammonia. The molar reactants ratio, TEOS:H<sub>2</sub>O:EtOH:NH<sub>3</sub> as 1:25:20:0.006 was rigorously kept constant. Actually, the supplied ammonia solution concentration was varied, such as, for each of samples of a series was prepared solution with different ammonia content: 25%, 12.5%, 6.25%, 3.125% NH<sub>3</sub>. The ammonia solution dilution was obtained by using necessary water fractions on the expenses of total water used in the reactant mixture, in order to keep constant, the hydrolysis ratio,  $r$ , TEOS: H<sub>2</sub>O =1:25. Consequently, the supplied ammonia solutions exhibited different pH values. We systematically have examined the influence of the using solutions with different base concentrations and pH on the morpho-textural properties of the obtained silica mesoporous matrices at 2 different temperatures. For the sonogels series synthesized at 35 °C, the pore diameter is decreasing with the ammonia concentration. In the other series of samples, synthesized at temperature of 45 °C, an opposite behaviour was observed. Specifically, the pore diameter decreased with the increasing of the ammonia supplied solution concentration. The specific BET surface areas, measured for both series of sonogels samples were in the range of 200÷330 m<sup>2</sup>/g. The total pore volume in the series of samples synthesized at 35°C, is increasing with the decreasing of the ammonia concentration; while, for the 45 °C series of samples it remains relatively constant. Within the series of samples synthesized at 35°C, the particle size of silica colloids increased with the ammonia concentrations, while the sonogel samples synthesized at 45 °C have an opposite behaviour. The sonogel sample with the highest free space, total pore volume= ~0.6 cm<sup>3</sup>/g, was obtained by using the 12.5% ammonia solution and 35 °C the synthesis temperature.

OA-114

**NUMERICAL CALCULATION OF EFFECTIVE ELASTIC AND THERMAL PROPERTIES OF OPEN-CELL MODEL FOAMS BASED ON COMPUTER-GENERATED DIGITAL MICROSTRUCTURES**T. Uhlířová, W. Pabst*Department of Glass and Ceramics, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28 Prague 6, Czech Republic*

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Porous ceramics and highly porous cellular ceramics (ceramic foams) have a broad variety of applications, ranging from filters and catalyst supports to lightweight kiln furniture, high-temperature insulation panels and furnace linings. Both the mechanical and thermal behavior is important for these applications, and the basic microstructure-dependent properties determining this behavior are elastic constants and thermal conductivity. It is well known that the elastic moduli and thermal conductivity decrease with increasing porosity, but the nature of this porosity dependence is influenced by other factors such as pore shape or pore surface curvature. Although model relations are available in the literature that can be tentatively used to describe this

porosity dependence they are not always reliable for predictive purposes. Even the famous and relatively successful Gibson-Ashby power-law relation for open-cell foams (in other contexts called differential model or differential scheme model) tends to overestimate the elastic moduli and thermal conductivity of real open-cell foams. However, in the case of real materials it can always be argued that additional microstructural defects are responsible for the low values measured. In this work, using numerical calculation of effective elastic and thermal properties based on computer-generated (and thus defect-free) digital microstructures, we show that this is not the case and thus the common argumentation is flawed. Four types of open-cell foams with solid struts have been constructed: cubic cell foams, Kelvin cell foams, random foams and a recently proposed new type of model foam based on the Gibson-Ashby cell. Numerical calculations have been performed using commercial software allowing the calculation of the effective thermal conductivity (second-order tensor) via the explicit-jump method and the effective elastic constants (fourth-order elasticity tensor or stiffness matrix) via a FFT-assisted iterative solution of the Lippmann-Schwinger equation. Periodic boundary conditions have been used throughout, and all calculations have been performed for sufficiently high phase property contrast, so that the results are valid irrespective of the solid phase. It is shown that, surprisingly, our new Gibson-Ashby-cell-based model foam does not at all obey the Gibson-Ashby power-law prediction. Actually only the Kelvin cell foams are really close to the Gibson-Ashby power-law prediction, while all other foams have significantly lower effective property values, with random foams being close to the Pabst-Gregorová exponential prediction for porosities below 30 % and the cubic cell foams and our new model foams based on the Gibson-Ashby cell are significantly below the exponential prediction for porosities smaller than 35 % and 50 %, respectively. Finally the results are compared with cross-property predictions and, for the cubic cell foams, with the results of analytical unit-cell calculations (Voigt-Reuss homogenization).

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OA-115

## CHALLENGES IN FABRICATION OF FERROELECTRIC THIN FILMS SUITABLE FOR USE IN TUNABLE MICROWAVE DEVICES

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Over decades ferroelectric materials are recognized as excellent candidates for numerous applications in microelectronic devices. Potential application of ferroelectrics for fabrication of tunable microwave devices becomes significant in last years. In contrast to semiconductors, MEMs and magnetic systems, ferroelectric thin films offer higher values of tunability, lower dielectric losses at low applied voltages and lower price of fabrication.

Barium titanate is one of the most investigated ferroelectric materials due to the high dielectric constant, low dielectric losses, and good piezoelectric and ferroelectric properties at room temperature. It is noticed that barium titanate based materials, where part of barium and/or titanium ions are substituted by elements as strontium and zirconium have huge potential application in microwave technologies.

BaTiO<sub>3</sub>, Ba<sub>1-x</sub>Sr<sub>x</sub>TiO<sub>3</sub> ( $x = 0.1, 0.2, 0.3, 0.4$ ) and BaTi<sub>1-x</sub>Zr<sub>x</sub>O<sub>3</sub> ( $x = 0.1, 0.2$ ) thin films were prepared by chemical deposition technique. As sources of Ba<sup>2+</sup>, Sr<sup>2+</sup> and Zr<sup>4+</sup> were used barium carbonate, strontium acetate and zirconium-oxychloride-octahydrate. Acetic acid and 2-metoxyethanol were used as solvents. Obtained solutions were deposited on previously cleaned platinumized silicon substrates by spin coating technique, and then sintered at different temperatures up to 1000 °C. Structure of materials was examined by XRD analysis, Raman spectroscopy and scanning electron microscopy. The gold electrodes for functional characterization (LCR, PFM) were prepared by sputtering.

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