11TH CONFERENCE FOR YOUNG SCIENTISTS IN CERAMICS

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Satellite event:
ESR COST IC1208 Workshop

BOOK OF ABSTRACTS

October 21-24, 2105
Faculty of Technology
Novi Sad, Serbia

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and
BOOK OF ABSTRACTS

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Preface

The 11^{th} Conference for Young Scientists in Ceramics is organized by the Department of Materials Engineering, Faculty of Technology Novi Sad, University of Novi Sad, Serbia (October 21-24, 2015) and it is followed with one Satellite Event: Early Stage Researchers Workshop of the COST Action IC1208 "Integrating devices and materials: a challenge for new instrumentation in ICT".

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 eighth 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 and COST IC1208 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 Secretary of science and technological development and we would like to thank them for their endorsement too. A total number of 110 presentations given by young researchers and 13 invited talks coming from 25 countries with multidisciplinary profiles will be presented during the conference. It should be emphasised that presented topics cover research subjects of the highest scientific interest: experimental, theoretical and applicative aspects of synthesis, processing, advanced nano/microscale and functional characterisation 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.

Editors

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PROGRAMME

WEDNESDAY, OCTOBER 21, 2015.

09.00 – 11.00 h – Registration (Conference desk)

11.00 – 11.30 h – Oppening (Rectorate-Amphitheater)

11.30 – 12.15 h – IT1 Invited lecture (Rectorate-Amphitheater)
Francis Cambier, Belgium, The use of lasers to obtain complex shape ceramics

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

13.15 – 14.00 h – IT2 Invited lecture (Rectorate-Amphitheater)
Paula Vilarinho, Portugal, Is potassium-sodium niobate a lead free alternative to PZT?

14.00 – 14.45 h – IT3 Invited lecture (Rectorate-Amphitheater)
Ákos Kukovecz, Hungary, Nanotechnology and sensors nanocomposites

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	**A1 – A. Kompch, *et al.*, Germany** Synthesis and structural analysis of Mn-doped ZnO nanoparticles
15.30 – 15.45 h	**A2 – A. Marzec, *et al.*, Poland** Hydrothermal synthesis of composite heterostructures in the TiO_2-SnO_2 system
15.45 – 16.00 h	**A3 – V. Nikolic, *et al.*, Serbia** Solvothermal synthesis of magnetite nanoparticles suitable for application in magnetic hyperthermia
16.00 – 16.15 h	**A4 – M. Piciorus, *et al.*, Romania** Spherical silica nanoparticles obtained by Stober process.

	Tetra-ethyl-orthosilicate concentration influence upon silica nanoparticles morphology
16.15 – 16.30 h	**A5 – J. Pantić, *et al.*, Serbia** Phase evolution of sphene based ceramics during annealing
16.30 – 16.45 h	**A6 – D. Nicheva, *et al.*, Bulgaria** Study of nickel-cobalt spinels prepared by Pechini method

Ceramic processing - 1 (Class Room, R10)

15.15 – 15.30 h	**A7 – A. Chmielarz, *et al.*, Poland** Ti_2AlC gel-cast foams-properties and characterization
15.30 – 15.45 h	**A8 – K. Wojciechowski, *et al.*, Poland** Translucent zirconia polycrystals prepared from nanometric powders
15.45 – 16.00 h	**C1 – J. Roleček, *et al.*, Czech Republic** Ice-templating of ceramics in industrial scale
16.00 – 16.15 h	**C2 – J. Zygmuntowicz, *et al.*, Poland** Alumina matrix ceramic-nickel composites formed by centrifugal slip casting
16.15 – 16.30 h	**A9 – A. Presenda, *et al.*, Spain** Low temperature degradation of zirconia materials sintered via microwave heating technology
16.30 – 16.45 h	**A10 – P. Ctibor, *et al.*, Czech Republic** Extremely thick coating prepared from TiO_2 by plasma spraying

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

17.00 – 18.15 h – Section 2

Ceramics for Energy - 1 (Blue Hall)

17.00 – 17.15 h	**C3** – **F. Ulu, *et al.*, Belgium** Development of core-shell structured metal oxide powders to be used as lithium ion battery cathode materials
17.15 – 17.30 h	**A11 – M. Botros, *et al.*, Germany** Aluminum-doped $Li_7La_3Zr_2O_{12}$ as a solid electrolyte for lithium-ion batteries
17.30 – 17.45 h	**A12 – D. Ciria, *et al.*, France** Mechanical properties of fully dense ceramic electrolytes for Solid Oxide Fuel Cells
17.45 – 18.00 h	**C4 – M. Plodinec, *et al.*, Croatia** Ceramic composites based on TiO_2 nanotubes for application in solar cells
18.00 – 18.15 h	**A13 – Z. Slavkova, *et al.*, Bulgaria** Characterization of $LiNaSO_4$ for batteries application

Engineering ceramics - 1 (Class Room, R10)

17.00 – 17.15 h	**A14** – **U. Akkasoglu, *et al*., Turkey** Pressureless sintering of SiAlON ceramics for cutting tool application
17.15 – 17.30 h	**C5** – **A. Dubiel, *et al*., Poland** Mechanical and thermal properties of silicon nitride-titanium nitride particulate composites
17.30 – 17.45 h	**C6** – **A. Wilk, *et al*., Poland** Aluminium oxynitride - hexagonal boron nitride composites with anisotropic properties
17.45 – 18.00 h	**C7 – O. Poliarus, *et al*., Ukraine** High-temperature oxidation character of NiAl-ZrB_2 composite materials
18.00 – 18.15 h	**C8 – V. Tsukrenko, *et al*., Ukraine** Ageing of ceramics in the ZrO_2–Y_2O_3–CeO_2–CoO–Al_2O_3 system

THURSDAY, OCTOBER 22, 2015.

09.15 – 11.15 h – Section 3

Synthesis of ceramic powders – 2 (Blue Hall)

09.15 – 09.30 h	**A15 – A. Ghafarinazari, *et al.*, Italy** Thermal oxidation mechanism of mesoporous silicon
09.30 – 09.45 h	**A16 – A. Levish, *et al.*, Germany** Chemical vapor synthesis of aluminum nitride nanoparticles from metalic aluminum
09.45 – 10.00 h	**A17 – R. Crisan, *et al.*, Romanial** Nano-meter sized maghemite with high surface area and superparamagnetic behavior synthesis by oxidation of magnetite
10.00 – 10.15 h	**A18 – S. Lukic, *et al.*, Germany** Chemical vapor synthesis (CVS) of Ga_2O_3 and GaN nanoparticles for water splitting

Electroceramics - 1 (Blue Hall)

10.15 – 10.30 h	**C9 – V. Tsygoda, *et al.*, Ukraine** Thermo-electromotive force of multicomponent composites based on the refractory oxygen-free compounds
10.30 – 10.45 h	**A19 – N. Kanas, *et al.*, Norwey** Ceramic processing of all-oxide ceramic thermoelectric module
10.45 – 11.00 h	**A20 – J. Lelièvre, *et al.*, France** New lead-free materials with $A_{1/2}Bi_{1/2}BO_3$ formula (A=Rb; B=Ti)
11.00 – 11.15 h	**A21 – J. Ćirković, *et al.*, Serbia** Structural and electrical properties of BST ceramics prepared by hydrothermally assisted complex polymerization method

Bioceramics - 1 (Class Room, R10)

09.15 – 09.30 h	**A22 – D. Larionov, *et al.*, Russia** Osteoconductive ceramics with a specified system of interconnected pores based on monophasic calcium phosphates
09.30 – 09.45 h	**A23 – M. Putz, *et al.*, Romania** Mixed cationic templates controlling ordered silica morphology
09.45 – 10.00 h	**C10 – N. Aničić, *et al.*, Slovenia** The influence of polymer characteristics and particle morphology on the elution control of vanadate ions from V_2O_5/polymer composites
10.00 – 10.15 h	**A24 – A. Wajda, *et al.*, Poland** The structure and textural characterization of zinc doped

	bioactive glasses from $NaCaPO_4$-SiO_2 system
10.15 – 10.30 h	**A25 – T. Đorđević, *et al*., Serbia** Evaluation of mesoporous silica and titanium dioxide as antiobiotic carriers in drug delivery systems
10.30 – 10.45 h	**A26 – A. Vladescu, *et al*., Romania** Improvement of the mechanical and antibacterial properties of hydroxyapatite
10.45 – 11.00 h	**A27 – M. Radović, *et al*., Serbia** Synthesis of alumina powders and their insecticidal effect against *Acanthoscelides obtectus* say
11.00 – 11.15 h	**A28 – P. Jeleń., Poland** Spectroscopic studies of bioactive coatings based on silicon oxycarbide glasses

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

11.45 – 12.30 h – IT4 Invited lecture (Rectorate-Amphitheater)
Anne Leriche, France, Comparison of two different methods to process macroporous scaffolds for bone substitution applications

12.30 – 13.15 h – IT5 Invited lecture (Rectorate-Amphitheater)
Markus Winterer, Germany, Routes to nanoparticles optimized for energy technology

13.15 – 14.45 h – Lunch (TF-Ceremonial Hall)

14.45 – 17.00 h – Section 4

COST Workshop - 1 (Blue Hall)

14.45 – 15.30 h	**IT6 Invited lecture – Offer Schwartzglass, Israel** Advanced ceramic material for efficient ultrasonic cleaning and micro blowers realization
15.30 – 15.45 h	**E1 – R.M. Oliveira Pinho, *et al*., Portugal** Effect of poling on dielectric, piezoelectric and ferroelectric properties of doped potassium sodium niobate
15.45 – 16.00 h	**E2 – J. Zaffran, *et al*., *Israel*** Improving NiOOH catalytic activity in electrochemical water splitting using transition metal dopants: A first-principles calculation based study
16.00 – 16.15 h	**E3 – O.A. Condurache, *et al*., Romania** Study of ferroelectric-relaxor $BaCe_xTi_{1-x}O_3$ ceramics
16.15 – 16.30 h	**E4 – N.I. Ilić, *et al*., Serbia** $BiFeO_3$ ceramics densification study
16.30 – 16.45 h	**E5 – L. Fulanović, *et al*., Slovenia** Characterization of $0.9Pb(Mg_{1/3}Nb_{2/3})O_3$-$0.1PbTiO_3$

	electrocaloric multilayered structures prepared by tape casting
16.45 – 17.00 h	**E6 – I. Turcan, *et al*., Romania** Investigation of BaSrTiO$_3$ porous ceramics

Catalists - 1 (Class Room, R10)

14.45 – 15.30 h	**IT7 Invited lecture – Andras Sapi, Hungary** 3D Mesoporous oxide supported platinum nanoparticles for heterogenous catalytic applications - Gas vs. liquid phase reactions

Bioceramics - 2 (Class Room, R10)

15.30 – 15.45 h	**A29 – A. Tikhonov, *et al*., Russia** Intercalation of layered calcium phosphate and synthesis of ceramics based on it

Optics - 1 (Class Room, R10)

15.45 – 16.00 h	**A30 – A. Sidorowicz, *et al*., Poland** Influence of thulium and holmium oxide powders morphology on properties of transparent Tm,Ho:YAG
16.00 – 16.15 h	**A31 – A. Bjelajac, *et al*., Serbia** Microwave assisted synthesis of CdS quantum dots in DMSO
16.15 – 16.30 h	**A32 – M. Nakielska, *et al*., Poland** Spectroscopic investigations of Tm,Ho:YAG ceramics for solid state laser applications
16.30 – 16.45 h	**A33 – I. Dinić, *et al*., Serbia** Hydrothermal synthesis of optically active rare earth fluorides
16.45 – 17.00 h	**A34 – M. Chaika, *et al*., Ukraine** Influence of Yb^{2+} on optical properties of YAG:Yb garnet

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

17.15 – 18.30 h – Section 5

COST Workshop - 2 (Blue Hall)

17.15 – 17.30 h	**E7 – B. Bajac, *et al*., Serbia** Structure and properties of multiferroic BaTiO$_3$/NiFe$_2$O$_4$ thin films obtained by solution deposition technique
17.30 – 17.45 h	**E8 – V.A. Lukacs, Romania** Biomorphic growth and functional properties of nickel oxide 1-D microstructures
17.45 – 18.00 h	**E9 – B. Belec, *et al*., Slovenia** Magnetic properties of plate-like composite nanoparticles combining soft-magnetic iron oxide with hard-magnetic barium hexaferrite
18.00 – 18.15 h	**E10 – I.V. Ciuchi, *et al*., Italy** Enhancement of the energy storage properties in PLZT

	ceramics with compositions across FE-AFE phase boundary
18.15 – 18.30 h	**E11 – E. Đurđić, *et al*., Serbia** The cation distribution in $NiFe_2O_4$ and $NiFe_{1.85}Y_{0.15}O_4$: Raman and X-ray diffraction studies

Engineering ceramics - 2 (Class Room, R10)

17.15 – 17.30 h	**A35** – **S. Ilic, *et al*., Serbia** Phase development and thermal behaviour of hybrid sol-gel derived mullite precursor
17.30 – 17.45 h	**A36** – **A. Dudek, *et al*., Poland** Subcritical crack growth in oxide and non-oxide ceramics using the Constant Stress Rate Test
17.45 – 18.00 h	**A37** – **T. Csanádi, *et al*., Slovakia** Micro-scale plasticity and elastic behaviour of ceramic crystals under micropillar compression
18.00 – 18.15 h	**A38 – K. Kornaus, *et al*., Poland** The influence of sintering temperature and additive on the microstructure of pressure-less sintered tungsten carbide
18.15 – 18.30 h	**C11 – E. Okur, *et al*., Turkey** Improving the thermal shock properties of Y-alpha-SiAlON/glass composite

20.00 h – Social Event

Restaurant “Fontana” *Meeting of Young Ceramist Network of the European Ceramic Society,* Invited speakers: Paula Vilarinho and Francis Cambier

FRIDAY, OCTOBER 23, 2015.

09.15 – 11.15 h – Section 6

COST Workshop – 3 (Blue Hall)

09.15 – 09.30 h	**E12 – A. Chandran, *et al*., Serbia** Synthesis and structural characterizations of SnO_2 thick films
09.30 – 09.45 h	**E13 – V. Preutu, *et al*., Romania** Preparation and properties of PCL-functional oxide composites
09.45 – 10.00 h	**E14 – A. Dzunuzovic, *et al*., Serbia** Properties of $BaTiO_3$ - $NiZnFe_2O_4$ multiferroic composites obtained by auto-combustion synthesis
10.00 – 10.15 h	**E15 – J. Vukmirovic, *et al*., Serbia** Fabrication of $BaTiO_3$ thin films by inkjet printing

Ceramics processing - 2 (Blue Hall)

10.15 – 10.30 h	**A39 – J. Hruby, *et al*., Czech Republic** Calculation of activation energy and its changes during sintering using MSC and Wang & Raj models
10.30 – 10.45 h	**C12 – A. Miazga *et al*., Poland** Graded ceramic/metal composites obtained by the centrifugal slip casting
10.45 – 11.00 h	**A40 – V. Mackert *et al*., Germany** UV laser sintering of SnO_2 and ZnO thin films produced by electrophoretic deposition
11.00 – 11.15 h	**A41 – R. Cabezas-Rodríguez, *et al*., Spain** Synthesis of yttrium silicate by solid-liquid state reaction for environmental barrier coatings

Bioceramics - 2 (Class Room, R10)

09.15 – 09.30 h	**A42 – S. Kurbatova, *et al*., Russia** Synthesis and characterization of resorbable calcium phosphate bioceramics with a ratio of $0,5 \leq Ca/P \leq 1$
09.30 – 09.45 h	**A43 – M.J. Lukić, *et al*., Serbia** Simultaneous thermal analysis and dilatometric study of HAp-$LiFePO_4$ system
09.45 – 10.00 h	**A44 – M. Slama, *et al*., Czech Republic** Effect of colloidal milling on the physical, mechanical and biological properties of hydroxyapatite monoliths prepared by electrophoretic deposition
10.00 – 10.15 h	**A45 – L. Stipniece, *et al*., Latvia** Synthesis and characterization of divalent cation substituted calcium phosphates
10.15 – 10.30 h	**A46 – M. Kuzina, *et al*., Russia**

Mixed-anionic calcium phosphate powders for bioresorbable ceramic

10.30 – 10.45 h **A47 – T. Maravić, *et al*., Serbia**
Influence of dental composite core material on biomedical properties of premolars restored with a zirconia full crown: A finite element analysis

10.45 – 11.00 h **A48 – T. Uhlířová, *et al*., Czech Republic**
Elastic properties of cellular alumina ceramics prepared by biological foaming

11.00 – 11.15 h **A49 – M. Mirković, *et al*., Serbia**
Synthesis of monetite ($CaHPO_4$) by mechanochemical treatment of brushite ($CaHPO_4 \cdot 2H_2O$)

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

11.45 – 12.30 h – IT8 Invited lecture (Rectorate-Amphitheater)
Lucian Pintilie, Romania, Polarization driven effects and the role of interfaces in ferroelectric thin films and heterostructures

12.30 – 13.15 h – IT9 Invited lecture (Rectorate-Amphitheater)
Endre Horváth, Hungary, From synthesis to application of photovoltaic perovskite nanowires

13.15 – 14.45 h – Lunch (TF-Ceremonial Hall)

14.45 – 17.00 h – Section 7

Popular science (Blue Hall)

14.45 – 15.15 h **IT10 Invited lecture – Roger Anderton, Great Britain**
Boscovich's Unification that came after Newton's Unification

15.15 – 15.45 h **IT11 Invited lecture – Dragoslav Stoiljkovic, Serbia**
From Boscovich's theory to modern quantum theory

Bioceramics - 3 (Blue Hall)

15.45 – 16.00 h **C13 – G. Kazakova, *et al*., Russia**
Resorbable bioceramics in $Ca_3(PO_4)_2$ – $Mg_2P_2O_7$ system

16.00 – 16.15 h **A50 – I. Narkevica, *et al*., Latvia**
Development of innovative 3D porous TiO_2 ceramic scaffolds for orthopaedic applications

16.15 – 16.30 h **A51 – J. Sekaninova, *et al*., Czech Republic**
Calcium partially stabilized ZrO_2 bioceramics nanocrystals

16.30 – 16.45 h **A52 – M. Prekajski, *et al*., Serbia**
Ouzo effect – as the new simple nanoemulsion method for synthesis of strontium hydroxyapatite nanospheres

16.45 – 17.00 h **A53 – M.D. Vranceanu, *et al*., Romania**
Calcium phosphate coatings deposited on Ti substrate using

	electrochemically assisted deposition

Equipment (Class Room, R10)

14.45 – 15.45 h	**Demonstration of JEOL microscopes** **Slavko Žižek, Slovenia**

Catalysts & ceramics for energy (Class Room, R10)

15.45 – 16.00 h	**A54 – T. Varga, *et al.*, Hungary** Synthesis, characterisation, and electrochemical properties of graphite oxide/vanadate nanowire composites
16.00 – 16.15 h	**A55 – N. Lysunenko, *et al.*, Ukraine** Electrical efficiency of SOFCs with 8YSZ and 10Sc1CeSZ electrolytes
16.15 – 16.30 h	**A56 – S. Dmitrović, *et al.*, Serbia** Synthesis and characterization of Ag doped ceria nanopowders
16.30 – 16.45 h	**A57 – K.L. Juhasz, *et al.*, Hungary** Synthesis and characterization of platinum nanoparticles with controlled size for heterogen catalytic processes
16.45 – 17.00 h	**C14 – J. Sroka, *et al.*, Poland** Improved properties of the epoxy – fly ash composites by silane treatment of the filler

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

17.15 – 18.30 h – Section 8

Electroceramics - 2 (Blue Hall)

17.15 – 17.30 h	**A58** – **M. Čebela, *et al.*, Serbia** Synthesis, optical and magnetic properties studies of multiferroic BiFeO$_3$
17.30 – 17.45 h	**A59 – C. Vlãdut, Romania** ZnO based films with sensing properties
17.45 – 18.00 h	**C15 – M. Drozdova, *et al.*, Estonia** Electrical behavior of zirconia-alumina nanofibers-graphene composites
18.00 – 18.15 h	**C16 – C. Ianasi, *et al.*, Romania** Fe$_2$O$_3$-SiO$_2$-PVA hybrid xerogels, precursors for superparamagnetic nanocomposites, potential candidates as MRI T2 contrast agents
18.15 – 18.30 h	**C17 – E. Pawlikowska, *et al.*, Poland** Ferroelectric barium-strontium titanate and ceramic-polymer composites based on BST in terahertz radiocommunication applications

Electroceramics - 3 (Class Room, R10)

17.15 – 17.30 h	**C18** – **A. Kukharchik, *et al*., Russia** Nano- and bio-structured materials: Surfaces and mesophase features
17.30 – 17.45 h	**C19** – **M. Pareiko, *et al*., Ukraine** Self-fluxing Fe-based alloy with TiB_2 additives for the spraying wear-resistant coatings
17.45 – 18.00 h	**C20** – **D. Németh, *et al*., Slovakia** FEM analysis of cracking around the indent in W-C coating
18.00 – 18.15 h	**C21 – K. Jach, Poland** Modification of quartz and ceramic substrates by deposition of tungsten layers
18.15 – 18.30 h	**C22 – I. Sytnyk, *et al*., Ukraine** The structure and properties of chromium carbide steels with titanium nitride coating

19.30 h – Social Event – Excursion

SATURDAY, OCTOBER 24, 2015.

09.15 – 10.15 h – Section 9

Synthesis of ceramic powders - 3 (Blue Hall)

09.15 – 09.30 h	**A60 – M. Nikolić, *et al*., Serbia** Synthesis and characterization of mesoporous and superparamagnetic bilayered-shell aroundsilica core particles
09.30 – 09.45 h	**A61 – S. Ognjanovic, *et al*., Germany** Characterization of aluminum nitride nanoparticles synthesized by chemical vapor synthesis
09.45 – 10.00 h	**C23 – S. Ilies, *et al*., Romania** Silver modified zeolite-multi-walled carbon nanotubes-epoxy composite electrode for electrochemical detection and degradation of ibuprofen in water
10.00 – 10.15 h	**C24 – T. Minović Arsić, *et al*., Serbia** Synthesis and characterization of ceria/carbon cryogel composite

Electroceramics - 3 (Class Room, R10)

09.15 – 09.30 h	**A62 – J. Stanojev, *et al*., Serbia** Dielectric properties of barium titanate based thin films

Traditional ceramics - 1 (Class Room, R10)

09.30 – 09.45 h	**T1 – M. Kavanová, *et al*., Czech Republic** Characterization of the interaction between glazes and ceramic bodies
09.45 – 10.00 h	**T2 – V. Topalović, *et al*., Serbia** Properties of sintered cordierite ceramics obtained by sol-gel methods of powder synthesis
10.00 – 10.15 h	**T3 – A. Abdelghany, *et al*., Egypt** Effect of transition metal addition in the bioactivity of borate bioglass: A descriptive correlational study

10.15 – 11.00 h – IT12 Invited lecture (Blue Hall)

Andreja Gajovic, Croatia, Raman spectroscopy technique and specific applications for study of ceramics

11.00 – 11.15 h – Coffe Break (Blue Hall)

11.15 – 12.00 h – IT13 Invited lecture (Blue Hall)

Kostantinos Giannakopulos, Greece, Structural characterisation of layers for advanced non-volatile memories

12.00 – 13.00 h – Section 10

Traditional ceramics - 2 (Blue Hall)

12.00 – 12.15 h	**T4 – K. Pasiut, *et al*., Poland** The influence of molar ratio Al_2O_3/SiO_2 on the structure of ceramic glazes
12.15 – 12.30 h	**T5 – A. Gerle, *et al*., Poland** Corrosion of $MgCr_2O_4$, $MgAl_2O_4$, $MgFe_2O_4$ spinels in SO_2–O_2–SO_3 atmoshere – thermodynamic evaluation
12.30 – 12.45 h	**T6 – O. Chudinovich, *et al*., Ukraine** Phase equilibria and properties of solid solutions in the La_2O_3-Yb_2O_3 and La_2O_3-Y_2O_3-Yb_2O_3 systems at 1500 °C
12.45 – 13.00 h	**T7 – M. Głuszek, *et al*., Poland** Preparation, properties and applications of shear thickening fluids based on silica, glycols and dopants

Ceramics processing - 2 (Class Room, R10)

12.00 – 12.15 h	**C25 – O. Kornienko, *et al*., Ukraine** Interaction cerium oxide with dysprosia at 1500 °C
12.15 – 12.30 h	**A26 – M. Marych, *et al*., Ukraine** Features of the structure and properties of ceramic composite system B_4C – eutectic alloy (B_4C-TiB_2)
12.30 – 12.45 h	**A27 – J. Shishkina, Ukraine** Fabrication of Ti_xAl_y/TiC based ceramic composite powder from the TiH_2-Al-C system

13.00 – 13.15 h – Closing (Blue Hall)

11th Conference for Young Scientists in Ceramics

SM-2015 & COST Workshop

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INVITED TALKS

IT1

THE USE OF LASERS TO OBTAIN COMPLEX SHAPE CERAMICS

Francis Cambier, E. Juste, C. Ott, F. Petit
Belgian Ceramic Research Center, 4 Avenue Gouverneur Cornez, 7000 Mons, Belgium

Even if the interaction between a laser beam and a ceramic is a complex phenomenon, one of the consequences of this interaction, if the ceramic absorbs the beam at the laser frequency, is a heat production inside the material. This heat can be used, either to machine the material, or to sinter or melt it.

Various laser wavelengths are currently commercially available (UV, visible, IR). It is difficult to have, at the same time, a high precision of the light beam (inversely proportional to the wavelength) and a low cost of the laser equipment. Today the best compromise is found with near IR lasers (in the range of ~ 1 µm). Non oxide ceramics (nitrides, carbides, etc.) absorb at such a wavelength, but it is not the case of oxide ceramics like alumina, zirconia, etc. For such materials, it is needed to add an absorption promoter.

In the talk, two different applications will be presented:

- the subtractive method, used to machine oxides at the green state, allowing to reach high precision of the component details, with a high speed processing time,
- the additive method (selective laser sintering – or melting), allowing to build up complex cm-size 3D shapes, starting from a powder bed.

The level of promoter addition and the mechanism of reaction of this absorbent with the laser beam, which are different in the two techniques will be discussed and illustrated by examples.

- For the subtractive technique, the development of the production of dental crowns made of zirconia (TZP) by machining of the desired shape from green blanks and the recycling of used blanks will be shown.
- For the additive technique, the production of alumina molds for metal investment casting will be illustrated.

IT2

IS POTASSIUM-SODIUM NIOBATE (KNN) A LEAD FREE ALTERNATIVE TO PZT?

Paula Maria Vilarinho

Department of Materials and Ceramics Engineering, CICECO – Aveiro Institute of Materials, University of Aveiro, Portugal

Will (K,Na)NbO$_3$-based system (KNN) ever be PbZrTiO$_3$ (PZT) - like?

This is the question to be addressed in this talk.

And why is this question important?

If it is possible predicting what is about to come, it is said that we will live in an ultradigitally responsive world, where more things are exponentially connected to the Internet than people [1]. From the smallest personal items to the largest continents, everything, everywhere will be digitally connected, and responsive to our wants and likes [1]. This is now called the Internet of Everything.

There are several technologies that will support these developments and these include Smart Devices, Sensors and Actuators and MicroElectroMechanical Systems (MEMs), that by themselves require functional Smart Materials as piezoelectrics. Piezoelectrics is indeed a huge market that enables technology for areas, such as microelectronics through positioning elements in photolithography, medical diagnostics through ultrasound imaging, sensors and actuators in automobile industry, and many others [2].

Among the most well know piezoelectrics are lead based perovskites, being typified by PZT in which the highest electromechanical coupling coefficients have been found. However, due to severe restrictions towards the use of lead and lead based materials, it is necessary to look for lead free piezoelectric alternatives. Within the several candidate families currently under consideration (K,Na)NbO3-based system deserves a special attention.

In this talk the state of the art on KNN based ceramics, thick films and single crystals will be presented. Processing limitations, behavioural differences between single crystals and polycrystals, role of specific dopants and microstructure designing strategies as texturing, will be reviewed [3–6]. The benchmark between KNN and PZT will be presented and possible applications for KNN based materials highlighted.

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IT3

NANOTECHNOLOGY AND SENSORS NANOCOMPOSITES

Ákos Kukovecz[1,2]

[1]Department of Applied and Environmental Chemistry, University of Szeged, Rerrich Béla tér 1., H-6720 Szeged, Hungary
[2]MTA-SZTE "Lendület" Porous Nanocomposites Research Group, Rerrich Béla tér 1., H-6720 Szeged, Hungary

Sensors are deeply woven into the fabric of our modern lifestyle today. As the Internet of Things gradually transforms from a sci-fi theme into physical reality around us, it is more important than ever to develop new sensors that can satisfy the following requirements: low cost, low energy consumption, long lifetime, high sensitivity, high selectivity, fast response, easy interfacing to existing semiconductor electronics.

Therefore, the sensor field of materials science is expanding rapidly, as demonstrated nicely by e.g. the 2015 launch of the new journal ACS Sensors. In this talk I will review briefly the history of sensors and indicators and highlight the importance of the field in general. Then we discuss the advantages/disadvantages of various sensor types and see that nanomaterials with typical crystallite size below the Debye length make excellent resistive sensors. One dimensional nanomaterials are particularly suitable for this purpose because the width of their conductive channel can be tuned finely by modifying their outer surface.

The talk will be concluded by showing recent research results from my own group in two specific topics: evaporation profile based liquid identification and improving the selectivity of gas sensors by finding and analyzing stationary noise patterns using Fluctuation Enhanced Sensing.

IT4

COMPARISON OF TWO DIFFERENT METHODS TO PROCESS MACROPOROUS SCAFFOLDS FOR BONE SUBSTITUTION APPLICATIONS

Anne Leriche

Laboratoire des Matériaux Céramiques et Procédés Associés (LMCPA), UVHC, Boulevard Charles de Gaulle, F-59600 Maubeuge, France

The aim of this presentation is to compare the characteristics of calcium phosphate porous ceramics scaffolds for bone substitute application obtained by two particular techniques.

The first method is the ceramic slurry infiltration of organic skeleton. It permits to obtain an isotropic spherical porosity with a close control of global porosity volume, of pore size and of interconnection size between the pores. The so obtained macroporosity is ranging between 60 and 75 vol.% with pore sizes between 200 and 3000 µm and interconnection sizes between 20 and 400 µm. By adding some additive, a microporosity can also be generated in the scaffold walls, from 0 to 40 vol.% within the micron range.

The second method is a casting method based on ceramic slurry ice, which produces tubular interconnected pores with ellipsoidal shape. The tubular porosity structure is supposed to enhance cell invasion. Several samples with pore size ranging from 50 µm to 350 µm and porosity from 30% to 70% were synthesized using different processing parameters: dry matter and binder content of slurry and freezing rate.

For both techniques, the macroporosity allows the cell proliferation inside the scaffolds. The analysis by scanning electron microscopy of cell invaded samples shows that the size of the pores of the ice-templated scaffolds is sufficiently large to enable fast osteoblastic cell colonisation. A microporosity added to the macroporosity allows a better control of resorbability but also can be used as supports for active biological species like antibiotics, phage…

Acknowledgement: The results presented in this paper are issued from a collaborative work involving LMCPA (E. Meurice, F. Bouchart, J.C. Hornez), BCRC (Belgium) (D. Hautcoeur, V. Lardot, F. Cambier) and 2 labs (LBMR and INEB) of the University of Porto (Portugal) (MH Fernandez, F Monteiro)

IT5

ROUTES TO NANOPARTICLES OPTIMIZED FOR ENERGY TECHNOLOGY

Markus Winterer

Nanoparticle Process Technology and CENIDE, University of Duisburg-Essen,
Lotharstr. 1, 47057 Duisburg, Germany

Designing materials based on nanoparticles may be divided in at least two steps: selection of materials with intrinsic properties meeting the requirements of the desired application and optimization of particle characteristics regarding their extrinsic properties that optimize processibility and maximize device performance. This is a big challenge since device performance and materials synthesis are far apart in the process chain. We use chemical vapor synthesis in which precursor molecules are supplied in a gas flow and react upon introduction of energy thereby forming small particles which grow by coagulation and coalescence into more or less agglomerated particles. Corresponding characteristic times determine the particle characteristics. Gas phase doping and functionalization of the nanoparticles is also possible.

A main characteristic of nanoparticles is their size and size distribution but also how they are assembled. Depending on process parameters hard or soft agglomerates of variable size are formed. Whereas size and size distribution influence processibility and device performance, agglomeration affects especially the processibility of the particles. The degree of crystallinity or absence of defects depends on the growth rate relative to the rate by which atoms find their ideal lattice position. Crystallinity is very important in applications where charge carrier transport or recombination play a role. One of the most important methods to adjust materials properties is doping. The distribution and location of the dopant atoms are important for their functionality and can be controlled by reactant mixing and diffusion. If the dopant is of limited solubility, segregation to the surface or second phase formation may be observed. We will illustrate how nanoparticle characteristics can be adjusted in CVS, how they can be quantified and how they influence the performance in solid state lighting, photocatalysis and thermoelectric conversion.

IT6

ADVANCED CERAMIC MATERIALS FOR EFFICIENT ULTRASONIC CLEANING AND MICRO BLOWERS REALIZATION

Offer Schwartsglass

EE department, Faculty of Engineering, Shenkar college of Engineering & Design, 12 Anna Frank St., Ramat Gan 52526, Israel

Piezoelectric composites comprising of ceramics like zinc oxide (ZnO) and aluminum nitride (AlN) and polymer phases are promising materials due to their superior features and easily tailored functionalities. Such composites can be used to realize miniature 2D/3D actuators arrays. Coherent stimulation of the array micro elements result in collective new features and macro functionality.

Integrating process steps and methodologies from the following disciplines:

- Low cost Microelectronics and MEMS planar thin films deposition and etching techniques
- Advanced ceramics composites fabrication techniques

will provide a cost effective solution for implementing a wide range of new actuators and applications.

Two examples for device and systems are given:

- Advanced Ultrasonic Cleaning
- Miniature Micro Blowers

Beyond their new functionality, small size and high efficiencies, these systems provide scalability in power consumption (input power) and their respective output action: precise control on direction and intensity of the mechanical vibration produced by the ultrasonic device and precise control on direction and air pressure intensity produced by the micro blower device. The actuators exploit the broad bandwidth (low mechanical quality factor), low acoustic impedance and high mechanical flexibility which are the tailored characteristics of the piezoelectric polymer structures.

IT7

3D MESOPOROUS OXIDE SUPPORTED PLATINUM NANOPARTICLES FOR HETEROGENOUS CATALYTIC APPLICATIONS – GAS vs. LIQUID PHASE REACTIONS

Andras Sapi[1,2], H. Wang[2], C. Thompson[2], K. Juhasz[1], D. Dobo[1],
M. Szabo[1], G.A. Somorjai[2], Z. Konya[1]

[1]Department of Applied and Environmental Chemistry, University of Szeged, 6723 Szeged Rerrich tér 1., Hungary
[2]Department of Chemistry, UC Berkeley, 94700, Hildebrand Hall, CA, USA

Keywords: Pt nanoparticles, controlled size, mesoporous oxide, hard template – replica method, heterogeneous catalysis

Platinum nanoparticles with controlled average sizes of 2, 4, 6 and 8 nm were synthesized and we use them as model catalysts to study alcohol oxidation reactions in both the liquid and gas phases at 60 °C. Different 3D mesoporous oxide materials (SiO_2 – MCF-17, SBA-15, Co_3O_4, MnO_2, Fe_2O_3, NiO, CeO_2) prepared by the soft and hard template (replica) method were used as catalyst supports.

The reaction at the solid/liquid interface is 2 orders of magnitude slower than that at the solid/gas interface, while catalytic activity increases with the size of platinum nanoparticles for both the liquid-phase and gas-phase reactions. The activation energy of the gas-phase reaction decreases with the platinum nanoparticle size and is in general much higher than that of the liquid-phase reaction which is largely insensitive to the size of catalyst nanoparticles. Water substantially promotes alcohol oxidation in the liquid phase. However, it inhibits the reaction in the gas phase. Co_3O_4 supported catalyst is 10 times more active compared to the SiO_2 supported Pt. MnO_2 supported catalysts has poor activity in the gas phase but the most active catalyst in the liquid phase.

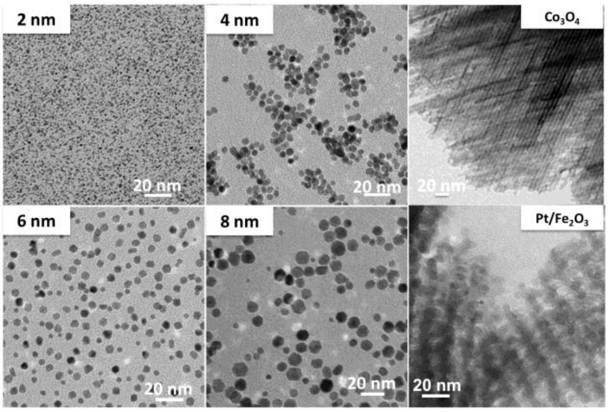

Figure 1. Pt Nanoparticles with controlled size (1-8 nm), 3D mesoporous Co_3O_4 and 2.5 nm Pt NP supported on 3D mesoporous Fe_2O_3

The kinetic results suggest different mechanisms between the liquid-phase and gas-phase reactions, correlating well with different orientations of alcohol surface species at the solid/liquid interface vs. the solid/gas interface as probed by sum frequency generation vibrational spectroscopy under reaction conditions and simulated by computational calculations. The usage of the 3D mesoporous oxide supports has great influence on the catalytic activity and selectivity.

References

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IT8

POLARIZATION DRIVEN EFFECTS AND THE ROLE OF INTERFACES IN FERROELECTRIC THIN FILMS AND HETEROSTRUCTURES

Lucian Pintilie
National Institute of Materials Physics, Atomistilor 105bis, Magurele, 077125, Romania

Ferroelectrics and multiferroics are attractive materials for various applications, as for example non-volatile memories, ultrasound transducers, pyroelectric detectors for IR, thermal imaging, tunable devices, etc. For a long time they were prepared in bulk form as crystals or ceramics. However, in the last decades the interest shifted more and more towards thin films and nanostructures, both for fundamental (e.g. study of size effects in ferroelectrics) and practical (e.g. the need of miniaturization and integration with semiconductor industry) reasons. The methods to deposit thin films are numerous, some of them being relatively not expensive and easy to implement (e.g. sol-gel), other requiring expensive equipment (e.g. pulsed laser deposition-PLD, molecular beam epitaxy and similar techniques involving high vacuum). At present it is certain that the macroscopic properties of ferroelectric thin films are strongly dependent on their structural quality. This is the reason why, in order to investigate the properties in samples having structural quality close to single crystals and to eliminate as much as possible extrinsic contribution coming from grain boundaries, parasitic phases, etc., efforts are made to grow epitaxial films of very good quality. One technique allowing the growth of the epitaxial ferroelectric films is PLD. Mastering the deposition parameters it is possible to obtain good heteroepitaxy, with smooth interfaces and surfaces. On such samples one can study the complex relation between ferroelectric and electronic properties of the material, and the effect of the interfaces on the macroscopic electrical properties. Therefore, the presentation will focus on the following aspects:

- Growth of ferroelectric heterostructures by PLD, giving examples of ferroelectric capacitors, ferroelectric multilayers and artificial multiferroic structures, and showing also the results of the macroscopic investigations perform to study the electric (magnetic) properties. The focus will be on epitaxial thin films, but examples will be given on polycrystalline (ceramic) films also for comparison.
- Electrode-ferroelectric interface; combined electrical, XPS, TEM investigations, all suggesting that the interface properties (e.g. the height of the potential barrier) in this case are controlled by the ferroelectric polarization and not by the work function difference as is the case for metal-semiconductor contacts. Recent theoretical simulations for the current-voltage characteristics will be presented, supporting the same hypothesis.
- Polarization induced self-doping in epitaxial films. The hypothesis is supported by complementary results of electrical, PFM, XPS and TEM investigations. Practically, it was found that the out-of-plane polarization promotes formation of specific charge defects in the films, namely vacancies, in order to compensate the depolarization field.

Potential applications will be discussed at the end, with focus on pyroelectric and photovoltaic effects in ferroelectric materials, others than $BiFeO_3$.

Acknowledgement: The authors acknowledge the financial support of the following projects: Idea-Complex Research Grant PN-II-ID-PCCE-2011-2-0006 (contract nr. 3/2012); FP7-IFOX; IFA-CEA (contract C4/2014).

IT9

FROM SYNTHESIS TO APPLICATION OF PHOTOVOLTAIC PEROVSKITE NANOWIRES

Endre Horváth, M. Spina, B. Náfrádi, L. Forró
Laboratory of Physics of Complex Matter, EPFL, 1015 Lausanne, Switzerland

In the last few years, the organolead halide perovskite $CH_3NH_3PbI_3$ and its derivatives have been found to be very efficient light harvesters and ambipolar semiconductors revolutionizing the field of solid-state solar cells. Its low temperature fabrication process allowed to design simpler solar cell structures (i.e. from mesoscopic to planar junctions) which yielded efficiencies approaching 20%.

We have shown that one can synthesize nanowires of this photovoltaic perovskite (Horváth *et al*, in Nano Letters DOI: 10.1021/nl5020684) which in association with carbon nanostructures (carbon nanotubes and graphene) makes outstanding composites with rapid and strong photoresponse. They can serve as conducting electrodes and as central components of sensors, detectors. The demonstration of the performance of several devices based on these composite structures will be given.

IT10

BOSCOVICH'S UNIFICATION THAT CAME AFTER NEWTON'S UNIFICATION

Roger J. Anderton
United Kingdom

Newton made a unification that is represented by his story of the apple. In the story of the apple: Newton observed an apple falling from a tree and realised that the same force acting on the apple was the same force acting on the Moon, i.e. gravity. In this way Newton made the unification of what until then was two different types of motion: terrestrial motion and celestial motion. However, this presented a mystery why was there two different types of forces: that of contact force and force that acted through a distance without contact. Boscovich then made the next major step of unification between non-contact force (i.e. action at a distance) and contact forces (e.g. such things as one object hitting another object and making it move). One of the major hurdles in getting Boscovich's theory accepted has been the mental difficulty of many generations of people have with accepting how contact and non-contact forces can be unified in Boscovich's theory, because his solution is that there is no contact forces; the appearance of contact forces is mere illusion, and all forces are really action-at-a-distance. At present I am engaged in trying to translate from Latin into English many of the works of Boscovich that have hitherto been untranslated.

IT11

FROM BOSCOVICH'S THEORY TO MODERN QUANTUM THEORY

Dragoslav Stoiljkovich[1], R.J. Anderton[2]
[1]*Faculty of Technology, University of Novi Sad, Serbia*
[2]*United Kingdom*

Boscovich (1711–1787) proposed that the basic elements of matter are non-extended indivisible points (a form of monads). Depending on the distance between two points the forces alternate several times from attractive to repulsive (Fig. 1). These elementary points are connected to the first order particles; these are connected forming the second order particles, etc. "All worlds of smaller dimensions, taken together, were like a single point in relation to the larger" world. Hence, his unique law of forces (Fig. 1) is valid for each pair of particles at any level of the hierarchy of matter. The number of arches, their size and shape can be different.

There are distances at which the repulsive and attractive forces are equal (Fig. 1). In cases E, I, N and R (**the cohesion limits)** the particles are in the stable equilibrium: if the distance accidentally increases or decreases, then an attractive or repulsive force appears bringing particles back to the previous distance. In cases G, L and P **(the non-cohesion limits)** the particles are in an unstable equilibrium because if the distance accidentally increases or decreases, the force that appears leads to a greater separation or greater approach. If the two points are located at focal points A and B near the centre D, the third point may be located anywhere on the ellipse at a distance corresponding to the cohesion limits (Fig. 2). There are as many ellipses as there are cohesion limits.

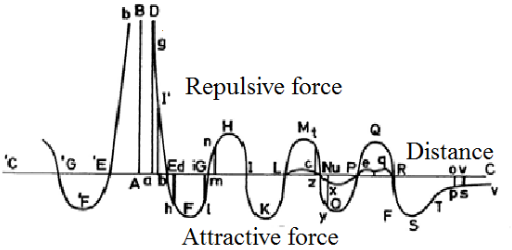

Figure 1. Boscovich's curve: Force vs. distance of two points (or particles) [1]

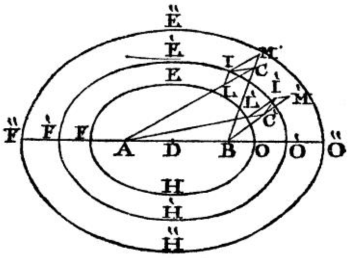

Figure 2. Boscovich's orbitals [1]

"In mechanics it is known that for a curve, whose abscissas represent distances and ordinates represent forces, then the area (delimited by the curve and abscissa) represents the increase or decrease of the square of velocity" of the particles [1]. It is known that $\Delta E=\int F\mathrm{d}r=m\Delta(v^2)/2$ wherein: force F, distance r, kinetic energy E of a particle of mass m moving at a speed v. Hence the area delimited by the arches and the abscissa in Fig. 1 represents energy. As a particle passes from one to the other cohesion limits (or orbitals), it loses or gains exactly a certain amount of energy ΔE, "quantum energy", as it is now called, which is equal to the difference between areas delimited by repulsive and attractive arches between the two cohesion limits.

In 1903–1907 J.J. Thomson concluded that only Boscovich's Theory could explain how electrons can move only at certain paths around the nucleus of atoms. In 1911 E. Rutherford, ex-student and collaborator to Thomson, experimentally confirmed this "planetary model of the atom". After several months spent with Thomson and Rutherford, N. Bohr in 1913 calculated the possible paths of electrons, taking into account that electrons can move from one orbital to another only if they receive or lose a certain amount of (quantum) energy - as Boscovich wrote since 1745 [2].

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IT12

RAMAN SPECTROSCOPY TECHNIQUE AND SPECIFIC APPLICATIONS FOR STUDY OF CERAMICS

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Basic principles of Raman spectroscopy (RS) will be explained and related to the other spectroscopy techniques. The selection rules for Raman scattering will be mentioned with the short explanation of physical background. The main specific data that can be accomplished by the technique will be discussed, in contrast or in synergy with the other main characterization techniques. Typical experimental setups of different Raman spectrometer will be explained and compared. Specific conditions of the recording Raman spectra necessary to achieve the reliable results will be clarified including the excitation lines and sample arrangement. The preparation of the samples for RS and some limits of the technique will be also considered.

Raman instrumentation in our laboratory comprises high class Raman spectrometer Jobin Yvon T64000 that is triple dispersive instruments and excitation by Coherent Inova Ar ion laser operated at 514 or 488 nm. The experimental possibilities including the additional instrumentation will be described. High and low temperature chamber “Linkam” working in temperature range 77 to 773 K, and “Leitz” high-temperature cell operating in temperature range 473 to 1773 K for work in micro-Raman set up will be revealed. The work in macro chamber and the cryostats that can be attached will be shortly mentioned (He close circuit cryostat operating from 10 K to room temperature and cryostat operating from 77 to 573 K).

The applications of the Raman spectroscopy as one of the powerful methods for solving the problems in materials science will be illustrated with the accent to the metal oxides based ceramic and nanostructures, while the application to one bio-ceramic system will be briefly mentioned. The examples from different own research topics will be shown for illustration. The observation of crystal structure, specific defects in nanostructures, mechanism of phase transition, solid state reactions during mechanochemical treatment and other studies of metal oxides will be presented with the accent to the specific RS results in comparison with XRD and TEM results. The observation and the recognition of the luminescence lines in the Raman spectrum that is important for proper interpretation of the RS results will be shown at the example of Y_2O_3 doped with rear earth elements. The use of RS for investigation of the amorphous and nearly amorphous materials with short range ordering will be shortly mentioned as well as the application of RS for the study of the calcium phosphate.

IT13

STRUCTURAL CHARACTERISATION OF LAYERS FOR ADVANCED NON-VOLATILE MEMORIES

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Non-volatile memory cells are the devices with the most aggressive scaling on the market. For this reason the accurate characterisation of their layer stacks is of great importance. We present a review of our recent work on a large variety of such stacks for charge-trap and resistive memories, which have been characterised structurally with Transmission Electron Microscopy and Conductive Atomic Force Microscopy (CAFM); the features of their structure on their function as memory elements will be discussed. In particular we will discuss the structural characterisation of floating gate memories where their metallic charge traps have been manufactured either by e-beam evaporation or with the use of a nanoparticle source. We will also discuss the effects of annealing on the properties of ALD-grown alumina control layers for MANOS (Metal/Alumina/silicon Nitride/silicon Oxide/Silicon) structures.

The study of resistive memories (layers of sub-stoichiometric TiO_2 on $Au/SiO_2/Si$) with the use of CAFM will also be presented, demonstrating the huge potential of this technique.

ADVANCED CERAMICS

A1

SYNTHESIS AND STRUCTURAL ANALYSIS OF Mn-DOPED ZnO NANOPARTICLES

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Nanostructured manganese oxide (MnO) is a potentially good catalyst material for water splitting [1]. Its large band gap of around 4eV however is a great disadvantage if applied in photocatalytic water splitting [2]. Zinc oxide with its band gap of around 3.4 eV would be more favorable in photocatalysis [3]. We therefore synthesized nanoparticles of zinc oxide (ZnO) doped with 0.5 and 1.0 per cent manganese by chemical vapor synthesis and investigated these samples by X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), UV-vis and FT-IR spectroscopy and electron paragmagnetic/spin resonance (ESR/EPR) [4].

The resulting nanoparticles are in the nanometer size range and of ZnO wurtzite structure. Their color is slightly yellow as opposed to pure nanoparticulate ZnO which is plain white. ESR/EPR as well as XAS in combination with Reverse Monte-Carlo simulations are used to investigate the local structure around the manganese atoms in order to identify the exact dopant location inside the wurtzite lattice and examine any structural distortion introduced by the dopant [5].

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A2

HYDROTHERMAL SYNTHESIS OF COMPOSITE HETEROSTRUCTURES IN THE TiO_2-SnO_2 SYSTEM

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Among the metal oxides, tin oxide and titanium dioxide belong to the group of materials with potentially the widest range of applications, due to their chemical and electrical properties. There are many studies relating to the synthesis and individual properties of TiO_2 and SnO_2. However, the hydrothermal synthesis and characteristics of composite nanoparticles in the TiO_2-SnO_2 system have not been much investigated. The work presents the synthesis and characterization of TiO_2-SnO_2 heterostructures obtained by the alkaline hydrothermal method. Applied experimental procedure allows to obtain mixtures of TiO_2 and SnO_2 crystals differs in size and shape.

The materials were characterized by X-ray diffraction (XRD), surface area estimated from the N_2 physisorption isotherm (BET), high-resolution transmission electron microscopy (HRTEM), scanning transmission electron microscopy (STEM).

X-ray diffraction measurements performed for nanocomposites TiO_2-SnO_2 reveal that nanomaterials are well crystallized and two polymorphic forms, anatase TiO_2 and cassiterite SnO_2 are presented. The obtained nanopowders are characterized by a significantly expanded specific surface area, which suggests low agglomeration of particles. The particle size of the composite nanoparticles was in the range of 5 nm (cassiterite) to 40 nm (anatase).

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A3

SOLVOTHERMAL SYNTHESIS OF MAGNETITE NANOPARTICLES SUITABLE FOR APPLICATION IN MAGNETIC HYPERTHERMIA

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In this paper, spherical Fe_3O_4@OA nanoparticles were prepared via solvothermal method, in which iron sulphate, sodium hydroxide and oleic acid were used as precursors. The main idea of this research was to develop the method for preparation of nanoparticles with magnetic properties suitable for application in magnetic hyperthermia. We have tried to tailor size, shape and the degree of interparticle interactions by varying experimental conditions during the synthesis route. Obtained samples were structurally and magnetically characterized by means of different experimental probes such as: X-ray diffraction, SQUID magnetometry, FTIR and Mossbauer spectroscopy. Structural characterization asserts that the obtained phase is magnetite, Fe_3O_4, while FTIR spectra support the presence of oleic acid coating on the particle surface. Detailed magnetic characterization done by SQUID measurements and Mossbauer spectroscopy confirmed the presence of the strong and weak interactions in the nanoparticles system, depending on the synthesis conditions.

A4

SPHERICAL SILICA NANOPARTICLES OBTAINED BY STÖBER PROCESS – TETRA-ETHYL-ORTHOSILICATE CONCENTRATION INFLUENCE UPON SILICA NANOPARTICLES MORPHOLOGY

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Spherical silica nanoparticles with controllable sizes was synthesized using tetra-ethyl-orthosilicate as starting material and ethanol as mutual solvent by base catalyzed sol-gel method. The influence of the precursor's concentration on the characteristics of the silica powders was investigated. Reactants mole ratio was nTEOS:H_2O:ETOH:NH_3 where $n = 0.022 \div 0.31 : 0.43 : 0.789 : 0.06$. Powders have spherical morphology, and diameter between 3 and 9 nm. Synthesized silica particles were characterized using SEM (Scanning Electron Microscopy). Textural properties of silica particles were investigated by BET technique. Results of BET specific surface area analysis were in 13–112 m^2/g range. The sol-gel route possesses advantage for synthesis of silica from silicon alkoxides, because of its low cost and environment friendly.

A5

PHASE EVOLUTION OF SPHENE BASED CERAMICS DURING ANNEALING

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Sphene ($CaTiSiO_5$) has been prepared from a powder mixture of $CaCO_3$, TiO_2 and SiO_2 using vibro-miling for homogenization and activation precursors. The mechanochemical process initially yielded amorphous powders, which on sintering at a temperature of 872 °C and 920 °C crystallized to form sphene ($CaTiSiO_5$) with minor amounts of secondary phases (perovskite and coesite). The evolution of the phase composition during thermal treatment was investigated by X-ray powder diffraction (XRPD) and thermal analyses (DTA-TGA). HSM results reveal that the maximum density is achieved at 1250 °C. The microstructure revealed the presence of densely packed grains in the sintered body. This simple method proved to be suitable to get sphene based ceramic at 1250 °C with a relatively high density (94%), which has potential for various applications.

A6

STUDY OF NICKEL-COBALT SPINELS PREPARED BY PECHINI METHOD

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The world-wide growing energy demand has boosted the deep research on alternative energy conversion and storage systems which are environmentally friendly, highly efficient and costly-effective. Rechargeable zinc-air batteries are promising power sources that use inexpensive and environmentally benign materials.

Nickel cobaltite, $NiCo_2O_4$, is a mixed-metal oxide spinel that has shown exceptional ability to serve as an oxygen evolution electrode and has been studied quite extensively by electrochemical methods for the purpose of the Zn-air batteries. However, the oxygen reduction reaction (ORR) on cathode is sluggish which limits the rate performance of the battery. Developing of highly active catalysts for ORR is necessary to improve the performance of zinc-air battery.

The aim of the present work is to study oxide materials from $NixCo_{3-x}O_4$ system,

where *x* = 0.7, 1.4, 2.1 as bifunctional catalysts for oxygen evolution and reduction electrodes.

The materials are prepared by means of Pechini method and characterized by X-ray diffraction (XRD), Infrared spectroscopy (IR), Differential thermal analysis (DTA) and Scanning electron microscope (SEM). The XRD analysis shows that the oxides crystallize in a cubic spinel phase. The SEM images reveal that the particles are mostly spherical in shape with size between 50–100 nm. The DTA results define the characteristics temperatures of the material.

A7

Ti_2AlC GEL-CAST FOAMS – PROPERTIES AND CHARACTERIZATION

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Ternary carbides called MAX phases have a layered structure in nanoscale with heterodesmic covalent chemical bonds. They have a $M_{n+1}AX_n$ composition, where M is early transition metal, A is an element of A group and X is carbon or/and nitrogen. They have unique properties laying between metal and ceramics such as low density, low thermal expansion coefficient, high modulus and high strength, good high-temperature oxidation resistance, good electrical and thermal conductors, machinability, tolerance to damage, and resistance to thermal shock. Those features make MAX phases very interesting and perspective novel materials.

In this work the foaming and gelling technology of ceramic suspensions containing Ti_2AlC powders was developed. Porous samples were manufactured with the use of agarose as a gelling agent. The rheological properties of the slurry as well as some of the mechanical and structural characteristics were investigated and described. The carbide preforms having interconnecting network of pores were found to be suitable for manufacturing metal-ceramic interpenetrating composites by metal melt infiltration.

A8

TRANSLUCENT ZIRCONIA POLYCRYSTALS PREPARED FROM NANOMETRIC POWDERS

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The zirconia-yttria solid solutions have been extensively studied due to their unique properties. In the case of material containing 8 mol% Y_2O_3, characterized by the cubic symmetry, its properties as the solid solution electrolyte proved to be useful. The pore-less or nearly pore-less polycrystals of this composition are interesting for their optical applications due to the very high refractive index (2.2) which has never been attained in optical glasses. The yttria zirconia solid solution polycrystal of 3 mol% Y_2O_3 and of tetragonal symmetry shows high fracture toughness due to the martensitic transformation of the tetragonal phase to its monoclinic form at the crack tip running through the material.

Relatively few investigations have been published on the fabrication methods of the theoretically dense zirconia polycrystals of 8 mol% Y_2O_3 content. In the majority cases the sub-micrometer, commercial powders were applied.

The aim of the present study was based on application of the really nanometric zirconia solid solution powders of yttria content of 8 mol% and 3 mol%. The powders were manufactured under hydrothermal conditions. The main problem in application of such powders is their tendency hard agglomerates forming. It will be shown how to overcome this obstacle.

The yttria-zirconia solid solution powders were prepared by the hydrothermal treatment of the co-precipitated gels. It resulted in the materials of particle sizes <10 nm. Their aqueous suspensions were subjected to the freeze drying. It leads to the powders composed of very weak agglomerates which break under pressure as low as ~1 MPa. The powders were characterized for their phase composition, specific surface area and pore size distribution of their compacts prepared by cold isostatic pressing under 250 MPa. They were pressure-less sintered in oxygen atmosphere to the state of closed porosity and then HIP-ed at 1300 °C. Microstructure investigation of the dense materials revealed their grain sizes and the grain size distribution. Grain sizes of the material of higher yttria content were about twice larger than those of the lower yttria concentration. Spectrophotometric investigation in the wave length range 190–1100 nm indicated high transparency of the 8 mol% Y_2O_3-ZrO_2 and much lower of the one of 3 mol% yttria content. Most probably it should be related to the birefringence phenomenon which occurs in the latter case due to the tetragonal symmetry of this material. In the polycrystal of 8 mol% Y_2O_3 concentration this phenomenon does not occur due to its cubic symmetry. Both materials were studied for their hardness and fracture toughness.

A9

LOW TEMPERATURE DEGRADATION OF ZIRCONIA MATERIALS SINTERED VIA MICROWAVE HEATING TECHNOLOGY

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Yttria (Y_2O_3) stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics have become significantly important as biomaterials for dental applications due to their superb mechanical properties, null interaction with human tissue and promising aesthetic characteristics [1]. Currently, sintering of Y-TZP materials is carried out in conventional heat furnaces that require long sintering times and high energy inputs. New heating technologies are being developed in order to reduce processing times and energy consumption to lower costs [2]. One of these techniques is microwave sintering. Microwave heating consists on the absorption of electromagnetic radiation by materials due to their intrinsic dielectric properties [3,4]. Ceramic materials such as Y-TZP are able to absorb microwave radiation due to the presence of molecular dipoles [3], in what is known as dielectric heating [5]. The purpose of this work is to have a better understanding of the effects of microwave heating in the sintering of Y-TZP ceramics and determine whether there can be an improvement in the quality of the consolidated material. A thorough characterization of the resulting microstructure, properties and behavior under simulated service conditions is carried out. This investigation consists of a comparative study of the resulting microstructure and mechanical properties between microwave and conventional sintering, evaluation of the effect of microwave sintering in low temperature degradation (LTD), and assessment of the wear behavior of sintered materials under wet and dry conditions. Figure 1 summarizes the different approaches and tests performed for characterizing the samples at each stage.

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A10

EXTREMELY THICK COATING PREPARED FROM TiO_2 BY PLASMA SPRAYING

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Rutile TiO_2 was sieved to obtain feedstock powder for plasma spraying (size 100–170 µm). Samples were produced using the water-stabilized plasma spray system WSP® 500 (Institute of Plasma Physics, Prague, Czech Republic). This system operates at about 160 kW arc power and can process high amounts of material per hour. The powder was fed in through two injectors and thickness of the produced coatings was as high as 15 mm. Aluminum blocks were used as substrates and compressed air as a powder feeding gas. Temperature was monitored by a two-color pyrometer, not to exceed 290 °C. The coatings were then stripped-off for further characterizations. This form of the plasma deposited material is called Free-Standing Plate (FSP).

A thermal post-treatment (annealing) was applied on selected samples. The samples were heated in a laboratory furnace in the air atmosphere with heating and cooling rates 6 °C/min and with the dwell time of 3 hours on the maximum temperature 1100 °C. Such a temperature is high enough for stoichiometry restoration and also for substantial changes in the microstructure, and in dielectric properties.

The material with this high thickness could be examined as a sintered bulk body, since geometrically is it fully comparable with them and not with a thin film or even thick film. The extremely high coating thickness enables us to cut the FSP into three parallel :horizontal” slices (i.e. cuts were perpendicullar to the spray direction): one was close the substrate, second one in the middle of the deposited mass and the third one close to the surface. This sectioning was performed on as-sprayed deposit as well as on thermally post-treated one. Usually the low coating thickness does not allow such a slicing.

The layers close to the surface of the post-treated FSP exhibited dielectric relaxation at frequency 100 kHz while the central slice and the layers close to the substrate exhibited tendency to relax at frequencies over 1 MHz. Typical relative permittivity of the annealed material was around 150 and loss tangent around 0,05. The volume resistivity in the order 10^9 Ωm and the surface resistance of the individual slices in the order 10^{12} Ω were measured. Before the thermal post-treatment the volume resistivity was in order of only 1 Ωm. This conductive character of the sample was due to the plasma-induced reduction of the TiO_2 ceramics into TiO_{2-x}, as reported by the authors in the past. For the restoration of stoichiometry and a return of a dielectric character of the material, the thermal annealing is necessary. The massive TiO_2 ceramic blocks produced by spraying and subsequent annealing were tested as a material filling a cavity in the waveguide applicator useful for a microwave hyperthermia therapy of cancer. The results showed that the massive FSP is prospective for the mentioned medical application.

A11

ALUMINUM-DOPED $Li_7La_3Zr_2O_{12}$ AS A SOLID ELECTROLYTE FOR LITHIUM-ION BATTERIES

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The synthesis and processing of fine-grained $Li_{7-3x}La_3Zr_2Al_xO_{12}$ (x = 0.17) solid electrolyte (LLZO) is performed for the first time using a combination of nebulized spray pyrolysis (NSP) and field assisted sintering technique (FAST). Using FAST, the grain growth is suppressed and highly dense ceramics with 93 % of the theoretical density are obtained. The total conductivity of the sample at room temperature is found to be 0.33 mS cm^{-1} with an activation energy of 0.38 eV, which is comparable to the literature values. Galvanostatic cycling of a symmetrical cell Li|LLZO|Li shows a good cycling stability over 100 h. The interfacial resistance in contact with Li-metal is determined using alternating current impedance spectroscopy to be 76 Ω cm^2.

A12

MECHANICAL PROPERTIES OF FULLY DENSE CERAMIC ELECTROLYTES FOR SOLID OXIDE CELLS

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Solid oxide fuel cells (SOFCs) technology offers a real alternative to classical technologies for the generation of electricity by clean, efficient and environmental-friendly means. Nevertheless, the main limitation of SOFCs lies in their efficiency losses with time or their breaking under temperature cycling. Nowadays there is an intense search for materials for SOFCs with the objective of improving these limitations. Among them, lanthanum silicates and yttrium-doped barium zirconates were presented as a potential alternative to be used as electrolyte material in solid oxide cells. While numerous studies have dealt with the influence of composition on conduction properties, very little is known about the mechanical properties of these compounds.

We have thus started an exhaustive study of mechanical properties of $La_{9.33+x}Si_6O_{26+3x/2}$ and $BaZr_{1-x}Y_xO_{3-x/2}$ sintered compounds, using known strategies to obtain dense compounds [1–4] and from our knowledge of oxide mechanical properties characterization [5–7]. We have performed both room temperature characterizations of mechanical properties through nano-indentation and elastic properties measurements. We also performed high temperature mechanical tests in conditions close to that of a working fuel cell.

In both cases, experiments were realized on high density samples (d > 98%) prepared through classical sintering. The young´s modulus and hardness of these two materials were first analyzed by nano-indendation and the results were interpreted by the Oliver and Pharr method. Mechanical compression tests were also performed at roomtemperature and compared to resonance spectroscopy measurements. High temperature (800 °C) tests were also performed which allowed testing the limit of failure of these materials in working conditions.

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A13

CHARACTERIZATION OF LiNaSO$_4$ FOR BATTERIES APPLICATION

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Solid ionic conductors are generally crystalline compounds in which electric current is carried by ions. They are promising materials for solid electrolytes application due to inflammability unlike the liquid electrolytes, which makes them safe and give high power density. We are interested in lithium sodium sulphate, LiNaSO$_4$ who belongs to the family of mixed sulphates of the LiMSO$_4$ (M = Na, K, Rb, NH$_4$, Ag) type, which exhibits interesting properties of ionic conduction. According to literature it has been used so far in electrochemistry as a solid electrolyte and as a supplement in a functional layer in medium temperature fuel cells. It is famous for its ionic superconductivity in the range of 500–700 °C.

Our goal is to study LiNaSO$_4$ for application as electrolyte in lithium batteries. We prepared our samples by means of mechano-chemistry and wet chemistry methods. The methods allow managing of the atmosphere and the synthesis at room temperature. The performed X-ray investigations confirmed that all characteristic peaks of LiNaSO$_4$ are present. The samples are homogenous with average particle size of 100 μm. The claim SEM images confirm the homogeneity on the surface and in the depths by the. The total conductivity of the samples with evaporated golden electrodes is measured by means of impedance spectroscopy method. The structure of the samples is characterized by using Infrared spectroscopy structurally and thermal behaviours are defined by DTA/TGA analysis.

A14

PRESSURELESS SINTERING OF SiAlON CERAMICS

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SiAlONs are ceramic materials with a range of technically important applications, from cutting tools to wear parts and the properties of SiAlONs can be tailored for specific applications. The main sintering method of SiAlON is gas pressure sintering. Gas pressure sintering is a rather costly method as a result of high investment and operation cost. Therefore, there is a need to develop pressureless sintering method of SiAlON ceramics to reduce their production cost and enable wider scale applications. Main drawback of pressureless sintering of SiAlON ceramics is the limitation of sintering temperature below 1800 °C in order to avoid Si_3N_4 decomposition. Therefore, rather reactive additive combinations and good processing conditions need to be developed. In this study, dense SiAlON ceramics were attempted to be produced by pressureless sintering of dry pressed compacts. With this respect, additive composition and amount, granule character, sintering temperatures etc. have been investigated as a way to achieve sintered densities over 99% and resultant properties are discussed.

A15

THERMAL OXIDATION MECHANISM OF MESOPOROUS SILICON

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We performed experiments and related analyses for obtaining mechanism of the oxidation reaction of mesoporous silicon (pSi) particle. The experiments were conducted on a pSi produced from silicon wafer by anodization and converted to micro-particle with sonication. Oxidation experiments were carried out at three different heating rates. The free model kinetics proposed by Ozawa-Flynn-Wall was used to determine the kinetic triplet for the pSi thermal oxidation. The reactions are determined to be a first oxidation at low temperature by removing surface moieties based on the Avrami-Erofeev model, followed by classical bulk oxidation according to three dimensional diffusion mechanism which controlled by Silicon oxide layer on the surface of pSi. However,

differential thermal analysis suggests that oxidation steps in this system are under control of phase transformation of silicon oxide.

A16

CHEMICAL VAPOR SYNTHESIS OF ALUMINUM NITRIDE NANOPARTICLES FROM METALIC ALUMINUM

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Chemical Vapor Synthesis (CVS) is a method to generate nanoparticles from the gas phase due to reaction of precursor substances. Narrow particle size distributions with homogeneus powder properties can be achieved once the process parameters are optimized.

Using a high frequency furnace allows the control of the time - temperature history of the particles by changing the geometry of the induction coil. Optimal time - temperature profiles enable the merging of the usual CVS process with metal evaporation as a precursor delivery method.

In this work aluminium nitride (AlN) was synthesized by evaporation of aluminium metal and subsequent reaction with ammonia. The synthesized AlN was characterized by X-ray diffraction, energy dispersive X-ray spectroscopy, scanning transmission electron microscopy and low temperature nitrogen adsorption.

A17

NANO-METER SIZED MAGHEMITE WITH HIGH SURFACE AREA AND SUPERPARAMAGNETIC BEHAVIOR SYNTHESIS BY OXIDATION OF MAGNETITE

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The synthesis of iron oxides that exhibit magnetic properties has been intensively studied for numerous applications in different fields, such as biomedical and technological areas. In this study, maghemite nanoparticles were synthesized in air atmosphere by chemical precipitation of Fe(III)Fe(II) precursors mixture, specifically $FeCl_3 \times 4H_2O$ and $FeSO_4 \times 7H_2O$ (2:1 molar ratio), in the presence of two different bases, KOH and NaOH. Two series of samples were prepared, being labeled as K series (KOH) and N series (NaOH), where the total amount of iron varied in reactant mixture (from 79.6×10^{-3} mol/L to 9.9×10^{-3} mol/L), while the concentration of supplied hydroxide solution remained constant (215×10^{-3} mol/L). Maghemite nanoparticles were obtained by air oxidation of as-synthesized fresh magnetite. It was assumed that the magnetite to maghemite completely oxidation occurred during drying and storage in air atmosphere, that was proved by Moessbauer measurements. We observed that crystallite size calculated from XRD patterns decreased as the iron concentration decreased. According to the magnetic measurements, all the samples present room temperature superparamagnetic behavior. As-prepared nanoparticles with spinel structure and spherical-like shape present relative high surface areas and porous structure, depending on synthesis conditions.

A18

CHEMICAL VAPOR SYNTHESIS (CVS) OF Ga_2O_3 AND GaN NANOPARTICLES FOR WATER SPLITTING

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In recent years, numerous attempts have been made for the development of photocatalysts that work under visible-light irradiation and efficiently utilize the solar energy. Photocatalytical activity of semiconducting materials, such as Ga_2O_3 and GaN, depend largely on their structural properties. Light absorption and charge carrier generation can be optimized by size effects and doping, charge separation by low defect density and band alignment through microstructural variation.

In contrast to conventional synthesis routes in the liquid phase, we want to exploit the advantages of Chemical Vapor Synthesis (CVS) method to meet these challenges. Nanoparticles are generated in the gas phase from inexpensive sources such as $Ga(acac)_3$ in a reaction with oxygen to form Ga_2O_3. By adding ammonia instead of oxygen as a reaction gas, GaN nanoparticles are synthesized. The objective of this work is to investigate the influence of process parameters, such as reaction temperature and system pressure on the characteristics of the final powders, in particular, particle size, crystallinity and agglomeration. It is found that temperatures between 1100 and 1200 °C and pressures of 300 mbar are optimal for producing highly crystalline materials with low defect density and high specific surface area, capable of performing overall water splitting. Gallium oxide structures, pure β-Ga_2O_3 phase, as well as GaN wurtzite structure are obtained according to X-Ray Diffraction analyzed by Rietveld refinement. Specific surface area and surface morphology are investigated using low nitrogen adsorption method and electron microscopy.

A19

CERAMIC PROCESSING OF ALL-OXIDE CERAMIC THERMOELECTRIC MODULE

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Thermoelectric (TE) oxides are promising candidates for energy conversion in a temperature range of 300–1200 K. These materials have many advantages like excellent high temperature stability, are environmental friendly and have low cost compared to non-oxide materials traditionally used as thermoelectric materials, but they also have disadvantages due to efficiency and processing. The efficiency of a TE material is described by the ZT or figure of merit which includes electric conductivity, Seebeck coefficient and the thermal conductivity. The module contains an n- and a p-type conductor separated by an insulator and connected with metal current collectors, regarding conventional TE module design. The metal layer limits the TE working temperature range because of melting.

For better utilization of the advantages with the oxides, a new design for an all-oxide TE device without metal current collectors is proposed (Fig. 1). Development of this new stack design requires many issues to be taken into consideration. This includes improving the efficiency of the oxide materials through designed microstructure followed by an advanced ceramics processing approach. The new technology can be applied towards ease of commercialization of TE modules.

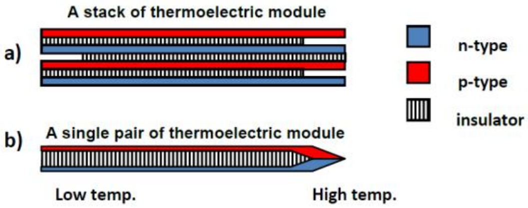

Figure 1. All-oxide TE module design

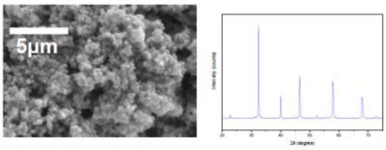

Figure 2. Synthesised powder of LSTO

For our all-oxide device we selected La-doped $SrTiO_3$ (LSTO), $Ca_3Co_4O_9$ (CCO) and $LaAlO_3$ (LAO) as n-type, p-type and insulator materials respectively, based on coefficients of thermal expansions. Each of these three oxides were synthetizing using solid state method or spray pyrolysis and the powders were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and dilatometry (DIL) (Fig. 2). Phase pure TE-materials were analysed to determine ZT, by measurements of Seebeck coefficient, electrical and thermal conductivity.

Using aqueous tape casting thick green tapes were prepared. Green tapes were thermally treated by co-sintering method. Chemical stability and compatibility of the three materials will be discussed. The work will report on the processing of the TE materials and their TE properties.

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A20

NEW LEAD-FREE MATERIALS WITH $A_{1/2}Bi_{1/2}BO_3$ FORMULA (A = Li, Rb; B = Ti)

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Nowadays, $PbZr_{(1-x)}Ti_xO_3$ compounds (PZT) are the most employed piezoelectric ceramics owing to their high electromechanical performance and low cost. However, because of the restrictions on the use of hazardous chemical elements, such as lead, the PZT materials must be substituted by lead-free compounds. These new materials are thus highly investigated for their piezoelectric properties. Actually, the most promising compounds are (i) alkaline-niobates, such as $(Na,K)BiO_3$, (ii) alkaline and bismuth titanates, such as $(Na, K)_{1/2}Bi_{1/2}TiO_3$ (NBT and KBT), and (iii) solid solutions based on these compounds such as $Na_{1/2}Bi_{1/2}TiO_3$-$BaTiO_3$.

This work is devoted to the search of new lead-free materials based on NBT and KBT. These two compounds, initially reported by Smolenskii *et al.* in 1961, show the possibility to replace lead cations of perovskite compound $PbTiO_3$ by a pseudo-ion alkali/bismuth (Na^+/Bi^{3+} or K^+/Bi^{3+}). If NBT and KBT have been largely investigated, there is no report yet about the formation of perovskite compounds with other alkali-elements like lithium and rubidium. Thus, the compositions $Li_{1/2}Bi_{1/2}TiO_3$ (LBT) and $Rb_{1/2}Bi_{1/2}TiO_3$ (RBT) have been synthesized and characterized by X-ray diffraction to identify the obtained phases. However, none of these compositions led to a perovskite structure.

With rubidium ion, the identification of the phases has not been possible as the ternary system Rb_2O-Bi_2O_3-TiO_2 has not been reported yet. In the case of lithium ion, we indexed all the phases, notably a pyrochlore isostructural to $Bi_2Ti_2O_7$. This phase is known as a metastable compound which decomposes above 650 °C, but stabilized in this work at 800 °C. It is also reported to be a promising material for Metal-Oxide-Semiconductor transistor as it has high permittivity and low dielectric loss. Thus, it seems interesting to study the stabilization of this pyrochlore by lithium ion.

We first study the stability of our compound as a function of temperature and check that the phase didn’t exist at 800 °C without addition of lithium. Then, we investigate the existence domain of this pyrochlore in the ternary system Li_2O-Bi_2O_3-TiO_2. Finally, we study the structure of this compound.

A21

STRUCTURAL AND ELECTRICAL PROPERTIES OF BST CERAMICS PREPARED BY HYDROTHERMALLY ASSISTED COMPLEX POLYMERIZATION METHOD

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Barium strontium titanate, $Ba_xSr_{1-x}TiO_3$, ceramics has been extensively used in microelectronics due to its excellent ferroelectric, dielectric, piezoelectric, and pyroelectric properties. In this work $Ba_{0.8}Sr_{0.2}TiO_3$ (BST) was obtained by hydrothermal treatment of precursor solution containing titanium citrate, previously prepared by complex polymerization method, and barium and strontium acetates. The powders were calcined at 700 °C, pressed into pellets and further sintered at 1280 °C for a various sintering times (from 1 to 32 h), in order to obtain dense ceramics. The sintering effect on structural parameters, phase composition, dielectric and ferroelectric properties was investigated. The phase compositions of the sintered samples were determined by X-ray diffraction (XRD) and EDS analysis. Microstructural properties were investigated by scanning electron microscopy. The phase transitions and dielectric properties were investigated by measuring dielectric permittivity (ε) and dielectric losses ($\tan\delta$) as a function of temperature. Ferroelectric properties such as remanent polarization (Pr) and coercitive field (Ec) were determinated by polarization-electric field (P-E) measurements. Rietveld refinement analysis of BST ceramics showed that predominant phase was tetragonal BST with monoclinic $Ba_6Ti_{17}O_{40}$ (B6T17) as a secondary phase. Phase contents determined by Rietveld refinement match well with the results of energy dispersive X-ray analysis. SEM micrographs of sintered samples revealed the abnormal grain growth where elongated twinned grains of BST grow in a matrix of smaller B6T17 grains. Dielectric and ferroelectric properties of BST ceramics have been correlated with density of sintered compacts, phase composition, structural parameters and microstructure.

A22

OSTEOCONDUCTIVE CERAMICS WITH A SPECIFIED SYSTEM OF INTERCONNECTED PORES BASED ON MONOPHASIC CALCIUM PHOSPHATES

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For the regenerative therapy techniques used in modern medicine, a calcium phosphate bone implants should have the following key properties: bioresorption (closely related with Ca/P ratio), osteoconduction, and sufficient strength. For permeability of the implant, a special architecture with interconnected pores with a size of at least 100 µm and an overall volume of more than 60% are required. However, the implant strength rapidly diminishes with increase of the porosity. The load on the implant can be eliminated in the initial period of therapy (therefore, a strength of 0.1–1 MPa is considered to be sufficient). A popular solution in the design of highly porous materials is the use of positive replica technique from open porosity polymeric sponge. The highly porous ceramics formed in this way has two serious drawbacks: low strength (below 0.1 MPa) and the absence of straight channels (related to the architecture of the initial sponge), resulting in relatively low permeability.

The goals of this study were to compare various industrial processes for the manufacture of highly resorbable ceramic implants with pre-determined Ca/P ratio between 0.5 and 1.5 based on having sufficient strength by means of three dimensional printing methods.

In this study, we compare several three-dimensional printing techniques mainly thermal extrusion 3D printing (fused filament fabrication, FFF process) and stereolithography (SLA process) using suspensions containing thermoplastic polymers and powders of pre-synthesized amorphous chemically mixed anionic precipitates or calcium ammonium pyrophosphates. The calcination of the preforms led to the formation of biphasic ceramic materials.

As a result, we have produced a number of biphasic ceramic materials based on calcium phosphates with pre-determined Ca/P ratio. Obtained samples were examined by XRD, scanning electron microscopy, thermal analysis, X-ray tomography, biomimetic and mechanical tests.

A23

MIXED CATIONIC TEMPLATES CONTROLLING ORDERED SILICA MORPHOLOGY

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MCM-41 type silica particles were obtained via base catalysed sol-gel route with Hexadecy lTrimethyl Ammonium Bromide (CTAB) and n-Dodecyl Trimethyl Ammonium Bromide (DTAB) equimolare mixture as template. It was investigated the each one and mixed cationic surfactants effect upon particle morphology depending on side chain length. When using equimolar mixture of two surfactants, the contribution to the porosity properties development was more significant in the case of longer side chain surfactant. Its influence dominate the resulting material pore structure evolution.After template removal by thermal treatment (~700 °C) porous silica nanoparticles have higher specific surface areas than xerogels, but it is occurring partial loses pore ordering. The morphology of particles was investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The images reveal that the particles are spherically shaped with radials oriented pores. The developed silica samples porosity and microstructure has been qualitatively evaluated using small angle neutron scattering (SANS) analysis which was revealed that the surface of the silica microspheres is smooth. The maximum value for the total pore volume was 0.743 cm^3/g obtained for the samples synthesized with CTAB, ammonia and calcinated at 500 °C; therefore the obtained mesoporous materials present most suitable properties in order to become drug carriers.

A24

THE STRUCTURE AND TEXTURE CHARACTERIZATION OF ZINC DOPED BIOACTIVE GLASSES FROM $NaCaPO_4$-SiO_2 SYSTEM

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Silicate-phosphate glasses and glass-ceramics materials from $NaCaPO_4$-SiO_2 system have an ability to form strong chemical bonds with living tissue. Their bioactivity is associated with the rapid formation of a thin layer of hydroxycarbonate apatite (similar to biological apatite) on their surface when the materials are implanted or in contact with biological fluids. The bioactive glasses and glass-ceramics have been recently applied in bone regeneration field, coating of implants and scaffolds. During implantation exists a risk of adherence and colonization of bacteria on the surface of the implants, what may lead to the uselessness of implants or to their failure. Bioactive materials containing addition of antibacterial ions can reduce microbial numbers on implants surface and kill bacteria or inhibit their growth. The choice of zinc was related to its antibacterial properties and fundamental role in bone formation.

Several compositions of zinc doped glass were synthesized by sol-gel method. Using X-ray, IR and Raman spectroscopy, the structure of amorphous state was characterized. The morphology and composition of the glasses were analyzed by scanning electron microscopy equipped with energy dispersive spectroscopy (EDS). The influence of zinc doping on the bioactivity of the glasses from $NaCaPO_4$-SiO_2 system was researched. All glasses were immersed in a simulated body fluid (SBF) at 37 °C for 30 days. Moreover thermal analysis was carried out to check the thermal properties of the glasses.

A25

EVALUATION OF MESOPOROUS SILICA AND TITANIUM DIOXIDE AS ANTIBIOTIC CARRIERS IN DRUG DELIVERY SYSTEMS

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Controlled delivery of drugs is an essential tool to improve drug delivery design and reduction in systemic toxicity. One of the strategies for controlled release of drugs could be development of biocompatible drug carriers. Mesoporous materials could overcome the disadvantages of traditional drug dosage forms offering possibilities to optimize drug delivery in appropriate dose to specific sites or to prolong delivery duration. Mesoporuos silica and titanium dioxide are valuable candidates for drug delivery carriers because they possess chemical and thermal stability, high biocompatibility and low toxicity.

Mesoporous silica and titanium dioxide were evaluated as gentamicin carriers for controlled drug delivery so their adsorption capacity for antibiotic was quantified at different pH values as well as kinetics of its release. Results showed that the highest amount of gentamicin, 80 mg per gram of material, was adsorbed by silica at pH 7.5 while 38 mg were desorbed at pH 5 during the first six hours. Two forms of nano-sized titanium dioxide were tested for their suitability as drug carriers. At pH 5 one-gram TiO_2 adsorbed 65 mg and 54.8 mg gentamicin when applied as powder and tableted powder, respectively. Tableted titanium dioxide enabled linear kinetic of gentamicin desorption at pH 4 during the first five hours which makes it a promising material for controlled drug release.

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A26

IMPROVEMENT OF THE MECHANICAL AND ANTIBACTERIAL PROPERTIES OF HYDROXYAPATITE

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In the last years, the bioactive coatings were frequently used to enhance adhesion between the bone and metallic implants. Among different types of bioactive coatings, HydroxyAPatite (HAP-$Ca_{10}(PO_4)_6(OH)_2$) is one of the most extensively used for bone repair and augmentation, as well as for implant coating, because of its chemical similarities to the bone and teeth components. However, the low mechanical strength of plain HAP ceramic and the relatively low bone bonding rate restrict its use as biomaterial.

The main goal of the presented work is the enhancement of the mechanical properties and of the resistance to the bacteria of the HAP deposited on Ti6Al4V alloy substrates. For this propose, SiC and Ag were added into the HAP structure. The coatings were deposited by co-sputtering of HAP, SiC and Ag targets, using an RF magnetron sputtering system. The power fed on Ag target was varied from 0.6 W to 1.2 W in order to prepare the coatings with different Ag contents. The SiC target was fed at constant RF (15 W). All the coatings, grown at 700 °C, were investigated in terms of elemental and phase composition, texture, morphology, mechanical properties (hardness, elastic modulus), corrosion resistance and antibacterial activity (*Staphylococcus aureus* MRSA), by EDS, XRD, FTIR, SEM and AFM techniques, nanoindentation, electrochemical tests and antimicrobial tests. The effects of the SiC addition and Ag contents on the film characteristics were also analyzed.

SiC addition determined no changes in HAP stoichiometry and crystalline structure. Also, no significant changes in phase composition and corrosion resistance of the films were observed as a result of different Ag concentrations in HAP films. The antibacterial abilities of HAP coatings were significantly improved by Ag addition, as observed after 24 h or 7 days of culture. The hardness, elastic modulus and antibacterial properties of the HAP coatings increased with increasing power fed on Ag target. On overall, improved mechanical and antibacterial properties were found by adding SiC and Ag to the basic HAP structure.

A27

SYNTHESIS OF ALUMINA POWDERS AND THEIR INSECTICIDAL EFFECT AGAINS *ACANTHOSCELIDES OBTECTUS* SAY

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Alumina powders were synthesized by citrate/nitrate autocombustion method with different citrate/nitrate molar ratios (0.5, 0.8 and 1.2). XRD analysis of the powders calcined at 1200 °C for 1 h showed only the presence of α–Al_2O_3 phase. Particle size of the powders determined by SEM analysis varied from nano- and submicron size of several µm. The powders were mixed with grains of bean to examine their insecticide effect against bean weevil (*Acanthoscelides obtectus* Say, 1831). Mortality of insects after seven days of exposure was the highest for the powder synthesized at 1.2 molar ratio, which contained the highest portion of submicron particles. This suggested that lager amount of citric acid could prevent higher degree of particles agglomeration. Mortality cause of insects is assumed to be related to the degree of powder coverage of the insects’ exoskeleton. The same powder also provided the best efficiency in suppressing the emerge of F_1 generation and seed protection even at lower concentrations.

A28

SPECTROSCOPIC STUDIES OF BIOACTIVE COATINGS BASED ON SILICON OXYCARBIDE GLASSES

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Silicon oxycarbide glasses or black glasses are materials of amorphous silica structure, where two O^{2-} ions are replaced with one C^{4-} ion. Due to local increase of bonds density they exhibit excellent mechanical, electrical properties as well as thermal resistance.

Glasses were prepared by sol-gel method. Ladder-like silsesquioxanes of strict T to D units ratio were used as a precursor. Glasses were burned at 800 °C in inert atmosphere. Conducted XRD and far infrared research showed that obtained material is fully amorphous. Detailed structural research in middle infrared allowed to state that obtained glasses of v-SiO_2 structure contain the Si–C bonds. SEM with EDX and Raman studies conducted after thermostating in simulated body fluid (SBF) showed presence of hydroxyapatite like phase.

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A29

INTERCALATION OF LAYERED CALCIUM PHOSPHATE AND SYNTHESIS OF CERAMICS BASED ON IT

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Over the world, people often face with damages and diseases of bone tissues. For a long time a material used extensively in medicine for restoring bone damage has been synthetic hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (HAp) because of its chemical and physical similarity to the inorganic constituent of bone tissue. The main disadvantage of these materials is the low resorption (dissolution) rate in tissue organism. Therefore, nowadays alternative materials are investigated for a bone tissue replacement, for example, resorbable implants based on homogeneous mixtures of two phases: calcium pyrophosphate $Ca_2P_2O_7$ (CPP) and tricalcium phosphate $Ca_3(PO_4)_2$ (TCP) or TCP/HAp. The precursors of these mixtures are layered calcium phosphates, in particular, octacalcium phosphate $Ca_8(HPO_4)_2(PO_4)_4 \cdot 5H_2O$ (OCP) and $Ca_8(HPO_4)_{2-x}R_x(PO_4)_4 \cdot 5H_2O$ (“intercalated”-OCP), in which a hydrophosphate-anion is substituted by a carboxylic acid-anion (R). Thus, the scope of this work consisted in making the precursors to fabricate biphasic bioceramics with a varying Ca/P ratio.

OCP became the object of the study due to its layered structure. It is composed of alternating apatitic and hydrated layers. Hydrophosphate groups in OCP hydrated layer can be replaced by dicarboxylate-anions. In this study substitution reactions of HPO4-anions in structure of brushite $CaHPO_4·2H_2O$ and OCP to acetic, succinic and citric-anions were carried out with pH-adjustment at different temperatures. Thus synthesized samples were characterized by powder X-ray diffraction (XRD), thermogravimetric analysis (TG) and scanning electron microscopy (SEM). The data were compared to that one of “pure”-OCP. Decomposition of “intercalated”-OCP occurs more slowly via of simultaneous liberation of CO_2 and H_2O from the structure of dicarboxylic-anion and hydrophosphate-anion, respectively. XRD data show that substituted OCP-phase was detected only in case of intercalation with succinic acid. Thermal decomposition of this phase leads to biphasic ceramics of different constitution according to substitution degree (x) of hydrophosphate-anion. Controling x-value one can vary the Ca/P ratio in Suc@OCP and, consequently, the resorption rate of biphasic ceramics.

A30

INFLUENCE OF THULIUM AND HOLMIUM OXIDE POWDERS MORPHOLOGY ON PROPERTIES OF TRANSPARENT Tm,Ho:YAG CERAMICS

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The aim of the work was to obtain transparent Tm,Ho:YAG ceramics (thulium doping range: 2.0–6.0 at.%, holmium doping range: 0.1–1.0 at.%) by reaction sintering using commercial powders and Tm_2O_3, Ho_2O_3 powders obtained by precipitation method.

Precipitation was conducted at various temperatures (25–70 °C) using 0.1–0.25 M water solutions of thulium and holmium nitrate and 1.5 M ammonium hydrogen carbonate water solution as a precipitation agent. The main aim of this work was to investigate the influence of precipitation parameters on the morphology, particle size and sinterability of final product.

The mixtures for reaction sintering of Tm,Ho:YAG, containing the powders produced and commercially available, were prepared and consolidated. Two types of granules were used: "manual" (obtained by sieving ceramic mas through a sieve) and granules obtained by the freeze granulation. The granulates were pressed (120 MPa) into pellets and the obtained samples were sintered in a vacuum at 1715 °C for 6 h. It has been proved that the crucial role for the optical quality of ceramics plays particle size, purity and degree of agglomeration of the powders used. Freeze granulation method allowed to obtain Tm,Ho:YAG ceramics with a high degree of microstructure homogeneity, which resulted in a high transmission values. As found, both concentration of holmium and thulium separately as well as the balance between them are of great importance for optical properties.

A31

MICROWAVE ASSISTED SYNTHESIS OF CdS QUANTUM DOTS IN DMSO

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There are many research studies investigating the wide range of application of cadmium sulfide (CdS) nanoparticles. By limiting the size of the nanoparticles to smaller than Borh radius, the quantization of the energy levels to discrete values causes the quantum confinement effect. The most remarkable feature of the quantum dots is the phenomena called multiplication by absorption of a single photon. Quantum dots of CdS prepared as aqueous sols have been reported to undergo photooxidation. Therefore, it was a challenge to synthesize CdS quantum dots in dimethyl sulfoxide (DMSO). This study shows that microwave radiation can affect CdS nanoparticles growth, since DMSO can act as a source of S^{2-} ions. The absorption onsets of the synthesized colloids are determined to be 395, 403 and 470 nm, for non-treated, pre-treated by microwave and post-treated colloids, respectively. The sizes of the nanoparticles were calculated by the effective mass model to be 3.01, 3.21 and 4.84 nm which was affirmed by transmission electron microscopy analysis.

A32

SPECTROSCOPIC INVESTIGATIONS OF Tm,Ho:YAG CERAMICS FOR SOLID STATE LASER APPLICATIONS

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Highly transparent ceramics have attracted a lot of interest due to their several interesting properties as low cost production and scalability to large size. These materials

find wide variety of industrial applications such as armors, lenses etc. Currently, ceramics is also very interesting material for active media of solid-state lasers. Since the 1990s the technology to generate a coherent beam from ceramic materials is improved and highly efficient lasers are investigated.

Trivalent thulium (Tm) and holmium (Ho) doped materials are suitable for generation of 2 μm emission in “eye-safe” wavelength region and can be used for biomedical, metrology and remote sensing applications. With novel freeze granulation method it is possible to obtain Tm,Ho:YAG ceramics with a high degree of microstructure homogeneity, which has resulted in high mechanical, thermal and optical properties equivalent to the best single crystals.

In this work we present the fluorescence properties in the “eye-safe” spectral range of YAG transparent Tm,Ho:YAG ceramics with various doping level of active ions. The unique set of transparent Tm,Ho:YAG ceramics with Tm concentrations ranging from 2 up to 6 at.% and Ho concentrations ranging from 0.1 up to 1 at.% enabled the investigation of dopants concentration influence on fluorescence properties. Careful spectroscopic characterization was performed, comprising concentration-dependent fluorescence spectra and fluorescence dynamics profiles under direct thulium and holmium ions excitation. All measurements were carried out at room temperature. Moreover, the energy transfer processes between Tm and Ho ions for both types of excitation (785 nm and 538 nm) has been demonstrated and discussed.

A33

HYDROTHERMAL SYNTHESIS OF OPTICALLY ACTIVE RARE EARTH FLUORIDES

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Hydrothermal method has great interest in recent years for synthesis of nano- and micro- crystals of upconverting rare earth (RE) fluorides, with controlled morphology and high purity, under high temperatures and pressures. Some surfactants (ethylenediaminetetraacetic acid (EDTA), polyvinylpyrrolidone (PVP), oleic acid (OA)) along with different type of solvents are added in order to control size, morphology and crystalline phases of particles. In this work Yb^{3+}/Er^{3+} co-doped YF_3 and $NaYF_4$ fine powders were synthesized using the hydro/solvo thermal method in the present of EDTA as a complexing agent. Effect of the processing parameters on the particles crystal structure, morphology and optical properties were estimated on the basis of X-ray diffractometry (XRPD), scanning electron microscopy (SEM) and photoluminescence measurement. It was shown that in terms of increased concentration of RE ions in aqueous solvent media the hexagonal β-$NaYF_4$:Yb^{3+}/Er^{3+} phase with the most efficient green emission were synthesized. On the other side, the occurrence of cubic α-

$NaYF_4$:Yb^{3+}/Er^{3+} and orthorhombic YF_3:Yb^{3+}/Er^{3+} were observed with a decrease of the RE ions when ethanol is used as a solvent. All of the samples provide intense green emission after been excited with infrared light (λ = 978 nm), which is assigned to the Er^{3+} ($^2H_{11/2}$, $^4S_{3/2}$) → $^4I_{15/2}$ electronic transitions.

A34

INFLUENCE OF Yb^{2+} ON OPTICAL PROPERTIES OF YAG:Yb GARNET

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Ceramic yttrium aluminum garnet (YAG) doped with Yb ions is one of promising material for solid state lasers due to the small quantum defect between the pump and the laser photons resulting in low thermal loading, long radiative lifetime of the upper laser level. Ytterbium ions in garnet can exist in two charge states 2^+ and 3^+. The presence of Yb^{2+} ion reduces fluorescence lifetime, and as result laser properties of ceramics. The purpose of this work is investigating charge valence transformation Yb ions in YAG.

To study the oxidation process ceramics of $Yb_{0,3}Y_{2,7}Al_5O_{12}$ was synthesized by reaction sintering. The synthesis was performed in a vacuum furnace at 1750 °C for 10 hours. After vacuum sintering ceramic YAG contain both Yb^{2+} and Yb^{3+} ions. Ceramics studied by means of XRD, scanning electron microscopy (SEM), optical absorption spectroscopy.

Synthesized ceramic consists of grains in range 3–40 nm. X-ray analysis reveals no impurity phase. Ytterbium ion can change valence state by means of oxidizing/reduction process. Under oxidation the oxygen is absorbed by ceramics, diffuses to Yb^{2+}, and oxidizes ones. This process is thermally activated.

Ceramic YAG:Yb was heat treatment at three different temperature 860 °C, 890 °C, 920 °C for different time. Monitoring the concentration of Yb^{2+} was performed with absorption spectra of Yb^{2+} and the ion concentration was calculated using the formula Smakula-Dexter.

The geometrical Yander model was used to describe dependence obtained. It was shown that process of changing valence of Yb ion limited by a diffusion mode. The value of activation energy of the oxidation process Yb^{2+} ions in ceramic YAG: Yb is estimated as 2.7 eV which is two times higher than one for single crystal of the same chemical composition that equals of 1.43 eV. This is most probably the fast that after mechanical treatment surface have different atomic defect, which are the centers of adsorption of molecules. This factor contributes to increasing rate of adsorption of oxygen onto the surface of the sample and consequently accelerated the whole process. Grains in ceramics possess atomic smooth surface, only grains located near the surface of the sample have the broken layer. Structural perfections of grain surface are the reason increasing activation energy process change valence transformation Yb^{2+} ion in YAG.

A35

PHASE DEVELOPMENT AND THERMAL BEHAVIOUR OF HYBRID SOL-GEL DERIVED MULLITE PRECURSOR

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The phase development of hybrid mullite precursor has been studied in the range of synthesized mullite gel to 1550 °C. The sol-gel derived mullite precursor powders were prepared using ethanol-water solutions of TEOS, Al(III) nitrate and urea. TGA/DSC, XRD and FTIR analyses were employed in order to follow structural development and phase transformation of as-prepared gel, combusted ash and calcined powders. Thermal analysis shows the two step mullitization path of synthesized mullite precursor occurred via spinel phase. The XRD patterns confirm that the crystalline mullite did not appear up to 1200 °C. The FTIR spectra of obtained mullite precursors and calcined mullite powders present the characteristic bands of Si–O–Si and Al–O–Al linkages up to 1200 °C indicating the diphasic nature of the examined precursors and powders. The SEM analysis reveals the irregularly agglomerated powder particles composed of smaller plate particles formed during combustion and calcination of the powder precursors.

A36

SUBCRITICAL CRACK GROWTH IN OXIDE AND NON-OXIDE CERAMICS USING THE CONSTANT STRESS RATE TEST

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Keywords: alumina, tetragonal zirconia, silicon carbide, silicon nitride, subcritical crack growth, constant stress rate

Fracture toughness is one of the most important parameters for ceramics description. In some cases, material failure occurs at lower stresses than described by K_{Ic} parameter. On these terms, determination of fracture toughness only, proves to be insufficient. This may be due to environmental factors, such as humidity, which might cause subcritical crack propagation in a material. Therefore, it is very important to estimate crack growth velocities to predict lifetime of ceramics used under specific conditions.

Constant Stress Rate Test is an indirect method of subcritical crack growth parameters estimation. Calculations are made using strength data, thus avoiding crack measurement. Expansion of flaws causes reduction of material strength. If subcritical

crack growth phenomenon occurs, critical value of crack lengths increases with decreasing stress rate due to longer time for flaw to grow before the critical crack propagation at K_{Ic} takes place.

Subcritical crack growth phenomenon is particularly dangerous for oxide ceramics due to chemical interactions occurring as a result of exposure to humidity. This paper presents results of Constant Stress Rate Test performed for alumina, zirconia, silicon carbide and silicon nitride in order to demonstrate the differences in subcritical crack propagation phenomenon course.

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A37

MICRO-SCALE PLASTICITY AND ELASTIC BEHAVIOUR OF CERAMIC CRYSTALS UNDER MICROPILLAR COMPRESSION

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Room temperature elastic and plastic behaviour of tungsten carbide (WC), β-silicon nitride (β-Si_3N_4) and zirconium diboride (ZrB_2) ceramics crystals were studied under micropillar compression. Pillars were fabricated by focused ion beam (FIB) in large grains, located close to the basal and prismatic orientations, selected by means of electron backscatter diffraction (EBSD). The micro-mechanical test and the damage characterization were performed by nanoindentation and scanning electron microscopy (SEM), respectively. In spite of the brittleness of these ceramics, considerable plasticity could be observed at certain orientations depending on the different slip activations of the materials. In WC and β-Si_3N_4 crystals the activation of the {10-10}⟨11-23⟩ and the {10-10}[0001] type slip systems were suggested, respectively. Moreover, significant elastic, plastic and fracture anisotropy was revealed showing different tendencies for Young's modulus, yield stress and rupture stress values which strongly influenced by the pillar orientation in case of each ceramic crystal.

A38

THE INFLUENCE OF SINTERING TEMPERATURE AND ADDITIVES ON THE MICROSTRUCTURE OF PRESSURE-LESS SINTERED TUNGSTEN CARBIDE

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The object of researches presented herein was polycrystalline tungsten carbide obtained by pressure-less sintering. According to the literature, too high sintering temperature or too large carbon addition lead to the phenomenon of excessive and discontinuous grain growth of WC. On the other hand, the addition of carbon facilitates sintering of WC and has a favorable influence on the thermal properties of polycrystalline WC. Within the framework of the carried out researches was sintering of: WC without additives (pure WC), WC containing carbon in an amount of 0.2 wt.% and 0.4 wt.%; WC containing tungsten in an amount of 0.5 wt.% and with the addition of both elements simultaneously in amounts of 0.5 wt.% W+ 0.2 wt.% C and 0.5 wt. %W + 0.4 wt.% C. Calculations of the amount of added carbon were based on the carbothermic reduction reaction of tungsten oxides (IV) and (VI) and amount of oxygen in the powder provided by the producer. The samples were sintered at a temperature range from 1800 to 2100 °C in the flow of argon. Dilatometric tests were also performed in order to register the sintering curves. Measurements of apparent density and porosity of the WC were also performed. An analysis of microstructures was done in terms of its correlation with the sintering temperature and used additives. Microstructures of the sinters were observed using a scanning electron microscope, and then the images of microstructures were characterized quantitatively by means of the Aphelion program.

A39

CALCULATION OF ACTIVATION ENERGY AND ITS CHANGES DURING SINTERING USING MSC AND WANG & RAJ MODELS

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Apparent sintering activation energy is useful for better understanding of the kinetics of sintering process. Calculation of this activation energy can be made using various approaches, the most commonly used methods are based on constant heating rate dilatometry measurements. These include the Master Sintering Curve model and Wang & Raj model. Both of these models were used to calculate the activation energy and its changes during sintering on the selection of ceramic materials (doped ZrO_2, Al_2O_3, …).

As each model has its limitations, same sets of dilatometry measurements were evaluated using both models. Obtained results were combined to get the dependency of the apparent sintering activation energy on relative density during sintering. Since manual calculation of these models is time consuming, specialized applications were developed and used in order to speed-up the whole measurement-calculation workflow. This enables effective determination of the effects of powder characteristics, green body processing and non-conventional sintering on the changes in the sintering activation energy.

A40

UV LASER SINTERING OF SnO_2 AND ZnO THIN FILMS PRODUCED BY ELECTROPHORETIC DEPOSITION

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Solution based processing and deposition of colloidal dispersions is suitable for economic and material-efficient production of thin films on large areas. Among the various colloidal processing techniques, electrophoretic deposition (EPD) enables the production of homogenous and dense films from dispersions with low particle loading.

In this work tin dioxide (SnO_2) and zinc oxide (ZnO) nanoparticles produced by chemical vapor synthesis are dispersed in aqueous media using ultrasonification. Stable dispersions are achieved by electrostatic and electrosteric stabilization. Colloidal properties of the dispersions are analyzed by dynamic light scattering and pH-dependent zeta potential measurements. Nanoparticulate films of SnO_2, ZnO and a SnO_2-ZnO mixture are deposited on silicon anodes. The influence of UV laser sintering on the microstructure is studied by varying the writing speed and laser power. Nanoparticulate films are characterized by high-resolution scanning electron microscopy and grazing incidence X-ray Diffraction. X-ray Diffraction and SEM results confirm the codeposition of SnO_2 and ZnO nanoparticles and the formation of zinc stannate (Zn_2SnO_4) after UV laser sintering.

A41

SYNTHESIS OF YTTRIUM SILICATE BY SOLID-LIQUID STATE REACTION FOR ENVIRONMENTAL BARRIER COATINGS

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Yttrium silicates are promising materials for improved oxidation and erosion protection due to their high melting point, low volatilization rate, low thermal expansion coefficient, and low oxygen permeation constant. Thus, yttrium silicate has potential applications as a high-temperature ceramic and environmental/thermal barrier coatings for structural Si-based ceramic materials (SiC, Si_3N_4).

However, due to the difficulties in the preparation and sintering of yttrium silicate polycrystals, their bulk properties have not yet been investigated. For that reason, fundamental data regarding high temperature mechanical behavior such as creep, strength, toughness or fatigue resistance are not yet available. This data is invaluable to assess the long-term behavior and lifetime of yttrium silicate based on environmental barrier coatings.

The objective of this work is to develop a feasible procedure to produce dense and single phase of Y_2SiO_5 polycrystals in order to evaluate these aforementioned properties. To this end, firstly we have synthesized Y_2SiO_5 by solid-liquid state reaction using appropriate amounts of SiO_2 and Y_2O_3 powders as precursors and studying the influence of different additives to enhance the synthesis and sintering process by means of the formation of transient liquid phase. Once achieved this aim, we will proceed to evaluate the thermo- mechanical properties.

The phase identification, morphology and microstructure observations of the resulting reaction products were performed using X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively.

A42

SYNTHESIS AND CHARACTERIZATION OF RESORBABLE CALCIUM PHOSPHATE BIOCERAMICS WITH A RATIO OF 0,5≤Ca/P≤1

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Modern regenerative medicine requires resorbable bioactive materials for bone implants. Resorption ability of materials based on calcium phosphates observes at Ca/P<1,67 and increases with a decrease of this ratio.

The aim of our research activity was to obtain and explore powders and ceramics based on calcium phosphates with a ratio of 0.5≤Ca/P≤1. Five powders were investigated in this range with a ratio of Ca/P=0.5; 0.625; 0.75; 0.875 and 1.

Solutions of poly- or/and pyrophosphoric acids were obtained from sodium poly- or/and pyrophosphates by means of the ion exchange. Then, calcium nitrate solution was added to these solutions of the acids. Afterward, ammonia solution was added to solutions containing the following ions: pyrophosphate ion, polyphosphate ion, NO_3^- and Ca^{2+}. In all cases pH-value of these solutions were adjusted to 10. This procedure resulted in formation of amorphous precipitates of hydrated calcium phosphates. Finally, dried and disaggregated powders were pressed into pellets, which were fired to obtain ceramics.

According to thermogravimetric analysis the total weight loss on heating from 20 to 1000 °C for all samples was in the range from 16 to 24%. The weight loss occurred in two stages: the first stage which took place from 40 °C up to 200 °C associated with the removing of adsorbed water and the second stage (from 200 to 600 °C), associated with the removing of crystallization water and decomposition of ammonium nitrate. Molecular formulas of synthesized hydrated phosphates were deduced from these data as $Ca(PO_3)_2 \cdot H_2O$ and $Ca_2P_2O_7 \cdot 2H_2O$.

According to XRD analysis of samples of ceramics, it has been found that for all examples, except the 100% content of pyrophosphate, the ratio between polyphosphate and pyrophosphate pointed before ion exchange was not saved after calcination. Amount of calcium pyrophosphate phase grew in comparison with expected values with increasing Ca/P in the samples. This phenomenon was probably associated with possible thermal hydrolysis of calcium polyphosphate during heat-treatment.

The density of the samples decreases, and the porosity increases with increasing of the content of calcium polyphosphate phase. Despite the low density, these samples demonstrated sufficient strength due to the presence of, calcium polyphosphate forming the melt during sintering. According to SEM micrographs the samples with the high content of polyphosphate had the network of interconnected pores.

The powders of calcium phosphates with a ratio of $0.5 \leq Ca/P \leq 1$, which were obtained by means of the wet precipitation method from solutions of condensed phosphate acids prepared by ion exchange method, had rather uniform composition. Such ceramics consisted of calcium poly- and pyrophosphates are suitable for making bone implants due to high resorption and osteoconductivity.

A43

SIMULTANEOUS THERMAL ANALYSIS AND DILATOMETRIC STUDY OF HAp-LiFePO$_4$ SYSTEM

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Sintered hydroxyapatite bioceramics have been widely studied as a potential material for bone tissue reparation, however, concerning its microstructural and mechanical properties some limits were achieved at the moment. Addition of other materials that could improve functionalities, while preserving inherent advantages of this bioactive ceramics is desirable strategy. In this work, a new idea of addition of lithium iron phosphate as hydroxyapatite sintering aid, provoking liquid phase sintering in the intermediate sintering phase, has been evaluated from the point of view of thermal and dilatometric studies in inert atmosphere, with coupled mass spectroscopy monitoring. Detailed characterization of prepared materials and sintered products is given, confirming the proof of concept. Sintering ability was significantly enhanced and important microstructural features were obtained.

A44

EFFECT OF COLLOIDAL MILLING ON THE PHYSICAL, MECHANICAL AND BIOLOGICAL PROPERTIES OF HYDROXYAPATITE MONOLITHS PREPARED BY ELECTROPHORETIC DEPOSITION

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In this work, the influence of colloidal milling of hydroxyapatite (HA) dispersion on the physical, mechanical and biological properties of monoliths prepared via electrophoretic deposition (EPD) was studied. Dispersions used for EPD were composed from HA ceramic load, monochloroacetic acid as stabilizer, lithium chloride as indifferent electrolyte and 2-propanol. Dispersions were colloidally milled in the range from 0 to 48 hours before the deposition. Applying colloidal milling caused two effects: i) the bimodal distribution of particles was created in comparison to commercially produced powder and ii) average particle size was reduced according to milling times. The reduction of average particle size influenced the deposition rate and deposition mass of EPD process. This two effects and sintering temperature mainly affected microstructure, mechanical and biological properties of HA monoliths. The longer milling times (smaller particle size in dispersions) reduced the porosity contained in the annealed and sintered deposited materials. It was found that sintering temperature can be reduced because the monolith sintered at 1250 °C had the same porosity level as monolith prepared from long time milled dispersion. Moreover, the presence of glass phase in structure appearing at 1250 °C can be eliminated. The hardness and Young´s modulus increased with milling time of dispersion but the fracture toughness remained almost the same. Biological properties of HA monoliths were evaluated by the SBF (Simulating Body Fluid) soaking test and the degradation test in Tris-HCl buffer solution. The main benefit of this study is the possibility to apply colloidal milling instead of high sintering temperature while appropriate mechanical and biological properties are maintained.

A45

SYNTHESIS AND CHARACTERIZATION OF DIVALENT CATION SUBSTITUTED CALCIUM PHOSPHATES

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The main inorganic component of bone is calcium phosphate (CaP) salts. Thereby synthetic CaPs, such as hydroxyapatite (HAp), β-tricalcium phosphate (β-TCP) due to their unique properties – biocompatibility, bioactivity and/or biodegradability, and osteoconductivity, are considered to be promising bone replacement materials. CaPs present themselves as prime materials for modifications and functionalizations, aiming for improvements on physicochemical and biological aspects of novel graft materials. Nowadays interest is turning towards modified synthetic CaPs involving the substitution of various ions, which possess antibacterial properties and are able to act on bone diseases. The incorporation of such ions have influence on the physical, chemical, and physiological properties of the CaPs products and consequently on the mineralization, demineralization, and remineralization of the calcified tissues.

Thus, the main goal of our work is to synthesize with divalent cations (*i.e.*, Mg, Sr and Zn) substituted CaPs through modified wet chemical precipitation method, and to evaluate the effect of the incorporated ions on the synthesis products in bulk and particle level. Various analytical methods, such as X-ray diffraction, Fourier transformation infrared spectroscopy, differential thermal analysis, atomic absorption spectrometry, Brunnauer-Emmett-Teller nitrogen adsorption, and field emission scanning electron microscopy were applied to characterize physicochemical properties of the studied samples.

According to the obtained results, as-synthesized powders containing various amounts (up to 5 wt.%) of (Mg^{2+}), (Sr^{2+}) or (Zn^{2+}) ions were characterized as low-crystalline apatite with the absence of additional phases. It was observed, that Mg, Sr and Zn incorporation reduce markedly the particle size of the apatite powders. Mg, Sr and Zn affect the structure when they partially replace Ca in the apatite crystal sites, which, further, reflects in the decrease of thermal stability of HAp. Overall, (Mg^{2+}), (Sr^{2+}) and (Zn^{2+}) ions containing CaPs with morphological features similar to that of bone mineral have been prepared for bone tissue engineering applications.

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A46

MIXED-ANIONIC CALCIUM PHOSPHATE POWDERS FOR BIORESORBABLE CERAMIC

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The fabrication of nanosized bioceramic particles is of great interest for many applications, including setting bone cements, high strength synthetic porous or non-porous bone grafts and other composites with complex structure. At the moment the idea of usage of materials with high resorption rate is becoming attractive to the researches. This challenge demands the synthesis of calcium phosphates with controllable Ca/P ratio less than 1.5. This approach is necessary to manipulate ceramics properties in a wide range, e.g. resorption rate, which is crucially important to design biomaterial for personalized care. Thus, the usage of condensed phosphates is required. Condensed ammonium phosphates are the most preferring ones while they are able to minimize the influence of precursor co-products on the ceramics structure and composition. However, the information about condensed ammonium phosphate preparation, purity and stability, presented in special reviews, turns out to be controversial, and, in addition, the thermal synthesis of individual condensed phosphates is impossible.

The main goal of this work was the synthesis of amorphous precursors for the biphasic ceramic with pre-determined Ca/P ratio between 0.5 and 1.5. In the course of the work we developed several routs of condensed ammonium phosphate synthesis for subsequent precursor obtainment and, finally, fabricated biphasic ceramics through thermal treatment of the precursors.

In the present work ammonium phosphates were synthesized by various methods with the help of ion-exchange resins and phosphates of nitrogen-containing organic bases. At the next step we obtained amorphous chemically mixed anionic precipitates by co-precipitation. Synthesis of the precursor was performed by pouring solution of calcium nitrate to solution of several condensed ammonium phosphates. The calcination of the precursor led to the formation of biphasic ceramic material characterized by submicron grain size.

As a result, we have produced a number of precursors for biphasic ceramics based on resorbable calcium phosphates with pre-determined Ca/P ratio. Obtained condensed ammonium phosphates were proved to be pure individual substances by XRD. The main aspects of sintering, microstructure and quality of the obtained material and amorphous precursors were analyzed by XRD, scanning electron microscopy, thermal analysis and mechanical tests. Thus, we can regulate composition of the ceramics at the stage of precursor synthesis and create the whole range of materials with variable Ca/P ratio and tunable properties.

A47

INFLUENCE OF DENTAL COMPOSITE CORE MATERIAL ON BIOMECHANICAL PROPERTIES OF PREMOLARS RESTORED WITH A ZIRCONIA FULL CROWN: A FINITE ELEMENT ANALYSIS

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Endodontically treated teeth with severe tooth tissue damage are often restored with a fiber-reinforced polymer composite post (FRC), a polymer-ceramic composite core and a full crown. Many current studies aim at defining what choice of reconstructive material would have the most optimal influence on the remaining tooth tissues, as well as on the longevity of the restoration.

A very useful method for this kind of analysis is the finite element analysis (FEA), which is a mathematical method of calculating the values and the distribution of stress and strain in complex three-dimensional (3D) models. The aim of this study was to examine the influence of dental polymer-ceramic composite core material on biomechanical properties of premolars restored with a zirconia full crown and a FRC post.

On a 3D tooth model created in SolidWorks 2014 software (Dassault Systemes SolidWorks Corp, USA), based on Computed Tomography (CT) scans of an extracted maxillary second premolar, the influence of two core materials (Gradia posterior, GC, Japan and Filtek Z250, 3M, ESPE, EUA) was tested.

The results showed that in the case where the core material with lower modulus of elasticity was used (Gradia posterior), lower von Mises stresses were distributed to the core itself, but they were found to be higher on the post and the post cement. This could indicate that a core material of much lower modulus of elasticity than the zirconia crown, transfers the stresses to the tissues below it, while the stiffer core material (Filtek Z250 in this study) might be able to receive a part of the generated stresses during mastication. The use of a stiffer core material could be favourable for the longevity of tooth tissues and the restoration.

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A48

ELASTIC PROPERTIES OF CELLULAR ALUMINA CERAMICS PREPARED BY BIOLOGICAL FOAMING

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Cellular alumina ceramics have been prepared by biological foaming method of alumina suspensions using baker's yeast Saccharomyces cerevisiae. This organism metabolizes sugars (glucose, fructose or sucrose) into ethanol and carbon dioxide (foaming agent). Starch was applied as a foam stiffener and pore-former and sugar as a fuel for fermentation. Samples were dried (8 h at 80 °C and 2 h at 105 °C) and subsequently fired at 1570 °C (heating rate 2 °C/min, dwell time 2 h). Disk-shaped samples (prepared with various process parameters – various starch and sugar types and contents in suspensions, various amounts of additional ethanol contents, several process variants) with different porosities (50–87 %) and different pore sizes (380–2150 μm) were prepared and their elastic properties were measured via the impulse excitation technique (according to ASTM E 1876-99 and ASTM C 1259-01 standards) using flexural and anti-flexural (torsional) vibrations in order to extract not only Young's moduli, but also the other elastic constants, including Poisson ratios. Elastic properties of alumina foams are in the following ranges: Young's modulus 2.0–43.4 GPa, shear modulus 1.0–17.5 GPa, bulk modulus 0.7–28.7 GPa, Poisson ratio 0.045–0.416 with an average value close to 0.2. While the absolute errors of Poisson ratios are too high to allow definitive conclusions concerning their agreement with one or the other model prediction, those of the elastic moduli are low enough to allow the conclusion that the elastic moduli of the most porous alumina foams closely follow the Gibson-Ashby power law, while the less porous samples are somewhere between the power-law and exponential prediction.

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A49

SYNTHESIS OF MONETITE ($CaHPO_4$) BY MECHANOCHEMICAL TREATMENT OF BRUSHITE ($CaHPO_4·2H_2O$)

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Synthesis of monetite ($CaHPO_4$) by means of mechanochemical treatment of brushite ($CaHPO_4·2H_2O$) was studied. Start sample (Brushite) was obtained by precipitation method at room temperature. Particle size of brushite was reduced using vibromilling. The powders were analyzed by X-ray powder diffraction (XRPD). Microstructure and morphology was determined by means of scanning electron microscopy (SEM). Brunauer-Emmett-Teller (BET) method was used for examining specific surface area of obtained powders. It was found that five minutes of milling induces brushite-monetite phase transformation. This type of synthesis is cost-effective compared to the other used methods for synthesis of monetite.

A50

DEVELOPMENT OF INNOVATIVE 3D POROUS TiO_2 CERAMIC SCAFFOLDS FOR ORTHOPAEDIC APPLICATIONS

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The development of scaffolds for replacement of injured and diseased hard tissues such as bones is highly desired in orthopaedic surgery. The porous structure of scaffold provide necessary framework for the bone cells to grow into the pores and integrate with host tissue, known as osteointegration. Thus porosity and pore size of biomaterial scaffolds play a critical role and also has great impact on mechanical properties. Particular attention has attracted TiO_2 ceramic scaffolds due to its excellent mechanical properties compared to other ceramic materials, biocompatibility and good osteoconductivity.

TiO_2 scaffolds were produced via polymer foam replica method. Commercially available anatase powder, polyvinyl alcohol solution, ethylene glycol, ammonia solution and deionised water were used as raw materials for ceramic slurry preparation. Homogenisation of the slurry was conducted by stirring for different period of time. Particle size distribution, viscosity and pH of titania slurry were monitored during stirring. Cylindrical polyurethane foams with fully interconnected pore structure serves as a sacrificial template for the ceramic coating. After drying, the polymer was slowly burned out and scaffolds were sintered in air at different temperatures (>1300°C) and holding times.

Increasing sintering temperature to 1300 °C complete phase transition from anatase to rutile crystalline modification takes place and stable rutile modification has been obtained. By optimising thermal treatment regimens grain growth and collapse of struts can be controlled that may result in higher compressive strength. Effect of slurry parameters (pH, viscosity, particle size, solid content etc.) on characteristics of produced titania ceramic scaffolds (porosity, pore size, mechanical strength) were evaluated.

Using replica method porous TiO_2 ceramic with porosity >85% and pore size >100 μm were obtained. Scaffolds showed fully open and interconnected pore structure. Obtained pore size and porosity is wide enough to ensure cell migration throughout scaffold structure and ensure vascularisation.

Acknowledgements: Support for this work was provided by the National Research Programme “Multifunctional Materials and composites, photonics and nanotechnology (IMIS^2)” Project No. 4 “Nanomaterials and nanotechnologies for medical applications”.

A51

CALCIA PARTIALLY STABILIZED ZrO_2 BIOCERAMICS NANOCRYSTALS

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Zirconia is well known ceramic material using in hard tissue engineering. It is mainly due to its bioinert properties and transformation toughening mechanisms acting in their microstructure, which can impart the components made out of them very interesting mechanical properties. The Y_2O_3 partially stabilized zirconia (Y-PSZ), together with Ce-ZrO_2 and Mg-ZrO_2, is the most typical and the most often studied for many decades. But nowadays, thanks to the new technologies, technics and available materials, we can substitute Y^{3+} (Ce^{4+}, Mg^{2+}) ions by biogenous Ca^{2+} ions and we can achieve more biologically active material while maintaining similar mechanical properties.

The aim of this study is preparation of a nanocrystalline partially CaO-stabilized ZrO_2 (Ca-PSZ) and to find the proper molar concentration of Ca^{2+} ions and solid-state temperature for the highest content of tetragonal phase in Ca-PSZ system using the sol-gel method.

By varying the calcium molar concentration (2.5, 5, 11, 17, 18.5 and 50 mol%) and solid-state reaction temperature (550–1200 °C) the percentage amount of crystalline phases (monoclinic, cubic, and tetragonal) was observed using the XRD analysis. Morphology and the chemical composition of the product were studied by the SEM/EDX analysis and the surface area was determined by the BET analysis. Cytotoxicity of materials was tested *in vitro* using direct contact assay test according to EN ISO 10993-5 specifications.

A52

OUZO EFFECT – AS THE NEW SIMPLE NANOEMULSION METHOD FOR SYNTHESIS OF STRONTIUM HYDROXYAPATITE NANOSPHERES

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Nanoemulsion technique was applied for synthesis of carbonated strontium hydroxyapatite (CSrHAp) at room temperature. X-ray powder diffraction analysis accompanied with Rietveld refinement reviled that synthesized powder were single-phase hydroxyapatite. Fourier transform infrared (FTIR) spectroscopy showed that the CSrHAp was A-type substitution. The carbonate amount substituting the hydroxide group in the synthesized apatite was estimated, from the corresponding CO_2 weight loss in the range 600–1100 °C. According to this empirical formula of as synthesized CHAp is $Sr_{10}(PO_4)_6(OH)_{0.60}(CO_3)_{0.70}$. These results were confirmed by the Rietveld refinement analysis. Using scanning electron microscopy analysis it was found that the synthesized CSrHAp particles were spherical in shape and that their sizes were in the nanometer range. Nanoemulsion strategy procedure provides a simple pathway to obtain single-phase CSrHAp.

A53

CALCIUM PHOSPHATE COATINGS DEPOSITED ON Ti SUBSTRATE USING ELECTROCHEMICALLY ASSISTED DEPOSITION

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Due to their characteristics, such as biocompatibility, corrosion resistance, titanium and its alloys have been widely used as implant materials. Despite its well-known ability to bond directly to the bone, Ti implants are affected by the unfavorable fixation. To overcome this problem, different coatings of a large variety of elements/materials can be used to improve the surface chemistry. Calcium phosphates (CaP) compounds can indeed improve the metallic implants osteoconductivity. Electrochemical deposition seems to be a good alternative because can be applied from aqueous solution, at room temperature, with a high control on deposition parameters and is cost effective.

In this study, CaP electrodeposition was carried out in a standard three electrode cell set-up in which an Ag/AgCl was used as a reference electrode, a platinum electrode as a counter electrode and the sample (Ti substrate) as a working electrode. The

electrolyte was prepared by dissolving $Ca(NO_3)_2$ and $NH_4H_2PO_4$, purchased from Sigma Aldrich, in ultrapure water.

A PARSTAT 4000 Potentiostat/Galvanostat (Princeton Applied Research, USA) operating in potentiodynamic mode was employed to a cathode potential of -1.4 V vs E_{ref} for 2 h, at different temperatures. Following electrodeposition, the samples were washed with ultrapure water and dried.

The obtained CaP coatings by electrodeposition technique were investigated. Surface morphology and chemical composition has been investigated by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The roughness and contact angle were also analyzed. X-Ray Diffraction and FTIR spectrum confirmed that CaP has been deposited on titanium substrate by electrochemical deposition. The linear polarization method was used to obtain the polarization resistance (R_p), corrosion current densities (i_{corr}), corrosion rates (CR) and Tafel slopes. The electrochemical parameters registered more favorable values for the coated samples.

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A54

SYNTHESIS, CHARACTERISATION AND ELECTROCHEMICAL PROPERTIES OF GRAPHITE OXIDE/VANADATE NANOWIRE COMPOSITES

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Keywords: nanocomposite, supercapacitor, specific capacity, energy density, charge-discharge cycle

There is a growing interest in composites composed of vanadium-oxides and carbon nanostructures due to their promising electrochemical properties. These composite materials are potential candidates for application as electrode materials in Li-ion batteries and supercapacitors. The latter have thousand times higher specific capacity than conventional electrolytic capacitors, while its performance deteriorates only slightly during hundreds of charge-discharge cycles.

In our study vanadate nanowire-graphite oxide composites were synthesized via the hydrothermal route. The influence of adding graphite-oxide to the composite on the electrochemical performances was investigated. The morphology, structure and interaction between components were studied by transmission and scanning electron microscopy (TEM, SEM), Raman spectroscopy, X-ray diffractometry (XRD) and energy dispersive X-ray spectrometry (EDS). The electrochemical properties were examined by the galvanostatic charge-discharge method, cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The specific capacity, energy and power density of the capacitor constructed from the vanadate/graphite-oxide composites were determined and their dependance on the amount of added graphite-oxide was discussed.

Results revealed that a hybrid type supercapacitor was formed. The specific capacities determined from cyclic voltammetry and galvanostatic charge-discharge method were in good agreement and were found to increase monotonously between 210 and 270 F/g with increasing amount of graphite-oxide. The energy density of the capacitor changed from 19 to 25 Wh/kg with the graphite-oxide content, however, power density turned out to be independent of the graphite-oxide amount at around 410 W/kg. These values are in good agreement with literature data. On the other hand, the mechanism responsible for the considerable deterioration of specific capacity during repeated charge-discharge cycles is yet to be resolved.

A55

ELECTRICAL EFFICIENCY OF SOFCs WITH 8YSZ AND 10Sc1CeSZ ELECTROLYTES

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Solid oxide fuel cells (SOFCs) are energy conversion devices that directly convert chemical energy into electrical energy with higher conversion efficiencies when compared to heat engines. Electrical efficiencies up to 65%, thermal efficiencies of ~30%, zero greenhouse gas emissions, low noise operation and variable fuel capability have made SOFCs an attractive technology for power and energy markets. While these advantages have made SOFCs one of the most sought after technologies, the high operation temperature (500–1000 °C) creates technical challenges that narrow their scope of use and creates economic barriers to widespread implementation. Developing SOFC systems that operate in lower temperature regions improves system stability, lowers materials degradation and extends the current reach of SOFC commercialization.

The standard SOFC electrolyte, zirconia-ZrO_2 doped with 8mol% yttria-Y_2O_3 (8YSZ) exhibits a high oxygen ion conductivity and exhibits stable behavior in both oxidizing and reducing environments. Despite YSZ being the most developed, commonly used electrolyte for SOFCs, a variety of oxide dopants with high solubility's in ZrO_2 can be used. Sc_2O_3 stabilized ZrO_2 electrolytes (ScSZ) are ideal candidates for SOFC operation in intermediate temperature ranges due to their high ionic conductivity at temperatures between 500–800 °C when compared to YSZ conductivities in the same range.

The focus of this work is to examine the performance of a ScSZ-based SOFC to measure its impact on cell performance and to determine its suitability as an alternative electrolyte material.

In this paper the cell performances of two different commercial cells tested under the same conditions are presented. Ni-YSZ anode supported cells with 8YSZ electrolyte and LSM-YSZ composite cathodes and ScSZ- electrolyte supported cells with Ni-ScSZ anode and LSCF-GDC cathodes were supplied by Ningbo Institute of Materials Technology and Engineering and used in this study. The cells were tested at 800 °C with 5 vol.% H_2-Ar as fuel and O_2 from ambient air as the oxidant. Despite of the thicker electrolyte, type 1 SOFC represent higher electrical performers than type 2 SOFC (Fig. 1).

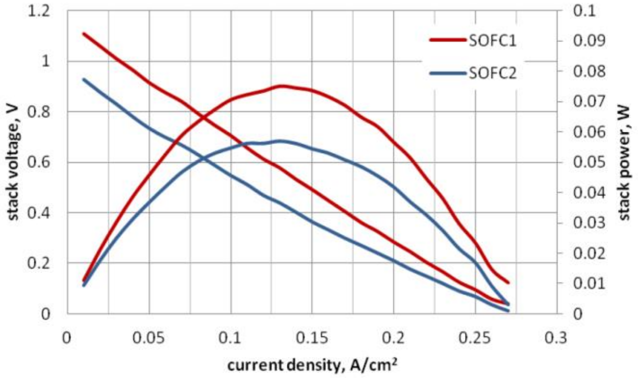

Figure 1. Electrical efficiency of different SOFC types

A56

SYNTHESIS AND CHARACTERIZATION OF Ag DOPED CERIA NANOPOWDERS

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Nanopowdered solid solution $Ce_{1-x}Ag_xO_{2-\delta}$ samples ($0 \leq x \leq 0.1$) were synthesized by a self-propagating room temperature synthesis (SPRT). XRD and Raman spectroscopy at room temperature were used to study the vibrational properties of these materials as well as the Ag solubility in ceria lattice. Results show that all obtained powders are solid solutions with a fluorite-type crystal structure and all powders were nanometric in size. The average size of $Ce_{1-x}Al_xO_{2-\delta}$ particles lies about 5 nm. The redux property of metal ions in ceria matrix render catalytic character to solid solution ($Ce_{1-x}Ag_xO_{2-\delta}$). The presence of Ag ions in $Ce_{1-x}Ag_xO_{2-\delta}$ causes different redux properties compared to the corresponding AgO or Ag_2O because of the influence of surrounding Ce ions in these compounds. In fact they are better catalysts than metal particles for the following reasons: although the adsorption probability of carbon monoxide over metal ion is about same as that on metal atom on a metal particle, in a nano metal particle only atoms on the surface of the particle are available for adsorption. Therefore, if metal particle of 5 nm size are present, than only one-fifth of the total metal atoms are available for absorption. On the contrary, when metal is incorporated in ceria matrix almost all the metal ions are available for adsorption as they are present mainly on the surfaces of $Ce_{1-x}Ag_xO_{2-\delta}$ powder. This results in five times more CO adsorption for the same amount of metal particles disperse over ceria. In addition, electronic interaction between Me ion and ceria enhaces the catalytic activity.

A57

SYNTHESIS AND CHARACTERIZATION OF PLATINUM NANOPARTICLES WITH CONTROLLED SIZE FOR HETEROGEN CATALYTIC PROCESSES

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In industrial applications, synthesis of monodisperse catalyst particles is problematic, therefore the effect rising from the size in catalytic processes remain unexploited. In this work, platinum nanoparticles with narrow size distribution were fabricated with a particular wet chemistry method from various platinum salt precursors, in elevated temperature, under argon atmosphere. In this process, 1–12 nm platinum nanoparticles were produced with controlled size.

The particles were embedded with low power sonication into 3-dimensional mesoporous silica supports. The free standing and supported catalysts were characterized by scanning- and transmission electron microscopy, X-ray diffraction, Raman spectroscopy and BET methods, and also with sum frequency generation spectroscopy for investigation under reaction conditions. These catalysts are tested in heterogenous catalytic reactions such as alcohol oxidation.

A58

SYNTHESIS, OPTICAL AND MAGNETIC PROPERTIES STUDIES OF MULTYFERROIC $BiFeO_3$

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Nanosized bismuth ferrite powder has a potential application in the production of lead free piezoelectric materials for actuators as well as magnetoelectric sensors. The simple, low-costing and energy-saving hydrothermal method has advantages over the conventional methods. $BiFeO_3$ powders were made using $Bi(NO_3)_3{\cdot}5H_2O$ and $Fe(NO_3)_3$ $\cdot 9H_2O$ as starting material and 8 M KOH as mineralizer. The particle size and morphology were analyzed using scanning electron microscopy (SEM). The phase composition of obtained samples was determined by X-ray diffraction (XRD) analysis. It revealed that synthesized material crystallize in space group R3c with cell parameters $a = b = 5.5780(10)$ Å and $c = 13,863(3)$ Å. IR and Raman spectroscopy have been performed on the synthesized bismuth ferrite (BFO) powders in order to confirm the formation of pure and well-crystallized BFO nanocrystallites. ^{57}Fe Mössbauer spectroscopy was performed in order to provide information on Fe cation arrangement in the $BiFeO_3$ phase. The magnetic and optical properties of properties of BFO samples were characterized by SQUID magnetometry, and ultraviolet–visible spectroscopy. Temperature dependence of magnetization shows antiferromagnetic-paramagnetic phase transition at $T_N = 220$ K, while below this temperature weak ferromagnetic ordering is detected.

A59

ZnO BASED FILMS WITH SENSING PROPERTIES

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ZnO and Zn-Sn-O films are playing an increasingly role in many applications as transparent electrode of solar cell, flat panel devices, infrared (IR) reflectors, organic light emitting diode (OLED), thin film transistor-liquid crystal display (TFT-LCD) and gas sensors. Sol-gel processes give the ability to produce ZnO and Zn-Sn-O films in a simple, low cost and highly controlled way. It is well known that the reagents, solvent,

concentration of precursors solution, chelating additives, thermal history are several factors to control morphology of the films.

In the present work we propose a systematic study of the influence of the chemical and physical parameters involved in the obtaining ZnO and Zn-Sn-O films by sol-gel dip coating method.

Soluble sols of ZnO and Zn-Sn-O (with atomic ratio of Zn:Sn=1:1 and Zn:Sn=2:1) were obtained starting from zinc acetate dehydrate and Sn(II) 2-ethylhexanoate (for Zn-Sn-O) in ethylic absolute alcohol (as solvent) and triethanolamine (as chelating agent). Multi-layers films were deposed on glass, ITO/glass and Si/SiO_2 substrates. The structural and morphological and optical characteristics of the consolidated films were investigated by X-Ray Diffraction and Scanning Electron Microscopy. The structure, morphology and optical properties of the obtained films depend on the composition, number of deposition and the thermal treatment temperature. Preliminary investigations of the resulted films as gas sensors were also realized, underlying promising properties for CO and propane detection.

A60

SYNTHESIS AND CHARACTERIZATION OF MESOPOROUS AND SUPERPARAMAGNETIC BYLAYERED-SHELL AROUND SILICA PARTICLES

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Mesoporous (SiO_2) and superparamagnetic (Fe_3O_4) bilayered-shell around monodispersed silica core particles was prepared by wet-chemical synthesis process. Ferrite nanoshell was synthesized by assembling of oppositely charged ferrite (Fe_3O_4) nanoparticles on the surface of the monodispersed silica core particles (having average size of ~0.5 μm and prepared by hydrolysis and condensation of tetraethylortosilicate). The obtained Fe_3O_4 layer was superparamagnetic and had very weakly developed pore structure. The second layer on the previously obtained particles with silica core and Fe_3O_4 shell was formed by deposition of silica nanoparticles from a silicate solution. To allow electrostatic assembling of the silica nanoparticles and formation of external silica layer around the silica/Fe_3O_4 particles, the latter were functionalized with poly(diallyldimethylammonium chloride) (PDDA). Obtained silica layer had well developed pore structure with average pore size of 10 nm. The obtained composite particles with two functional layers can be used in bioengineering for immobilization of

enzymes inside external mesoporous silica layer while internal superparamagnetic ferrite layer would allow magnetic separation from reaction mixture.

A61

CHARACTERIZATION OF ALUMINUM NITRIDE NANOPARTICLES SYNTHESIZED BY CHEMICAL VAPOR SYNTHESIS

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Phosphor converted light-emitting diodes (pc-LEDs), based on III-V semiconductors are widely commercially available. Phosphors are needed to convert the blue light, from the most often used InGaN LEDs, to white light; however, the phosphor synthesis can still be improved. Increasing the reactivity of the starting materials, such as AlN in case of aluminum based phosphors, might be one promising way to accomplish this. The activation energy required for the phosphor synthesis is expected to decrease with decreasing particle size and narrowing particle size distribution of the starting materials. An improved synthesis using AlN nanoparticles might also affect the phase purity and conversion efficiency of the resulting phosphors in a positive way. Chemical Vapor Synthesis (CVS) is one of the methods to produce such nanoparticles by facilitating and controlling the gas phase reaction between triethylaluminum (TEAl) and ammonia. The purity of the nanoparticles is analyzed by EDS, while powder X-ray diffraction is used for the determination of the crystal structure. The local structure is investigated by XAFS spectroscopy and FEFF9 simulations. Moreover, the degree of agglomeration of the nanoparticles determined from low temperature nitrogen adsorption and powder X-ray diffraction further complements the characterization.

A62

DIELECTRIC PROPERTIES OF BARIUM TITANATE BASED THIN FILMS

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The development of microelectronics is in close connection with the development of new ceramic materials especially considering miniaturization and multifunctionality of microelectronic components. In recent years, barium titanate based thin films, as well as multilayer multiferroic thin films, have attracted the close attention. Their potential

application includes the field of sensors, transducers, actuators, memory devices etc. This research was based on developing the method for measuring dielectric properties of barium titanate based thin films and investigation of their dielectric properties. Processing of barium titanate based films consisted of making sols via sol-gel technique and their deposition on substrate by spin coating method. Thin films were deposited on silicon substrates with conductive layer of platinum ($Si/SiO_2/TiO_2/Pt$) and sintered in tube furnace at 750 and 900 °C. Dielectric properties of samples were measured in temperature range from 50 to 200 °C, at frequencies from 20 Hz up to 1 MHz. The obtained results were analysed and connected with structural characteristics of the thin barium titanate based films determined by XRD analysis, Raman spectroscopy, SEM and AFM. Structural characterization has confirmed expected phase composition and nanostructured grains. The value of dielectric constant for thin films (from 50 up to 600) was dependent of the sintering temperature, grain size and phase composition of films.

CERAMIC COMPOSITES

C1

ICE-TEMPLATING OF CERAMICS IN INDUSTRIAL SCALE

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Ice-templating, also known as freeze-casting, is a relatively simple, inexpensive, and very versatile technique to fabricate bulk porous scaffolds. The basic idea of ice-templating is to obtain porosity that is replica of ice crystals, by freezing suspension and subsequently removing the ice crystals by sublimation of the solvent (most commonly water is used as a solvent). The solid phase in the suspension can be of practically any nature, from ceramic to metal particles or polymer, but ceramic particles are the most used. Porous ceramic scaffolds can be obtained by controlled freezing of ceramic slurries, which is followed by sublimation of water and densification (sintering). In ice-templating, the particles in ceramic slurry are ejected from the moving solidification front and pile up between growing ice crystals creating multilayer porous ceramic structures with well-defined architecture. The process is based on the very low solubility of the second phase in the solvent, which ensures its segregation during solidification. Typical process of ice-templating is shown in the Fig. 1. [1].

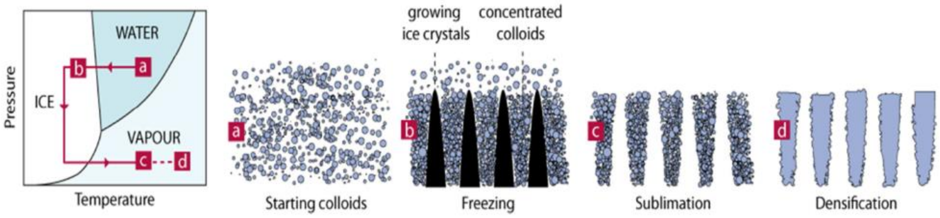

Figure 1. Schematic diagram of the ice-templating method [1]

This research is focused on the problem of scaling up the ceramic samples prepared by ice-templating method for hybrid ceramic composites. The main problem with large samples is achieving of controlled ice crystals growth throughout the whole sample volume. This phenomenon is caused by loss of sufficient temperature gradient in the ceramic slurry as the solidification front moves away from the cooling plate. Ceramic suspension containing alumina in water is after ice-templating partially sintered to obtain laminated ceramic structure.

Proposed solution for improvement of controlled ice crystals growth is to design and manufacture special vessels for ice-tempalting with different heat transfer in various directions. Our preliminary results demonstrate that shape of vessel for ice-templating has a significant impact on the appearance and direction of growth of ice lamellae. New design of vessel for homogeneous lamellae formation in large scale is proposed and will be verified.

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C2

ALUMINA MATRIX CERAMIC NIKEL COMPOSITES FORMED BY CENTRIFUGAL SLIP CASTING

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The paper is focused on the possibility of the fabricating the alumina matrix ceramic-nickel composites with gradient concentration of metal particles. As the method of composite fabrication, centrifugal slip casting was chosen. This method allows to fabricate the graded distribution of nickel particles in the hollow cylinder composites. The horizontal rotation axis was applied.

In the experiments the following powders were used: α-Al_2O_3 TM-DAR from Taimei Chemicals (Japan) of an average particle size 133 nm and density 3.96 g/cm^3, Ni powder from Sigma-Aldrich of an average particle size 3 µm and density 8.9 g/cm^3. Aqueous based slurries (with 50 vol.% content of solid phase) containing alumina and nickel powder (10 vol.%) were tested. A composition of deflocculates i.e. diammonium citrate (p.a., Aldrich) and citric acid (p.a., POCH Gliwice) were used. Final sintering was conducted on all the specimens at 1400 °C in reducing atmosphere (N_2/H_2).

The obtained samples were characterized by X-ray diffraction studies (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDS). Quantitative description of the microstructure of the composites was made on the basis of SEM images using computer image analysis.

The macroscopic as well SEM observations of composites confirmed the gradient concentration of Ni particles in the composites material. Application of the centrifugal slip casting method allows for the graded distribution of metal particles in the samples.

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C3

DEVELOPMENT OF CORE-SHELL STRUCTURED METAL OXIDE POWDERS TO BE USED AS LITHIUM ION BATTERY CATHODE MATERIALS

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Lithium Manganese Oxide ($LiMn_2O_4$, LMO) (4.1 V, 148 mAh/g) and Lithium Nickel Manganese Oxide ($Li_2NiMn_3O_8$, LMNO) (4.7 V, 148 mAh/g) are high voltage

cathode materials aiming at high energy density battery applications like electric vehicles [1]. However, their applicability in actual devices is limited due to their low electrochemical stability with most of the commercial electrolytes at high potentials. The purpose of this research is to introduce a core-shell structure for the battery cathode using LM(N)O as the core and TiO_2 as the shell material to prevent the electrolyte decomposition on the surface of the LM(N)O particles. A kinetics controlled coating technique [2] mainly based on hydrolysis and condensation reactions of titanium butoxide (TBOT) was used to initiate heterogenous nucleation and growth of TiO_2 on commercial LM(N)O particles in an ethanol based environment. The effect of different synthesis parameters; such as the LM(N)O amount, the TBOT amount and the reaction time on the shell formation was examined using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). SEM images revealed changes in surface morphology when compared to the bare, commercial LMO powders. Titanium and oxygen signals revealed by EDS also pointed out possible formation of a thin TiO_2 layer on LMO particles. However, powder agglomeration was observed via SEM after coating process. To avoid agglomeration, studies were focused on increasing the zeta-potential value of the core particles by surface functionalization. Citric acid functionalized [3] LMNO particles revealed stable dispersions at a pH of 10 with a zeta-potential value of about -54 mV in aqueous medium while the non-functionalized LMNO particles revealed about -16 mV at the same pH. Effect of using the citric acid functionalized starting powders in the ethanol based actual shell synthesis medium is further to be investigated.

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C4

CERAMIC COMPOSITES BASED ON TiO_2 NANOTUBES FOR APPLICATION IN SOLAR CELLS

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Application of ceramic materials is restricted due to their low fracture toughness. One of the ways to increase the mechanical properties of ceramics is insertion of metallic ductile phase, creating ceramic/metal composites. One group of such materials are functional gradient materials (FGM) which some properties (such as hardness) change as a function of distance from the surface of the material.

TiO_2 is known as material with excellent chemical and photochemical stability was found that anatase is photocatalytically more active than rutile due to its large surface area. Ordered TiO_2 nanostructures, including nanoparticles, nanotubes, and nanorods have garnered much research for their use in solar energy applications. In hybrid solar cells, the titania nanostructures accept electrons from photoexcited dye molecules or polymers adsorbed to the surface and direct the electrons into an external circuit. Hybrid solar cells with titania nanotubes have several advantages over other nanostructures and planar solar cells. The high surface area of nanotubes, compared to nanorods or flat surfaces, allows for more adsorption by electron donors such as molecular dyes and polymers, thus increasing solar photon absorption and charge collection (Roy 2011) [1].

In this research we will study different ways of modification of TiO_2 nanotubes with the aim to increase solar absorbance and efficiency for applications in solar cells. The nanotubes will be synthesized by electrochemical oxidation of Ti-foil. TiO_2 nanotube arrays on substrates provide a superior platform for building up ordered heterojunctions for enhanced charge separation and electron transportation. The parameters of the TiO_2 nanotubes will be finely tuned, allowing the optimization of device fabrication for different types of solar cells, such as DSSCs, hybrid solar cells.To obtained desired properties of the nanotubes, the nanotubes will be decorated with silver nanoparticles, $BaTiO_3$ nanoparticles and dyes. For the structural characterization of all the titanate nanostructures we will used conventional and analytical transmission electron microscopy (TEM) techniques, scanning electron microscopy, XRD and Raman spectroscopy, and UV-Vis spectroscopy for measuring solar absorbance.

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C5

MECHANICAL AND THERMAL PROPERTIES OF SILICON NITRIDE-TITANIUM NITRIDE PARTICULATE COMPOSITES

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The influence of titanium nitride addition on mechanical and thermal properties of silicon nitride based composites was checked. Samples with varying amounts of fine or coarse titanium nitride particles were hot-pressed out of commercial powders. The microstructure and phase composition of the composites were analyzed using SEM and XRD methods. Fracture toughness, bending strength and hardness of materials were

examined. Thermal diffusivity and thermal conductivity of composites was measured using flash analysis method. Thermal expansion of samples was examined.

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C6

ALUMINIUM OXYNITRIDE – HEXAGONAL BORON NITRIDE COMPOSITES WITH ANISOTROPIC PROPERTIES

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Ceramic materials based on polycrystalline nitrides, considering their high thermal conductivity, good dielectric properties, outstanding chemical stability, high melting point and excellent corrosion resistance to molten-metal, have great potential applications in various fields of high-temperature industries like power industry, electrical applications, metallurgy or even molding industry. Due to the high bending strength, AlON is hardly machinable using conventional devices and it is difficult to achieve complex-shaped components. Growing industry's expectations create a necessity to modify a phase composition, microstructure and to tailor selected properties. Introduction of dispersed h-BN as a second phase with layered structure, distinguished by a good machinability and thermal properties could be a solution to this problem. On the other hand, covalent bonding of nitrides and resulting sintering difficulties should be concerned. Thermal diffusivity and thermal conductivity are one of the critical parameters, which decide about nitrides materials' applications. That is why extensively investigated thermal properties of AlON-h-BN composites are a major component of this work.

The aim of the present work was to obtain aluminium oxynitride-hexagonal boron nitride composites with anisotropic properties using SHS-derived powders. Mixtures of aluminium, aluminium oxide and amorphous boron powders (with different weight proportions between the substrates) were combusted in nitrogen atmosphere. After crushing and grounding, the SHS prepared powders were hot-pressed in nitrogen atmosphere to obtain dense composite materials, which were composed mainly of aluminium oxynitride AlON and hexagonal boron nitride h-BN. X-ray analysis showed very strong orientation of platelets h-BN grains with flat surface corresponding to the (0001) plane of the h-BN structure. The h-BN grains were orientated in-plane, perpendicular to hot-pressing direction. The composites demonstrated strong anisotropy of such thermal properties; thermal conductivity was few times higher in the direction perpendicular to hot-pressing force and parallel to the longer diameter of h-BN grains.

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C7

HIGH-TEMPERATURE OXIDATION CHARACTER OF NiAl-ZrB_2 COMPOSITE MATERIALS

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Keywords: intermetallic, zirconium boride, composite, high-temperature oxidation, oxide, tribosynthesis.

The steady development of the current science and engineering requires both further improvement of traditional materials and design of novel ones capable to operate under the conditions of high temperatures and resist to the destructive action of aggressive forces. Today the development of composite materials based on the intermetallic NiAl, which exhibits high operation characteristics, is very important. Such materials are of particular interest in view of their application as high-temperature-oxidation-resistant protective coatings on parts of gas turbine engines.

Before, the authors have been shown that tribological characteristics of intermetallics under high-temperature friction conditions may be markedly improved owing to introduction of refractory borides into their composition. Refractory boride inclusions strengthen the intermetallic matrix, which results in increasing the wear resistance of material. Moreover, oxides formed on the surfaces of boride grains during tribosynthesis are suggested to act as a solid lubricant, which protects the contact surfaces against setting and so promotes a decrease in the wear of the tribopair.

In this work the effect of the testing temperature on the mechanism of high-temperature oxidation has been investigated for composite materials of the NiAl-ZrB_2 system. The composites were oxidized at 500, 800 and 1000 °C for 1 and 90 min in air. The composition and thickness of oxide films formed on composite surface were examined using the Auger analysis. It was established that all of the investigated composites oxidized selectively. Herein the intensity of formation and chemical composition of the oxide phases formed on the diboride grains and the intermetallic matrix differ for different composites and depend on the temperature of testing.

The work demonstrates that the development of novel composite materials of the NiAl-ZrB_2 system is a promising trend in the field of tribological material science.

C8

AGEING OF CERAMICS IN THE ZrO_2–Y_2O_3–CeO_2–CoO–Al_2O_3 SYSTEM

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Y-TZP ceramics are characterized by high chemical resistance and bioinertness and are perspective for orthopedic and dental restorative applications. However it undergoes low temperature ageing in humid environments. The degradation is caused by the spontaneous tetragonal → monoclinic (t→m) transformation of ZrO_2 grains, which is accompanied by the volume increasing. It leads to the formation of micro-cracks and the composite strength decreasing. Therefore, the researches of bioinert material based on ZrO_2 are directed to improving the phase stability of zirconia in an aggressive environment of living organism.

It is known that CeO_2 and Al_2O_3 additions into Y-TZP ceramics increase resistance to low temperature aging in the living organism environment. The micro additive of CoO provides colorations of ceramics.

The aim of this investigation is to evaluate the ageing resistance and mechanical properties of ceramics in the ZrO_2-Y_2O_3-CeO_2-CoO-Al_2O_3 system with different amounts of Al_2O_3.

The starting nanocrystalline powders with composition (mol.%): $(96{,}2\text{-}x)ZrO_2\text{-}2.8Y_2O_3\text{-}0.7CeO_2\text{-}0.3CoO\text{-}xAl_2O_3$, where x = 1, 2.5 and 10, were produced by hydrothermal synthesis in an alkaline medium (pH > 9). Specimens were formed by cold uniaxial pressing and sintered at 1300 °C in air, for 1.5 hours.

Accelerated ageing *in vitro* in autoclave was used to determine the ageing resistance of specimens. It is accepted that ageing of samples under hydrothermal conditions (134 °C, 7 hours) is equivalent to 20 years of bioimplants location in human body [1]. The bending strength of produced ceramics was determined by the 3-point bending method and varied from 400 to 550 MPa.

Before hydrothermal degradation it was identified only t-ZrO_2 according to the XRD results. After hydrothermal degradation the traces of monoclinic phase of ZrO_2 (m-ZrO_2) were detected. Since the amount of m-ZrO_2 after ageing was less than 5 % the investigated ceramic composite has good resistance to the aging in the humid environments.

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C9

THERMO-ELECTROMOTIVE FORCE OF MULTICOMPONENT COMPOSITES BASED ON THE REFRACTORY OXYGEN-FREE COMPOUNDS

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The possibility of use of thermoelectric transducers based on the refractory oxygen-free compounds, which have linear dependence of the thermo-electromotive force from temperature in the temperature range from 900 °C to 1750 °C, is investigated in the work for the first time. At maximum temperature thermo-electromotive force coefficient of these transducers is 10 times higher than coefficient of high-temperature metallic thermocouples. Thus, ceramic thermocouples are resistant to various gas environments (oxidation, renewable, neutral), molten metals, salts, slags.

It was found, that type of material of additive and morphology of composite microstructure are essential for a value of functional parameter. For materials with approximately equal thermo-electromotive force coefficient (TiB_2, TaN), which are used as additives of the negative branch, in contact with composites of positive branch (fillers: soot and carbon fiber), thermo-electromotive force value is determined by microstructure of positive branch morphology: so when carbon fiber is used as filter, thermo-electromotive force of functional element is 3 times higher. If fillers with thermo-electromotive force coefficients which differ by about 5 times (TaN i ZrC) are used in negative branch and thermo-electromotive force coefficients of fillers in the positive branch are approximately equal but have different morphology (carbon fiber and boron carbide powder) – resulting values of thermo-electromotive force coefficients of functional element coincide accurate to the measurement error.

C10

THE INFLUENCE OF POLYMER CHARACTERISTICS AND PARTICLE MORPHOLOGY ON THE ELUTION CONTROL OF VANADATE IONS FROM V_2O_5/POLYMER COMPOSITES

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Vanadate ions exhibit insulin-mimicking action and *in vitro* studies confirmed proliferative effect of micromolar vanadate solutions on human cells [1]. In addition, *in vivo* studies showed acceleration of healing process in both, soft and hard tissue repair [2]. Therefore, an implant coating containing vanadium pentoxide, as a source of

vanadate ions, could show bioactive properties and might be used in regenerative tissue engineering. However, according to the literature, the solubility of vanadium pentoxide is several orders of magnitude higher than the boundary between bioactivity and cytotoxicity of vanadate ions[1]. Hence, the necessity of an effective solubility control of V_2O_5 requires cautious design of the implant coating.

The embedding of 1D nanoparticulate vanadium pentoxide in a polymer matrix could lead to a composite material with optimal solubility control. The elution of active material from polymer matrix, in general, depends on solute diffusion, polymeric matrix swelling and material degradation[3]. In order to determine influence of all three factors on vanadate elution, 1D nanowires and 0D microparticles of V_2O_5 were incorporated into different polymeric matrices (Table 1). As prepared V_2O_5/polymer composites were exposed to PBS medium at temperature of 37 °C and shaking rate of 60 rpm in order to test short-term and long-term vanadate elution kinetics.

Table 1. The polymer characteristics

Polymer	PLGA*	Chitosan	PCL*	Polystyrene
degradation	< 3 months	> 1 year	> 1 year	not observed
water absorption	2.5 %	70 %	0.5 %	0
molar mass	60 000	100 000	80 000	20 000

* PLGA - poly(lactic-co-glycolic) acid; PCL- poly-ε-caprolactone

We have demonstrated that control of vanadate elution from a polymer matrix containing 1D nanoparticles is better than from matrix containing 0D microparticles. These results indicate better retention of 1D nanoparticles inside matrix due to their high specific surface area. Furthermore, short-term elution studies reveal that PLGA is superior carrier for vanadium pentoxide nanostructures able to keep the level of released ions within bioactive limit. However, long-term elution study showed that PLGA's degradation causes burst of vanadate ions into solution induced by degradation of the matrix. Chitosan is inappropriate due to high water absorption. Furthermore, the physical interactions between V_2O_5 particles and either polystyrene or PCL are not sufficiently strong to retain particles inside matrix. In this contribution, implications of V_2O_5 morphology and polymer properties to the controlled delivery of vanadate ions from the V_2O_5/polymer composites will be discussed in detail.

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C11

IMPROVING THE THERMAL SHOCK PROPERTIES OF Y-α-SiAlON/GLASS COMPOSITE

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The thermal shock resistance is the ability of materials to resist damage when they are subjected to sudden changes in temperature during heating or cooling. SiAlON ceramics are good candidate for engineering application due to its good mechanical properties (hardness, excellent oxidation) at high temperature. However, its thermal shock resistance needs to be improved for these application areas. Production of glass-ceramic is the one way for the improving of thermal shock resistance of ceramic materials. However, the studies about the improvement of thermal shock properties of SiAlON ceramics', which has widespread structural application area and as well as keeping the high temperature mechanical properties is seen that limited. At this study glass phase systems, RO-Al_2O_3-SiO_2-B_2O_3 (R=Ba and Ca), that liquid phase be formed (using Y^{3+} and La^{3+} cations) in and that provide the development of microstructure in needlelike shape. It was designed in Y-doped α-SiAlON systems with better microstructural properties by initial composition optimization which is alternative to present glass system with lower coefficient of thermal expansion. The results showed that the type of metal cations and rare earth in glass composition significantly affect the microstructure, mechanical and thermal shock resistance of α-SiAlON ceramics related with the grain morphology and grain boundary phase.

C12

GRADED CERAMIC-METAL COMPOSITES OBTAINED BY THE CENTRIFUGAL SLIP CASTING

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Keywords: centrifugal slip casting, functional graded material (FGM)

Centrifugal slip casting is a method for powder processing that combines the effects of centrifugation and slip casting. If the particles in the slurry will have different densities, due to centrifugal force we can obtain a functionally gradient material (FGM).

In the experiment the following materials were used: alumina powder (TM-DAR, *d* = 140 nm) and iron powder of an average particles size of 3 µm. Composite water-based slurries were prepared with 10 vol.% iron powder with respect to the total solid volume. After homogenization the slurry were cast into a plaster mold and spin with three different rotation speed. For comparative purposes, a sample was cast without the action of centrifugal force. Subsequently the samples were dried and sintered in reducing atmosphere.

In order to investigate the stability of slurries the sedimentation study were performed for slips with different sold content. Microstructural observation and EDS analysis performed the distribution of metallic particles in ceramic matrix. Quantitative description of the microstructure of the graded region in the composites was made based on SEM images using computer analysis.

The research showed that the gradient composite affects both speed used in the centrifugal process and the solid content in the slurry.

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C13

RESORBABLE BIOCERAMICS IN $Ca_3(PO_4)_2$–$Mg_2P_2O_7$ SYSTEM

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Composite materials containing phases of tricalcium phosphate $Ca_3(PO_4)_2$ (TCP) and/or hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (HA) have been approved and authorized for clinical use in the most countries of the world. The main advantages of these materials consist in good biocompatibility and osteoconductivity in vivo. Chemical composition of ceramic materials based on HA and TCP are very similar to those of bones of animals and humans. These materials are not toxic and they do not cause allergic reactions. However, they have the following disadvantages: low speed of bioresorption, weak stimulatory effect on the growth of new bone tissue (osteoinduction), low fracture toughness and low fatigue strength under physiological conditions.

Being integrated into the lattice of hydroxyapatite bone tissue magnesium ion is an important factor for bone metabolism, for the bone matrix formation and its mineralization. It can also affect the activity of osteoblasts and osteoclasts, i.e. the rate of bone growth. Manmade ceramic material preparing based on calcium and magnesium phosphates can be an active source of chemical elements, which are essential for the new bone formation. The enhancement of the bioactivity of calcium phosphate ceramics can be achieved by adding the magnesium phosphate.

The aim of our research activity was to obtain resorbable bioceramic in $Ca_3(PO_4)_2$-$Mg_2P_2O_7$ system, based on synthetic powders of calcium and magnesium phosphates. The selection of various calcium and magnesium salts as initial ingredients allowed us to control a predetermined pH level in the reaction zone. Fine calcium and magnesium phosphate powders were synthesized by means of wet precipitation from aqueous solutions of ammonium hydrogenphosphate and calcium and magnesium nitrates/ acetate/ saccharates. The phase composition of synthetic powders was presented with the calcium and magnesium phosphates, i.e. HA - $Ca_{10}(PO_4)_6(OH)_2$, struvite -

$MgNH_4PO_4(H_2O)_6$, or a mixture of HA and struvite. The content of struvite increased with increasing magnesium content of the initial solution. Samples molded from synthetic powder were fired with a heating rate of 5 °C/min, holding for 2 hours at a final temperature in the range 900–1200 °C. According to XRD analysis the phase composition of ceramic materials, was consisted of tricalcium phosphate, magnesium pyrophosphate, solid solutions of calcium/magnesium orthophosphate and calcium/magnesium pyrophosphate.

The obtained ceramic materials can be used for regeneration of damaged bone tissue.

C14

IMPROVED PROPERTIES OF THE EPOXY-FLY ASH COMPOSITES BY SILANE TREATMENT OF THE FILLER

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Epoxy resin and its composites are widely used in many fields, for example: automotive, aerospace, construction or electronics. Properties of epoxy materials can be improved by filling resin matrix with fillers. Different branches of industry use different fillers in epoxy resin systems in order to get composites with the enhanced parameters. Quartz powder is one of the most popular epoxy resin filler. In the presented research work, the possibility of a quartz flour replacement by fly ash was investigated. Due to lower viscosity the processing of the fly ash composite is easier, e.g. filling of the mold. However, properties of fly ash composite such as mechanical properties and thermal conductivity, are inferior to that obtained on standard system with quartz powder. Therefore, in order to improve this two parameters with fly ash as a filler, the fly ash particles were modified by a silanization process. Four different conditions of silanization process and two different concentrations of the silane were performed. The presence of silane on the surface of fly ash particles was confirmed by two characterization methods: Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The set of samples based on the epoxy resin composites filled with modified particles was prepared. The properties of samples were investigated by means of mechanical tests, such as tensile strength and fracture toughness. Also the thermal conductivity measurements were performed on the prepared composites. The enhancement of the tested parameters was observed for all the epoxy resin samples filled with the modified fly ash in comparison to epoxy resin filled with untreated fly ash. The obtained results show possibility of application of the functionalized fly ash in epoxy composites used in industrial systems.

C15

ELECTRICAL BEHAVIOUR OF ZIRCONIA-ALUMINA NANOFIBERS-GRAPHENE COMPOSITES

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Yttria stabilized zirconia (3Y-TZP) composites with different contents of the cost-effective multi-layered graphene coated alumina nanofibers (ANFC) were prepared by spark plasma sintering (SPS). The samples were produced by powder processing and densified using SPS at 1250 °C with 40 MPa pressure for 5 min. Scanning electron microscopy observation of the microstructures showed that ANFCs were homogeneously dispersed in the matrix. The effect of ANFC on the electrical and dielectric properties of the composites at room temperature was studied. The composties demostrated typical percolation behavior with a very low percolation treshold between 0.7 and 2.0 wt.% ANFC content (corresponding to ~0.1 and 0.2 wt.% of graphene content). Addition of ANFC resulted in slight decrease in mechanical properties, but the electrical resistivity dropped significantly when the ANFC concentration exceeded percolation treshold. For the sample with 3.3 wt. % ANFC conductivity increased up to 11 orders of magnitude compared to monolithic zirconia, exhibiting 57.7 $S\cdot m^{-1}$, satisfying the required condition for the Electro-Discharge Machining (EDM).

C16

Fe_2O_3-SiO_2-PVA HYBRID XEROGELS, PRECURSORS FOR SUPERPARAMAGNETIC NANOCOMPOSITES, POTENTIAL CANDIDATES AS MRI-T2 CONTRAST AGENTS

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Superparamagnetic nanocomposites are susceptible to be applied as MRI-T2 contrast agents due to their properties, among which the high colloidal stability, size-dependent properties and long blood circulation for in vivo applications. PVA-SiO_2-

Fe_2O_3 nanocomposite sample has been synthesized via sol-gel method using tetraethilortosilicate, and Iron (III) acetylacetonate as precursors and polyvinyl alcohol (145000 molecular weight). The used $Fe_2O_3/(Fe_2O_3+SiO_2)$ ratio was 20 (wt.%). The obtained xerogel sample was subsequently thermally treated at different temperatures, specifically: 60, 120, 180, 220, 260, 300, 400 and 500 °C. In order to observe the system evolution during thermal treatment, the obtained samples were characterized by TGA, FTIR, EDAX, BET, SEM, TEM, XRD, Mossbauer spectroscopy and magnetic measurements. XRD and Mossbauer measurements indicate the presence of maghemite unique crystalline phase starting from 180 °C and keeping its stability up to 300 °C. Further increasing the calcination temperature, the transformation of maghemite toward most stable hematite was observed, in 300–400 °C temperature range the two crystalline phases care coexisting. At 400 °C it was found 35% hematite. Completion of phase transition was attained at 500 °C, when all maghemite was converted into hematite. Based on crystalline and magnetic structures evolutions, the sample that was thermally treated at 260 °C presents the optimum properties. It is represented by maghemite unique phase, it have superparamagnetic behavior presenting a small symmetric hysteresis, with remnant magnetization (σr) of 1.45 emu/g. and saturation magnetization (σsat) of approximately 48.10 emu/g. Obtained materials suitability for biomedical applications was discussed and specific potential application as contrast agents for MRI was evaluated. The maghemite nanoparticles were dispersed in gels corresponding to 10^{-3}, 10^{-4} and 10^{-5} % Fe_2O_3 concentrations and tested in phantoms.

C17

FERROELECTRIC BARIUM-STRONTIUM TITANATE AND CERAMIC-POLYMER COMPOSITES BASED ON BST IN TERAHERTZ RADIOCOMMUNICATION APPLICATIONS

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In the last century, ferroelectric materials have become an integral part of electrical engineering. The rapid development of radio communication systems, radio navigation and broadband radioaccess systems dictates the need to work at higher frequency bands. Great majority of these systems are currently working in a wide range of microwave spectrum. This implies the need for development of new technological solutions, new microwave components such as "smart" antenna systems.

The scientists dealing with these questions predict that significant progress will be achieved by using a new generation of ferroelectric materials. It is possible to produce these materials as thin composite tapes, which will eliminate previous drawbacks - such as exfoliation of elements in green state. They are therefore considered to be very attractive for applications in microwave band.

The aim of research was the selection of components for ceramic slurry which enabled to obtain thin films by tape-casting with the use of appropriately selected water-soluble polymer dispersions. Ferroelectric ceramic powder used in the study was barium strontium titanate (BST) doped by appropriate amount of, for example: nickel oxide.

Over the years engineers use this process to create ceramic capacitors that serve as electrical insulators in packaging, battery separator films, nanotechnology, smart materials, and biomaterials products. Tape casting is a casting process used in the manufacture of ceramic tapes from ceramic slurry. This forming technique is widely used in the production of thin sheets of flexible tape.

C18

NANO- AND BIO-STRUCTURED MATERIALS: SURFACES AND MESOPHASE FEATURES

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Surface and photorefractive properties of the inorganic and organic materials modified under nanostructurization process are discussed according our previous [1–5] and obtained recent results.

Some accent are given to show the advantage of the nanostructurization process to decrease the resistivity of the conducting inorganic structures based on ITO and to increase the wetting angle of different inorganic materials operated in the broad spectral range.

Special role of the dipole moment increasing is indicated as a macroscopic parameter of the nanostructured organic medium responsible for a relationship between the photorefraction and the photoconductivity characteristics. Basic polymer, namely, polyimide as well as the liquid crystal matrixes from cyanobiphenyl groups doped with unique nanoparticles, is chosen as the good model to increase the local volume polarizability via intermolecular charge transfer complex formation.

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C19

SELF-FLUXING Fe-BASED ALLOY WITH TiB$_2$ ADDITIVES FOR THE SPRAYING WEAR-RESISTANT COATINGS

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Intensive wear of machine parts, such as pump parts (sealing, impellers, bodies, etc.), hydraulic pistons, shafts and journal bearings leads to the development of new wear-resistant materials and coatings.

Self-fluxing alloys belongs to the eutectic ones and is recognized as candidates for wear resistance application. In order to improve alloy operating properties, in particular tribological characteristic, hard grains of refractory borides and carbides are added into alloy.

The goal of this study is to develop self-fluxing alloy-based composite powder materials for the spraying wear resistant coatings.

In the study commercially available self-fluxing alloy of FeNiCrSiBC system (Fe-base, Ni-37%, Cr-14%, Si-2.5%, C-1.4%, B-2.2%; 30–32 μm) was chosen as the base for composite material. Investigation of the interfacial interactions, such as wetting and chemical reactions in TiC-, CrB$_2$-, TiB$_2$-FeNiCrBSiC systems at high-temperature, shows that titanium diboride is expected to be one of the best reinforcements for self-fluxing alloy.

In the experiment composite powder material of FeNiCrSiBC-20wt.%TiB$_2$ system was produced. Developed composite material and FeNiCrSiBC self-fluxing alloy were applied on steel samples by plasma spraying.

Investigation of coatings wear behavior was carried out in dry sliding conditions at room temperature. Steel pins coated with FeNiCrSiBC-20wt.%TiB$_2$ composite material and FeNiCrSiBC self-fluxing alloy were used as samples. As well as stainless steel disc coated with self-fluxing alloy FeNiCrSiBC was applied as the counter body. It has been determined that developed composite materials have lower wear values compared with the initial FeNiCrSiBC coating, and can be recommended for the industrial use.

C20

FEM ANALYSIS OF CRACKING AROUND THE INDENT IN W-C COATING

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Finite element method (FEM) combined with experimental observations have been to study the cracking during nanoindentation in W-C coatings in the composite system hard brittle (WC) coating on softer steel (100Cr6) substrate. Nanoindentation was performed with Berkovich indenter using single load and continuous stiffness measurement (CSM) modes. The maximum penetration depth was around 2000 nm with the corresponding load of ~580 mN. Focused ion beam technique with scanning electron microscopy was applied to study topography and the cross-section of the indents. The experimental results showed that cracks appeared in the sink-in zone and at the apex of the indent on the interface between the coating and the substrate. These locations are in good agreement with the prediction of the FEM analysis based on bilinear model indicating the largest probability for the crack formation where principal tensile stresses are the highest. The distance from the indenter tip to the first crack combined with the calibration curve obtained from the FEM of the maximum tensile stress location in sink-in zone can be used as a simple method for the determination of the critical tensile stress at the maximum depth for W-C coating.

C21

MODIFICATION OF QUARTZ AND CERAMIC SUBSTRATES BY DEPOSITION OF TUNGSTEN LAYERS

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Thin films of tungsten oxide have many interesting properties. They were thoroughly investigated as photo- and electrochromic materials and found use in "smart windows", anti-dazzle mirrors and displays information. Futhermore, WO_3 is promising candidate material for applications in gas sensing, lithium batteries, catalysts and solid lubricants. Another possibility is to use them to modify ceramic surfaces. Ceramic materials are not wetted by metals. This is a big problem during manufacturing of ceramic-metal composites, especially by a pressure-less sintering method. One of the solution is the use of an intermediate layer of high-melting metal. This layer of one side should have adhesion to the ceramic surface, on the other hand should be wetted by the metal. Tungsten layer fulfills these conditions.

In this work WO_3 thin films deposition using CVD method at atmospheric pressure has been developed. Tungsten (V) ethoxide were used as precursor. Influence of the process parameters, such as gas flow rate and substrate temperature on selected properties of obtained coatings were examined. Thin films were deposited on quartz and ceramic substrates. They were subsequently annealed in a protective atmosphere and in a next step reduced to the metal. The resulting layers both before and after reduction was subjected to X-ray phase analysis (XRD), microscopic observation (SEM) and spectroscopic measurements (FT-IR). Finally, to study surface modification of alumina ceramics wetting test with copper was performed and the contact angle measurements were made.

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C22

THE STRUCTURE AND PROPERTIES OF CHROMIUM CARBIDE STEELS WITH TITANIUM NITRIDE COATING

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Thin film coating in thickness from 2 till 10 µm is applied on the surface of the edged and proven tools made of high speed steel, sintered-hard alloy and ceramics. It improves greatly a set of their exploitation characteristics and enables improvement of material processing by cutting.

We analyzed the impact of conditions of coating application through PVD method from (Ti,Zr)N on the surface of the powder chromic steel Cr13Mo2 and Cr17Ni2. The impact of the duration of deposition, heat-treatment temperature on the features of the surface of chromic carbide steel with coating (Ti,Zr)N was investigated. Wear resistance of the obtained samples was determined. It was shown that samples with Cr17Ni2 – 30 vol.% Cr_3C_2, which were in the process of deposition for 40 minutes, had the largest wear resistance. Microstructure and phase composition of the border coating – bearing plate was analyzed. Active interaction of the coating elements and bearing plate with forming of conversion zone was found. The dependence of the coating adhesive strength with the base on the time of the surface deposition was determined. It was shown that it decreased with the increase of the duration of deposition.

Investigations have shown that average destruction of the surface with the increase of the distance of coating was likely to decrease. It results in worsening of the coverage adhesive strength with base coat. It can be explained by the increase of the concentration of structural defects on the substrate film border due to significant difference between thermal expansion coefficients of the materials of the film and the base. Optimal values of the deposition parameters were found: nitrogen pressure P= 2 MPa, deposition distance d = 230 mm and duration of deposition T = 30 – 40 min. In such conditions the best mechanical and tribotechnical coating characteristics of titanium-zirconium double nitride are observed.

Thus, ion-plasma coatings of titanium-zirconium nitride enable to improve significantly mechanical-and-physical properties of chromium carbide steel, which open prospects for its usage as cutting tools and wear and corrosion products.

C23

SILVER MODIFIED ZEOLITE-MULTI-WALLED CARBON NANOTUBES-EPOXY COMPOSITE ELECTRODE FOR ELECTROCHEMICAL DETECTION AND DEGRADATION OF IBUPROFEN IN WATER

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The aim of this study is to explore the dual character of the electrode materials and electrochemical techniques in pharmaceuticals-containing water treatment and control applications. In general it is noteworthy that electrode materials for water quality monitoring are conceptually similar in different applications regarding degradation of pollutants from water. Thus, the development of suitable electrode materials and electrochemical techniques involves actually relatively easy adaptation from a field to another, taking into account the specific application peculiarities (*e.g.*, electrode geometry, design and operating conditions for degradation application).

The electrochemical interaction of ibuprofen (IBP) on two types of multi-walled carbon nanotubes based composite electrodes, i.e., multi-walled carbon nanotubes-epoxy (MWCNT) and silver-modified zeolite-multi-walled carbon nanotubes-epoxy (AgZMWCNT) composites electrodes are described.

The composite electrodes were obtained using two-roll mill procedure. SEM images of surfaces of the composites revealed a homogeneous distribution of the composite components within the epoxy matrix. Ag functionalized ZMWCNT composite material was obtained in order to improve electrode electrical conductivity and electroactive surface area.

The electrochemical determination of ibuprofen (IBP) was achieved using AgZMWCNT by cyclic voltammetry, differential-pulsed voltammetry, square-wave voltammetry and chronoamperometry. The IBP degradation occurred on both composite electrodes under controlled electrolysis at 1.2 and 1.75 V vs. Ag/AgCl, and IBP concentration was determined comparatively by differential-pulsed voltammetry, under optimized conditions using AgZMWCNT electrode and UV-Vis spectrophotometry methods to determine the IBP degradation performance related to non-functionalized electrode (MWCNT). AgZMWCNT electrode exhibited a dual functionality allowing both IBP detection and degradation, actually its control.

C24

SYNTHESIS AND CHARACTERIZATION OF CERIA/CARBON CRYOGEL COMPOSITE

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Ceria was synthesized by self-propagating room temperature method. Carbon cryogel was synthesized by sol-gel method, followed by freeze-drying and carbonization. Ceria/carbon cryogel composite was synthesized by simple hand mixing of ceria and carbon cryogel in mortar. The obtained material was characterized using following methods: nitrogen adsorption-desorption measurements, field emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR), temperature programmed desorption (TPD) and Raman spectroscopy. Also, isoelectric point (IEP) and point of zero charge (PZC) were determined. The application of composite in water purification processes was investigated in batch adsorption experiments.

C25

INTERACTION CERIUM OXIDE WITH DYSPROSIA AT 1500 °C

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For many years, CeO_2 - based materials have been mainly used as catalysts and pigments in ceramics or glasses. Today ceria is also applied as an active component in the so-called three-way catalysts (oxidation of CO, oxidation of CnHm, reduction of NO_x) for car exhaust. They are also promising materials for gas sensor applications in the automotive industry. Rare earth-doped ceria has higher values of ionic conductivity than fully yttria-stabilized zirconia (8YSZ), in the 600–800 °C temperature range. Better ionic conductivity can lead to decreased operational temperatures, increases in life-span as well as greater range of choices for constituting material for electrochemical devices such as solid oxide fuel cells and gas sensor. The main drawback of these electrolytes is the increased electronic conduction under low oxygen partial pressure that is accompanied by reduction of Ce^{4+} to Ce^{3+}. It has been reported that the reduction of ceria-based materials can be neglected at lower temperature

around 600–700 °C. However, such low temperature is not suitable for singly doped-ceria operations, as SOFC's electrolyte material has a high resistance. Structural modification of ceria solid solutions is one possible ways to improve their electrical conductivity.

The objective of this work is to investigate the phase relations in the binary system CeO_2-DyO_3 at 1500 °C in air in the whole concentration range. Powders of $CeO(NO_3)_2$ and Dy_2O_3 (99.99 %) were used as raw materials. The samples were prepared in steps 5 mol% from nitrate solutions with their subsequent evaporation and decomposition at 1000 °C for 2 h. Thermal treatment of as-prepared samples was carried out in to stages: at 1100 °C (452 h) and then at 1500 °C (150 h) in the furnaces with heating elements based on Fecral (H23U5T) and Supercantal ($MoSi_2$), respectively. The heating rate was 3.5 °C/min. The phase composition of the samples was investigated by X-ray (DRON-1.5, Burevestnik, Leningrad), petrographic (MIN-8, optical microscope, LOMO, Leningrad).

The study of solid state reaction of CeO_2 (fluorite-type, F) and Dy_2O_3 (cubic modification of rare-earth oxides, type C) at 1500 °C showed that two types of solid solutions based on cubic modifications of F-CeO_2 and C-Dy_2O_3 in the CeO_2-Dy_2O_3 system. These solid solution regimes were separated from end to end with the two-phase field: (F+C).

The boundaries of the homogeneity fields for the solid solutions based on F-CeO_2 and C-Er_2O_3, as well as lattice parameters of the unit cells for solid solutions F-CeO_2 and C-Er_2O_3 were determined. The solubility of Dy_2O_3 in F- modification of CeO_2 is about 20 mol % at 1500 °C. The lattice parameter of the unit cell decreased from a = 0.5409 nm in pure CeO_2 to a = 0.5398 nm for the solid solution of boundary composition. The solubility of CeO_2 in cubic C- erbium oxide attain ~10 mol%. The lattice parameters of the unit cell C phase varies from a = 1.065 nm in pure Dy_2O_3 to a = 1.066 nm for the solid solution of boundary composition.

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C26

FEATURES OF THE STRUCTURE AND PROPERTIES OF CERAMIC COMPOSITE B_4C–EUTECTIC ALLOY (B_4C-TIB_2) SYSTEM

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In this paper by combination of methods of crucibleless zone melting and spark plasma sintering (SPS) was obtained a new polycrystalline ceramic composite material of B_4C–E(B_4C-TiB_2) system. For its production were developed eutectic powders B_4C-TiB_2 with modified surface morphology, which mixed with the boron carbide powder and compacting by SPS. The unique surface morphology is that the reinforcement fiber and plate TiB_2 appear on the surface of each particle (Fig. 1), creating the effect of traction. SPS was conducted in an atmosphere of nitrogen and argon. The relative density of the samples after sintering was 98 %.

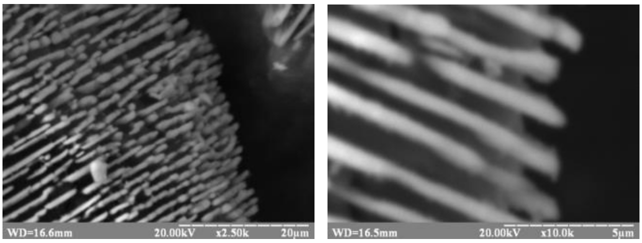

Figure. 1 Surface morphology of eutectic powders of B_4C-TiB_2 system

The microstructure of the composite is a matrix of boron carbide and evenly distributed in the matrix of eutectic grains. The grains boundaries of in the sintered samples are the perfect joints eutectic grains without visible defects and pores that may indicate their relatively high strength.

This composite showed sufficiently high mechanical properties. Bending strength at room temperature reaches 387.2 MPa, which exceeds the strength of directionally solidification eutectic alloy B_4C-TiB_2 (190 MPa) in 2 times. Bending strength at 1600 °C respectively amounted to 399.8 MPa. The unique morphology of the particle surface can increase adhesion eutectic powder B_4C-TiB_2 with matrix B_4C. By the results of X-ray diffraction and Electron studies it is no interactions between the matrix and eutectic grains are observed. This fact indicates good grip of eutectic grains with matrix and indicates an increase of flexural strength due to increase content of eutectic grains in the material. The strength of composites of B_4C–E(B_4C-TiB_2) sintered in a nitrogen atmosphere is up to 16 % higher than strength of samples sintered in argon atmosphere, that can explain by the formation of nitride phases, creating the effect of rigidity.

TRADITIONAL CERAMICS

T1

CHARACTERIZATION OF THE INTERACTION BETWEEN GLAZES AND CERAMIC BODIES

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The main reason for the use of these glassy coatings is to increase the resistance of ceramics to various types of corrosive environments without loss of properties.

Glazes and ceramic bodies are in close contact and react chemically and physically during firing. Chemical reactions of individual components take place, change the chemical composition and if the firing process is long enough lead to the formation of interlayers on interfaces. Characterization of the interaction between glazes and ceramic bodies can provide information about application features, firing processes and range and sign of stress in the glaze layer. Glaze defects are caused by improper stresses within glaze layers (tension, compression) or by changes in ceramic bodies (moisture expansion, rehydration) and glaze layers during usage or improper storage of ceramic products.

The study of the interaction between glazes and ceramic bodies was focused on the evaluation of surface defects, determination of the coefficients of the thermal expansion measured by dilatometer and characterization of glaze–body interaction problems.

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T2

PROPERTIES OF SINTERED CORDIERITE CERAMICS OBTAINED BY SOL-GEL METHODS OF POWDER SYNTHESIS

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Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) as a ceramic material has great importance in materials science and wide application in industry thanks to its low thermal expansion and dielectric constant, high chemical and thermal stability and high electrical resistivity. Cordierite ceramics is used for producing supports for electric heaters, ceramic fire resistant materials for furnaces, catalyst supports for the complete combustion of exhaust gases of internal combustion engines, insulation material in high frequency electronics, as well as substrates for integrated circuits and electronic modules.

Properties of sintered cordierite ceramics, where cordierite powders were obtained by alkoxide hydrolitic and non-hydrolitic method, colloidal method and starting from silicic acid, were studied and compared. The properties of sintered materials were observed by SEM of the fracture surfaces and polished and thermally etched samples. The mechanical properties of obtained materials, microhardness and indentation fracture toughness, were determined by microindentation method. The best sinterability showed powder obtained by colloidal method (sintering temperature of 1370 °C for 2 h), and powder obtained by Si-acid method (sintering temperature of 1400 °C for 2 h).

T3

EFFECT OF TRANSITION METAL ADDITION IN THE BIOACTIVITY OF BORATE BIOGLASS: A DESCRIPTIVE CORRELATIONAL STUDY

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Keywords: bioactive borate, biocompatibility, solubility and bone repair.

The basic structure and some physical properties of a bioactive borate glasses containing different types of transition metals were prepared and investigated with an in vitro analysis with the same molar composition as 45S5 glass but with all the silicon dioxide (SiO_2) replaced by boron trioxide (B_2O_3). The biodegradability was explored and estimated by soaking the samples into simulated body fluid (SBF) at 37 °C for fixed periods of time up to 30 days. The pH change of SBF was measured during the soaking periods in order to explore the ionic change processes. The structural changes in the crystalline phases obtained in the glass matrix before and after immersion in SBF were recognized by means of x-ray diffraction (XRD), Fourier transform infrared analysis (FTIR) and scanning electron microscope (SEM). All obtained results support the formation of hydroxyapatite (HA) layer with different amounts that depend on the type of transition metal added to the glass matrix. The biodegradation data and spectral analysis of different samples may consider the material to be a good candidate for use as a bone substituent.

T4

THE INFLUENCE OF MOLAR RATIO Al_2O_3/SiO_2 ON THE STRUCTURE OF CERAMIC GLAZES

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Ceramic glazes are various chemical and phase composition enamels. This multiplicity of glazes has influence on the structure, and consequently, on technological and using parameters. Molar ratio of silica oxide (as basis forming glass-oxide) to alumina oxide (modifier and stabilizer of glazes structure) determines the different kinds of glazes structures and the way of reaction between the structure of glazes and the other oxides.

In this paper was determined the influence of molar ratio Al_2O_3/SiO_2 on the structure of ceramic glazes which were intended for sanitary ware. The tested glazes had constant and unchangeable molar ratio alkali oxides (Na_2O/K_2O) and alkaline earth oxides (CaO/MgO). The value of molar ratio Al_2O_3/SiO_2 was the only variable in the examined glazes and it stayed in range of 5 to 11. The structure in these glazes was described based on the results from X-ray analysis and infrared spectroscopy. The result of this paper are numerous and very interesting dependences, on which the changing molar ratio Al_2O_3/SiO_2 had impact.

T5

CORROSION OF $MgCr_2O_4$, $MgAl_2O_4$, $MgFe_2O_4$ SPINELS IN SO_2–O_2–SO_3 ATMOSHERE – THERMODYNAMIC EVALUATION

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Presented results are connected with high temperature sulfate corrosion of basic refractory ceramics containing magnesium spinels: $MgAl_2O_4$, $MgFe_2O_4$ (magnesioferrite), $MgCr_2O_4$ (magnesiochromite) and their solid solutions. Basic refractory ceramics containing magnesium spinels and theirs solid solutions are widely use in metallurgy, chemical, ceramic and glass industry. This group of refractories, during working campaign, is exposed to a several destructive factors. One of such factor is gas corrosion caused by sulfur oxides. However the gas sulfate corrosion of basic refractory materials containing magnesium spinels have great practical meaning the corrosion resistance of the material main components are not sufficiently examined. This work presents thermodynamic analysis of the system: ($MgCr_2O_4$, $MgAl_2O_4$, $MgFe_2O_4$) – SO_2–O_2–SO_3 aimed on: 1) calculation of the standard free enthalpy of the chemical reactions at temperature range 573–1273 K in order to determine their thermodynamic spontaneity; 2) calculation of the equilibrium composition of the gas mixture initially containing 13.0 vol.% SO_2 and 18.3 vol.% O_2 in the temperature range 573–1273 K; 3) calculation of equilibrium sulfates ($MgSO_4$, $Al_2(SO_4)_3$, $Cr_2(SO_4)_3$, $Fe_2(SO_4)_3$) decomposition pressure and equilibrium partial pressure for the reaction of SO_3 with spinels for prediction temperature range of reaction products stability. Thermochemical calculation gives information about equilibrium state in analyzed system. In real condition equilibrium state do not have to be achieved. From this reason results of calculations with experimental data were compared. Good agreement between theoretical prediction and experiment was achieved

T6

PHASE EQUILIBRIA AND PROPERTIES OF SOLID SOLUTIONS IN THE La_2O_3-Yb_2O_3 AND La_2O_3-Y_2O_3-Yb_2O_3 SYSTEMS AT 1500 °C

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Systems with yttria, lanthana and ytterbia 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 La_2O_3-Yb_2O_3 and ternary La_2O_3-Y_2O_3-Yb_2O_3 systems at 1500 °C in air in the whole concentration range. Powders of La_2O_3, Yb_2O_3 and Y_2O_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 120 h and at 1500 °C for 50 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 La_2O_3 and Yb_2O_3 at temperature 1500 °C showed that three types of solid solutions based on hexagonal modification of A-La_2O_3, cubic modification of C-Yb_2O_3 and intermediate phase with perovskite-type structure of $LaYbO_3$ (R) with rhombic distortions in the La_2O_3-Yb_2O_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-La_2O_3, C-Yb_2O_3 and $LaYbO_3$, as well as lattice parameters for solid solutions were determined.

The refined solubility of Yb_2O_3 in A- modification of La_2O_3 is about 9 mol% at 1500 °C (50 h). The lattice parameter of the unit cell varies from a = 0.6523 nm, c = 0.3855 nm in pure

La(OH)$_3$ to *a* = 0.6557 nm, *c* = 0.3864 nm in the sample containing 55 mol% La_2O_3-45 mol% Yb_2O_3. The solubility of La_2O_3 in cubic C- modification ytterbium oxide attains ~2 mol% (1500 °C). The lattice parameters of the unit cell C phase varies from *a* = 1.0432 nm in pure Yb_2O_3 to *a* = 1.0453 nm for the solid solution of boundary composition. It has been found that the boundaries of the R- phase homogeneity field in the concentration range of 48–56 mol% Yb_2O_3. The lattice parameters of the unit cell R phase varies from *a* = 0.6015, *b* = 0. 5805, *c* = 0.8438 nm in single-phase sample, containing 50 mol % La_2O_3-50 mol % Yb_2O_3 to *a* = 0.6033, *b* = 0. 5846, *c* = 0.8395 nm in two-phase sample (R + C), containing 40 mol% La_2O_3-60 mol% Yb_2O_3. In the ternary system La_2O_3-Y_2O_3-Yb_2O_3 continues solid solutions based on C modifications of yttria and ytterbia, as well as intermediate phases $LaYO_3$ ($LaYbO_3$), and boundary fields of solid solutions based on hexagonal (A) and monoclinic (B) modifications La_2O_3 at 1500 °C were determined.

Acknowledgement: This work has been funded by project (AFOSR) STCU P513 (2012-2014).

T7

PREPARATION, PROPERTIES AND APPLICATIONS OF SHEAR THICKENING FLUIDS BASED ON SILICA POWDER, GLYCOLS AND DOPANTS

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In recent decades, an immense development of design of composite materials has been observed. This movement is beneficial because combined constituent materials have significantly different and sophisticated properties than the individual components. Ceramic-polymer composites based on shear thickening fluids (STF) have a great potential in systems used for human body protection. That non-Newtonian liquid, also known as dilatant, is characterized by an increase of viscosity as a function of shear rate [1]. It can be explained by a few theories. According to the particle flocculation mechanism, polymer chains adsorb onto the surface of powder particles. A three-dimensional network, formed by the connections between the carrier fluid molecules and the powder particles, is created under the influence of high shear rate. Bridges are made between the polymer and the powder and consequently viscosity is increased. This process is reversible [2].

As a result of latest research, increase of viscosity is determined as a very good value. Materials and devices based on STF can dissipate energy associated to shocks, impacts and vibrations. Combining chemistry of polymers, colloids, nanotechnology and ceramics, scientists aim to produce a reliable liquid materials used to protect the human body, mainly dedicated for representatives of the uniformed services and sportsmen [3].

This thesis is devoted to the development of composition and analysis of the rheological properties STF based on silica dispersed in various organic liquids. The aim of the research was to verify applicability of the final suspensions to produce liquid armor. Herein we present the studies on the effects of the molecular weight of a dispersing liquid, the concentration of the silica and the higher temperature on the rheological properties of suspension.

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ESR WORKSHOP
COST IC1208

E1

EFFECT OF POLING CONDITIONS ON DIELECTRIC, PIEZOELECTRIC AND FERROELECTRIC PROPERTIES IN DOPED POTASSIUM SODIUM NIOBATE

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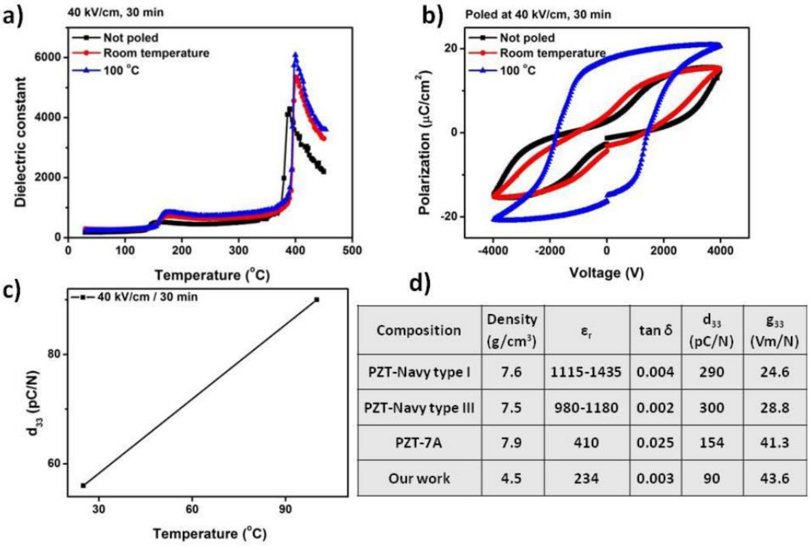
Potassium sodium niobate (KNN) is one of the most promising lead-free systems. However, undoped KNN has poor sinterability and by conventional sintering results in inadequate densification and, consequently, in inferior physical and electrical properties. CuO and Li_2O have been used in KNN and others systems to improve densification and electrical properties. CuO as dopant improves densification and grain growth, which is attributed to a liquid phase formation [1]. On the other hand doping with Li_2O enhances the densification and stabilization of dielectric properties [2]. To maximize the piezoelectric properties of piezoceramics the alignment of ferroelectric domains is required via a poling process [3]. In this work, the dielectric, piezoelectric and ferroelectric properties, of $(K_{0.49}Na_{0.49}Li_{0.02})(Nb_{0.985}Cu_{0.015})O_3$ (KNNLC) are investigated as function of the poling conditions (field, temperature and time). It was observed that: i) the dielectric constant at room temperature increases from 179 for unpoled KNNLC



Composition	Density (g/cm³)	ε_r	tan δ	d_{33} (pC/N)	g_{33} (Vm/N)
PZT-Navy type I	7.6	1115-1435	0.004	290	24.6
PZT-Navy type III	7.5	980-1180	0.002	300	28.8
PZT-7A	7.9	410	0.025	154	41.3
Our work	4.5	234	0.003	90	43.6

Figure 1. a) Dielectric constant versus temperature, b) hysteresis loops and c) piezoelectric coefficient versus poling temperature of unpoled KNNLC ceramics compared with poled KNNLC poled ceramics. d) Comparison between the electromechanical properties commercial PZT and this work.

ceramics to 234 for poled ones (40 kV/cm, 100 °C and 30 min); ii) the Curie temperature do not change after poling (T_C = 396±6 °C) (Fig. 1a); iii) the piezoelectric coefficient increases ~ 40% from d_{33} = 56 pC/N to d_{33} = 91 pC/N with poling temperature (Fig. 1c); iv) P_r increases from 2.6 μC/cm^2 to 18.2 μC/cm^2, for unpoled KNNLC ceramics and KNNLC ceramics poled at 40 kV/cm, 30 min and 100 °C, respectively and v) the pinched ferroelectric loops of unpoled KNNLC ceramics disappear after poling at 100 °C (Fig. 1b).

The relation between the observed poling behavior and the defect structure of these ceramics is established.

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E2

IMPROVING NiOOH CATALYTIC ACTIVITY IN ELECTRO-CHEMICAL WATER SPLITTING USING TRANSITION METAL DOPANTS: A FIRST PRINCIPLES CALCULATION BASED STUDY

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Due to its massive industrialization, modern society is more and more concerned with energetic issues. While fossil resources have been so far the main source of energy, their availability is limited in the time and their utilization is responsible for various pollutants. Hence, it is necessary to look for alternative energies. Photoelectrochemical cells are an attractive solution [1].

Such devices aim at converting solar energy into chemical fuels. However, this technology suffers from a major limitation due to water oxidization (Oxygen Evolution Reaction – O.E.R) at the anode electrode. Indeed, this reaction requires highly active catalysts often based on noble metals, and thus economically non-viable for intensive industrial exploitation. Besides, they are not stable in operating conditions and thus have a short life span [2].

Various oxide and hydroxide of earth-abundant metals have already been proposed. Among them, nickel oxihydroxyde (NiOOH) exhibits outstanding properties, especially owing to its high chemical stability. However, its catalytic activity is extremely dependent on impurities and doping. Indeed, while Fe doped NiOOH is known to be remarkably active, pure NiOOH is only poorly efficient.3 Using density functional theory (DFT), we studied properties of doped and undoped NiOOH. For the first time in a theoretical work, we present here a systematic scan of various metallic dopants. According to bulk and slab calculations, we were able to show very different trends in catalytic activity between them. Owing to our results, we expect to give interesting guidelines for experimentalists and industrials in order to design more efficient photoelectrochemical cells.

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E3

STUDY OF FERROELECTRIC-RELAXOR $BaCe_xTi_{1-x}O_3$ CERAMICS

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$BaTiO_3$-based ceramics are attractive as Pb-free relaxors with applications in microelectronics and wireless communications. Among the dopant ions, cerium is a special element with two oxidation states: Ce^{3+} and Ce^{4+}, that can be incorporated at Ba-site (as Ce^{3+}), while Ce^{4+} will be preferentially incorporated on Ti-sites.

In the present paper, single phase dense (98-99% relative density) $BaCe_xTi_{1-x}O_3$ (x = 0.02; 0.05; 0.10 and 0.20) ceramics were prepared by a conventional ceramic processing consisting of four fundamentals steps: preparation of the powder by solid state reaction, consolidation of the powder by isostatic pressing, sintering of the powder compact into a dense polycrystalline ceramic, cutting and polishing. The mixed powders have been calcined at 1000 °C for 4h and sintered at 1450 °C for 4h, resulting in dense single phase ceramics with homogeneous microstructures. The sample with 20% of Ce needed a sintering treatment at 1550 °C to become dense enough. X-ray diffraction on calcinated powders and sintered ceramics showed the formation of perovskite single phase $BaTi_{1-x}Ce_xO_3$. The microstructural features (density, grain size and grain size distribution) of the dense ceramic were determined from SEM images.

Impedance spectroscopy in the temperature range of (20 to 180) °C shows a composition-induced ferroelectric-to-relaxor crossover with compositional-dependent shifts of the structural transition temperatures by comparison with ones of the pure $BaTiO_3$. The dielectric characteristics indicate the possibility of either superposition of phases and/or the possible partial doping of Ce^{3+} on A positions. The nonlinear dielectric properties of ceramic samples were investigated at room temperature and the results were discussed in term of ferroelectric-relaxor crossover induced by composition.

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E4

$BiFeO_3$ CERAMICS DENSIFICATION STUDY

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Bismuth ferrite ($BiFeO_3$) is one of the very few materials possessing ordered both electrical dipoles and magnetic moments in very wide range of temperatures. That gives it tremendous opportunities for research and application.

$BiFeO_3$ precursor powders were prepared by auto-combustion and soft chemical synthesis methods using different organic compounds as fuels and complexing agents. They were calcined at 600 °C for 2 h, grinded, pressed and sintered at 800 °C for 1 h with quenching. XRD and SEM confirmed formation of very pure $BiFeO_3$ perovskite phase, but problem with low density, which is characteristic for $BiFeO_3$ ceramics, still remained. It doesn't affect magnetic properties, but has strong influence on electrical and, through them, on multiferroic properties. To define actual, intrinsic properties of material, it is very important to obtain ceramics with densities close to theoretical. For this reason, many treatments were tried in order to raise samples' densities: powder milling and homogenization, ultrasound, different pressures, various thermal treatments. Powders and ceramic samples were then characterized (particle size analysis, XRD, SEM). The densities of sintered $BiFeO_3$ samples were measured. All the treatments had impact on ceramics density, but some also introduced secondary phases.

E5

CHARACTERIZATION OF $0.9Pb(Mg_{1/3}Nb_{2/3})O_3$-$0.1PbTiO_3$ ELECTROCALORIC MULTILAYERED STRUCTURES PREPARED BY TAPE CASTING

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The electrocaloric (EC) effect is an adiabatic and reversible temperature change, due to reorientation of dipoles in a polar material induced by an electric field. Lead-based relaxor ferroelectrics, such as $PbMg_{1/3}Nb_{2/3}O_3$-$PbTiO_3$ (PMN-PT), exhibit high dielectric permittivity and polarization, and a large change of polarization with temperature resulting in large EC temperature changes (ΔT_{EC}). The largest ΔT_{EC} has been

measured in thin ceramic or polymer films, e.g. 40 K at the electric field amplitude of 1.2 MV/cm [1]. Although these values exceed the highest reported values measured in bulk ceramics, the EC coefficients ($\Delta T_{EC}/\Delta E$) of bulk ceramics are much higher than those of the thin films. Bulk ceramics have larger cooling power and are more appropriate for exploitations in cooling applications [2,3]. Due to the fact that the electric field amplitudes needed to achieve ΔT_{EC} of a few K are in the range of 100 kV/cm, the thickness of ceramic elements should be as low as possible.

The aim of this work was to prepare multilayer ceramic elements by tape casting technology, which could be considered in cooling applications. As the thickness of each layer is reduced in comparison with thickness of bulk pellets, high electric fields can be applied at such multilayer element. The $0.9Pb(Mg_{1/3}Nb_{2/3})O_3$-$0.1PbTiO_3$ ceramic powder was prepared by mechanochemical activation of the powder mixture. The formulation of the slurry and the casting conditions were optimised. Ceramic elements were prepared by atmosphere sintering at temperatures between 900 and 1000 °C. In the contribution we report the results of microstructural analysis of the sintered elements, their dielectric properties, and the results of indirect electrocaloric measurements, and compare them with the properties of ceramics shaped by dry compaction of the powder.

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E6

INVESTIGATION OF $BaSrTiO_3$ POROUS CERAMICS

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Barium strontium titanate ($Ba_{1-x}Sr_xTiO_3$) ceramics are among the most promising candidates for applications in capacitors, infrared detectors, microwave devices and dynamic random access memories, due to its high permittivity, low dielectric loss, nonlinear variation of permittivity with the applied electric field, ferroelectric and pyroelectric properties [1]. The value of Curie temperature (T_C) of $Ba_{1-x}Sr_xTiO_3$ (BST) can be varied by adjusting the Ba/Sr ratio. In case of phase shifting, it is desired to operate the device in paraelectric region, hence the need for low value of T_C.

In this study, we have chosen a BST composition with a Ba/Sr ratio of 0.65/0.35 ($x = 0.35$) because of its attributes: low T_C (~ 19 °C), high permittivity, low dielectric loss and high tunability values. Therefore, the aim was to prepare and to analyze the role of porosity on the functional properties of BST65/35 ceramics with various degrees of porosity. The porous ceramics were prepared through the addition of lamellar graphite with different concentrations (0, 10, 20 and 35 vol.%), in BST65/35 powders [2]. The phase purity of these samples was analyzed by X-Ray Diffraction (XRD) and the microstructure was investigated using Scanning Electron Microscopy (SEM).

Impedance Spectroscopy analysis has been performed at a temperature range of 0–200°C and frequency range of 20 Hz – 2 MHz. The permittivity of porous $Ba_{0.65}Sr_{0.35}TiO_3$ ceramics decreases with increasing porosity and remains constant at high frequencies for all the investigated samples. The temperature dependence of dielectric properties revealed a high value of permittivity (ε = 15024 for dense BST65/35 and of 450 for the highest porosity level in BST65/35) at the phase transition (T_C ~ 19 °C). The field dependency of permittivity and relative dc–tunability of the $Ba_{0.65}Sr_{0.35}TiO_3$ ceramics with various degrees of porosity were investigated at room temperature. The porous BST65/35 ceramics were characterized in terms of their dielectric properties and the results were correlated with their microstructural characteristics.

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E7

STRUCTURE AND PROPERTIES OF MULTYFERROIC $BaTiO_3/NiFe_2O_4$ THIN FILMS OBTAINED BY SOLUTION DEPOSITION TECNIQUE

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In recent years, novel multiferroic materials have attracted close attention in developing microelectronic industry. These materials are unique group of advanced materials with the ability to exibit more than one ferroic ability. Multiferroic thin films offer few advantages compared to bulk, most of all smaller size and lower power consumption, being aware that processing of thin films may pose a challenge. In this research, phase composition, dielectric and magnetic properties of multilayer $BaTiO_3/NiFe_2O_4$ multiferroic thin films have been investigated. Different multilayer thin films structures were obtained by spin coating of ferromagnetic and ferroelectric layers in alternating order. Obtained thin films were crack free, with no secondary phases, with total thickness up to around 700 nm. Interesting magnetic magnetic behavior was observed, while dielectric properties were limited and strongly influenced by the introduction of magnetic layers. By increasing the titanate/ferrite layer thickness with defferent order of layer deposition, better crystallization was achieved, which was confirmed by X-ray diffraction and Raman analysis, with higher value of dielectric constant and better ferromagnetic saturation and remanent magnetization.

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E8

BIOMORPHIC GROWTH AND FUNCTIONAL PROPERTIES OF NICKEL OXIDE 1-D MICROSTRUCTURES

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Natural evolution has provided numerous examples of exceptional building materials. Therefore, a variety of bio-inspired morphology synthesis strategies are being explored through processes of *in situ* modification with bio-templates because of their structural and compositional hierarchical order. Nickel oxide is an important semiconductor and antiferromagnetic material, widely used in electrochemical, optical and magnetic applications and its functionality greatly depends on its nano-/microscale [1]. 1-D structures are receiving significant attention because of their potential applications in energy conversion, separation science, environmental protection and chemical sensors [2].

The aim of this work was to prepare pure phase biomorphic 1-D NiO microtubes by using natural fibers as bio-template and to study the effects of the synthesis parameters (bio-template, calcination temperature, precursor concentration) on their microstructure and functional properties. The final products were obtained by infiltration of biotemplates (Cotton - Gossypium, Hemp - Cannabis sativa and Flax - Linum usitatissimu, Fig. 1) with nickel nitrate solution at different concentrations, followed by calcination in air, at different temperatures. The phase formation and morphologies have been investigated by using X-ray diffraction (XRD) and scanning electron microscopy (SEM) (Fig. 1). The influence of the synthesis parameters and templates on the functional characteristics of the 1-D NiO samples was determined and interpreted in terms of their nano/microstructural properties.

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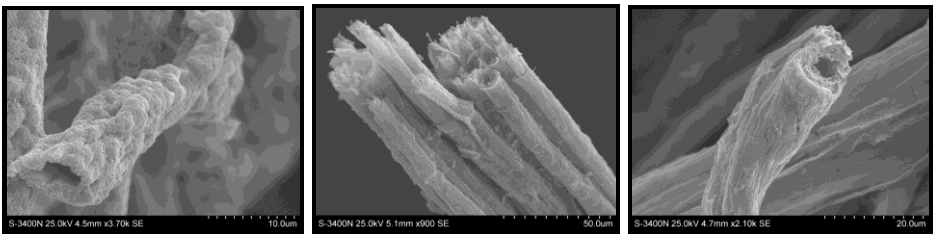

a) b) c)

Figure 1. SEM image of biomorphic NiO produced by using cotton (a), hemp (b) and flax (c) as biotemplates

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E9

MAGNETIC PROPERTIES OF PLATE-LIKE COMPOSITE NANOPARTICLES COMBINING SOFT-MAGNETIC IRON OXIDE WITH HARD-MAGNETIC BARIUM HEXAFERRITE

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Magnetic properties of bi-magnetic sandwich-like composite nanoparticles composed of a hard-magnetic barium hexaferrite ($BaFe_{12}O_{19}$) platelet core positioned in between two soft-magnetic spinel iron oxide – maghemite (γ-Fe_2O_3) layers will be presented. Composite nanoparticles were prepared with simple method, based on co-precipitation of Fe^{3+}/Fe^{2+} ions in colloidal suspension of the core nanoparticles [1]. The spinel was formed exclusively as layers at the core nanoparticles. The magnetic properties of the composite nanoparticles were measured using vibrating sample magnetometer perpendicular and parallel to their preferential magnetic orientation. For this purpose, the nanoparticles were hydrophobized with dodecylbenzene sulphonic acid (DBSa) [2], dispersed in a hot wax and magnetically oriented in a homogeneous magnetic field of 1T. The magnetic orientation was preserved by cooling and solidification of the wax. The composite nanoparticles exhibit single-phase magnetic hysteresis, suggesting that the two phases are exchanged coupled[3]. The difference between in-plane and out-of-plane magnetic hysteresis showed relatively good magnetic orientation of the nanoparticles. The comparison of the in-plane magnetic hysteresis of the core and the corresponding composite nanoparticles shows a large increase in saturation magnetization (M_S) and remanence (M_R), and decrease in coercivity (H_C) for the composite nanoparticles.

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E10

ENHANCEMENT OF THE ENERGY STORAGE PROPERTIES IN PLZT CERAMICS WITH COMPOSITIONS ACROSS FE-AFE PHASE BOUNDARY

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Antiferroelectrics (AFEs) are promising materials for application in pulsed capacitors with high energy-storage density due to their field-induced switching between AFE and ferroelectric (FE) phase. Generally, the free energy difference between AFE and FE states around morphotropic phase boundary is smaller. This allows the AFE-FE phase transition to be easily induced by an electric field, mechanical pressure or a temperature change, accompanied with other excellent properties like high polarization and enormous strain. Once the AFE-FE transition is the key for high energy storage properties, in order to develop antiferroelectric materials for energy storage, is better to search for AFE-FE phase boundaries compositions. Motivated by these factors we investigated the energy-storage properties of $Pb_{1-x}La_x(Zr_{0.9}Ti_{0.1})_{1-x/4}O_3$ (PLZT x/90/10) in the compositional range around the AFE/FE phase boundary ($0<x<0.04$). The recoverable energy density was enhanced in the composition range where booth ferroelectric and antiferroelectric phases coexist and a maximum value of 1.85 J/cm^3 at 65 kV/cm was obtained at room temperature for composition 3.5/90/10. All compositions from the range of phase superposition show high dielectric permittivity (~800) and low dielectric loss (lower than 0.04) in a large range of frequency. These properties indicate that PLZT ceramics might be potential candidates AFE materials for energy-storage capacitor applications.

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E11

THE CATION DISTRIBUTION IN $NiFe_2O_4$ AND $NiFe_{1.85}Y_{0.15}O_4$: RAMAN AND X-RAY DIFFRACTION STUDIES

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The cation distribution of $NiFe_2O_4$ and $NiFe_{2.85}Y_{0.15}O_4$ samples was investigated by Raman and X-ray studies. The investigated samples are prepared by procedure described in [1] and for the purpose of further research they are annealed at 750 °C, The diffraction patterns indicate that samples present the spinel-type structure, without any additional phases. Cation distribution was investigated by refinement of the site occupancies of Fe^{3+}, Ni^{2+} and Y^{3+} ions at the tetrahedral 8a an octahedral 16d sites (space group $Fd\bar{3}m$). The Rietveld refinement procedure showed that $NiFe_2O_4$ sample has a completely inverted spinel structure, while $NiFe_{2.85}Y_{0.15}O_4$ has a partially inverse spinel structure.

The Raman spectra collected in the wave number range of 100–1000 cm^{-1} showed five predicted modes for the spinel structure. The observed splitting of A_{1g} Raman mode into two energy values attributed to peaks belonging to each ion (Ni^{2+} and Fe^{3+}) in the tetrahedral positions. Since both Ni and Fe ions can occupy the tetrahedral site in $NiFe_2O_4$, it is expected for A_{1g} mode to split into two branches ($A_{1g}^{(1)}$ and $A_{1g}^{(2)}$), where the lower wave number $A_{1g}^{(2)}$ mode would be attributed to the Ni-O bond due to the larger mass of Ni relative to Fe.

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E12

SYNTHESIS AND STRUCTURAL CHARACTERIZATIONS OF SnO_2 THICK FILMS

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For any semiconducting metal oxides nanomaterials, dielectric properties of material play an important role, sensing, catalytic activity, adsorption, etc. mainly depends upon dielectric and its surface properties. These properties depend on the chemical composition, method of synthesis and surface characteristics. Various physical and chemical properties of nanomaterials can tune by choosing various chemical treatments or novel synthetic routes. Among various metal oxide semiconductors, SnO_2 is extensively used for gas sensing applications SnO_2 semiconductor is one of the well-known metal oxides used for sensors and catalytic application. The electrical properties have one of the key roles in the performance of metal oxide semiconductors. Moreover, we can tune these properties by applying doping and controlling the particle size of the material. Here we explain synthesis of SnO_2 Nanopowder by following co-precipitation method and structural characterization and morphology studies of SnO_2 thick film have been done by using XRD, Raman spectroscopy. Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Impedance Analysis

E13

PREPARATION AND PROPERTIES OF PCL-FUNCTIONAL OXIDE COMPOSITES

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The aim of this work was the preparation and the properties investigation of some PCL-functional oxide composites. We used poly-ε-caprolactone (PCL) as polymeric matrix for composites filled with magnetic ($CoFe_2O_4$) or ferroelectric ($BaTiO_3$) nanoparticles.

Poly-ε-caprolactone (PCL) was chosen due to its attractive properties such as low melting point, non-toxicity, slow degradation and drug permeability. Being also a biocompatible, biodegradable and bioresorbable polymer it has many possible application in flexible electronics, medical devices, drug delivery and tissue engineering [1].

For preparation we used a solvent casting method wich implied the use of relatively low temperatures, simple aparature and which leads to composites films with a good distribution of filler in the polymeric matrix.

After the preparation, the composites formation and their characteristic as structure and microstructure were investigated by XRD and SEM. The XRD analyses confirmed the formation of biphasic composites (spinel-polimer/perovskit-polimer) with no other secondary phases. The SEM images showed a very good distribution of the ferroelectric/magnetic nanoparticles in the polymeric matrix.

The frequency dependence of dielectric properties at room temperature have been investigated for all the samples, and discussed in correlation with the microstructural data and a theoretical model based on the microstructural formation.

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E14

PROPERTIES OF $BaTiO_3$-$NiZnFe_2O_4$ MULTIFERROIC COMPOSITES OBTAINED BY AUTO-COMBUSTION SYNTHESIS

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Nickel zinc ferrite (NZF(70-30)) and barium titanate (BT) nanosized powders were synthesised by auto-combustion method. Multiferroic composites with the general formula $x\mathrm{Ni}_{0.7}\mathrm{Zn}_{0.3}\mathrm{Fe}_2\mathrm{O}_4$–$(1\text{-}x)\mathrm{BaTiO}_3$ (x = 0.1, 0.3, 0.5, 0.7, 0.9) were prepared from nickel zinc ferrite and barium titanate powders by mixing in planetary ball mill for 24 h. Pellets were sintered at different temperatures in order to get dense, two phased composites. XRD characterization showed the formation of nickel zinc ferrite spinel structure and perovskite barium titanate structure, without presence of secondary phases. SEM images at the free surface indicated the formation of two types of grain morphology: polygonal grains typical for NZF phase and rounded grains typical for BT phase, both nanosized.

Magnetic measurements of all sintered composites were carried out and presented in Fig. 1. Saturation magnetization moment decreases in comparison with pure NZF, because of non-magnetic barium titanate phase. Coercive field was higher for composites with regard to pure NZF, which can be explained with fact that the composite possesses a higher anisotropy field than the NZF at the same applied field. The fields at which saturation occur was almost the same for all materials

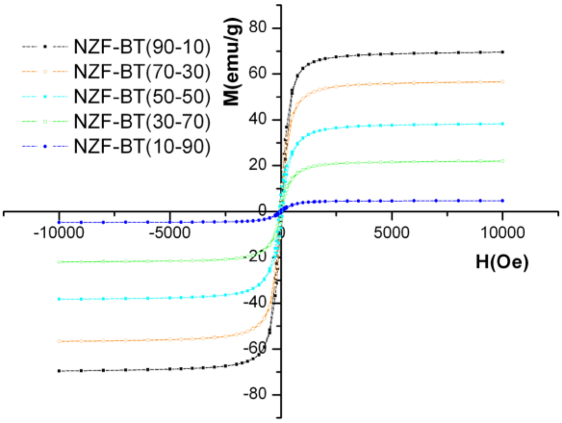

Figure 1. Magnetic measurements of $xNi_{0.7}Zn_{0.3}Fe_2O_4$*–*$(1\text{-}x)BaTiO_3$ *ceramics*

E15

FABRICATION OF $BaTiO_3$ THIN FILMS BY INKJET PRINTING

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Barium titanate in thin film form is one of the most investigated ferroelectric materials for microelectronics application. The properties of barium titanate thin films are dependent of many factors such as synthesis method, deposition technique, substrate selection, thermal treatment, etc. It has been a challenge to adjust these factors and obtain thin films with required microstructure and functional properties.

In this research, $BaTiO_3$ films were prepared with inkjet printing technique (Dimatix DMP 3000 and Epson XP-202 printer). Inkjet printing is one of the most popular method in thin film production. Although inkjet printing is simple and cheap technique, it is necessary to achieve requiered rheological properties of inks for successful printing process (viscosity, surface tension, particle size). Barium titanate inks were prepared with sol gel technique, where manipulation with rheological parameters is relatively easy. After examination of rheological parameters, inks were deposited on silicon substrates and calcinated at 750 °C. Microstructure of obtained films was investigated by scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction and Raman spectroscopy. In addtioin, $BaTiO_3$ thin films were prepared with spin coating method. Thus, advantages and disadvantages of the deposition techniques in production of sol gel prepared barium titanate thin films were investigated.

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