The Seventh Students' Meeting

PROCESSING AND APPLICATION OF CERAMICS

PROGRAMME and BOOK OF ABSTRACTS

SM-2007

Novi Sad
December 6-8, 2007

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Preface

The Seventh Students' Meeting, SM-2007, "Processing and Application of Ceramics", is organized by the Department of Materials Engineering, Faculty of Technology, University of Novi Sad. The Meeting will be held in Novi Sad, Serbia on December 6-8, 2007.

The idea of this kind of meeting appeared in 1998, when group of people from the Department of Materials Engineering gathered together and established the meeting which is today well recognized and greatly appreciated among the students and scientists from all over the Europe. The Students' Meeting started first as a national meeting, but with patient work and strong effort we succeeded to raise the quality up to the standards of today's International Meeting. The main goals of this traditional Meeting are the promotion of the work in the field of ceramics done by young researchers and closer international contacts between students from different universities and institutes, through exchange of knowledge, ideas and experience. Considering the aims and the significance it has, The Students' Meeting got the support from the European Ceramic Society.

Following the concept of the previous meeting, the students will have an opportunity to present their work in one of the four topics:

- Advanced Ceramics
- Ceramics Composites
- Traditional Ceramics
- Culture Heritage

All information related to The Seventh Students' Meeting, SM-2007, "Processing and Application of Ceramics" can be found on the web site:
http://tehnol.ns.ac.yu/www/dokumenta/Sm-2007/index.html

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PROGRAMME

THURSDAY, DECEMBER 6, 2007.

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10.00 – 10.30 h – Oppening

10.30 – 11.45 h

Section ***Ceramics Composites*** (Blue Hall)

10.30 – 10.45 h	**A1** – **J. Bezjak, et al., Slovenia** The synthesis and phase transitions of $Ba_4Nb_2O_9$ polymorphs
10.45 – 11.00 h	**A2** – **J. Suffner, et al., Germany** Vapor Phase Synthesis of Fluorine-doped SnO_2 Nanoparticles
11.00 – 11.15 h	**A3** – **L. Hegedusova, et al., Slovakia** Contact strength of Si_3N_4 and SiC materials
11.15 – 11.30 h	**A4** – **V. Lukac, et al., Czech Republic, Slovenia** Preparation potassium tantalate thin films through chemical solution deposition and their characterization
11.30 – 11.45 h	**A5** – **B. Vlad Oros, et al., Romania** Efficient entrapment of amylases by sol-gel technique in silica hybrid matrices

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13.30 – 14.45 h

Section ***Ceramics Composites*** (Blue Hall)

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13.45 – 14.00 h	**C2 – R. Smajda, et al., Hungary** Temperature response of carbon nanotube films modified with pyroelectric materials
14.00 – 14.15 h	**C3 – A. Maglica, et al., Slovenia** Preparation and properties of β-SiAlON/ZrN nano-composites from ZrO_2-coated Si_3N_4 powder
14.15 – 14.30 h	**C4 – S. Lojanova, et al., Slovakia** Effect of the rare-earth oxide additives on mechanical properties of Si_3N_4/SiC micro/nanocomposite
14.30 – 14.45 h	**C5 – O. Koszor, et al., Hungary** Processing, mechanical and thermophysical properties of silicon nitride based composites with carbon nanotubes and graphene

Section ***Advanced Ceramics*** (Class Room)

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13.45 – 14.00 h	**A7 – U. Lacnjevac, et al., Serbia** Electrochemical Deposition and Characterisation of Ni-Mo Powders
14.00 – 14.15 h	**A8 – O.A. Kornienko, et al., Ukraine** Interacon cerium oxide with zirconia, hafnia and lanthanids
14.15 – 14.30 h	**A9 – S. Turbinsky, et al., Belarus** The simulation of stress-deformed state of high-pressure apparatuses for the sintering of the Si_3N_4 ceramics by the finite element method
14.30 – 14.45 h	**A10 – O.V. Boytsova, et al., Russia** Thermal expansion of (Mg,Cu)O, (Mg,Ni)O and (Mg,Zn)O solid solutions with the rock-salt structure

14.45-15.15 h – Coffe Break

15.15 – 16.45 h

Section ***Traditional Ceramics & Culture Heritage*** (Blue Hall)

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15.30 – 15.45 h	**T2 – R. Adziski, et al., Macedonia** Fabrication of slag-glass composite with controlled porosity
15.45 – 16.00 h	**H1 – S. Kramar, et al., Slovenia** Study of sandstone deterioration of the historical buildings (Slovenia)
1600 – 16.15 h	**H2 – S. Petrovic, et al., Serbia, Macedonia** Historical materials from the medieval fortress Bac
16.15 – 16.30 h	**H3 – Nedučin, et al., Serbia** Genesis of international approach in protection of world heritage during 20th century – A review
16.30 – 16.45 h	**H4 – D. Cvetković, et al., Serbia, Italy** Determination of wall-painting techniques of Bodjani monastery

Section ***Advanced Ceramics*** (Class Room)

15.15 – 15.30 h	**A11 – M. Mikoczyova, et al., Slovakia** The influence of forming method on densification and final microstructure of submicrometre alumina ceramics
15.30 – 15.45 h	**A12 – K. Marinkovic, et al., Serbia** Morphological features of Y_2O_3:Eu particles obtained through twin fluid and ultrasonic atomization
15.45 – 16.00 h	**A13 – L.-P. Curecheriu, Romania** Tunability modelling and experiments for $BaTiO_3$- based solid solutions
16.00 – 16.15 h	**A14 – O. Frolova, et al., Ukraine** The injection molding processes simulation for manufacture ceramics
16.15 – 16.30 h	**A15 – M. Posarac, et al., Serbia** Thermal Shock Behavior of Nano-Spinels

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FRIDAY, DECEMBER 7, 2007.

09.00 – 10.30 h

Section ***Advanced Ceramics*** (Blue Hall)

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Effect of particles size on the structure and the properties of Si_3N_4 ceramics sintered at high pressures

09.15 – 09.30 h **A17 – M. Maletin, et al., Serbia, Greece**
Synthesis, stucture and magnetic properties of In-doped $ZnFe_2O_4$ as powder nanoparticles and dispersed in polymer matrix

09.30 – 09.45 h **A18 – J. Konig, et al., Slovenia**
Influence of axial pressure on dielectric properties of $Na_{0.5}Bi_{0.5}TiO_3$-$NaTaO_3$ ceramics

09.45 – 10.00 h **A19 – K. Đuriš, et al., Serbia**
Influence of Ca^{2+} and Sr^{2+} dopants on properties of $LaMnO_3$ prepared by polymerizable complex method

10.00 – 10.15 h **A20 – A.J. Darbandi, et al., Germany**
Synthesis of Nanocrystalline Strontium lanthanum manganite with high specific surface area via Spray pyrolysis technique

10.15 – 10.30 h **A21 – M. Ninic, et al., Serbia**
Synthesis of nanometric powders based on cerium oxide

Section ***Advanced Ceramics*** (Class Room)

09.00 – 09.15 h **A22 – Z. Dudas, et al., Romania, Hungary**
Hybrid silica xerogels containig microbial hydrolases

09.15 – 09.30 h **A23 – A. Egelja, et al., Serbia**
Synthesis and characterisation of biomorphic cellular SiC ceramics

09.30 – 09.45 h **A24 – M. Radovanovic, et al., Serbia**
Investigation of electrical parameters of nanostructured titania coatings deposited on interdigitated electrode system

09.45 – 10.00 h **A25 – C. Andronescu, et al., Romania**
Thermal and optical properties of PbI_2 intercalated with polyethylene glycol

10.00 – 10.15 h **A26 – M. Logar, et al., Slovenia**
Thin film fabrication by modified sol-gel process

10.15 – 10.30 h **A27 – S. Jankov, et al., Serbia**
Dielectric Properties of Nanosized $ZnFe_2O_4$

10.30-11.00 h – Coffe Break

11.00 – 12.30 h

Section ***Ceramics Composites*** (Blue Hall)

11.00 – 11.15 h	**C6 – L. Hirc, et al., Slovakia** Preparation of SiC based ceramic materials with unconventional sintering aids
11.15 – 11.30 h	**C7 – M. Daranjyi, et al., Hungary** Characterization of sandwiched coating layers consisting of titanate nanowires
11.30 – 11.45 h	**C8 – R.M. Nowak, et al., Poland** Influence of nitrogen on the tribological properties of a-C:H layers on the polycarbonate substrates
11.45 – 12.00 h	**C9 – G.P. Kysla, et al., Ukraine** Ceramics materials of quasibinary system LaB_6-MoB_2
12.00 – 12.15 h	**C10 – B. Feneyi, et al., Hungary** DC conductivity of silicon nitride based carbon-ceramic composites
12.15 – 12.30 h	**C11 – L. Kipsova, et al., Slovakia** Synthesis and Thermo-Mechanical Properties of $MgSiN_2$ and $LaSi_3N_5$

Section ***Advanced Ceramics*** (Class Room)

11.00 – 11.15 h	**A28 – M. Pocuca, et al., Serbia** The influence of the thermal treatment conditions on morphology and orientation of LNO thin films
11.15 – 11.30 h	**A29 – E. Bartonichova, et al., Czech Republic** Ultrasound and microwave synthesis of bismuth oxide for catalytic application
11.30 – 11.45 h	**A30 – P.P. Varga, et al., Hungary** Function of Magnesium Nitrate Dope in the Production of Polycrystalline Alumina Ceramics
11.45 – 12.00 h	**A31 – K. Vojisavljevic, et al., Serbia** The impedance analysis of the zinc oxide ceramics
12.00 – 12.15 h	**A32 – M. Perusic, et al., Bosnia & Herzegovina** The Influence of Crystal Admixture on Zeolite Quality Production
12.15 – 12.30 h	**A33 – S. Ianosev, et al., Romania** Cordierite synthesis ($2MgO.2Al_2O_3.5SiO_2$) by unconventional methods

12.30-15.30 h – Novi Sad bus-tour, Visiting of labs. & Lunch

15.30 – 17.00 h

Section ***Advanced Ceramics*** (Blue Hall)

15.30 – 15.45 h	**A34 – R. Djenadic, et al., Germany** Chemical Vapor Synthesis of Nanocrystalline Anatase (TiO_2) - Time-Temperature Profile Influence on Powder Characteristics
15.45 – 16.00 h	**A35 – M. Zarzecka-Napierała, et al., Poland** Comparison of the yttrium aluminium garnet (YAG) nanopowder preparation methods
16.00 – 16.15 h	**A36 – M. Vijatovic, et al., Serbia** Effect of synthesis method on $BaTiO_3$ properties
16.15 – 16.30 h	**A37 – E. Eurnal, et al., Germany** Structural characterization of Cu^{2+} and Fe^{3+} functional centers in 'lead-free' $K_yNa_{1-y}NbO_3$ piezoelectrics
16.30 – 16.45 h	**A38 – A.V. Kapylou, et al., Belarus** Effect of High Pressure Sintering temperature on microstructure and physical-mechanical properties of TiB_2 ceramics
16.45 – 17.00 h	**A39 – Z. Escedi, et al., Romania** Synthesis of Mesoporous Alumina using Polyvinil-alcohol as Porosity Control Additive

Section ***Ceramics Composites*** (Class Room)

15.30 – 15.45 h	**C12 – I. Khobta, et al., Ukraine** Synthesis of composite TiN-TiB_2 by reactionary electric discharge sintering method
15.45 – 16.00 h	**C13 – M.-R. Chirita, et al., Romania** Ceramic Micropackages for MEMS Application
16.00 – 16.15 h	**C14 – M. Mrazova, et al., Czech Republic** The preparation of dental raw materials with controlled fraction of leucite crystals
16.15 – 16.30 h	**C15 – G. Kozma, et al., Hungary** Mechanochemical synthesis of mixed metal oxide phases
16.30 – 16.45 h	**C16 – O.A. Tokarev, et al., Ukraine** The structure and mechanical properties of multilayer nanocrystalline TiN/ZrN condensates obtained by vacuum-arc deposition
16.45 – 17.00 h	**C17 – R. Geber, et al., Hungary** Investigation of mineral fillers for the utilization opportunities in the road construction

20.30 h – Social Event

SATURDAY, DECEMBER 8, 2007.

09.30 – 11.00 h

Section ***Advanced Ceramics*** (Blue Hall)

09.30 – 09.45 h	**A40 – L. Gal, Hungary** Synthesis of ferrites in radiofrequency thermal plasma reactor
09.45 – 10.00 h	**A41 – S. Mashuga, et al., Ukraine** Synthesis and properties of $BaTiO_3$/Ni nanocomposite
10.00 – 10.15 h	**A42 – Z. Zivcova, et al., Czech Republic** Alumina ceramics prepared with new pore-forming agents
10.15 – 10.30 h	**A43 – A. Kocjan, et al., Slovenia** The preparation of Al_2O_3 coatings on ZrO_2 ceramic substrates and their influence on the adhesion with luting cement
10.30 – 10.45 h	**A44 – A. Stankovic, et al., Serbia** Synthesis of ZnO nanocrystal s trough surfactant-assisted mechanochemical process
10.45 – 11.00 h	**A45 – I. Stijepovic, et al., Serbia, Romania, Lithuania** Structural and functional characterization of doped $LaGaO_3$

Section ***Traditional Ceramics*** (Class Room)

09.30 – 09.45 h	**T3 – M. Arsenovic, et al., Serbia** Additives in clay brick industry: Porosity improving
09.45 – 10.00 h	**T4 – V. Jovanov, et al., Macedonia, Serbia** Biocorrosion of composite materials based on fly ash and clay materials
10.00 – 10.15 h	**T5 – C. Paroczai, et al., Hungary** Magnetic cleaning of the Glass Industry's Raw-Materials
10.15 – 10.30 h	**T6 – R. Rekecki, et al., Serbia** Design of ceramic microstructures based on waste materials
10.30 – 10.45 h	**T7 – B. Angjuseva, et al., Macedonia** Dense ceramics materials obtained from fly ash
10.45 – 11.00 h	**T8 – M. Hadnadjev, et al., Serbia** Investigation of photocatalytic activity on clay roofing tile

11.00-11.30 h – Coffe Break

11.30 – 13.00 h

Section ***Ceramics Composites & Advanced Ceramics*** (Blue Hall)

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11.45 – 12.00 h	**C18 – O. Sych, et al., Ukraine** Biocomposites based on calcium phosphates: preparation and properties
12.00 – 12.15 h	**C19 – Y. Kismir, et al., Turkey, Serbia** Thermal stability of chitosan/bentonite nanocomposites
12.15 – 12.30 h	**C20 – Y. Lebedov, et al., Ukraine** Microstructure and dielectric properties bechavior of Si_3N_4-SiC hot pressing composite
12.30 – 12.45 h	**A46 – L. Brazda, et al., Czech Republic** Kinetics of dissolution of calcium phosphate (Ca-P) bio-ceramics
12.45 – 13.00 h	**A47 – I.M. Szilagyi, et al., Hungary** Controlling the composition of nanosize h-WO_3 for gas sensors

Section ***Advanced Ceramics*** (Class Room)

11.30 – 11.45 h	**A48 – D. Demyrskyi, et al., Ukraine** Microwave sintering of ceramics
11.45 – 12.00 h	**A49 – R. Filipovic, et al., Bosnia & Herzegovina** Posibility of aluminate cements production by ablation in alumina production process
12.00 – 12.15 h	**A50 – R. Dzakula, et al., Serbia** Investigation of electrical characteristics of different ceramic samples using Hall effect measurement
12.15 – 12.30 h	**A51 – K. Kozhemyachenko, et al., Russia** Investigations of oxygen permeability in doped strontium cobaltites
12.30 – 12.45 h	**A52 – Z. Lazarevic, et al., Serbia** Microstructure development of ceramic powders with perovskite structure prepared by mechanochemical synthesis

13.00 h – Cloasing of the Meeting

ADVANCED CERAMICS

A1

X-RAY DIFFRACTION ANALYSES OF THE POLYMORPHIC PHASE TRANSITIONS OF $Ba_4NB_2O_9$

J. Bezjak, B. Jančar, D. Suvorov

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Recently, B-site cation-deficient hexagonal perovskites, containing mixed cubic hexagonal stacking sequences of AO_3 layers along $[0001]_H$ have started to attract attention due to their low dielectric losses and their high quality factor at microwave frequencies. Studies of hexagonal perovskites within the $BaO–WO_3–Nb_2O_5$ ternary system have revealed similarities in the X-ray powder-diffraction spectra between the ternary compound $Ba_6WNb_2O_{14}$ and the binary compound $Ba_4Nb_2O_9$. It was supposed that $Ba_6WNb_2O_{14}$ crystallizes in a structure similar to that of $Ba_6WTa_2O_{14}$ (P3m1), an already known hexagonal compound. However, refining the assumed structure using the data of $Ba_6WTa_2O_{14}$ showed the necessity to use the modified P3m1 structural model, as some of the reflections were not refined. In order to understand the crystal chemistry governing the formation of $Ba_6WNb_2O_{14}$ the $Ba_4Nb_2O_9$ ceramic was prepared with a conventional solid-state reaction and analyzed by means of X-ray diffraction and scanning electron microscopy. From the inspection of the diffraction data three modifications related to two reversible phase transitions below 1200°C were isolated.

A2

VAPOR PHASE SYNTHESIS OF FLUORINE-DOPED SnO_2 NANOPARTICLES

Jens Suffner[1], Jens Kling[2], Horst Hahn[1,3]

[1]*Joint Research Laboratory Nanomaterials, FZ Karlsruhe, Germany*
[2]*Structural Research Division, TU Darmstadt, Darmstadt, Germany*
[3]*Institute of Nanotechnology, FZ Karlsruhe, Germany*

Novel electro-optic applications like solar cells, light emitting diodes, and displays require transparent conducting materials as front electrodes. Wide band gap oxides like tin oxide (doped with fluorine or antimony) or indium oxide (doped with tin oxide) are utilized as transparent conducting oxides (TCO). The emergence of printable electronics uses dispersions of nanopowders applied via inkjet printed. We present the preparation of nanoscale SnO_2 powders by Chemical Vapor Synthesis (CVS) starting from tetramethyltin. Doping the particles with fluorine could be obtained by feeding particles are well crystalline, owing an average particle size of about 6-7 nm as obtained

difluoromethane together with the oxide precursor to the hot wall reactor. The formed from XRD line broadening, transmissions electron microscopy, and Brunauer-Emmett-Teller (BET) surface area analysis. The structure of the formed particles has been studied by FT-IR spectroscopy and X-ray photoelectron spectroscopy (XPS). The Sn/F ratio can easily be adjusted by controlling the mass flow rate of difluoromethane as determined by XPS. The influence of the doping on the optical properties has been measured by UV/VIS-Spectroscopy.

A3

CONTACT STRENGTH OF Si_3N_4 AND SIC MATERIALS

L. Hegedüsová, J. Dusza

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The paper deals with an investigation of strength of Si_3N_4 and SiC based ceramics using two measuring techniques to include the opposite-sphere contact and four-point bending strength tests. The Si_3N_4 ceramics exhibits higher characteristic strength within the both techniques along with a significant difference compared to the SiC ceramics within the bending test. With regard to the Weibull's modulus, experimental results of the contact test for the investigated materials are mutually similar. Considering the bending mode, the Weibull's modulus of SiC ceramics is significantly lower compared to that of Si_3N_4 ceramics. Additionally, cone cracks with similar size, studied by the optical microscopy, are formed during the contact strength test to control and the Weibull's parameter and fracture. Lower radius of the spheres results in longer cone cracks and lower angle between the cracks and a sample surface, and vice versa. Finally, the Weibull parameter within the bending test is controlled by volume defects to result in significant strength degradation exhibiting mainly in the SiC ceramics.

A4

POTASSIUM TANTALATE THIN FILMS PREPARED THROUGH CHEMICAL SOLUTION DEPOSITION

Viktor Lukáč[1], Josef Buršík[1], Maxim Savinov[2], Přemysl Vaněk[2], Radomir Kužel[3], Andrea Bencan Golob[4]

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[2]*Institute of Physics, Academy of Sciences, Prague, Czech Republic*
[3]*Charles University, Faculty of Mathematics and Physics, Prague, Czech Republic*
[4]*Jozef Stefan Institute, Ljubljana, Slovenia*

Potassium tantalate (KT) $KTaO_3$ thin films of both pyrochlore and perovskite cubic structures were prepared from a solution of potassium iso-butoxide and tantalum iso-butoxide in absolute iso-butanol with an additive of diethanolamine as a stabilizer through the chemical solution deposition (CSD) on platinized Si(100) substrates. The effects of K:Ta sol stoichiometry, reaction atmosphere and annealing regime on the kinetics of pyrochlore to perovskite transition has been studied by means of powder XRD, SEM, TEM, DTA/TG and FTIR. Optimum conditions for perovskite phase crystallization are using oxidizing atmosphere, rapid thermal annealing modus and crystallization at temperatures higher then 700°C. The measurements of dielectric dispersion confirm the character of material to be incipient ferroelectrics and were comparable with the results gained on KT single crystal.

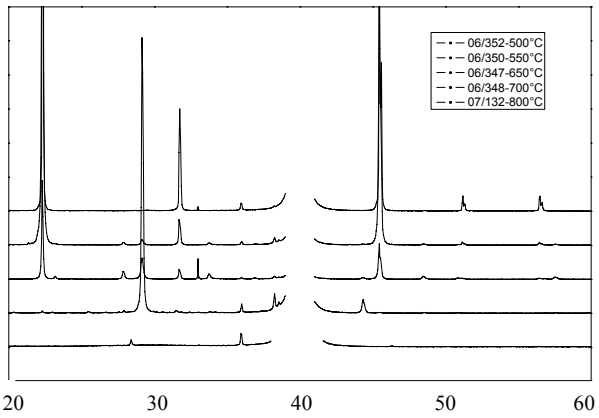

Figure 1. Powder XRD patterns of KT films heat treated at different temperatures.

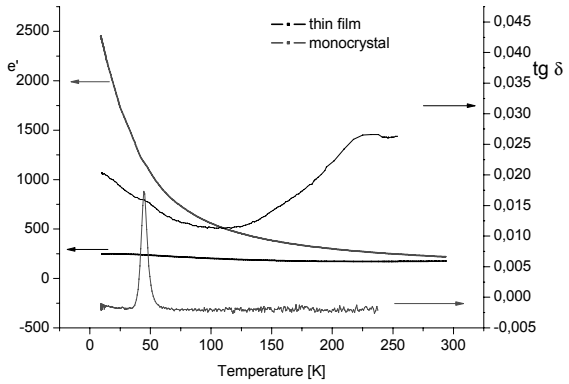

Figure 2. Room temperature frequency dispersion curve.

A5

EFFICIENT ENTRAPMENT OF AMYLASES BY SOL-GEL TECHNIQUE IN SILICA HYBRID MATRICES

B. Vlad-Oros[1], G. Preda[1], Z. Dudas[1], M. Dragomirescu[2], A. Chiriac[1]

[1]West University of Timisoara, Faculty of Chemistry, Biology, Geography, Dep. of Chemistry & [2]Banat University of Agricultural Sciences and Veterinary Medicine, Faculty of Animal Science and Biotechnologies, Timisoara, Romania

An enzyme widely used in fermentative industry, amyloglucosidase (AMG), was used to prepare enzyme-doped silica powders and to investigate the effect of silica as host matrix on enzyme kinetics. The biomaterials were prepared by the sol-gel method, using different alkoxysilane, in different ratios, as precursors. Encapsulated enzymes followed Michaelis-Menten kinetics and maintained good catalytic activity. The Michaelis constant (KM) and the maximum rate of starch hydrolysis reaction (Vmax) were calculated according to the Michaelis-Menten and Lineweaver-Burk plots. The values of the Michaelis constant (KM) of the encapsulated enzymes were higher than that of the free enzyme, indicating the presence of partitioning and diffusional effects in the pores of the sol-gel matrix. The temperature and pH influence on the activity of free and immobilized amyloglucosidase were also compared. The results of this study show that the enzyme immobilized in organic/inorganic hybrid silica matrices, by the sol-gel method, are suitable for many different applications (medicinal, clinical, analytical) allowing the entrapped amyloglucosidase to retain its whole biological activity.

A6

A NOVEL CERAMIC MATERIAL WITH MEDICAL APPLICATION

Joanna Podporska, Marta Błażewicz, Barbara Trybalska

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Wollastonite ($CaSiO_3$) is a ceramic, bioactive material used in bone tissue regeneration. Up till now the basic methods used in wollastonite manufacturing have been chemical (melting together with glass crystallization process, chemical coprecipitation) and sol – gel methods.

A new, and promising way of fabrication of wollastonite-containing ceramics is controlled pyrolysis of polysiloxane precursors with inorganic fillers. Heat treatment of such mixtures (appropriate type of polymeric precursor, fillers and the final heat treatment temperature) leads to the formation of wollastonite-containing ceramic material already at about 1000^{o}C [1,2]. This is a relatively inexpensive and efficient method which enables to obtain complex shapes of the samples.

The aim of this work was to obtain sintered, wollastonite-containing bioceramics and determine its bioactive features. The material was manufactured by thermal treatment of polisiloxane resin with inorganic fillers ($Ca(OH)_2$ and nano-grain sized silica). Obtained powders were grounded and then sintered at three different temperatures. Then the samples were investigated by the "in vitro" bioactivity test in simulated body fluid (SBF).

Samples were characterized by the FTIR, XRD and SEM-EDS methods. On the basis of the achieved results, it can be assumed that the obtained material possesses bioactive features and can be the potential material for hard tissue regeneration application.

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A7

ELECTROCHEMICAL DEPOSITION AND CHARACTERIZATION OF Ni-Mo POWDERS

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Electrodeposition of Ni-Mo alloy powders of different compositions has been performed from the solutions containing either ammonium sulphate or ammonium chloride as a supporting electrolyte (1 M). The concentration ratio of Ni/Mo varied from

1/6 to 2/1, with the concentration of nickel ions being 0.1 M. Polarization curves were recorded with IR drop compensation and powders were deposited at the values of limiting current density.

The deposited powders were investigated by SEM, EDAX and X-ray techniques. It is shown by SEM analysis that such powders contain mainly cauliflower like agglomerates of the size varying from 10 μm to 400 μm. EDAX analysis showed the presence of 5 - 20 at.% Ni, 15 - 35 at.% Mo and 50 - 65 at.% O, depending on the alloy powder composition (i.e. Ni/Mo ratio). X-ray analysis confirmed nano-crystalline deposit, since no peaks on the diffractograms of as deposited powders could be detected. DTA analysis confirmed that the recrystallization temperature is about 585^{o}C. In order to increase crystallites, powders were kept in the furnace at 600 0C for 2 h in the atmosphere of N_2. After such treatment X-ray analysis showed that the main phase in the powders is $NiMoO_4$, which was present in all investigated powders. Among this phase a small amount of $Ni_2Mo_3O_8$ phase was detected in the powders obtained at the Ni/Mo ratio 1/1. These two phases were present in the powders deposited from the solution containing Ni/Mo ratio equal or lower than 1/3. With increasing the Ni/Mo ratio in the solution (1/1 and 2/1) the amount of MoO_3 in the powders also increased. After recristallization well defined crystals were detected by SEM for all investigated samples.

A8

INTERACTION CERIUM OXIDE WITH ZIRCONIA, HAFNIA AND LANTHANIDES

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Phase equilibria in the ternary systems ZrO_2-HfO_2-CeO_2 and ZrO_2-CeO_2-La_2O_3 as well as phase relations in the boundary binary system CeO_2-La_2O_3 were studied at 1500°C in air in the whole concentration range. X-ray diffraction and electron microprobe X-ray diffraction and petrography were used to determine phase contents. The microstructures of the sintered ceramic samples were examined by using the scanning electron microscopy (SEM). The structure of the boundary binary systems defines the phase equilibria in the ternary systems.

Solid solutions based on tetragonal (T) ZrO_2, cubic (F) CeO_2 were revealed to form wide phase fields and narrow ones of monoclinic (M) HfO_2 in the ZrO_2-HfO_2-CeO_2 system (1500°C). In the subsolidus area of the phase diagram ZrO_2-CeO_2-La_2O_3 (1500^{o}C) solid solutions based on tetragonal ZrO_2 and cubic with fluorite-type structure CeO_2, hexagonal La_2O_3, as well as intermediate phase with pyrochlore-type structure $La_2Zr_2O_7$ were determined. Hexagonal (A) and cubic (F) solid solutions based on lanthana and ceria are in equilibrium at 1500^{o}C in the boundary binary system CeO_2-La_2O_3. The ceria solubility in hexagonal lanthana is around 20 mol %. The lanthana solubility in the F-CeO_2 was found to be around 50 mol %. The isothermal sections at 1500^{o}C for the ternary systems ZrO_2-HfO_2-CeO_2 and ZrO_2-CeO_2-La_2O_3 were developed.

A9

THE SIMULATION OF STRESS-DEFORMED STATE OF HIGH PRESSURE APPARATA FOR THE SINTERING OF Si_3N_4-BASED CERAMICS

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High pressure sintering is an effective method for the obtaining of highly dense ceramics based on high-melting point compounds, for example, silicon nitride. For sintering various constructions of high pressure apparatus (HPA) are used. In many respects their characteristics determine the properties of sintered ceramics and its production reliability and profitability. "Anvils with cavities" HPA type are simplest and convenient. As a rule the operating efficiency of HPA and the service time are determined by its constructive features and the character of stress-deformed state of hard alloy anvils during device exploitation.

In order to optimize the elements of the construction and to decrease the level of stresses in most loaded sites the simulation of stress-deformed state of HPA anvils used by us for the sintering of the ceramics based on the silicon nitride has been done. Finite element method and the software developed by us are used for the calculations. The distribution of stresses and deformations in volume and on surface of the anvil of various materials, the hard alloy WC - 6% Co and P6M5 steel, is studied. Different profiles of the active surface of the anvils are reviewed and the effect of curvature radius of the bottom and the cavity side surface coupling on the stress distribution in the anvil is investigated.

The zones with highest level of breaking stresses are determined and the dependencies of extreme equivalent stresses on anvil geometric parameters are obtained at various states of HPA: "assembly", "loading" and "unloading" of HPA. It has been shown that due to the changing of the cavity profile, it is possible to decrease the level of stresses in HPA anvils in two times. It allows increasing their service time. It makes it possible to employ the steel anvils instead of the hard alloy ones and provides the expenses decrease for manufacturing silicon nitride ceramic products.

A10

THERMAL EXPANSION OF (Mg,Cu)O, (Mg,Ni)O AND (Mg,Zn)O SOLID SOLUTIONS WITH THE ROCK-SALT STRUCTURE

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The mismatch of thermal expansion coefficients (TEC) of different materials in layered structures often leads to destruction of system integrity (cracking, delamination, etc.), if temperature is varied. This poses a serious problem in technology of solid oxide fuel cells, thermal barrier coating and other important material classes. The problem appears also in emerging field of superconducting oxide coatings on long flexible textured nickel alloy tapes, since such tapes undergo a sequence of high temperature deposition processes (> 600°C) followed by the practical use at temperature below 100K. MgO is often used as a buffer layer between the metal substrate and superconducting coating. Its TEC is approx. 20-30% lower than that of a metal tape, that may lead to crack formation under heating. We have tried to increase the TEC of the buffer layer material by doping of MgO with CuO, NiO and ZnO. The approach is based on the well known empirical relation between TEC and the melting point: the lower is the melting point, the higher is the TEC. The increasing content of dopant in MgO causes gradual decrease of the melting point of solid solution and makes it possible to modify the coefficient of linear thermal expansion. Investigated materials were solid solutions of $Mg_{1-x}Cu_xO$ with x = 0-0.2 and $Mg_{1-x}Zn_xO$ (x = 0-0.5). Sintered ceramics have been characterized by XRD, electron microscopy and EDX. Dilatometry and high-temperature X-ray diffraction were used for CTE measurements. The dependence of TEC on composition of these solid solutions will be discussed.

A11

THE INFLUENCE OF FORMING METHOD ON DENSIFICATION AND FINAL MICROSTRUCTURE OF SUBMICROMETRE ALUMINA

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Conditions of green body preparation and consequently the conditions of sintering are prerequisite for the preparation of dense samples of Al_2O_3 with superior optical and mechanical properties. The goal of this work was determination of forming conditions for preparation of green body, and to find out optimal sintering regime facilitating the preparation of ultra-fine grained high purity alumina with maximal density, the finest microstructure and the lowest pore size.

A12

MORPHOLOGICAL FEATURES OF Y_2O_3:Eu PARTICLES OBTAINED THROUGH TWIN FLUID AND ULTRASONIC ATOMIZATION

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Modern display devices such as plasma display and field emission display employ advanced Eu-doped yttrium oxide material, a well-known red phosphor. Utilization in such devices requires particles with spherical shape, narrow size distribution and non-aggregation characteristics since this ensures high resolution and improved brightness. Spray pyrolysis is a feasible method for obtaining the needed phosphor particle characteristics in view of the fact that atomized precursor solution, when fed into furnace, leads to the successive solvent evaporation, drying, solute precipitation and chemical decomposition on a droplet level, ensuring the formation of particles with required compositional and structural characteristics. Atomization process can be established by two different principles: disintegration of liquid jet by an air flow in case of twin-fluid atomizers and by liquid sonication. The latter phenomenon occurs when a liquid is placed on a smooth surface that is set into vibrating motion perpendicular to the surface. When the amplitude of the underlying vibration increases to the critical value the generated standing waves start to collapse and tiny drops of liquid are ejected from the top of the degenerating waves normal to the atomizing surface.

In this work yttrium oxide doped with 5 at% of europium was directly prepared by spray pyrolysis at 900°C and the effect of the type of atomization process on the

morphology and the size of the particles was investigated by using RBI's ultrasonic atomizer (frequency 1.3 MHz) and a TSI 3079 Model twin-fluid atomizer. Under the frequency of 1.3 MHz generated droplets have the mean size around 3 μm. In general it is considered that droplets produced by ultrasonic atomization have a relatively narrow size distribution and smaller particle size than twin-fluid, but depending on the specific nozzle and the type of liquid delivery system employed in twin-fluid atomization submicronic droplets can be generated as well. The twin-fluid used in this work uses a compressed air atomizer with a stainless-steel twin-stream injection nozzle producing a polydisperse aerosol with a mean droplet diameter of 0.3 μm. Since the properties of advanced materials significantly depend on particle size, shape and morphology it is of great interest to find out the correlation between those characteristics and processing parameters. Detail characterization of produced powders was carried out by means of XRD, SEM/EDS and TEM analysis.

A13

TUNABILITY IN $BaTiO_3$-BASED SOLID SOLUTIONS: MODELLING AND EXPERIMENT

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The ferroelectrics show a strong nonlinearity under the electrical field. In the last few years, electric field-tunable dielectrics have attracted much interest for their potential applications as variable capacitors, phase shifters, tunable filters and voltage-controlled oscillators [1], particularly in circuits and devices needed by the wireless communications industry, for scientific, space, commercial and military use. The electric field-induced tunability describes the ability of a material to change its permittivity by the electric field.

In the present work, the dc-electric field dependence of the permittivity ε_r (E) in polar dielectrics was theoretically studied and compared with experimental data of some $BaTiO_3$-based solid solutions. The Landau-Ginzburg-Devonshire (LGD) theory and its approximate treatments (Johnson's relation) in case of a single polarization mechanism in dielectrics were firstly used [2]: ε_r (E) was calculated by using the Johnson's relation, the even-power equation, and the LGD theory (Fig. 1, a). The experimental results for $BaTiO_3$-based ceramics were fitted with these models and a good agreement was obtained, particularly at low fields. The experimental dependence in $Ba(Zr,Ti)O_3$ ceramics is well described by the Johnson eq. (Fig.1 b). By using the fitting results, the outputs of circuits containing tunable ferroelectrics can be simulated.

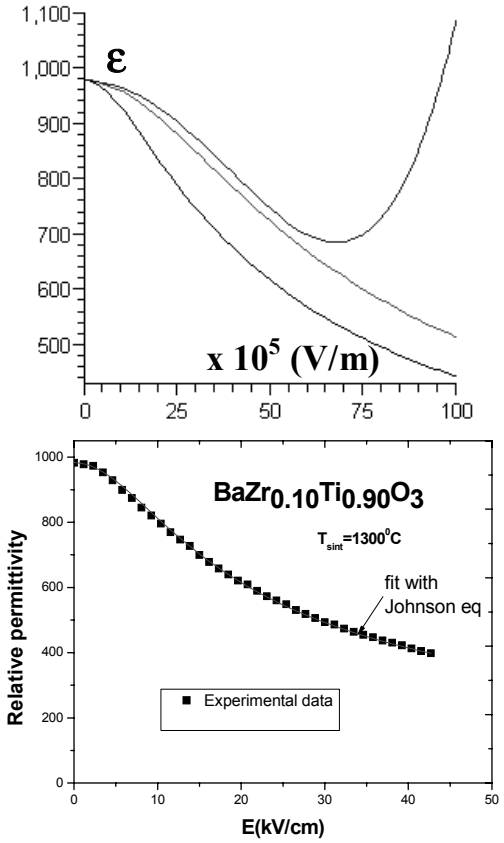

Figure 1. (a) Simulation of the field-dependence of the permittivity with eqs.: Johnson (blue), even-power (red), LGD (black); (b) Experimental data for BZT fitted with Johson's relation.

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A14

THE INJECTION MOLDING PROCESSES SIMULATION FOR CERAMICS MANUFACTURE

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Today the injection molding is considered to be the most effective of the powder metallurgy technology for the manufacture of high-precision items with small dimensions. This technology permits producing parts of complicated shapes with large as well as small dimensions from almost all types of powder materials including metal, ceramics and composite materials. Actually the injection molding is the only technology that allows manufacturing the parts of complicated shapes from structural ceramics. The ceramic parts, obtained by the injection molding, are used in many branches of industry such as automotive, chemical, aerospace, equipment facilities, hardware support, military, electrical engineering and electronics.

One of the steps of the injection molding process, that is the feedstock injection into the complicated shape mould, is examined. The feedstock is considered as a microheterogeneous medium where the matrix is linear or nonlinear viscous phase and the filler is a lot of solid incompressible particles. The feedstock flowing is studied using the model of nonlinear viscous flowing with the shear viscosity coefficient. This coefficient depends on the solid phase concentration and depends nonlinearly on the shear strain velocity. In contrast to other investigations we succeeded in connecting of the velocity sensibility of the shear viscosity coefficient of the binder-ceramic powder system with the powder concentration. Using the Finite Element Method the field of feedstock flowing velocities with the nonlinear shear viscosity coefficient for different powder concentrations has been obtained. On the first step during feedstock flowing simulation the effective viscosity coefficient was calculated for the different concentrations of the powder phase. Using this result as an initial viscosity we have studied the feedstock behavior as a uniform material on the second step.

Analyzing the obtained viscosity field we can give indirect recommendations about initial parameters of the feedstock flowing for the best solid particle distribution in the liquid phase. It is an important question for manufacturing of high-quality production.

A15

THERMAL SHOCK BEHAVIOR OF NANO-SPINELS

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This work deals with synthesis and properties of nano-spinels obtained by self propagation room temperature method (SPRT) and modified glycine nitrate procedure (MGNP). Behavior of the nano-spinels powders during the heating process was monitored by thermal microscopy, X – ray powder diffraction (XRD), scanning electron microscopy (SEM) and differential thermal analysis (DTA). Sintering parameters such as shrinking, expansion, softening, melting as well as phase composition were determined. Behavior of sintered samples after thermal shock treatments was investigated. Thermal shock of the samples was measured using standard laboratory procedure, water quench test. Level of surface deterioration before and during quenching was monitoring by image analysis. Dynamic Young modulus of elasticity and strength degradation were determined by ultrasonic measurements.

A16

EFFECT OF PARTICLES SIZE ON THE STRUCTURE AND THE PROPERTIES OF Si_3N_4 CERAMICS SINTERED AT HIGH PRESSURES

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Silicon nitride is the perspective composition for the producing of high-strength heat-resistant ceramic materials used as a cutting tool or as a construction delail. It is perspectively to produce high-dense and high-strength ceramic materials of silicon nitride without additives under high pressures and high temperatures. High pressure sintering intensifies the sintering process and provides a small-flake structure to the obtained material. It provides the increasing of the physical mechanical characteristics.

The effect of particles size of starting powder on the microstructure, the density, the microhardness, the fracture toughness and the lattice parameters of specimens sintered at the pressures up to 4.0 GPa and at the temperatures up to 2000 °C have been

studied at this paper. Powders of Si_3N_4 with particles size of 50 nm to 400 nm have been used as the starting powders.

The sintering has been carried out in the high pressure apparatus of anvils with cavities type. The composites structure has been studied by XRD and SEM. The dependences of physical mechanical properties of Si_3N_4 on particles size of starting powder are defined and the sintering kinetic and the kinetic of $\alpha \rightarrow \beta$ polymorphic transformation in Si_3N_4 are studied. The obtained results are being analysed and compared with the literature data of properties of Si_3N_4 produced by another methods.

A17

SYNTHESIS AND STRUCTURE OF FERRITE POLYMER NANOCOMPOSITES

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Magnetic nanoparticles and their unique properties make them attractive, both from the scientific value of understanding their properties and the technological significance of enhancing the performance of the existing materials. Therefore synthesis and application of magnetic nanoparticles is a subject of intense research. Our interest is in developing a convenient method to incorporate ferrite nanoparticles in polymer matrix in order to evaluate their structure and magnetic behavior. In this paper, a procedure for the incorporation of nanocrystalline In-doped $ZnFe_2O_4$ in polystirene (PS) matrix via in situ polymerization will be presented. The structure of ferrite-PS nanocomposite will be characterized by differential scanning calorimetry (DSC), X - ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

A18

INFLUENCE OF AXIAL PRESSURE ON DIELECTRIC PROPERTIES OF $Na_{0.5}Bi_{0.5}TiO_3$–$NaTaO_3$ CERAMICS

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$Na_{0.5}Bi_{0.5}TiO_3$ is one of the most interesting lead-free ferroelectric materials due to its unique and diverse phase transitions. Studies of the influence of mechanical forces on the electrical properties of $Na_{0.5}Bi_{0.5}TiO_3$ have shown that the dielectric constant decreases under an applied axial pressure. The reduction of the permittivity is the largest

in the region of the dielectric maximum and is much smaller at room temperature, which is connected with the large mechanical coercive field, closely linked with the electrical coercive field (73 kV/cm). The pressure's influence on permittivity can be increased by an appropriate choice of modifying material. In our study we investigated the influence of $NaTaO_3$ additions on the properties of $Na_{0.5}Bi_{0.5}TiO_3$.

The axial pressure dependence of permittivity of the materials from the $Na_{0.5}Bi_{0.5}TiO_3$ –$NaTaO_3$ solid-solution system will be presented. With the addition of $NaTaO_3$ the pressure dependence of the permittivity increases as the dielectric anomalies are shifted towards lower temperatures and the coercive field is lowered. The maximum of the pressure dependence of the permittivity was achieved in a sample with 15 mol% of $NaTaO_3$. The dielectric properties of the materials are time dependent, and are not fully reversible when the pressure is removed. However, after annealing the samples at 600°C the initial dielectric properties are recovered.

A19

INFLUENCE OF Ca^{2+} AND Sr^{2+} DOPANTS ON PROPERTIES OF $LaMnO_3$ PREPARED BY POLYMERIZABLE COMPLEX METHOD

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Substituted lanthanide manganites with the general formula $A_{1-x}B_xMnO_3$ (A=La, B=Ca, Sr…) have attracted a lot of attention due to its colossal magneto-resistance properties, but also as good conducting materials and catalysts. In this work, pure and Ca^{2+}, Sr^{2+}-doped $LaMnO_3$ (LMO) was synthesised with polymerizable complex method, starting from lanthanum and manganese citrates, calcium acetate and strontium nitrate. Three compositions of doped manganites were prepared with the following concentrations of dopants: 30% Ca^{2+} (LCMO); 30% Sr^{2+} (LSMO); and 15% Ca^{2+} + 15% Sr^{2+} (LCSMO) (in mol. %). After drying, the gel was further calcined at 800°C for 2h in O_2 or air atmosphere, according to TG/DTA results. Obtained powders were characterised using XRD, BET and SEM analysis, and stoichiometry was confirmed by ICP/AS analysis. Powders were pressed and sintered at 1350°C for 4h. Grain size of sintered samples was determined using SEM, and chemical composition was investigated using EDS analysis. It was found that dopants have great influence on densification and morphology. LMO and LCSMO sintered samples showed dense structure with regularly shaped grains, but LCMO and LSMO were porous, with non-defined grain boundaries. The influence of dopants on magnetization and ferromagnetic phase transition was studied on powders and sintered samples in the field cooled (FC) and zero-field cooled (ZFC) conditions.

A20

SYNTHESIS OF NANOCRYSTALLINE STRONTIUM LANTHANUM MANGANITE WITH HIGH SPECIFIC SURFACE AREA VIA SPRAY PYROLYSIS TECHNIQUE

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Among the different cell components of Solid Oxide Fuel Cell, till today, Sr-doped lanthanum manganite (LSM) is considered as the preferred cathode material for the modern high-temperature SOFC. The electrochemical properties of LSM cathode layers depend largely on the microscopic features of the triple point boundaries (TPB), which in turn depend on the starting powder characteristics such as particle size, shape, and their distribution. Hence, optimization of the structure and composition of the cathode layer is of prime importance in enhancing the electrochemical performance of the cathode and thereby the overall performance of the cell.

Nanocrystalline, single phase, strontium lanthanum manganite (LSM) powder was synthesized by a unique spray pyrolysis process of aqueous precursor solutions for solid oxide fuel cell applications. Optimization of operational synthesis parameters and precursor has facilitated the synthesis of LSM powder with higher specific surface area containing new morphology compared to conventional LSM powder. Nanocrystalline powder has been characterized by TG/DTA, In situ-XRD, ICP, HRSEM, nitrogen adsorption. Formation of the perovskite structure in amorphous powder has been studied both by in-situ X-ray diffraction and thermogravimetric analyses. Reduction of specific surface area and crystalline growth of synthesized powder has been investigated after various heat treatments by nitrogen adsorption and in-situ-XRD.

A21

SYNTHESIS OF NANOMETRIC POWDERS BASED ON CERIUM OXIDE

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Nanometric powders of solid solutions of cerium oxide were obtained by modified glycine nitrate procedure (MGNP). Solid solutions of the host compound of cerium oxide - $Ce_{1-x}Me_xO_2$ - with one or more dopants in the lattice were synthesized. Rare earths cations (Me = Yb, Gd, and Sm) as dopants, were added to ceria in total concentration x = 0.2 that was kept constant. The criterion for the fractions of dopants was to keep the value of lattice parameter of cerium oxide unchanged as much as possible, regardless of the presence of dopants. In accordance with this, nominal compositions of multiple doped solid solutions were calculated on the basis of the previously obtained values of the lattice parameters. The lattice parameters were calculated by using the model that takes into the account the existence of oxygen vacancies in the structure.

The results have shown that the measured values of the lattice parameters and the values calculated using the model, were in very good agreement. On the basis of our data it was, also, found that the value of the critical ionic radius was 1.024 Å.

A22

HYBRID SILICA XEROGELS CONTAINING MICROBIAL HYDROLASES

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Enzymes are ubiquitous natural biocatalysts of nanometer scale. Practical use of enzymes has been realized in various industrial processes and products including laundry detergents, and is being expanded in new fields: fine-chemical synthesis, pharmaceuticals, biosensors, bioremediation, biobleaching, polymerase chain reaction, protein digestion in proteomic analysis, and biofuel cells [1,2]. The main goal of the immobilization of proteins and enzymes is the re-utilization of bioadsorbents/ biocatalysts for extended periods of time under industrial conditions. Thus, the

development of simple protocols for the immobilization of proteins on inert supports that can permit dramatic increases on protein stability still remains a key issue in bio-affinity chromatography and biocatalysis [3,4].

Biocatalysts were obtained with encapsulated Alcalase in silica gels produced by acid catalyzed hydrolysis of silane compounds such as methyltriethoxysilane (MTES), dimethyldiethoxysilane (DMDES) and tetraethoxysilane (TEOS), with or without additives addition. These gels were characterized with regard to mean pore diameter, specific surface area, pore size distribution (B.E.T. method), weight loss upon heating (TGA) and chemical composition (FTIR). The understanding of relationships between the reaction parameters, enzyme activity and physicochemical characteristics of the support is very important for the improvement of future biocatalysts production.

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A23

SYNTHESIS AND CHARACTERIZATION OF BIOMORPHIC CELLULAR SIC CERAMICS

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In this study porous silicon carbide ceramics have been obtained from two types of wood - dogwood (hardwood) and linden (softwood). Biomorphic cellular silicon carbide (SiC) ceramics with woodlike microstructure has been prepared by carbothermal reduction reactions of wood/TEOS composite at 1600 ºC. Wood specimens were carbonized at 1000ºC in Ar atmosphere for 2 hours. The porous carbon preform was infiltrated with TEOS ($Si(OC_2H_5)_4$), as a source of silica, without pressure at 25 ºC. The morphology of resulting porous SiC ceramics have been investigated by scanning electron microscopy (SEM/EDX) and X-ray diffraction (XRD). Experimental results show that the biomorphic cellular morphology of wood is preserved in the porous SiC ceramics that consists of β-SiC phase. Biomorphic SiC ceramics obtained from these two types of wood show difference in porosity, microstructure and mechanical properties.

A24

INVESTIGATION OF ELECTRICAL PARAMETERS OF NANOSTRUCTURED TITANIA COATINGS DEPOSITED ON INTERDIGITATED ELECTRODE SYSTEM

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The recent years have seen an increasing interest in the interdigitated electrode system. The most widely used applications of interdigitated electrodes include photo-sensitive detectors, surface acoustic wave filters and sensors for chemicals and gasses. In this paper, we will report electrical properties of titanium – dioxide (TiO_2) thin films deposited on alumina substrate with gold electrode in the interdigitated form to obtain appropriate devices for sensor application.

Nanostructured titania coatings were obtained by the sol–gel method, starting from stable and transparent titania sol.

The thickness of the gold electrode layer is 0.85 μm. All the structures were fabricated on the ceramic Al_2O_3 substrate which is 500 μm thick. The geometrical dimensions of all the structures are the same width and spacing between the fingers s = w = 1 μm. The number of fingers for all the analyzed structures is n = 4 and the length of a finger is l = 5 μm.

In order to obtain values of electrical parameters first were measured impedance Z, phase angle θ, capacitance C_p and resistivity R_p. The measurements were performed using an Agilent RF Impedance Analyzer 4191A in the frequency range from 1 MHz to 1 GHz. We have also developed a software tool for calculation of important electrical parameters from above-mentioned measured data. The results for conductivity δ and permittivity ε as a function of the frequency will be presented in this paper and corresponding conclusions will be given.

A25

THERMAL AND OPTICAL PROPERTIES OF PbI_2 INTERCALATED WITH POLYETHYLENE GLYCOL

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PbI_2 intercalated with polyethylene glycol (PEG) have been studied by X-ray diffraction, thermo-gravimetric analysis, FTIR spectroscopy, Raman scattering and low temperature photoluminescence (PL). The intercalated compound was prepared by chemical reaction between KI and $Pb(NO_3)_2$ in aqueous polyethylene glycol solution. The Raman and PL spectra of PbI_2 intercalated with PEG are quite different from those of pure PbI_2 crystalline powder. The difference results from a compressing effect produced by the insertion of polymer guest species into the PbI_2 host layers. Thermal analyses and infrared absorption spectroscopy have been used to study desorption of polymer from PbI_2 in order to evaluate the stability range and morphological changes with temperature.

A26

THIN FILM FABRICATION BY MODIFIED SOL-GEL PROCESS

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Crystalline titanium dioxide (TiO_2) and zirconium dioxide (ZrO_2) films are the promising materials for various applications such as optical devices, dye-sensitized solar cells and photo-catalysts. Therefore control over the thickness, structure and morphology of the films with the large coating area is important.

Thin inorganic films with defined thickness were fabricated by a new route that combines layer by layer (LbL) templating and an in-situ sol-gel process. In order to create a template for the inorganic film fabrication, polyelectrolyte multilayers (PEMs) of polyallylamine (PAH) and polyacrylic acid (PAA) were assembled on hydrophilic silicon single-crystal (100) wafers by alternately dipping the substrates into polyanion and polycation aqueous solutions. The thickness of the PEM is tuned at the nanometer level, depending on the number of polyelectrolyte layers deposited. PEM templates for the in-situ sol-gel reaction were then exposed to the anhydrous precursor solution. The hydrolysis and condensation of the precursor occurred upon interaction with the water adsorbed in the PE layer, causing its gelation and the formation of thin, inorganic/PE hybrid coatings of defined thicknesses. Upon calcinations at 500°C for 2 hours an inorganic thin film is obtained.

A27

DIELECTRIC PROPERTIES OF NANOSIZED $ZnFe_2O_4$

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In this paper we present the results concerning the dielectric properties of the nanosized $ZnFe_2O_4$. Dielectric permittivity, the loss factor, as well as the conductivity were measured in 300 K – 630 K temperature range and at 1 Hz, 10 Hz, 100 Hz, 1 kHz and 10 kHz frequencies. As a consequence of complex structure, dielectric behaviour of ferrites is explained by interface polarization. This type of polarization is dominant at frequencies below 10 kHz. The conducting process is explained by hopping mechanism between the Fe^{2+} and Fe^{3+} ions at octahedral sites. Increasing trend of electrical conductivity σ, and decreasing trend of dielectrical permittivity ε with increasing frequency can be explained by phenomenological Koops theory [1], in which dielectric materials are treated as two layer structure of Maxwell-Wagner type [2]. In this model the grains constitute conducting layer, while grain boundaries represented poorly conducting layer. The grains have small value of dielectric constant and have dominant role at high frequencies. The grain boundaries have high value of dielectric constant, and mainly influence the dielectric properties at low frequencies.

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A28

THE INFLUENCE OF THE THERMAL TREATMENT CONDITIONS ON MORPHOLOGY AND ORIENTATION OF LnO THIN FILMS

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Because of its metallic conductivity in wide temperature range and perovskite structure with lattice parameter similar to most ferroelectric perovskites, lanthanum nickelate ($LaNiO_3$, LNO) stands out as a good candidate for conducting layer in ferroelectric memories. The most of physical methods, like PLD, MBE or sputtering are efficient in preparation of epitaxial thin films. On the other hand, chemical methods also show some advantages, such as reproducibility, good control over the product

stoichiometry, the easier deposition of thin films over the large surface and reduced production cost.

In this paper the influence of the thermal treatment conditions on microstructural parameters of LNO thin films was investigated. LNO precursor solution was synthesized by chemical method (modified Pechini method), from the polymeric citrate precursors. The starting solution was spin-coated onto Si(100) substrates. Film thickness was controlled with number of deposited layers. Deposited layers were thermally treated with one of the two different heating rates: 1°/min (Process A) and 20°/min (Process B), and annealed at 700°C. The microstructure of LNO films was characterized by AFM and X-ray diffraction analysis. Smooth and crack-free thin films without pores were obtained. It is shown that characteristic microstructural parameters like surface roughness and shape and size of the grains strongly depend on heating rate during thermal treatment. LNO thin films obtained through different thermal processes have completely different structures, from polycrystalline (Process B) to well-crystallized and highly oriented (Process A).

A29

ULTRASOUND AND MICROWAVE SYNTHESIS OF BISMUTH OXIDE FOR CATALYTIC APPLICATION

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Bismuth oxide was synthesized via microwave-assisted hydrothermal and sonochemical method. The influence of microwave and ultrasound field on the reaction mechanism was studied. The effect of reaction parameters (ultrasound and microwave power, temperature, pressure and pH) on the product phase composition and morphology was discussed. The transformation of bismuth hydroxide to bismuth oxide was controlled by pH value and it was accelerated by the increase of time, the ultrasound and microwave power. The composition of reaction products was strongly depended on pH value. The amorphous products were obtained at acidic pH conditions and crystalline phase α-Bi_2O_3 was obtained at pH = 12. The particle size of prepared powders could be controlled by the pH value and by addition of a chelating agent. The particle size was reduced from micrometric to nanometric size in the presence of the chelating agent. The $Bi(OH)_3$ to Bi_2O_3 transformation mechanism was proposed consisting polycondensation of Bi – OH to Bi – O – Bi and crystallization of Bi_2O_3.

A30

FUNCTION OF MAGNESIUM NITRATE DOPE IN THE PRODUCTION OF POLYCRYSTALLINE ALUMINA CERAMICS

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Polycrystalline alumina (PCA) is a widely used ceramic material due to the excellent mechanical, optical properties and resistance to chemical corrosion. Binders of the PCA sintering process usually contain magnesium nitrate, mainly known as agent of burning organics to ash. During the sintering process, decomposition products (especially oxygen evolved) from magnesium nitrate may initialize the burning of organic type other additives used. Complete removal of residues of organic origin, even at low temperature, is essential for the final quality of polycrystalline ceramics used in lighting technology. On the other hand the released volatile gaseous products (including nitrogen oxides) are subject of environmental concerns.

We have analyzed and monitored the thermally evolved gases released from $[Mg(H_2O)_6](NO_3)_2$ and organic binders used in manufactural processes of PCA ceramics in flowing air and pure nitrogen atmosphere by simultaneous thermogravimetry and differential thermal analysis coupled online with mass spectrometry (TG/DTA-MS) and with FTIR spectroscopic gas cell (TG-FTIR) up to 700°C.

Our measurements characterized the technological steps of alumina ceramic discharge tubes from the aspect of analytical chemistry. We simulated the pre-sintering process of alumina with thermal analytical methods. It was proved that magnesium nitrate added to organic binders improves the efficiency of pre-sintering by evolved oxygen derived from the thermal decomposition.

A31

THE IMPEDANCE ANALYSIS OF ZINC OXIDE CERAMICS

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Investigations focused on zinc oxide in different forms, such as single crystal, powders, thin and thick films or nanostructures, goes back many decades due to its different technological applications. One of the mainly used techniques for powders preparation is the mechanical activation in high-energy mills. Mechanical activation is very complex process responsible for different changes on milling material. For this

study, zinc oxide powder was submitted to mechanical treatment in vibro and planetary-ball mills. Having in mind that the contribution of the internal strain induced by severe mechanical deformation is different in these mills, it can be expected that the macro and microstructural transformation of prepared zinc oxide powders and sintered bodies made from them are globally dissimilar.

To understand the importance of mechanical activation in process of obtainment ZnO ceramics it is necessary to examine some of its basic physical properties, specially the electrical properties. Since the grain size and the porosity are the major terms for the quantitative analysis of microstructure, we have been investigated electrical properties, densification and grain growth during sintering of mechanically activated zinc oxide. SEM micrographs of sintered samples were used to follow the microstructural changes and for assessment of mean grain size. Electrical properties, i.e. resistance and capacitance of the grain boundary region were measured using the ac impedance spectroscopy. The apparent activation energies for charge transport through the grain boundaries were calculated from the Arrhenius equation. Donor densities were obtained from Mott-Schottky measurements.

It was shown that mechanical activation contributes to a gradual modification of the microstructure and fine defect structure of zinc oxide powders and finely of electrical properties. Also, incorporation of impurities from grinding bodies and vials in ZnO powders during the milling, even in minor quantities, can be responsible for different electrical properties of ZnO ceramics prepared from powders mechanically treated in vibro and planetary-ball mills.

A32

THE INFLUENCE OF CRYSTAL ADMIXTURE TO QUALITY OF ZEOLITES PRODUCED

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Zeolites are micro porous crystalline solids with well-defined structures. Generally zeolites contain silicon, aluminium and oxygen in their framework and cautions, water and/or other molecules within their pores. It is possible that usually describes zeolites by empirical formula $M_{2/n}O{\cdot}Al_2O_3{\cdot}xSiO_2{\cdot}yH_2O$, but their structure determination is more complicate, because that formed area of polyhedrons generate characteristic vacancies and tunnels which give them specific properties. Properties of zeolites and their specific physical and chemical characteristics provide usage by large number of industries, as are: chemical technology, materials and process chemical engineering, etc. In the world, major uses are in petrochemical cracking, ion-exchange and in the separation and removal of gases and solvents. Other applications are in

agriculture and construction. Good understanding of interior and surface particle effects we need because knowledge of molecular and crystal structure of zeolites. This paper could give unpretentious contribution to knowledge about influence of impurities which are present into alumina solution during industry production of zeolites. Particularly, some factories of alumina use sodium-aluminate as raw material for production of zeolites and their production could to be more difficult because large species of impurities.

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A33

CORDIERITE SYNTHESIS ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) BY UNCONVENTIONAL METHODS

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Thanks to his valorous properties: extremely low dielectric constant and a low thermal expansion coefficient, the cordierite synthesis makes the object of a great number of scientific works.

The paper presents cordierite synthesis using three different methods:

a) the classic synthesis method, based on the calcinations of mechanical mixtures of $MgCO_3$, SiO_2 (quartz) and $Al(OH)_3$;
b) the hydrosilicates precursors synthesis method based on the precipitation of $MgO{\cdot}SiO_2{\cdot}xH_2O$ in the presence of Al_2O_3;
c) thermal conversion of the heteropolynuclear complex combination resulted from the oxidation reaction of 1,2-ethanediol with Mg and Al nitrates. The complex combination formation takes place in the presence of SiO_2 (aerosil), resulting a composition with advanced homogeneity;

The obtained results prove that the cordierite formation takes place with some difficulties:

- using the classic synthesis method the cordierite phase is not present in the samples prepared without mineralizer and annealed at 1200ºC for one hour, (there are present only: spinel ($MgO \cdot Al_2O_3$), α-Al_2O_3 and quartz); using 2%Li_2O as mineralizer, the main phase resulted at 1200ºC is μ-$2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$;
- in the case of the hydrosilicates precursors method, after annealing the reactant mixtures at 1200ºC, the main phases are enstatite ($MgO \cdot SiO_2$) and spinel with low crystallinity (the cordierite phase is not present). Using 2% Li_2O as mineralizer, μ-cordierite is obtained as main phase;
- using the complex combination method it was observed that in the absence of Li_2O, the spinel phase formation takes place at 600ºC, followed by its reaction with SiO_2 resulting α-cordierite. In the samples annealed at 1200ºC alongside the α-cordierite, the presence of spinel and α-cristobalite was evidenced. The thermal behavior of the samples prepared with 2% Li_2O and annealed at 800ºC is essentially different than those prepared by a) and b) methods; in this case the main phase at 800ºC is μ-cordierite and at 1200ºC the main phases are μ-cordierite and α-cordierite alongside a small proportion of spinel and α-cristobalite.

These results prove the advantages of the thermal conversion of the complex combinations in mixture with SiO_2, which allows the cordierite synthesis temperature induction; using the Li_2O as mineralizer enhances the formation of μ-cordierite modification.

A34

CHEMICAL VAPOR SYNTHESIS OF NANOCRYSTALLINE ANATASE (TiO_2) - TIME-TEMPERATURE PROFILE INFLUENCE ON POWDER CHARACTERISTICS

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Chemical Vapor Synthesis (CVS) is the conversion of molecular species into nanocrystalline particles (NPs) by chemical reactions in a gas flow reactor. The time-temperature profile in the gas phase of the reactor has a profound influence on the particle characteristics such as particle microstructure and surface chemistry and, therefore, on the quality of the powder consisting of NPs. Pure anatase NPs are generated in a hot wall reactor from titanium isopropoxide (TTIP) using different time-temperature profiles. The powder characteristics are analyzed in detail using nitrogen adsorption, X-ray diffraction, dynamic light scattering, FTIR spectroscopy and transmission electron microscopy. The powders show very high degree of crystallinity, small particle size and a low degree of agglomeration.

A35

COMPARISON OF THE YTTRIUM ALUMINIUM GARNET (YAG) NANOPOWDER PREPARATION METHODS

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This paper presents results of investigation of YAG powders synthesis process based on complexing properties of citric acid. Influence of citric acid estrification induced by propanol, or ethylene glycol on the system homogenity was investigated. These reagents were introduced to water solution of yttrium and aluminum nitrates. A variety of powders from Al_2O_3-Y_2O_3 system with different phase composition were obtained by altering the citrate to nitrate ratio. Evolution of the powders phase composition vs. temperature was investigated using DTA/TG, XRD, and FT-IR methods. The most interesting results were observed in case of citric acid – propanol – relative nitrates system. Mole ratio of these reagents equal 1:2.5:2.5 (nitrates (Al, Y) : citric acid : propanol) allowed to synthesize pure YAG phase powders at temperature as low as 950°C.

A36

EFFECT OF SYNTHESIS METHOD ON $BaTiO_3$ PROPERTIES

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Barium titanate ($BaTiO_3$) has been of practical interest for more than 60 years because of its attractive properties. $BaTiO_3$ can be prepared using different methods. It was detected a significant influence of used method on structure and properties of barium titanate materials.

In this paper powder of $BaTiO_3$ was prepared by two methods. The first one was synthesis from polymeric precursors through Pechini process (soft chemistry-PPM) which was carried out as a three–stage process from oganometallic complex [1]. The second one was a mechanochemical synthesis from powder mixture of BaO and TiO_2 [2]. In both cases $BaTiO_3$ was sintered for 2h at 1300^{o}C without pre-calcination step. The formations of phase and crystal structure of $BaTiO_3$ prepared by both methods were carried out by XRD analysis. The morphology and microstructure of obtained powders and sintered samples were examined by SEM method.

The XRD results of powders obtained by both methods indicate the formation of cubic phase of $BaTiO_3$ and tetragonal phase in sintered samples. $BaTiO_3$ powder prepared by PPM was well crystallized but significant amount of amorphous phase was detected for other method. SEM micrographs indicate that the morphology of the powders consists of particles and its agglomerates and their dimensions depend of the synthesis method. The powder prepared mechanochemically possesses higher number of agglomerates, particles are bigger and with irregular shape. Average particle size is about 100 nm and 250 nm for Pechini and mechanochemical process, respectively. Two types of domain configuration were observed in samples sintered at 1300°C for 2h and prepared from powders obtained by PPM.

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A37

STRUCTURAL CHARACTERIZATION OF Cu^{2+} AND Fe^{3+} FUNCTIONAL CENTERS IN 'LEAD-FREE' $K_YNA_{(1-Y)}NbO_3$ PIEZOELECTRICS

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The volatile and toxic nature of PbO in PZT ($Pb(Zr_xTi_{1-x})O_3$) ceramics causes not only health but also environmental problems as being disposal and even during their processing. Therefore, although these ceramics are widely used in piezoelectric transducers, transformers, sensors and etc., lead-free alternative materials are currently investigated. Among these alternatives, the alkali niobate ferroelectrics ($(K_yNa_{1-y})NbO_3$, KNN) have been reported as one of the most promising materials due to their high Curie Temperature and electrical properties. In order to obtain dense compounds with decent properties, doping with different elements has to be performed. Though some dopants have good influences on the KNN perovskite structure, some do not. The aim of this study is to figure out the structure of Cu^{2+} and Fe^{3+} doped KNN ceramics via different EPR techniques since these dopants show paramagnetic characteristics. Further insight into the ferroelectric properties of such materials may be obtained by systematically characterizing their defect chemistry.

A38

EFFECT OF HIGH-PRESSURE SINTERING TEMPERATURE ON MICROSTRUCTURE, PHYSICAL AND MECHANICAL PROPERTIES OF TiB_2 CERAMICS

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Titanium diboride ceramics have excellent physical and chemical properties such as high melting point, high hardness, and good corrosion resistance; moreover, TiB_2 ceramics have an excellent electric conductivity. However, up to now, the applications of TiB_2 ceramics are rather limited due to the difficulties that exist in producing fully dense materials. The high-pressure sintering (HPS) has been considered as an effective candidate sintering process for TiB_2 ceramics. The main features of HPS include lower sintering temperature and short sintering time (less than 5 min). It has been shown that HPS increased the density of TiB_2 ceramics significantly.

In this paper, the effect of high-pressure sintering temperature on the microstructure, physical and mechanical properties of TiB_2 ceramics has been investigated. Pure TiB_2 powder with an average particle size of 5 μm was sintered in a modified high-pressure anvil-type apparatus under static pressure of 4 GPa in the temperature range 1400-1800^{o}C.

Microstructure analysis showed that the HPS process allows preparing TiB_2 ceramics with full-dense fine-grained structure. The mean grain size appeared to be less than 10 μm. Insignificant grain growth is observed with increasing the sintering temperature while the structural inhomogeneity is reduced.

It is shown that the relative density of TiB_2 samples rises with increasing the sintering temperature and reaches the maximal value of 99.3 % at 1700-1800^{o}C. At the same time, the highest possible microhardness (about 33 GPa) was achieved in the temperature range 1500-1600^{o}C. This fact can be explained using the data obtained by XRD analysis. The evolution of half-broadening of the (211) peak with raising the sintering temperature demonstrates that the increase in microhardness is accompanied by the lattice deformation, which is caused by an increase of the level of internal stresses in the samples. Then, the reduction in microhardness is related to the onset of the recrystallization process and relaxation of internal stresses.

Thus HPS permits preparing fully dense TiB_2 ceramics with fine-grained structure. The density of samples rises with increasing the sintering temperature up to 1800^{o}C while the maximal microhardness is observed in samples sintered in the range 1500-1600^{o}C. The XRD analysis has shown that this fact can be ascribed to an increase of the level of internal stresses in these samples.

A39

SYNTHESIS OF MESOPOROUS ALUMINA USING POLYVINYL ALCOHOL TEMPLATE AS POROSITY CONTROL ADDITIVE

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As consequence of the rapid development of high technology industries, in the last years the production of homogeneous and nanoscale ordered structure materials with accurate shapes and dimensions become a priority for research and industry. The current preoccupations in the field of sol-gel synthesis implementation in technology are multiple because of its advantages in the production of materials with tailored properties.

This paper summarizes the study about production and textural characterization of ceramic materials with tailored nanoporous structure, as thin films, nonsupported membranes and as bulk gel. The ceramic materials were prepared by the sol-gel method using alcoxide precursors, based on hydrolysis and condensation reaction of the aluminium triisopropylate. The sol-gel process has been applied to prepare amorphous and poorly crystalline materials, as nanoporous ceramics with high surface areas and small pore sizes. The effects of polyvinyl alcohol (PVA) template and calcinations temperatures on the characteristics of the prepared materials were investigated. The variation of mesoporous texture, pore size and morphology, were determined by nitrogen adsorbtion/desorbtion analysis and TEM. Macroporous size distribution was determined using mercury porosimetry and SEM was used to observe the pore structure of these samples. The evolution of the phase composition of the samples has been monitored by XRD.

Experimental observation after drying and annealing shows that it is possible to produce nanoporous alumina ceramics using polyvinyl alcohol template. The obtained alumina samples presents macroporous texture with the average pore radius rav = 15.65 μm, for sample prepared with 1.25 g PVA addition and annealed at 1000ºC and also mesoporosity with the average pore diameter Dav = 14.04 nm, for the same sample.

A40

SYNTHESIS OF ZINC FERRITES IN RADIOFREQUENCY THERMAL PLASMA REACTOR

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Zinc ferrites crystallize in face centred cubic spinel structure. In the normal zinc ferrite structure, the Zn^{2+} cations located in tetrahedral, while the Fe^{3+} ions are in octahedral positions. These ferrites exhibit paramagnetic properties. In special conditions, such as rapid quenching, position of Zn^{2+} and Fe^{3+} cations in the crystal structure can be partially or even completely reversed. The resulted inverse spinel structure is ferrimagnetic with remarkable saturation magnetization. Thus, inverse zinc ferrites can be used as electrical and magnetic materials and also as carriers for targeted drug release. Inverse ferrite spinels with ultrafine particle size can be produced in RF thermal plasma conditions due to high processing temperature and the rapid quenching of the formed, meta-stable phases. In addition, thermal plasma synthesis makes production of ferrite powders from corresponding nitrates or oxides simpler. The aim of this work was investigation of the formation of normal and inverse zinc ferrite spinels from different precursors in RF thermal plasma conditions.

The ferrites were synthesised from corresponding nitrates and oxides in RF thermal plasma reactor. Argon was used as plasma and carrier gas. The sheath gas was a mixture of Ar and O_2, respectively. Effects of stoichiometric ratios, position of feeding points and plasma power on product properties were investigated. Particle- and domain sizes, saturation magnetisation and phase composition were studied in details. In all cases both micro- and nanosized ferrite particles were formed. However, the nanoparticles coating the surface of larger particles were detected in a greater share. Mainly spinels of targeted ferrite type were formed. However, more or less magnetite ($FeO \cdot Fe_2O_3$) and other corresponding oxides (ZnO, Fe_2O_3) also observed.

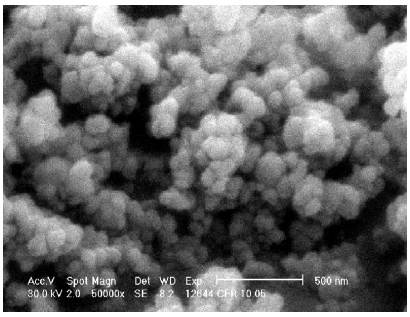

Figure 1. SEM micrograph of RF plasma produced zinc ferrite powder.

A41

SYNTHESIS AND PROPERTIES OF $BaTiO_3$/Ni NANOCOMPOSITE

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In this work powders of nanocomposite barium titanate – nickel with different nickel content were obtained. Properties of these powders were investigated. Curves of compressibility, particles size distribution data and specific surface, results of HR TEM are presented. The dielectric properties of obtained powders were measured. The analysis of finding showed that there is threshold concentration limit of nickel in barium titanate. Material has high dielectric permittivity near threshold concentration limit and if nickel concentration is higher than threshold concentration limit material loses its dielectric properties and becomes conductor. It was shown that value of threshold concentration limit depends on dielectric/metal particles size ratio.

A42

ALUMINA CERAMICS PREPARED WITH NEW PORE-FORMING AGENTS

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Porous ceramics have a wide range of applications at all length scales, ranging from filtration membranes and catalyst supports to biomaterials (scaffolds for bone ingrowths) and thermally or acoustically insulating bulk materials or coating layers. Organic pore-forming agents (PFAs) of biological origin can be used to control porosity, pore size and pore shape. This work concerns the characterization and testing of several less common pore-forming agents (lycopodium, coffee, flour and semolina, poppy seed), which are of potential interest from the viewpoint of size, shape or availability. The performance of these new PFAs is compared to that of starch, which has become a rather popular PFA for ceramics during the last decade. The PFAs investigated in this work are in the size range from 5 μm (rice starch) to approx. 1 mm (poppy seed), all with more or less isometric shape. The burnout behavior of PFAs is studied by thermal analysis, i.e. thermogravimetry and differential thermal analysis. For the preparation of porous alumina ceramics from alumina suspensions containing PFAs traditional slip casting (into plaster molds) and starch consolidation casting (using metal molds) are used in this work. The resulting microstructures are investigated using optical microscopy, combined with image analysis, as well as other methods (Archimedes method of double-weighing in water, mercury intrusion porosimetry).

A43

THE PREPARATION OF Al_2O_3 COATINGS ON ZrO_2 CERAMIC SUBSTRATES AND THEIR INFLUENCE ON THE ADHESION WITH LUTING CEMENT

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In dentistry numerous attempts have been made to develop all-ceramic systems that eliminate metal-core structures. Stabilized tetragonal zirconia (Y-TZP) is a promising material that has become increasingly popular as an alternative, high-toughness core material in dental restorations because of its biocompatibility, attractive mechanical properties and its superior natural appearance compared with metal dental restorations. However, the adhesion of the luting cement to the zirconia surface is poor and, therefore, sandblasting is clinically used to increase the surface roughness of ceramics. As an alternative, chemical modifications of the zirconia surface, i.e., silanization and other, are being investigated to improve the binding between the luting cement and the zirconia surface, which are usually complex and frequently ineffective in long term applications.

The precipitation of nanosized alumina coatings on zirconia ceramics using aluminum nitride (AlN) powder hydrolysis was investigated in this work in order to improve the adhesion of the luting cement to the zirconia surface. The hydrolysis of the AlN powder follows the reaction scheme:

$$AlN + 2H_2O \rightarrow AlOOH_{(amorph)} + NH_3$$
$$NH3 + H_2O \leftrightarrow NH_4^+ + OH^-$$
$$AlOOH_{(amorph)} + H_2O \rightarrow Al(OH)_{3(xstal)}$$

It has been suggested that crystalline aluminum hydroxides are formed by a dissolution/precipitation process, similar to biomimetic apatite formation in simulated body fluid. The first reaction product is amorphous aluminum hydroxide, which then ages to various crystalline products, depending on the time, the temperature and the pH. These hydroxides were expected to form a uniform thin coating on the zirconia substrate, exhibiting a high specific surface area. The influence of precipitation conditions, such as time, temperature, pH and AlN concentration, on the morphology of the precipitated aluminum hydroxide was studied and the crystal structure and morphology of the alumina coating after thermal treatment were determined. The adhesion of the luting cement on the coated zirconia substrates was also measured with a shear test and the results were compared to those obtained with uncoated zirconia substrates. It was found that the alumina coatings on zirconia ceramics significantly improve the adhesion with the luting cement.

A44

SYNTHESIS OF ZnO NANOCRYSTALS THROUGH SURFACTANT-ASSISTED MECHANOCHEMICAL PROCESS

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The scope of this work was to synthesize and characterize nanocrystalline ZnO powders by the use of a wet mechanochemical process and thermal treatment. Initial precursors used in these experiments were zinc chloride and zinc acetate dihydrate. The influence of the surfactants – sodium dodecyl sulphate (SDS), polyvinyl pyrrolidone (PVP) and oxalic acid – on the size, structural and morphological characteristics of ZnO particles have been investigated. In addition, $CaCl_2$, an inert salt matrix, was also tested for its influence on the particle properties. Prepared powders were characterized in terms of their crystalline structure by the X-ray diffraction; morphology and size of prepared particles were determined by scanning electron microscopy.
Results demonstrate that pure ZnO powders were obtained after mechanochemical synthesis. Organic and polymeric surfactants – PVP and SDS – lead to more uniform and homogeneously dispersed ZnO particles. On the other hand, hard agglomeration of ZnO particles was observed in the case of inert $CaCl_2$ salt matrix.

A45

STRUCTURAL AND FUNCTIONAL CHARACTERIZATION OF DOPED $LaGaO_3$

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In this work lanthanum-gallate based powders with composition $La_{1-x}Sr_xGa_{1-y}Mg_yO_{3-\delta}$, ($0 \leq x, y \leq 0.2$) were prepared using citrate sol-gel method. As-synthesized powders were calcinated at 900°C, pressed and sintered at different temperatures (up to 1450°C). Powder characterization showed that calcinated powders were agglomerated and consisted of primary particles with average size of ~100 nm. Pure $LaGaO_3$ powder has dominant cubic perovskite phase, with a small amount of orthorhombic phase. The amount of the secondary phases is much higher in the powders in which La and Ga were substituted with Sr and Mg. Sintered samples contained cubic perovskite phase while only pure $LaGaO_3$ had small amount of the secondary

orthorhombic phase. It has been shown that sintering temperature of 1450°C is appropriate temperature in order to obtain microstructure with close porosity. Under these conditions the obtained densities had values higher than 95% TD, with exception of pure $LaGaO_3$. In this way it has been shown that substitution of La and Ga with Sr and Mg, not only stabilizes cubic perovskite phase but also improves powder sinterability. Electrical measurements confirmed that obtained LSGM ceramics could be applied as electrolite in fuel cells with working temperature between 600°C and 700°C.

A46

KINETICS OF DISSOLUTION OF CALCIUM PHOSPHATE (Ca-P) BIO-CERAMICS

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Hydroxypatite (HAp) $Ca_{10}(PO_4)_6(OH)_2$ as well as β-tricalcium phosphate (β-TCP) $Ca_3(PO_4)_2$ belongs to the group of the most important bioceramics used for medical or dental applications. These bioactive materials have an important role in hard tissue repairs because of their good resorbability (β-TCP) or ability to bond with surrounding bone tissue (HAp) in body fluid environment.

This work deals with dissolution kinetics of HAp and β-TCP, both prepared synthetically. The aim was to observe relationship between quality of the Ca-P material (their porosity as well as the way of their preparation) and the rate of the Ca-P dissolution. For the in vitro experiments we used several solutions. In vitro tests are useful in finding of a dissolution mechanism of the Ca-P material resulting in a healing time.

Firstly were tested HAp or β-TCP granules in the various corrosive solutions (pH value was changed) under static conditions. Concentration of Ca^{2+} and $(PO_4)^{3-}$ ions and pH value were analyzed in the leached solutions. As well as weight changes of the samples were measured. Results indicated significant shortening of the in vitro test at lower pH value of testing solution and no significant effect of the way of material preparation. The kinetics of the Ca-P materials dissolution was designed for the various testing conditions. In the next part of the work, which is proceeding, samples are tested under dynamic conditions, where still fresh corrosive solution continually flows along the sample situated in a flow cell.

A47

CONTROLLING THE COMPOSITION OF NANOSIZE h-WO_3 FOR GAS SENSORS

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For applications in photoelectrochromical cells and chromogenic (electro-, photo- and thermochromic) devices, gas sensors and catalysts, tungsten oxides have attracted much attention in the past decades. The hexagonal tungsten trioxide, h-WO3, is one of the most researched tungsten oxides due to its open-tunnel structure and intercalation chemistry. We studied its preparation through the annealing hexagonal ammonium tungsten bronze (HATB), $(NH_4)_{0.33-x}WO_{3-y}$. We have chosen this way to prepare h-WO_3, because we thought that by this route the composition of h-WO_3 could be easily controlled in a great range.

The formation of h-WO_3 was investigated by simultaneous TG/DTA and on-line evolved gas analysis (TG/DTA-MS) in oxidative (air), inert (N_2, He) and reductive (10 % H_2/Ar) atmospheres. Intermediate solid products were analyzed by powder X-ray diffraction (XRD), scanning electron microscopy (SEM), Raman, solid state 1H-MAS NMR and X-ray photoelectron (XPS) spectroscopy.

We obtained new structural information about both the precursor HATB and the product h-WO_3. We could detect three different positions of ammonium ions in HATB that can be situated on the surface of the particles, between the crystallites, or in the hexagonal channels of crystallites. The ammonium ions in the hexagonal channels seem to be vital for stabilizing h-WO_3: when they are completely released, the hexagonal framework collapses in an exothermic reaction. We suppose that the structure of h-WO_3 can not be maintained without some stabilizing ions or molecules in the hexagonal channels.

As shown by the various analytical methods, through adjusting the temperature and atmosphere of annealing of HATB, we could control the composition (W oxidation state, residual NH^{4+} content) of the product h-WO_3 particles. The as-produced h-WO_3 samples with different (oxidized or reduced) composition were tested as gas sensors, and the effect of the composition of h-WO_3 samples on their gas sensing properties was studied.

A48

MICROWAVE SINTERING OF CERAMICS

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The main benefits of exploiting microwave energy in thermally activated processes arise from the specificity of microwave energy absorption. In past decades much efforts has been made in this field, however no universal model for process description has been proposed yet. To fill in a gap in understanding of microwave sintering process nature, model experiments on powders particles were held by both microwave and conventional sintering. The exponential model was used for description of initial stage sintering of group of particles. Phenomenon of thermal runaway during microwave heating was observed. Shrinkage studies were performed. Comparison of microwave and conventionally sintered samples has been made.

A49

POSIBILITY OF ALUMINATE CEMENT PRODUCTION BY ABLATION IN ALUMINA PRODUCTION PROCESS

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Alumina Factory "Birac" in Zvornik has good predisposition for production of aluminate cements, since it has already available all components for its production (Na-aluminate, bauxite, lime, hydrate). Also, there is a larger quantity of equipment which could be used for that purpose.

Main goal was to determine optimal value of basic parameters for producing aluminate cements which are following: chemical composition (content of Al_2O_3), duration of synthesis and temperature of sintering.

Analysis enabled defining of aluminate cement quality dependency in structural view by analyzed parameters. On this way, a significant contribution of determining its optimal quality was made, as well managing of the technological process of aluminate cement production. For the characterization of starting components (Na-aluminate, bauxite, lime), as well for final product classic analytic methods have been used as: gravimetry and volumetry, optical methods (UV-VIS spectrophotometry, atomic absorption spectrophotometry, XRD-fluorescent spectrometry), thermal analysis (DTA-DTG-TG); electroanalytic method – potentiometric titration; laser determination of particle size and for processing obtained data modern computer equipment was used.

Charges are prepared depending of cement type that should be produced. Syntheses were performed in reaction vessel on 90°C in certain time period. Afterwards solid phase was separated from liquid by filtration, was dried and ignition until sintering. Sintering of different composition charges was performed in laboratory high-temperature kiln and it was determined that temperature interval of sintering was from 1280 to 1440°C, for time period of 30 min.

We have obtained different types of aluminate cements, with adequate characteristics, depending on starting components and sintering conditions.

A50

INVESTIGATION OF ELECTRICAL CHARACTERISTICS OF DIFFERENT CERAMIC SAMPLES USING HALL EFFECT MEASUREMENT

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Hall effect is a very popular technique and is widely used to quantify important electrical parameters such as carrier concentration, resistivity, mobility and Hall coefficient of different types and shapes of samples.

In this paper, electrical characteristics of different samples will be analyzed using a Hall effect measurement system (Ecopia, HMS-3000), which is based on the van der Pauw method. This system has a 0.37 Tesla permanent magnet. Hall measurements can be performed by altering the incoming current until the voltage produced is near some target voltage. In order for a highly resistive sample to obtain the target voltages, the current applied must be very small.

It is possible to test various sample using two kinds of sample board (20mm x 20mm or 6mm x 6mm). Samples need to be soldered in four points edge. Contacts were provided by copper wires soldered with indium/tin pellets to the corners of the sample. The size of the contact spots was made as small as possible, less than 0.5 mm, to increase the accuracy of measurements which drops as the ratio of the contact area to the sample area increases. All samples will be measured at room temperature. The sample board needs to be inserted into magnet set lid. After closing the lid of magnet set, we can start the PC program and enter input data (applied magnetic flux density, thickness of the sample, input current...).

In this paper, measured results for carrier concentration, resistivity, mobility and Hall coefficient will be presented and by using graph the specifications of current-voltage and current-resistance between terminals of four point contact of different samples will be demonstrated.

CERAMICS COMPOSITES

C1

PROCESSING AND CHARACTERIZATION OF GLASS REINFORCED BIOGENIC HYDROXYAPATITE COMPOSITES WITH FERROMAGNETIC ADDITIVES

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In the present study, biogenic hydroxyapatite-glass materials were fabricated with Fe and Fe_3O_4 powders at different sintering temperatures. It has been established that the magnetic susceptibility of doped composites depends on their sintering conditions. The composites with 2 mass % of Fe or Fe_3O_4 sintered at above 500ºC under the usual atmospheric conditions have a magnetic susceptibility $\leq 1.3 \cdot 10^{-3}$ cm^3/g. It increases to $3 \cdot 10^{-3}$ cm^3/g for the specimens with 1%mas of additives sintered at 500ºC in vacuum. The influence of ferromagnetic additives on the degradation composites has been studied in vitro. Their presence leads to an increase in the degradation rate within the first 40 minutes of the composite soaking. Short-term processing specimens by the magnetic field, lead to increase in the initial degradation rate, but insignificantly influence it within more prolonged soaking. The results are of importance for creation of bone implant with ferromagnetic properties in order to improve treatment of bone defects.

C2

PYROELECTRIC TEMPERATURE SENSING WITH MULTI-WALL CARBON NANOTUBE FILMS

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Self supporting mats of entangled carbon nanotubes (buckypapers) are nowadays utilized in an increasing number of applications including nanoactuators, artificial muscles and sensors. Self-supporting, randomly aligned multi-wall carbon nanotube films (Fig. 1a) (buckypapers) were converted into resistive temperature measurement devices working at ambient pressure in air with ±1ºC accuracy in the 25–70ºC range. Thermosensibilization was achieved by decorating (Fig. 1b) the surface of

the films with pyroelectric $CsNO_3$ or $LiNbO_3$ crystals. The temperature response was linear and free of hysteresis with temperature coefficient of resistance values of –0.15798 %·ºC^{-1} and –0.2457 %·ºC^{-1} for $CsNO_3$ and $LiNbO_3$ doped films, respectively. It is anticipated that similar devices could also be built with piezoelectric and ferroelectric crystals. Thus, the described method opens a simple and inexpensive way towards preparing buckypaper based sensors for measuring temperature, pressure and electromagnetic field strength.

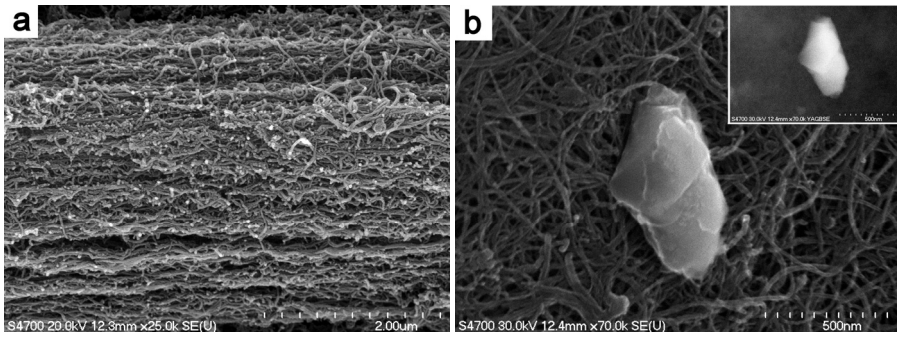

Figure 1. Characteristic SEM image of a studied MWCNT film.

C3

PREPARATION AND PROPERTIES OF β-SiAlON/ZrN NANO-COMPOSITES FROM ZrO_2-COATED Si_3N_4 POWDER

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Recently much attention has been devoted to the production of particulate reinforced silicon nitride and SiAlON materials not only because of improved fracture toughness, strength and mechanical reliability, but also because of their potential multifunctionality, especially electrical conductivity, which can be obtained by the incorporation of electrically conductive particles into the matrix phase. Because of their excellent mechanical properties and good oxidation and corrosion resistance, such composite materials are potentially interesting for the production of glow plugs for diesel engines, heaters, igniters, etc.

In this work we report on the preparation and properties of β-SiAlON/ZrN electro-conductive nano-composites from ZrO_2-coated Si_3N_4 powder. The silicon nitride powder was coated with nano-sized zirconia particles. This was achieved by the homogenous precipitation of ZrO_2 from the zirconium acetate solution using urea as the precipitating agent. For the preparation of sintered β-SiAlON/ZrN composites two different approaches were used. In the first one the ZrO_2-coated Si_3N_4 powder was

mixed with the appropriate sintering additives (Al_2O_3, Y_2O_3 and AlN) and reaction sintered, while in the second approach the coated powder was first calcined at 1600°C to prepare ZrN coated Si_3N_4 powder that was subsequently mixed with the sintering additives and sintered. For comparison, the composites with the same composition were also prepared by mixing Si_3N_4 and ZrO_2 powders with sintering additives and sintering. During thermal treatment and/or sintering of Si_3N_4/ZrO_2/AlN powder mixtures zirconia reacts with silicon nitride and aluminum nitride to form zirconium nitride. Dense β-SiAlON/ZrN composites were successfully prepared by pressureless sintering at 1850°C for 2 h in nitrogen atmosphere. The resultant compositions and microstructures were investigated using XRD, SEM and TEM. Additionally, the electrical resistivity and mechanical properties of composites were measured. It will be shown that dense composites with attractive mechanical and electrical properties can be prepared by reaction sintering of ZrO_2-coated Si_3N_4 powder choosing the right amount and composition of sintering additives.

C4

EFFECT OF THE RARE-EARTH OXIDE ADDITIVES ON MECHANICAL PROPERTIES OF Si_3N_4/SiC MICRO/NANOCOMPOSITE

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Submitted work deals with the preparation of Si_3N_4/SiC micro/nanocomposite where SiC phase formation is achieved by in situ chemical reaction between SiO_2 and C during the sintering process. Different rare earth oxides have been used as sintering additives and their effect on the microstructure (shape and particle-size distribution) and mechanical properties (hardness and fracture toughness) was investigated. Except of the influence of sintering aids on the properties of Si_3N_4/SiC micro/nanocomposites also the effect of high temperature treatment at 1750°C for 4, 26 and 70 hours were studied.

The fracture toughness and Vickers hardness of the reference monolithic Si_3N_4 decrease with the increasing ionic radius of used rare earth oxides. The fracture toughness of composite material is lower in comparison to the reference monolithic silicon nitride, which might be influence of the finer grains. The trend for fracture toughness with the increasing ionic radius of rare earth is not the same as it is for monolithic silicon nitride. The presence of SiC inclusions diminishes the influence of the rare earths on the fracture toughness.

The average grain diameter distribution has been significantly changed after 26 hours of heat treatment. The Vickers hardness and fracture toughness of heat treated Si_3N_4/SiC micro/nanocomposite decreases with the prolonged time of high temperature treatment HTT.

C5

PROCESSING, MECHANICAL AND THERMOPHYSICAL PROPERTIES OF SILICON NITRIDE BASED COMPOSITES WITH CARBON NANOTUBES AND GRAPHENE

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Silicon nitride based composites with different amount (1, 2 and 3 wt%) of multiwall and singlewall carbon nanotubes, and graphene have been prepared. Optimisation of the manufacturing processes has been conducted to preserve the carbon nanotubes in composites and to avoid damaging during high temperature processing.

The carbon nanotubes have been produced by CVD (Chemical Vapor Deposition) processes at Szeged University (multiwall carbon nanotubes -MWNT) and some comercial samples have been obtained from Nanocyl S.A. (multiwall and singlewall nanotubes -MWNT and SWNT). The proper separation and dispersion of carbon nanotubes proved to be a difficult task of the composite preparation. For a better homogeneity, after ball milling the ultrasonic agitation of MWNT-powder mixtures have been performed. By increasing the sonication time some advances have been made, but the general tendency of nanotubes (derived from the high specific surface area), the strong adherence and linking behavior to each other could not be totally suppressed. High efficient attritor mill was also employed, for distribution of carbon nanotubes in ceramic matrix. To preserve the carbon nanotubes in composites and to avoid damaging during high temperature processing, the optimisation of the manufacturing processes has been conducted. A suitable bonding between carbon nanotube and silicon nitride have been also monitored. Scanning electron microscopy showed, that there is a good contact between carbon nanotubes and the surface of silicon nitride grains. The CNTs are located mainly in the inter-granular places and they are well attached to the silicon nitride grains. X-ray charactrization, mechanical and thermophisical properties of composites will be presented. It was found that microstructure features achieved by properly designed sintering parameters are the main responsible factors for the strength improvements.

C6

PREPARATION OF SIC-BASED CERAMIC MATERIALS WITH UNCONVENTIONAL SINTERING AIDS

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Present work is focused on the preparation of SiC/SiOC nanocomposite. Polymer derived SiOC is used as a sintering additive for densification of SiC. The expected benefit of using silicon oxycarbides as sintering aid are their excellent high temperature properties (creep resistance), which might be comparable with recently used Lu_2O_3, exhibiting the best creep resistance of Si_3N_4 based materials.

The starting powders were mixed in a ratio 30 wt% SiOC : 70 wt% SiC. The samples were hot pressed in graphite resistance furnace using 30 MPa mechanical pressure and $CO+N_2$ atmosphere. Four different heat treatment conditions were used for the densification of samples. Relatively high density (91-95%) was obtained after sintering at low temperature ~1600^{o}C. However, the Vickers hardness of the samples was low (5-6 GPa), comparable to the hardness of SiOC glass. The TEM observation of SiOC phase showed the formation of SiC nanocrystals at this temperature.

Sintering at higher temperatures (1850^{o}C) followed by crystallization under pressure resulted in higher density (2.5-2.7 g/cm^3) and increased hardness (12-16 GPa). The indentation fracture resistance varied from 2.40 to 4.02 MPa.m$^{1/2}$, depending on the microstructure of samples. Although the sintering conditions should be further optimised the results show that SiC/SiOC nanocomposites can be prepared by hot-pressing in relatively short time, at low temperatures, and the mechanical properties are also promising.

C7

CHARACTERIZATION OF SANDWICHED COATING LAYERS CONSISTING OF TITANATE NANOWIRES

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Nanotechnology and building of thin layers are more and more widespread nowadays since it is considered to be the solution to several technical problems. The thickness of thin films falls into the range where materials have no bulk character any more, but rather, they exhibit special qualities instead. Provided we produce these thin films from nanoparticles, nanotubes and nanowires, we anticipate further advantageous qualities. During my experiments I built thin films from multiwalled carbon nanotubes and titania nanowires by the help of dip coater and spin coater.

Having prepared suitable suspensions from the starting material, I investigated the factors influencing the quality of the coating. The optimal values of the following parameters: dipping time, dipping repetition number, surfactant quantity, surface modification, When spin-coating, I have found the optimal rps value, the optimal number of drops with a given concentration of solution and the possibilities of surface pre-modification.

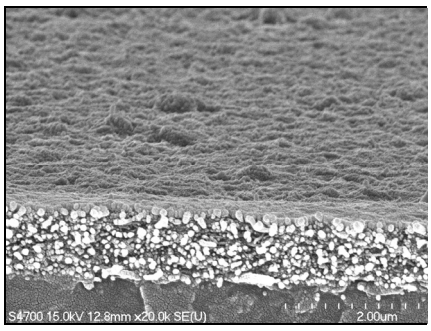

Figure 1. TiONW-MWCNT layer.

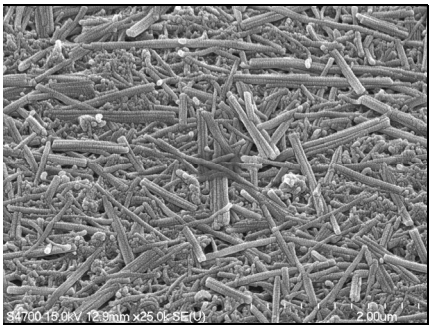

Figure 2. TiONW-MWCNT-TiONW.

Knowing the optimal experimental parameters I built different thin films with two methods. The titania, titania–carbon nanotube and titania–carbon nanotube–titania coatings were dried and studied by a Hitachi S-4700 scanning electron microscope.

The measurements revealed that dip coating is the more target based and useful procedure out of the two methods to produce thin films from nanowires and nanotubes. The samples from the spin coating procedure show a more uneven surface which is deteriorating even more when new layers are applied and during the use of surfactant.

C8

INFLUENCE OF NITROGEN ON THE TRIBOLOGICAL PROPERTIES OF a-C:H LAYERS ON THE POLYCARBONATE SUBSTRATES

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Polycarbonates (PC) posses many commercial applications. However, PC is still limited to non-abrasive and chemical-free environments due to its low hardness, low scratching resistance and high susceptibility to chemical attacks. To overcome this limitation, PC can coat by hydrogenated amorphous carbon layers. The a-C:H layers have very attractive properties such as high hardness, infrared transparency, chemical inertness, low friction coefficients, and biocompatibility. Addition of nitrogen on the structure allows lowering internal stress and improve tribological properties of a-C:H layers.

In this work, a-C:N:H layers were deposited from mixture CH_4/N_2 gases by Plasma Enhanced Chemical Vapor Deposition (RF CVD 13,56 MHz). Effects of the nitrogen incorporation on structure and tribological properties of deposited layers were investigated. The structure of layers were characterized by X-ray Photoelectron Spectroscopy and Fourier Transform Infrared spectroscopy (FTIR). The friction coefficient, wear resistance as well as the surface topography of a-C:H:N layers were estimated by tribometer in ball-on-disc configuration and atomic force microscopy (AFM), respectively. Results from the measurement indicate that incorporated nitrogen content has considerable effects on film properties. The IR spectra of the obtained layers have demonstrated a presence of nitrogen bonded both to carbon and to hydrogen. A formation of the following bonds has been confirmed: $-C\equiv N$, $-NH_2$, $-C-NH_2$, $>C=NH$. All they are typical for a-C:N:H layers. The surface roughness of layers estimated by AFM seems to be more smooth with the increase of the N/C fraction. The tribological tests have shown that the layers reduce the friction coefficient of the polycarbonate (up to 50 %) and considerably improve wear resistance.

C9

CERAMICS MATERIALS OF QUASI-BINARY LaB_6-MoB_2 SYSTEM

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Ceramic materials based on lanthanum hexaboride are ideal cathodes materials providing for development of up-to-date electron-beam technology for spraying, welding and dimensional processing of metal and alloys.

Main shortage of boride-based materials is low impact strength 2-5 $MPa \cdot m^{1/2}$, which can be improved through armoring with fibers or by synthesizing eutectic composition materials which usually are considered to be natural composites.

In presented work the quasi-binary LaB_6-MoB_2 system ceramic material are studied for the firs time. Materials were obtained using the method of crucibles zone melting of pressed blanks.

Regularity of changing of melting temperature and microstructure of various composition alloys indicates for eutectic character of alloys crystallization. Phases do not interact among themselves since their properties do not change along with phase change, grid parameters remain unchanged. Eutectic has a fine-grain structure consisting of lanthanum hexaboride and molybdenum diboride layers. Leading role in formation of eutectic colony is played by LaB_6, which initiate eutectic crystallization. Eutectic colonies crystallize on primary single crystals - LaB_6 cubes. This is regular as this phase has relativity high melting entropy. The colony contains of six LaB_6 cubic crystal-based tetrahedral pyramids. At that the eutectic pyramids are divided by lanthanum hexaboride plates that are formed as result of growth of base LaB_6 cube, plate structure eutectic grows on sections of eutectic pyramids faces. And as a whole, the eutectic colony is a two-phase cube i.e. a structure of base crystal with high melting entropy.

The results obtained indicated that LaB_6-MoB_2 alloys status diagram has the eutectic character with coordinates of eutectic point T melt. eut. 2400 K and 74.4 mol. % MoB_2, i.e. it produces quasi-binary eutectic in a triple La-Mo-B system. Eutectic has a plat structure. Eutectic colonies grow in the form of tetrahedral pyramids on planes (001) of base LaB_6 crystal. Micro hardness of LaB6 does not change in alloys. Micro hardness of MoB_2 phase in connection with its decay by cooling increases with alloys melting temperature increases because with rapid cooling in the course of zone melting the higher the alloy melting temperature the higher the degree of such transformation.

C10

DC CONDUCTIVITY OF SILICON NITRIDE BASED CARBON-CERAMIC COMPOSITES

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The silicon nitride ceramics are usually known as strongly refractory and enduring materials and they have typical electrically insulating properties. If the reinforcing phase of ceramic composite that have been mainly put in the material because of the improvement mechanical parameters is a good electrical conductor, in that case it is worth to investigate the composite in electrical aspect. In our case carbon nanotubes, black-carbon and graphite were added to the basic silicon nitride ceramic. In this work the electrical conductivity of carbon-ceramic composites was determined. The conductivity according to the type and concentration of the carbon additives was observed by applying four point DC resistance measurements. Insulator and conductor composites in a wide conductivity range can be produced depending on the type and quantity of the additives. The additive types as well as the sintering parameters have influenced the basic electrical properties of the conductor composites.

C11

SYNTHESIS AND THERMO-MECHANICAL PROPERTIES OF $MgSiN_2$ AND $LaSi_3N_5$

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Several ternary nitrides have been investigated intensively for substrate applications because of their potentially high thermal conductivity in combination with a high electrical resistivity, or as candidates for long lasting ceramic phosphors. Among others $MgSiN_2$ has been considered to be potentially interesting for substrate applications, and together with $LaSi_3N_5$ also as a host lattice for luminescent materials. Although the calculated thermal conductivity of $MgSiN_2$ is 75 $W \cdot m^{-1}K^{-1}$, the reported experimental values are in the range 17–25 $W \cdot m^{-1}K^{-1}$. $MgSiN_2$ has also high hardness (20 GPa), reasonable strength (280 MPa), and fracture toughness (3 $MPa \cdot m^{1/2}$), which makes it suitable for some engineering applications. There is a lack of data about $LaSi_3N_5$. The purpose of this work is to produce $MgSiN_2$ and $LaSi_3N_5$ powders from a

complex mixture of metal silicide, silicon, and Si3N4 starting materials with an intention to combine the already known advantages of binary systems and make the synthesis cheaper. The heat treatment schedule was optimized on the base of DTA-TG analysis.

The Vickers hardness, indentation fracture toughness, microstructure and the thermal conductivity of dense $MgSiN_2$ and $LaSi_3N_5$ will be evaluated.

C12

SYNTHESIS OF COMPOSITE TiN-TiB_2 BY REACTIONARY ELECTRIC DISCHARGE SINTERING METHOD

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At the present work the research of synthesis processes of ceramic composite material (CCM) on the basis of TiN – TiB_2 as the material for cutting tools by processing of the powder mixture TiH_2 (TiH_x)-BN and Ti-BN under reactionary electric discharge sintering method was carried out. CCM on the basis of TiN – TiB_2 was obtained in graphite die under direct passing of superposition of direct electric current and alternating one of higher frequency (about 5 kHz). At the synthesized composite the phase proportion of TiN:TiB_2 was changed in the wide range (from 20% to 80% each phases) by means to addition of active boron or inert titanium nitride in powders initial mixtures. The microstructure, physical and mechanical properties of synthesized composites was investigated. It was assigned that increasing of the direct current density during sintering of samples from tetragonal hydride adjusted to increase of relative density and microhardness, crack growth resistance and abrasive wear-resistance correspondingly. Application of the tetragonal titanium hydride as a starting material is more preferably in comparison with the cubic one and the metallic titanium. At the same time the highest properties (γ_{relat} = 98,50 %, H_v = 23,5 GPa, K_{1c} = 5,3 mPa·$m^{1/2}$) were obtained from mixture on the basis of the tetragonal titanium hydride under the density direct electric current j = 5×10^6A/m^2 that was related with higher purity of starting titanium hydride. This in turn determines the rise of hydrogen partial pressure in the synthesis reaction zone and increases reaction rate.

C13

CERAMIC MICROPACKAGES FOR MEMS APPLICATION

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MEMS technology can be used to integrate ceramics with semiconductors and metals to create systems that serve specific functions.

The RF-MEMS components mounted profiled ceramic micropackages were used on a large scale because of the advantages they have such as their own capacitances and inductances (fraction of pF and nH respectively), reduced microwave losses, very small dimensions, mechanical robustness and environment attack resistance. Ceramic technology provides excellent mechanical strength, thermal conductivity, heat durability, electrical performance with TCE matching to silicon and boro-silicate glass.

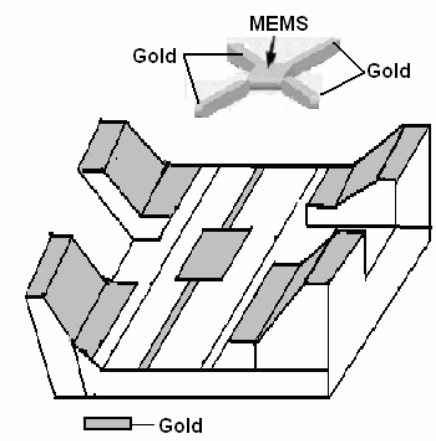

Figure 1. Ceramic micropackages (LID - leadless inverted devices).

The ceramic substrate used in manufacturing of profiled (LID) micropackages has a content of alumina of 99.6%. By using technological processes specific to electronic microtechnologies (metallic thin film deposition, photolithography photoresist, mask etching) ceramic structures with a certain metallization configuration and an exceptional dimensional precision, dimensional tolerance being of the order of microns, are obtained. The selection of a ceramic material for applications in microelectronics and MEMS (FIg.1) application is usually a compromise, according to the importance attached to the different properties of the final product within the low and high frequence applications, respectively.

Profiled and planar ceramic substrates must be physical, chemical and electrical compatible with thin metallic films deposed on them (in a geometrical configuration) and these must be compatible with the mounting components. In fact there is a long list of necessary proprieties of plane and profiled ceramic substrate: very good planarity, low flexure distortions, good adhesion of metallic films, heat shock resistance, thermal coefficients closed from the film ones, high purity, absence of porosity, uniform microstructure low surface's rugosity, very strictly tolerances, possibility of modern mounting technology applying, high friability and low cost.

C14

THE PREPARATION OF DENTAL RAW MATERIALS WITH CONTROLLED FRACTION OF LEUCITE CRYSTALS

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This work is dealing with synthesis of leucite material, which can be used for the preparation of the dental composite by subsequent thermal processing. High value of the thermal expansion coefficient makes possible for application of prepared composite in metal-ceramics restorations. New preparation procedure of dental materials is based on syntheses of two separation compounds: crystalline leucite phase prepared at relatively low temperature and matrix.

Previously, hydrothermal synthesis of tetragonal leucite ($KAlSi_2O_6$) was developed in our laboratory. The optimal conditions bring the particles sizes of about 3 μm. The presence of agglomerates in the range of 5-30 μm was demonstrated by optical microscope and measurement of particle size by laser method.

Leucite dental materials were prepared by mixing of different amount of synthetic tetragonal leucite with commercial matrix. Dental composites were prepared from dental material by pressing and firing up to 960°C. Pure matrix was prepared as a reference sample at the same conditions as the composites. Dilatometric measurements demonstrated, that the coefficient of thermal expansion increases by 32 % when 20 wt. % of tetragonal leucite was added into basic matrix. Obtained results indicate a possibility to control both final the composition of composite and the value of its thermal expansion by the addition of tetragonal leucite into matrix.

C15

SYNTHESIS AND CHARACTERIZATION OF Cr DOPED SnO2 NANOPARTICLES USING MECHANOCHEMISTRY

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There is growing interest in synthesizing new semi-conductor materials. Due to its semi-conductor character and simple synthesis, tin-oxide is widely used in the electronic industry for the production of sensors, photocells and batteries. That's why we made CrO_x-SnO_2 composites from $CrCl_2$ and $SnCl_2$ by mechanochemical procedure. We used a FRITSCH Pulverisette-6 type planetary ball mill. In this article we demonstrate the method of synthesis and the results of UV-Vis and mid-IR spectroscopy as well as XPS, TEM, SEM and GTM (a method for monitoring the pressure changes online in the milling drum during the mechanochemical reaction) examinations.

For milling process we used a 250 cm^3 stainless steel milling drum with 40 stainless steel balls of 10 mm diameter. The milling time was 75 minutes at 400 rpm. Samples were calcined in air at 600°C for 2 h unless stated otherwise. The starting materials were 8.3711 g $SnCl_2$, 3.933 g Na_2CO_3, for matrix 13.024 g NaCl and different amount of anhydrous $CrCl_2$. The chromium content of the samples was varied between 10:0.1 to 10:2.0 Sn:Cr ratio in a series of experiments, while in another set the Sn:Cr ratio was fixed at 10:1.0 and the calcination temperature was varied between 200°C and 800°C. After calcination NaCl was removed by washing with distilled water in an ultrasonic bath. The final preparation steps were filtration and drying. We used electron microscopy (TEM, SEM) to characterize the morphology of the nanoparticles and examined the aggregation behavior and average diameter of the SnO_2 and CrO_x nanoparticles as a function of calcination temperature Sn:Cr ratio. The composition of the samples was analyzed by spectroscopic techniques. The pressure vs. milling time curves were used to study the formation kinetics of the nanoparticles in the mechnochemical process.

C16

THE STRUCTURE AND MECHANICAL PROPERTIES OF MULTILAYER NANOCRYSTALLINE TiN/ZrN CONDENSATES OBTAINED BY VACUUM-ARC DEPOSITION

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The TiN/ZrN multilayered condensates were produced by the vacuum-arc deposition technique on stainless steel flat 100×100×0,3 mm substrates, using Ti and Zr plasma flows in reactive nitrogen gas medium with its working pressure $6{,}6 \cdot 10^{-1}$ Pa. The TiN/ZrN multilayered condensates consist of TiN and ZrN sublayers, which have different thickness controlled by predetermined technique parameters. High hardness (45 GPa) and Young's modulus (320 GPa) were obtained for such composites. Obtained experimental data show that physical-mechanical characteristics of such composites considerably differ from separate material properties of which multilayered composites consist. The dependence of hardness and Young's modulus of the composites on sublayers thickness within a range of 100 nm was determined. The researched structure and mechanical properties of the condensates allow to find out their industrial application as coatings of different purposes.

C17

INVESTIGATION OF MINERAL FILLERS FOR THE UTILIZATION OPPORTUNITIES IN THE ROAD CONSTRUCTION

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In this article the author presents some results of those investigations, which were done at the University of Miskolc, Department of Ceramic and Silicate Engineering. In these investigations there were examined 4 different mineral fillers (limestone filler, andesite and basalt fillers), which are the most used fillers in the road constructions in Hungary. During the investigations microstructural tests (grain structure, specifical surface tests and scanning electronmicroscopic test) and lifetime tests (sedimentation test /to define the hydrophile coefficient/), swelling test on standard asphalt specimens) were executed. The author try to find a relationship between the measured parametres (specifical surface,hydrophile coefficient, swelling value). In the knowledge of the results it is possible to select those fillers, which are convenient for road construction. Estabilished by the results, there is a relationship between hydrophile coefficient and specifical surface of the fillers. Therefor it is important – before using these fillers – to examine the hydrophile coefficient and specifical surface of the fillers, and the presence and proportion of submicronal grains.

C18

BIOCOMPOSITES BASED ON CALCIUM PHOSPHATES: PREPARATION AND PROPERTIES

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Calcium phosphates hold great promise as implantation biomaterials since they have the ability to bond to bone. Hydroxyapatite is one of the most promising materials for clinical use due to the fact its chemical and crystallographic structures are similar to that of bone mineral.

The aim of the present study was the comparative investigation of the microstructure and properties of two types of composites based on calcium phosphates prepared at the same conditions.

The porous composites based on synthetic calcium phosphates and biogenic hydroxyapatite with addition of (35.5-41.0) wt % SiO_2-Na_2O-B_2O_3 glass were fabricated through two-stage sintering technique (T_1 = 1100ºC and T_2 = 800°C).

Morphology, density, porosity, mechanical properties and solubility in vitro of prepared composites have been investigated. The materials obtained were analyzed using SEM, TEM, chemical methods, XRD, IR-, and UV-spectroscopy. The presence of monocrystalline blocks 0.4-2.2 μm in a cross-section and 2.9-7.6 μm in a length for composites based on synthetic calcium phosphates and 0.5-1.2 μm in a cross-section and 2.1-12.6 μm in a length for composites based on biogenic hydroxyapatite were established. These blocks form due to recrystallization of solid state during liquid phase sintering of polycomponent system from nanopowders of synthetic calcium phosphates (105-305 nm) and biogenic hydroxyapatite (85-140 nm). The samples investigated have a density of about 2.77-2.92 g/cm^3 and show a porosity of about 25-30 %, thus exhibit a compression strength of about 108-141 MPa. All properties were showed to depend on the amount of glass and type of introduced phosphates.

The investigations in vitro have shown that the porous composites based on synthetic calcium phosphates have a significant higher dissolution in physiological solution in comparison with composites based on biogenic hydroxyapatite.

The results obtained have revealed that the prepared composites combine good mechanical properties with sufficient solubility. The researched materials may be potential candidates as bioactive implant materials.

C19

THERMAL STABILITY OF CHITOSAN/BENTONITE NANOCOMPOSITES

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The most appealing aspect of polymers is the diversity of accessible molecular compositions. As well as having a choice of monomers, this includes molecular architectures such as branched chains, copolymers, polymer blends, and composites with inorganic fillers with different scale size. Polymer composites are playing an ever increasing role in modern society, replacing inorganic and metallic materials in many applications, and also enabling many entirely new purposes. Polymer/clay nanocomposites exhibit many excellent properties (mechanical, thermal, physical and electrical) compared to those of the pure polymer and conventional composites. These kinds of material are superior adsorbents in colored waste water treatment. Bio-polymer nanocomposites are often used as adsorbents, due to their biodegradable and nontoxic nature. It has been found that the thermal stability of some polymers is affected by the type of nano particles. In this study chitosan/bentonite nanocomposites were produced by adding clay into the polymer using solution technique. Bentonite was modified before composite preparation. The glass transition temperature of the samples (as one of the most important properties in determining the applicability of polymeric material in engineering) was investigated by DSC. The effect of the clay content on the acid blue adsorption was determined. Thermal stability was checked by simultaneous DSC/TGA measurement within the temperature range from 30°C to 300°C. The morphology of the composites was studied by SEM. It was estimated that finely dispersed clay particles in the polymer matrix effects the adsorption behaviour. Taking into consideration the area of application, the chitosan seems to be very promising material as the component in nanocomposites for the adsorption of dye from waste water

C20

MICROSTRUCTURE AND DIELECTRIC PROPERTIES BECHAVIOR OF Si_3N_4-SiC HOT PRESSING COMPOSITE

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In present work has been investigated the influence of hot pressing temperature on properties behavior of ceramic matrix composite Si_3N_4 – 10 vol % SiC. The specimens were prepared in inductance oven in low reducing environment (CO) at different isothermal temperatures. The electrical properties and microstructure were measured in directions parallel (I) and normal (II) to hot-pressing axis, some mechanical properties were measured along hot-pressing direction.

High homogeneity of microstructure of material was obtained at two different temperatures – T_1 and T_2. Below T_1 almost identical electrical properties were observed for different measurement directions (Fig. 1) and we assume forming SiC clusters in direction I. Above T_1 and below T_2 we observed deterioration of electrical properties in direction II. Also we suppose that rotation and enlargement of clusters occurs in this temperature interval. Above T_2 electrical properties coming identical again for different measurement directions and in our opinion rotation and disintegration clusters occurs.

Obtained at optimal temperature T_1 specimens are characterized by isotropic structure, homogenization of grain boundary phase, good electrical properties, material consolidation completion, low values of hardness and fracture toughness. Obtained at optimal temperature T_2 specimens are characterized by intensive growth of mass loss of the sample, homogenization of matrix phase, high values of hardness and fracture toughness, high level of anisotropy: bad electrical properties in direction normal to the hot-pressing direction.

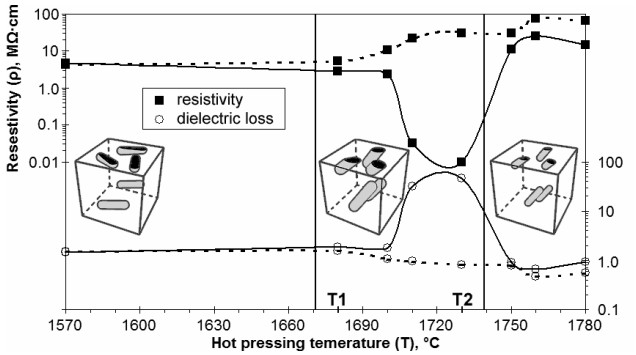

Figure 1. Electrical properties vs. hot-pressing temperature in directions parallel (- - -) and normal (—) to hot-pressing axis.

It has been proved that obtained at optimal temperature T_1 specimens satisfy the requirements and can be used as a high thermal conductivity and dielectric substrate for flip-chip technology.

TRADITIONAL CERAMICS

T1

ENHANCEMENT OF INSULATING PROPERTIES OF BRICK CLAY BY RENEWABLE BIO-MATERIALS

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The use of technologically byproduct bio-materials in various segments of the brick and tile industry increases continuously. During the process of firing the additives previously mixed into the raw clay ignite, thus providing extra thermal energy inside the product decreasing the required external energy need. Beside this effect, the combustion of the bio-materials increases the porosity resulting in an enhanced thermal insulation. We have investigated the effect of some common, agricultural byproducts on the thermal properties of brick products. We added industry relevant amounts of additives (sawdust, rice-peel, seed-shell) to the basic clay composition forming 0, 4, 7 percentage by weight mixtures. The samples were produced, dried and fired using industry standard procedures. To gather precise thermal conductivity data, all the different samples were measured using a RAPID-K type static thermal conductivity measuring instrument. The results show that increasing the quantity of bio-materials in the clay mixture significantly decreases the thermal conductivity. Thus, improving the insulation capability of commercial brick products, while there is only a minor reduction in the mechanical strength. We also ranked the investigated agricultural byproducts for their pore forming properties. It was found that the rice-peel additive decreases the thermal conductivity the most. We have found that the sawdust caused the least improvement in the thermal insulation.

T2

FABRICATION OF SLAG-GLASS COMPOSITE WITH CONTROLLED POROSITY

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The preparation and performance of porous ceramics made from waste materials were investigated. Slag from thermal electrical plant Kakanj, Bosnia & Herzegovina with granulation 0.125+0.063 mm and 20 wt% waste TV glass with granulation < 0.063 mm were used for obtaining of composite slag-glass, with controlled porosity. The obtaining of this composite was realized at 950^{o}C/2h. The open porosity of this composite was 26.8 ± 1.0 %, and the pores were interconnected with size of 250-400 μm. This material possesses E-modulus and bending strength of 10.6 GPa and 45.7 MPa, respectively. Technical coefficient of thermal expansion was $8.47 \cdot 10^{-6}/^{o}$C. Weight lost in 0.1M HCl

after 30 days was 1.2 wt%. The permeability and the form coefficient of the porous composite were $K_0 = 0.12$ Da and $C_0 = 4.53 \cdot 10^5$ m^{-1}, respectively. The porous composite shows great potential for use as filters, diffusers for water aeration, dust collectors, acoustic absorbers, etc.

T3

ADDITIVES IN CLAY BRICK INDUSTRY: POROSITY IMPROVING

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There are many different organic and inorganic additives which may be used in clay brick industry. These substances are used to improve brick porosity, while decreasing drying time and firing energy consumption. This way, reductions of both products volume mass and thermal conductivity occur.

Problems which may befall are clay plasticity decreasing and overfilling with organic additives which can lead to uncontrolled energy releasing during firing. Generally, every density reduction involves remission of compressive strength and thermal conductivity. That is why additives composition should be precisely examined by identifying their effects on porosity improving.

The main objective of this study is to investigate utilization potential of some organic additives in clay bricks. Concrete, the effects of soy crust, wood cutting and coal chat material addition on both durability and mechanical properties of the bricks were investigated. Different ratios of the clay deposit were added in raw-brick clay. The samples (plates, bricks and cubes) were tested by using the standard test methods and compared.

As a result, it was concluded that these materials can be used in building bricks by taking advantage of low cost and environmental protection, whereby thermal conductivity decreases.

T4

BIOCORROSION OF COMPOSITE MATERIALS BASED ON FLY ASH AND CLAY MATERIALS

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Fly ash obtained as waste material from the thermal powder plants is considered a potential raw material due to the fact that its chemical composition is similar to the clay materials. Fly ash is widely used in the design of concrete characteristics, but the new potential for its usage has been the focus of the interest of scientists recently.

The aim of this paper was to design composite materials based on fly ash of defined characteristics and clay materials with the aim to investigate their destruction as the result of microorganisms attacks i.e. biocorossion or ageing process. The fly ash was obtained from thermal power plant REK Bitola, Macedonia. The used clay material was based on illite clay minerals originating from the region of Bitola. The raw materials were characterized from chemical, physical and structural aspect. In order to increase geometrical factor and the activity of materials theirs granulation less than 0.063 mm was used. The content of fly ash varied from 10 to 90 wt.%. Pressing and sintering processes were applied for consolidation process of the powered systems. The sintering process was realized under the following conditions : T= 900, 1000, 1050 and 1100^{o}C/1h using heating rate of 3 and 10^{o}C/min. From the spectrum of the obtained composites the composition made of 60 wt% clay and 40 wt% fly ash showed thermal stability and optimal mechanical properties. The further biocorrosion investigation was focused on the compacts of: fly ash, clay material and composite based on these two materials (40% fly ash and 60% clay) in laboratory conditions using Aspergillus niger fungus. The pore size distribution as well as the appearance of new phases will be followed as they are reliable tools for the texture and structure changes of the fly ash/clay materials compacts.

T5

MAGNETIC CLEANING OF THE GLASS INDUSTRY'S RAW MATERIALS

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In this report we show the effect of magnetic cleaning. We cleaned the quartz-sand, dolomite, feldspar and limestone with Eriez CMT-5 Magnetic Trap, presented in Figure 1.

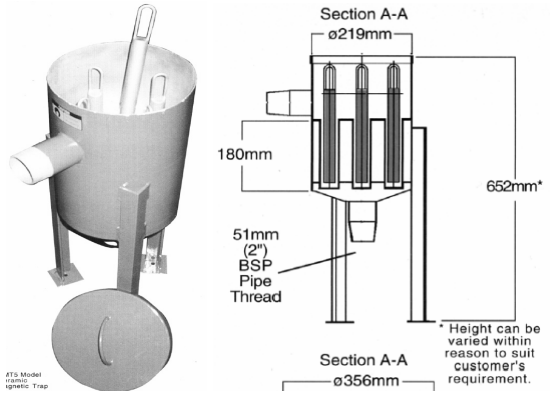

Figure 1. Eriez CMT 5 Magnetic Trap.

Every raw-materials was cleaned for 10 times. After the compare we found that the dolomite and the feldspar together contain more contaminant then the quartz-sand alone (Fig. 2.).

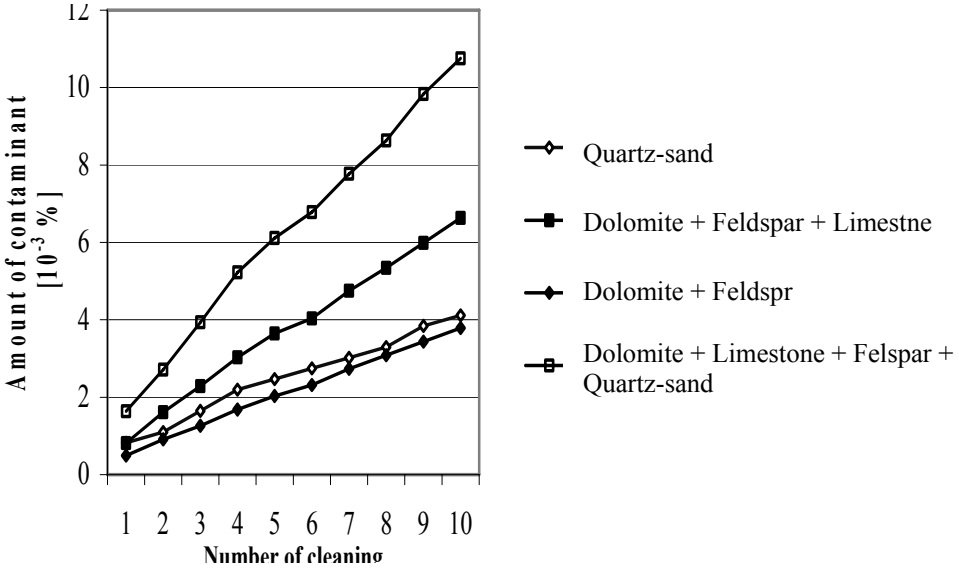

Figure 2. Compare of the results of magnetic cleaning.

T6

DESIGN OF CERAMIC MICROSTRUCTURES BASED ON WASTE MATERIALS

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The progressive changes in ceramic raw materials during firing processes are a complex area. This is partly due to the large number of raw material characteristics, primarily mineral composition, and partly to the relatively inadequate particle distribution in the unfired clay body. The most important starting point is always the optimal raw material composition which should give appropriate physical-mechanical characteristics to the final products, after firing processes, and should provide an efficient and economical production.

The paper analyzes the influence of some additives (fly ashes and waste glass materials) on the development of the ceramic roofing tile microstructure during the thermal treatment. The analyzed raw material mixtures were: the standard raw material mixture from Kanjiza (Northern part Serbia) and the one whose design was based on the standard raw mixture and the additives. The silica phase obtained during the thermal collapse of the clay minerals, in the presence of the glass additive, bounded better CaO and MgO components released from the carbonates. The crystalline phases like plagioclases were performed in a considerable quantity and the products with new physical characteristics were formed.

T7

DENSE CERAMICS MATERIALS OBTAINED FROM FLY ASH

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The aim of this investigation was the production of dense ceramics materials suitable for building materials by using only coal fly ash received as by-products from the power plant REK Bitola, Macedonia. Five types of fly ashes were investigated depending on sizes of fly ash particles. These particle sizes depend on the zone of electro filtration (four zones of electro filter and one from the collection zone). Fly ashes were

characterized from chemical, physical and structural aspects. The fly ashes were granulated using certain amount of water followed by pressing. Sintering of the green samples was realized in the temperature interval from 950°C to 1100°C and one hour holding time at maximal temperature, using heating rates of 3°C/min and 10°C/min in the dynamic part of the thermal treatment. The obtained dense ceramics materials were characterized from the aspects of their thermal, physical and mechanical properties. The highest values of the bending strength, E-modulus and compressive strength were achieved on compacts made from fly ash from IV zone at 1100°C. The bending strength and E-modulus were 47.01 ± 2 MPa, and 22.23 ± 1 GPa, respectively, and the compressive strength was 94 ± 5 MPa. For the same samples water absorption shows the value 2.39 %. The chemical and physical properties of the dense materials make them suitable for a potential wide range of application in the building industry.

T8

INVESTIGATION ON PHOTOCATALYTIC ACTIVITY ON CLAY ROOFING TILE

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Heterogenous photocatalysis is a useful technique for the degradation of many contaminants in air, water, or on solid surfaces. The general scheme for the photocatalytic reaction of organic compounds involves the excitation of the semiconductor by irradation with supraband gap photons and migration of the electron-hole pairs to the surface of photocatalyst, where the holes may react with adsorbed H_2O or OH^- to form hydroxil radicals. TiO_2 is one of the most studied because of its stability and photosensitivity. Photocatalytic activity of TiO_2 film would be more accessible to assessment if it were associated with a colour change of model pollutant. One of the most used azo-dyes, methylene blue, has been chosen as a model compound. The aim of this paper was to investigate the application of methylene blue reaction in order to estimate the photocatalytic activity of TiO_2 film on clay roofing tile. Conditions which are used on the nonporous surfaces, such as glass, are simulated. Reduction of methylene blue by photocatalytic activity of TiO_2 with or without preadsorption of dye is mesured. For the clay roofing tile with porous surface it is necessary to define the steady-state of dye preadsorption.

T9

SEVNB METHOD FOR FRACTURE TOUGHNESS ON TRADITIONAL CERAMICS

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Fracture toughness is the measure of resistance to brittle fracture when a crack is present in material. The reliable value of fracture toughness is of great importance especially in the purpose of selection and application of materials. There are many methods currently used for fracture toughness determination of ceramics, although, in some cases, during realization, difficulties are present and the obtained results are often unreliable. Recently, Single-Edge-V-Notched Beam (SEVNB) as a new method for advanced ceramics was introduced. However, the application on traditional ceramics is not yet established. For that reason, in this paper, SEVNB method was applied on traditional ceramic material in order to determine applicability of the method.

In this study, a clay roofing tile system material in green and fired condition, was used. The raw material was industrial design mixture of two clay materials, consisted of illite-kaolinite minerals and the carbonates. The roofing tiles were produced in standard industrial conditions by extrusion and firing at T_{max} = 960°C, τ = 24h. After firing, roofing tiles were consisted of quartz, plagioclase, gelenite and diopside minerals. In addition, the total porosity was diminished by firing process from 29.31% to 20.31%.

For testing, two sample sizes were used, the recommended 3×4×45 mm and the larger one 10×10×45 mm. The samples were cut in direction parallel and normal to extrusion, while notches were cut on the sample surface and on the sample thickness. Notches were prepared manually by a razor blade, inside a pre-cut shallow slot. After fracture, the samples were examined by JEOL 6460LV scanning electron microscope (SEM) operating at 25 kV.

The obtained results for samples with 3×4 and 10×10 mm cross section revealed significant difference between KIC values. For 10×10 mm samples they were higher than corresponding 3×4 mm. This observation could be explained by: i) lamellar texture initiated during extrusion and firing; and ii) razor blade handling which may cause chipping on notch edges. Those effects had a higher influence on the smaller samples resulting in decreasing of KIC values.

Finally, the results shown, that for traditional ceramic SEVNB method can be used successfully and that the larger cross section samples are more preferable

CULTURE HERITAGE

H1

STUDY OF SANDSTONE DETERIORATION OF THE HISTORICAL BUILDINGS (SLOVENIA)

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Proper knowledge of stone properties and understanding of degradation causes is necessary for successful maintenance, protection and restoration work of buildings made of natural stone. There is a range of degradation phenomena related to sandstone weathering. Examples of some common sandstone weathering mechanisms are granular disintegration, scaling, delamination, fissures due to mechanical stresses and biodeterioration. Present study is focused on identifying the petrographycal factors which affect the durability of the sandstone which were determined by X-ray diffraction, optical and scanning electron microscopy. Some petrophysical properties of sandstone like porosity, capilarity and hydric dilatation were determined. An attempt is made to quantify the influence of microorganisms within the general frame of rock decay mechanisms. Using different techniques we have isolated diverse filamentous fungi from the sandstone. The main mechanisms of sandstone deterioration are mineral dissolution, secondary minerals and soluble salt crystallization. It is possible to observe the variety in porosity between fresh and deteriorated stone as well.

H2

HISTORICAL MATERIALS FROM THE MEDIEVAL FORTRESS BAČ

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Conservation and restoration of cultural heritage are the objects of great interest worldwide. For setting the correct methodology for the procedures of the restoration it is very important to have the right information about the state of the object and the characteristics of the original materials. The basis of our examinations were clay products (samples of bricks, terracotta and clay roof tile) from the Middle ages, the

fortress in Bač. The following methods were used: x-ray diffraction, classic chemical analysis, SEM-EDS, Hg-porosimetry and dilatometry. Based on the used methods, mineral composition, temperature and regime of firing and textural properties of the examined materials were determined. The degree of destruction of examined materials was also identified, in order to find compatible materials for future techniques of conservation and restoration.

H3

GENESIS OF INTERNATIONAL APPROACH IN PROTECTION OF WORLD HERITAGE DURING 20th CENTURY – A REVIEW

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The recognition of versatile values of the world heritage, both in aesthetic and social sense, and its cumulative contribution to understanding of mankind development are the result of an international approach that was developed during the 20th century. The modern conservation theory is based on a critical historical evaluation of wide range of values, as reflected in various international conventions and documents. A review of the main international documents and institutions in the field of world heritage treatment provides a reliable source for better perception of genesis of multidisciplinary approach to preservation and conservation theory and practice during the 20th century on local, national and international level.

H4

DETERMINATION OF WALL-PAINTING TECHNIQUES OF BODJANI MONASTERY

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Two representative groups of mortar samples with pigments from Bodjani monastery (North of Serbia, 15th centuries) were studied. The main aim of this work was the determination and scientific confirmation of the painting technique used in Bodjani monastery. The basic samples of both groups were made based on lime mortar with thin

colored layers. Two common models of painting technique were determined : *al Fresco* and *al Secco*. The *al Fresco* technique was a technique with direct deposition of paint on the fresh mortar, while the *al Secco* technique was the painting of the already dried mortar with pigments in the presence of different organic binders.

The determination of the used technique, as well as the used pigments, were carried out by use of the following methods: optical microscopy of the cross section, Micro Ramman Spectroscopy and SEM/EDS techniques and Gas chromatography.

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