Faculty of Technology
University of Novi Sad

9th Students' Meeting
and
2nd ESR COST MP0904 Workshop

Book of Abstracts

SM 2011
COST SIMUFER

Novi Sad, Serbia, November 16-18, 2011

CONFERENCE for YOUNG SCIENTISTS

The Ninth Students' Meeting, SM-2011
The Second ESR Workshop, COST MP0904

PROGRAMME
and
BOOK OF ABSTRACTS

November 16-18, 2011
Novi Sad, Serbia

Programme and Book of Abstracts of the Conference for Young Scientists (The Ninth Students' Meeting - SM-2011, and The Second ESR Workshop, COST MP0904) publishes abstracts from the field of ceramics, which are presented at traditional international the Conference for Young Scientists.

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Preface

The Conference for Young Scientists is organized by the Department of Materials Engineering, Faculty of Technology, University of Novi Sad, Serbia (November 16-18, 2011). This year it consists of two events The Ninth Students' Meeting, SM-2011, "Processing and Application of Ceramics" and The Second Early Stage Researchers Workshop of the COST Action MP0904 "Single- and multiphase ferroics and multiferroics with restricted geometries".

The Students' Meeting started first as a national meeting in 1998, 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 early stage researchers, being MSc and PhD students or young doctors. Additionally, major general topics of broad interest will be presented by experienced scientists through the invited lectures and active discussions are expected in the specific domains of the oral presentations. We strongly hope that the overall activities during this event will create for the young researchers a fruitful platform for finding new topics, ideas and approaches for their scientific research and an excellent opportunity for establishing connections and finding proposals for collaborations.

It is our deepest belief that by gathering the young human resources in Europe trough such events, an active European scientific network can be created, for the general benefit of R&D in Europe. Therefore, we strongly appreciate that the European Ceramic Society recognized the efforts and the enthusiasm we have put into this idea of building the bridge between young researchers and we truly hope that the European Ceramic Society will support this initiative in the future. Special thanks to the JECS Trust Fund and COST MP0904 for strong financial support of the Meeting.

A total number of 112 presentations given by young researchers and 11 invited talks coming from 27 countries with multidisciplinary profiles will be presented during the conference. It should be emphasised that presented topics cover research subjects of the highest scientific interest: experimental, theoretical and applicative aspects of synthesis, processing, advanced nano/microscale and functional characterisation of various types of structures and ceramic materials.

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

On behalf of the organising committee, a warm welcome to all participants of our Conference and hope you will have a successful Meeting with many interesting discussions. Have a good and fruitful time in Novi Sad!

Editors

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WEDNESDAY, NOVEMBER 16, 2011.

08.00 – 10.00 h – Registration (Conference desk)

10.00 – 10.30 h – Oppening (Amphitheater)

10.30 – 11.15 h – IA1 Invited lecture (Amphitheater)
Teodor Atanackovic, Serbia, Hooke's law and its generalization

11.15 – 12.00 h – IA2 Invited lecture (Amphitheater)
Louis Winnubst, The Netherlands, Fabrication, microstructure, transport properties of ceramic membranes

12.00 – 13.30 h – Welcome Party (Ceremonial Hall)

13.30 – 15.15 h – Section 1

Advanced Ceramics (Blue Hall)

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13.45 – 14.00 h	**A2 – V. Kalyani, et al., Italy** Synthesis of perovskite nanostructure via simple hydrothermal process
14.00 – 14.15 h	**A3 – P. Pusztai, et al., Hungary** Synthesis and characterization of one-dimensional cerium-phosphate nanostructures
14.15 – 14.30 h	**A4 – J. Lukovic, et al., Serbia** Synthesis of nanocrystalline tungsten carbide
14.30 – 14.45 h	**A5 – M. Fang, et al., Sweden** Rapid mixing: a new route to synthesize magnetite nanoparticles with high magnetization at room temperature
14.45 – 15.00 h	**A6 – V. Tsukrenko, et al., Ukraine** Hydrothermal synthesis of nanocrystalline zirconia powders
15.00 – 15.15 h	**A7 – I. Dugandžić, et al., Serbia** Assembling and spheroidization of colloidal TiO_2 nanoparticles via aerosol route

Ceramic Composites (Class Room, R10)

13.30 – 13.45 h	**C1 – A. Mielewczyk-Gryń, et al., Poland** Lanthanum ortho-niobate based composite material for IT-SOFC anode supports
13.45 – 14.00 h	**C2 – R. Poreba, et al., Czech Republic** The influence of silica particles and layered nanosilicates on properties of organic-inorganic nanocomposites
14.00 – 14.15 h	**C3 – S. Matijašević, et al., Serbia** Glass-ceramics prepared from $Li_2O•Nb_2O_5•SiO_2$ glass containing TiO_2
14.15 – 14.30 h	**C4 – Y. Damaschek, et al., Germany** Study on low-temperature printable ferroelectric capacitors based on $BaTiO_3$ - polymer nanocomposites
14.30 – 14.45 h	**C5 – E. Markiewicz, et al., Poland** Pyroelectric response of polymer-ceramics composites of 0-3 connectivity
14.45 – 15.00 h	**C6 – D. Dobó, et al., Hungary** Morphological characterization of inorganic nanocomposites
15.00 – 15.15 h	**C7 – C. Ciobanu, et al., Romania** The influence of some additives containing SiO_2 on physico-mechanical properties of dry mortars

15.15 – 16.00 h – IM1 Invited lecture (Amphitheater)

Liliana Mitoseriu, Romania, Size and interface effects in nanostructured dense ferroelectric ceramics

16.00 – 16.15 h – Coffe Break

16.15 – 18.30 h – Section 2

Ferroics and Multiferroics (Blue Hall)

16.15 – 17.00 h	**IA3 Invited lecture – B. Malič, et al., Slovenia** Solution-derived functional oxide thin films: Crystallization-microstructure-functional properties
17.00 – 17.15 h	**M1 – N. Horchidan, et al., Romania** Dielectric and nonlinear properties of $BaTi_{1-x}Sn_xO_3$ ceramics prepared by solid state method
17.15 – 17.30 h	**M2 – R. Frunza, et al., Slovenia** Transparent oxide thin films from solutions processed at low temperatures
17.30 – 17.45 h	**M3 – R. Lowndes, et al., United Kingdom** Control of the microwave dielectric properties of $Ca_{(1-x)}Nd_{2x/3}TiO_3$

17.45 – 18.00 h	**M4 – M. Kachlik, et al., Czech Repblic** Processing of bulk $Sr_{0,3}Ba_{0,7}TiO_3$ ceramic
18.00 – 18.15 h	**M5 – M. Ivanov, et al., Lithuania** Dielectric spectroscopy of nanograin PSN ceramics
18.15 – 18.30 h	**M6 – M. Vijatović Petrović, et al., Serbia** Barium titanate properties enhanced by attrition milling

Advanced Ceramics (Class Room, R10)

16.00 – 16.15 h	**A8 – L. Benešová, et al., Czech Republic** Effect of calcination temperature on microstructure and mechanical behaviour of aluminosilicate inorganic polymers
16.15 – 16.30 h	**A9 – A. Dubnika, et al., Latvia** Preparation and characterization of silver doped hydroxyapatite scaffolds
16.30 – 16.45 h	**A10 – R. State, et al., Romania** Theoretical approach of oxide hardness
16.45 – 17.00 h	**A11 – L. Kobera, et al., Czech Republic** Solid state NMR spectroscopy of aluminosilicate inorganic polymer
17.15 – 17.30 h	**A12 – O. Tokariev, et al., Germany** Mechanical property characterization of transparent $MgAl_2O_4$
17.30 – 17.45 h	**A13 – D. Tsoukleris, et al., Greece** Chemical and mechanical stability of nanoengineered titania thin films
17.45 – 18.00 h	**A14 – P. Horňák, et al., Slovakia** The effect of deposition conditions of different magnetron sputtering mode on nanohardness of WC/C coatings
18.00 – 18.15 h	**A15 – J. Sułowska, et al., Poland** Structural role of Cu in soil active glasses
18.15 – 18.30 h	**A16 – O. Kornienko, et al., Ukraine** Interaction of cerium oxide with erbia at 1100°C

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THURSDAY, NOVEMBER 17, 2011.

09.00 – 10.30 h – Section 3

Ferroics and Multiferroics (Blue Hall)

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09.15 – 09.30 h	**M8 – J. Griffiths, et al., United Kingdom** P-Type $Na_xCo_2O_4$-based thermoelectric ceramics for energy generation from waste heat
09.30 – 09.45 h	**M9 – G. Stojanović, et al., Serbia** Ink-jet printing on flexibile substrates and sensors fabrication
09.45 – 10.00 h	**M10 – P. Heijboer, et al., France** TTB crystal growth by the vertical optical floating zone method: optimization of rod sintering and growth parameters
10.00 – 10.15 h	**M11 – C. Larosa, et al., Italy** Cobalt nanostructures by electroless reduction: The role of surfactants and complexating anions
10.15 – 10.30 h	**M12 – L. Curecheriu, et al., Romania** dc-electric-field dependence of dielectric constant in ferroelectric systems

Traditional Ceramics (Class Room, R10)

09.00 – 09.15 h	**T1 – P. Zemenová, et al., Czech Republic** Ageing of samples of low-firing ceramics in hydrothermal conditions
09.15 – 09.30 h	**T2 – O.C. Mocioiu, et al., Romania** Improvement of resistance at wearthering of glasses which reproduced historical items by protective coating
09.30 – 09.45 h	**T3 – D. Despotović, Serbia** Different methods in the examination of the movable cultural heritage in the activities of the conservation department of the Gallery of the Matica srpska
09.45 – 10.00 h	**T4 – M. Kuneš, et al., Czech Republic** Characterization and evaluation of the system ceramic body - glaze
10.00 – 10.15 h	**T5 – M.Taykurt Daday, et al., Turkey** The investigation of new opaque glazes for floor tile
10.15 – 10.30 h	**T6 – M. Arsenović, et al., Serbia** Encapsulating sludges in brick structure

10.30 - 10.45 h – Coffe Break

10.45 – 11.45 h – Section 4

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11.00 – 11.15 h	**C9 – R. Bystricky, et al., Slovakia** Role of sintering additives on SiC formation in Si_3N_4 ceramics
11.15 – 11.30 h	**C10 – D. Deschuyteneer, et al., Belgium** Laser cladding of metal/ceramic composites for wear applications
11.30 – 11.45 h	**C11 – O. Hanzel, et al., Slovakia** Influence of carbon allotropes on the preparation and mechanical properties of Al_2O_3-C composites

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11.00 – 11.15 h	**A18 – A. Peltekov, et al., Bulgaria** **S**ynthesis of ferrites of the system MeO (MeO – Zn, Cd) and study of their solubility in solutions of H_2SO_4 after preliminary treatment
11.15 – 11.30 h	**A19 – N. Paunović, et al., Serbia** Magnetic properties of Fe^{2+}/Fe^{3+} doped CeO_2 nanocrystals
11.30 – 11.45 h	**A20 – B.M. Fraygola, et al., Brazil** Magnetic and ferroelectric phase coexistence and magnetoelectric proprieties in multiferroics PFW-PT ceramic solid solution

11.45 – 12.30 h – IA4 Invited lecture (Amphitheater)

Paula Vilarinho, Portugal, Thick films by field assisted solution processing: fundamentals application and opportunities

12.30 – 13.15 h – IA5 Invited lecture (Amphitheater)

Marco Deluca, Austria, Micro-Raman spectroscopy applied to the characterisation of ceramic materials

13.15 – 14.45 h – Lunch (Ceremonial Hall)

14.45 – 15.30 h – IA6 Invited lecture (Amphitheater)

Markus Winterer, Germany, Towards functional nanoparticles: Gas-phase doping

15.30 – 17.00 h – Section 5

Advanced Ceramics (Blue Hall)

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15.45 – 16.00 h	**A22 – M. Busch, et al., Germany** Synthesis of zeolite-based catalysts by chemical vapor infiltration for nitrogen oxide conversion
16.00 – 16.15 h	**A23 – J. Heinecke, et al., Switzerland** Fabrication of multilayered cellular ceramics via radiation curable dispersions
16.15 – 16.30 h	**A24 – I. Zake, et al., Latvia** Porous alumina ceramics modified with SiC nanopowder
16.30 – 16.45 h	**A25 – D. Hautcoeur, et al., Belgium** Porous ceramics by ice templating: Freezing characteristics, rheological and microstructure properties
16.45 – 17.00 h	**A26 – A. Iatsenko, Ukraine** Development of permeable highly porous cellular glass ceramics for sorption-reduction Fe-containing materials water remediation

Ferroics and Multiferroics (Class Room, R10)

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15.45 – 16.00 h	**M14 – C.S. Olariu, et al., Romania** Modeling methods for composites electromagnetic properties
16.00 – 16.15 h	**M15 – M. Albino, et al., France** Original dielectric behaviors related to the Tetragonal Tungsten Bronze structure
16.15 – 16.30 h	**M16 – Ž. Cvejić, et al., Serbia** Electrical conductivity and magnetic properties of nanocrystalline In-doped zinc ferrites
16.30 – 16.45 h	**M17 – J. Olšanová, et al., Slovakia** Synthesis, sintering and characterisation of strontium doped $K_{0,5}Na_{0,5}NbO_3$ceramics
16.45 – 17.00 h	**M18 – F. Gheorghiu, et al., Romania** Investigation of functional properties of $BiFeO_3$ ceramics prepared by one-step sintering method

17.00 – 17.15 h – Coffe Break

17.15 – 18.45 h – Section 6

Advanced Ceramics (Blue Hall)

17.15 – 17.45 h	**IA7 Invited lecture – K. Giannakopoulos, Greece** Field emission scanning electron microscopy (FE SEM): Capabilities and limitations
17.45 – 18.00 h	**A27 – L. Mahnicka, et al., Latvia** Porous mullite ceramics formation and modification with some additives
18.00 – 18.15 h	**A28 – P. Gdaniec, et al., Poland** Application of various pore-formers for ceramic materials
18.15 – 18.30 h	**A29 – M. Kodols, et al., Latvia** Bi_2WO_6 photocatalyst nanopowder synthesis and photodegradation of MB
18.30 – 18.45 h	**A30 – J. Pantić, et al., Serbia** Sphene based ceramics

Advanced Ceramics (Class Room, R10)

17.15 – 17.30 h	**A31 – K. Gdula-Kasica, et al., Poland** Characterization of doping influence on barium cerate properties
17.30 – 17.45 h	**A32 – Z.V. Mocanu, et al., Romania** Investigation of La-doped $BaTiO_3$ ceramics prepared by alternative methods
17.45 – 18.00 h	**A33 – J. Bobic, et al., Serbia** Niobium doped barium bismuth-titanate ceramics
18.00 – 18.15 h	**A34 – T. Kainz, et al., Austria** Study of theformation reaction of lead-free (1-x)BNT - xBKT ceramic
18.15 – 18.30 h	**A35 – M. Vuković, et al., Serbia** Fine grained varistors prepared from ZnO nanoparticles
18.30 – 18.45 h	**A36 – N. Horchidan, et al., Romania** Contributions to the study of non-linear dielectric properties of $BaTi_{1-x}Sn_xO_3$ ceramics

20.00 h – Social Event

FRIDAY, NOVEMBER 18, 2011.

09.00 – 10.30 h – Section 7

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10.00 – 10.15 h	**A41 – S. Perko, et al. Slovenia** Densification and strength of porous Y-TZP ceramics
10.15 – 10.30 h	**A42 – V. Pouchly, et al., Czech Republic** Master Sintering Curve applied on Two Step Sintering

Ferroics and Multiferroics (Class Room, R10)

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09.15 – 09.30 h	**M20 – N. Pavlović, et al., Belgium** $BiFeO_3$ thin films: Between decomposition and phase stability
09.30 – 09.45 h	**M21 – L. Padurariu, et al., Romania** Ferroelectric-relaxor crossover of $BaTiO_3$-based solid solutions and grain size effect in $BaTiO_3$ described by Monte Carlo simulations
09.45 – 10.00 h	**M22 – Š Bagdzevičius et al., Lithuamia** Broadband dielectric investigation of bismuth modified lead-free strontium titanate ceramic
10.00 – 10.15 h	**M23 – Z.V. Mocanu, et al., Romania** Functional properties of $Mg_xNi_{1-x}Fe_2O_4$ ferrite
10.15 – 10.30 h	**M24 – B. Mojić, et al., Serbia** Preparation of multiferroic titanate/ferrite composite ceramics with core/shell structure

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Advanced Ceramics (Blue Hall)

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11.15 – 11.30 h	**A45 – M. Perušić, et al., Bosnia & Herzegovina** Specific of the results of bauxite thermal analysis
11.30 – 11.45 h	**A46 – L. Gorjan, et al., Slovenia** Strength of wick-debinded ceramic parts prepared by low pressure injection molding

Advanced Ceramics (Class Room, R10)

10.45 – 11.00 h	**A47 – B. Čolović, et al., Serbia** Influence of hydroxyapatite pore geometry on drug release kinetics
11.00 – 11.15 h	**A48 – M. Miljevic, et al., Serbia** Synthesis and characterization of bio-calcium phosphates
11.15 – 11.30 h	**A49 – S. Ilić, et al., Serbia** Synthesis and characterization of mullite
11.30 – 11.45 h	**A50 – S. Stojanović, et al., Serbia** Application of MTT viability test for biocompatibility examination of N-CP/DLPLG nanoparticles on cell cultures

11.45 – 12.30 h – IM2 Invited lecture (Amphitheater)

Mario Maglione, France, Integration of ferroelectric materials: a long-term view

12.30 – 13.15 h – IM3 Invited lecture (Amphitheater)

Marlies Van Bael, An Hardy, Belgium, Solution based synthesis of electroceramic nanostructured materials

13.15 – 14.45 h – Lunch (Ceremonial Hall)

14.45 – 15.30 h – IM4 Invited lecture (Amphitheater)

Juras Banys, Lithuania, Broadband dielectric spectroscopy of ferroelectrics and related materials

15.30 – 17.00 h – Section 9

Advanced Ceramics (Blue Hall)

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16.00 – 16.15 h	**A53 – C. Benel, et al., Germany** Nanostructured thin film $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ synthesized via spray pyrolysis for micro-SOFC application
16.15 – 16.30 h	**A54 – G. Stanciu, et al., Romania** Structural and electrical properties of $YBa_2Cu_3O_{7-x}$ thin films deposited by PLD and RF assisted PLD
16.30 – 16.45 h	**A55 – N. Kugyela, et al., Hungary** Photosensitization of titanate nanowires with core-shell quantum dots
16.45 – 17.00 h	**A56 – D. Stepanovs, et al., Latvia** Semiconducting properties of titanium oxide containing ceramics

Ceramic Composites (Class Room, R10)

15.30 – 15.45 h	**C12 – A. Butlers, et al., Latvia** Synthesis and properties of titania/alumina ceramic foam formed by catalytic hydrogen release
15.45 – 16.00 h	**C13 – B. Bochentyn, et al., Poland** Microstructure and electrical properties of $SrTi_{0.98}Nb_{0.02}O_{3-\delta}$-based composites applied as porous layers
16.00 – 16.15 h	**C14 – N. Laufer, et al., Hungary** Dielectric relaxation spectroscopy of titanate nanostructures
16.15 – 16.30 h	**C15 – I. Cherniakova, et al., Ukraine** Effect of the grain size of conductive particles on microstructure and electrical properties of Si_3N_4-ZrC ceramics composites
16.30 – 16.45 h	**C16 – I.V. Ciuchi, et al., Romania** Determination of the Compact Bone Mineral Volume Fraction by Dielectric Investigations
16.45 – 17.00 h	**C17 – S. Likhomanova, et al., Russia** Mechanisms of optical limiting in a COANP solution doped with fullerenes C_{70}: Applicability for the optoelectronic devices

17.00 – 17.15 h – Coffe Break

17.15 – 18.45 h – Section 10

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18.00 – 18.15 h	**C19 – M. Štefanič, et al., Slovenia** Synthesis of bioactive calcium phosphate coatings on ZrO_2 (Y-TZP) ceramics for applications in medicine
18.15 – 18.30 h	**C20 – R. Halgaš, et al., Slovakia** Cross-section hardness maping of human teeth
18.30 – 18.45 h	**C21 – N. Petrović, et al., Serbia** Antimicrobial properties of ceramic based nanocomposite biomaterials

Advanced Ceramics (Class Room, R10)

17.15 – 17.30 h	**A57 – B. Bajac, et al., Serbia** Bismuth titanate thin films prepared by wet-chemical technique
17.30 – 17.45 h	**A58 – B. Fórizs, et. al., Hungary** Gas sensors based on WO_3 nanoparticles, WO_3 and WO_3/TiO_2 nanowires
17.45 – 18.00 h	**A59 – N. Lazarević, et al., Serbia** Phonon-phonon and electron-phonon interaction in pure and doped $FeSb_2$ single crystals
18.00 – 18.15 h	**A60 – Z.I. Dudás, et al., Romania** The influence of the nature of matrices and of the embedded species upon the physicochemical properties of final sol-gel derived materials
18.15 – 18.30 h	**A61 – M. Nikolic, et al., Serbia** Effect of surface functionalization and process parameters on synthesis of mesoporous silica core-shell particles

18.45 h – Closing of the Meeting (Blue Hall)

The Ninth Students' Meeting, SM-2011
PROCESSING AND APPLICATION OF CERAMICS

The Ninth Students' Meeting, SM-2011
PROCESSING AND APPLICATION OF CERAMICS

Organizer

Department of Materials Engineering, Faculty of Technology,
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INVITED TALKS
ADVANCED CERAMICS

IA1

HOOKE’S LAW AND ITS GENERALIZATION

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We present Hooke’s law and its various of generalizations. Special attention will be paid to generalizations that describe visco-elastic and visco-inertial bodies. Application of fractional calculus (i.e., use of derivatives of real order) to model dissipative behavior of materials will be presented. The restrictions following from the Second law of thermodynamics on the form of constitutive equations will be analyzed and one version of a non-local Hooke’s law and its application will be discussed.

IA2

FABRICATION, MICROSTRUCTURE, TRANSPORT PROPERTIES OF CERAMIC MEMBRANES

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Research in the inorganic membrane group of the University of Twente encompasses the development of new membrane materials, fundamental understanding of (multi-component) transport mechanisms, and the design of membrane processes and membrane reactors. The ceramic materials studied concern porous membranes with pore sizes down to a few Ångstroms as well as dense, oxygen ion conducting, membranes.

Dense and porous ceramic membranes find applications in an expanding range of industries including pharmaceutical, food and beverage, chemical and petrochemical industry, metal finishing and power generation. Challenges that relate to reproducibility in fabrication and a good understanding in the transport versus microstructure relationship needs to be overcome. In this lecture the focal point will be in treating relations between fabrication process, microstructure and transport properties.

Knowledge and control of the microstructure during fabrication is the only way to obtain ceramic materials and devices with special, unique and reproducible properties. It is the aim to develop new, tuneable materials for several technological applications, which requires flexible routes. Examples will be given on from membrane fabrication by e.g sol-gel chemistry and grafting of polymers on ceramic membranes as well as several transport properties.

IA3

SOLUTION-DERIVED FUNCTIONAL OXIDE THIN FILMS: CRYSTALLIZATION - MICROSTRUCTURE - FUNCTIONAL PROPERTIES

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Ceramic functional-oxide thin films with enhanced functional properties, such as dielectric permittivity, voltage tunability, remanent polarisation, or electrocaloric effect, to name only a few, have been studied for different microelectronic applications, including thin film capacitors, memories, sensors, actuators, tunable microwave devices or micro heating/cooling devices.

Thin film microstructure critically influences the functional properties. In case of Chemical Solution Deposition (CSD), the crystallisation and evolution of the film microstructure depend on, and may therefore be tailored by the chemistry of the sol, the choice of the substrate, and by the processing conditions, mainly by the temperatures and times of the individual heat treatment steps - drying, pyrolysis and annealing. For example, the dielectric permittivity and voltage tunability of solution-derived $Ba_{0.3}Sr_{0.7}TiO_3$ (BST) thin films were almost doubled, namely from 345 and 1.47, to 722 and 1.93, as the grain size was increased from 40 nm to 80 nm [1].

The contribution addresses the crystallization and evolution microstructure of selected functional oxide thin films: lead lanthanum zirconate titante, and lead-free perovskites: potassium tantalate and tantalate-niobate, and potassium sodium niobate; and the correlation between the film microstructure and respective functional properties.

In $KTa_{0.6}Nb_{0.4}O_3$, structural analogue of $(Ba,Sr)TiO_3$, strongly enhanced dielectric permittivity and its voltage tunablity could be obtained by optimizing the solution synthesis conditions and the amount of potassium excess, which both contributed to a uniform microstructure of the single-phase perovskite films [2]. Following a similar approach, phase-pure $KTaO_3$ films with a uniform microstructure exhibited dielectric permittivity and dielectric losses value of 230 and 0.017 at 300 K and 14.8 GHz.

In the $(K_{0.5}Na_{0.5})NbO_3$ thin films the amount of alkali excess in solutions influenced the degree of {100} orientation of the perovskite phase, the monoclinic distortion of the unit cell, and the densification, nucleation and growth processes of the films. The films with the granular microstructure, prepared from the solutions with the sodium excess, had the room temperature values of dielectric permittivity and losses of 470 and 0.02, respectively, at 1 kHz [3].

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IA4

THICK FILMS BY FIELD ASSISTED SOLUTION PROCESSING: FUNDAMENTALS, APPLICATIONS AND OPPORTUNITIES

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The state of the art and trends in the processing of thick films by field assisted solution processing is reviewed in this talk. New results on dielectric thick films will be presented and their importance framed within the existing and future needs. This talk covers a few concepts that should be of interest for the functional ceramics community.

The use of thick films is becoming more and more important for applications in which miniaturization is required, as for microelectronic applications. A number of established and emerging thick film deposition techniques is currently available. Among them is the electrophoretic deposition (EPD). As a simple and cost effective process, besides allowing products of different geometries to be processed, EPD has been widely used for the fabrication of high-performance functional thick/thin films.

This talk provides a review on the most important techniques for the deposition and patterning of ceramic thick films, and in particular on EPD. The fabrication by EPD includes the preparation of charged suspensions that under an electric field are attracted and deposited onto a conductive and oppositely charged electrode and a final sintering step. The fundamentals of EPD, including mechanisms, suspension properties and zeta potential, kinetic aspects and deposition parameters will be discussed. Numerous applications for EPD thick films, including coatings, laminated or graded materials, infiltration in porous matrix, etc., will be described as well. Finally the demonstration that the control of substrate constraint and sintering conditions of thick films can be used to control grain anisotropy and thus final properties of functional materials will be presented.

IA5

MICRO-RAMAN SPECTROSCOPY APPLIED TO THE CHARACTERIZATION OF CERAMIC MATERIALS

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In recent years, there has been a significant revival of interest in Raman spectroscopy among the community of material science and engineering. This trend is motivated by the increasing miniaturisation of materials and their increasing structural complexity (layered, hierarchical structures and patterns) on the micro- to nanoscale, designs that are aimed to achieve multiple functionalities within a material system. In order to optimise the design of such materials and devices, a thorough control of the governing physical quantities as well as of the integrity of the overall structure is clearly needed. Advanced characterisation methods must thus provide access to the properties at the lattice level, and must possess an "open architecture" allowing in-situ characterisation in dependence of the relevant physical quantities.

Raman spectroscopy potentially fulfils these needs. Being based on the Raman effect, which is the inelastic scattering of light by matter, it enables obtaining information on (i) the presence of chemical species and phases; (ii) the symmetry, orientation and distribution of crystalline phases; (iii) the order/disorder characteristics of materials in their solid or liquid state and (iv) the (residual) deformation (stress) of the crystalline lattice in solids. Its micro scale spatial resolution, the ease of use and the little need for sample preparation, together with its non-destructiveness, are at the basis of the recent popularity of this technique.

In this talk, the basics of Raman spectroscopy will be presented with an eye on its most common applications in the field of advanced ceramic materials. The potentialities of the technique will be explored and discussed through a series of examples and experimental results involving (i) the study of phase distributions and phase transitions in ceramics; (ii) the study of ferroelectric domain orientation with polarised Raman spectroscopy and (iii) the measurement of residual stress by exploiting the piezo-spectroscopic effect.

IA6

TOWARDS FUNCTIONAL NANOPARTCLES: GAS-PHASE DOPING

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Dopants are often required to generate functional materials, for example yttrium doped zirconia is an ionic conductor, transition metals in zinc oxide introduce magnetism or group III elements in zinc oxide generate electrons as charge carriers. We will discuss the challenges of generating doped nanoparticles, their preparation by chemical vapor synthesis. In order to be functional, the dopants have to be on specific sites in the host lattice. This local structure can be investigated using EXAFS spectroscopy. EXAFS contains element specific information of the local structure through the photoelectron generated by X-ray absorption at the characteristic absorption edge of the element and the amplitude and phase modulation of the signal by the backscattering atoms. The detailed analysis of EXAFS spectra using Reverse Monte Carlo (RMC) simulations makes it possible to obtain information of the location of dopant atoms in the host material.

IA7

FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FE SEM): CAPABILITIES AND LIMITATIONS

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Scanning electron microscopy (SEM) is one of the most valuable techniques for the study of a vast variety of materials and structures. The operation of an SEM will be described, together with the basic principles of the interaction of electrons with the materials; SEM's main limitations will be highlighted. In most of the modern SEMs, the use of Field Emission (FE) electron sources does not simply enhance the limits of operation (with the exploitation for example of Low-Voltage imaging, electron beam deceleration and energy filtering of the detected electrons), but it also offers new options such as the Voltage Contrast Imaging. Examples of work with FE-SEM will be presented in parallel to comparisons with other characterization techniques.

IA8

CERAMIC MATERIALS IN $CaO-P_2O_5$ SYSTEM

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A variety of novel calcium phosphate-based materials for medical applications (CPs) have been developed recently. These materials are biocompatible due to their similarity to chemical composition of inorganic component of the mammalian bones.

According to known phase diagrams and experience of different research groups the use of custom scheme of ceramic production permits to produce ceramic materials or composites for a just restricted number of crystalline phases. These are hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$with Ca/P = 1.67 (HAP), tricalciumphosphate $Ca_3(PO_4)_2$with Ca/P = 1.5 (TCP), pyrophosphate $Ca_2P_2O_7$with Ca/P = 1 (CPP) and polyphosphate or metaphosphate $Ca(PO_3)_2$with Ca/P = 0.5. Sometimes it is necessary to use bioresistive materials such as HAP. The materials with Ca/P ratio below 1.67 are bioresorbable and dissolve in body fluids when implanted. In particular, upon construction of drug delivery systems or tissue engineering constructions for bone regeneration, it is necessary to use bioresorbable materials.

Modern powder technologies, including technology of ceramic composites, need fine powders of high quality. It is preferably to use powders made *via* chemical methods, which exploit the well-known "bottom up" approach. The method of solution precipitation is the most convenient and popular if reactive powders are targeted.

HAP is the only substance from the above list of CPs which can be synthesized *via* precipitation from aqua solutions. Other CPs can form only upon thermal conversion of the following wet-synthesized CPs: Ca-deficient HAP $Ca_{10-x}(HPO_4)_x(PO_4)_{6-x}(OH)_{2-2x}$; amorphous CPs like $3Ca_3(PO_4)_2 \times xH_2O$ and $Ca_2P_2O_7 \times xH_2O$, brushite $CaHPO_4 \times 2H_2O$, monetite $CaHPO_4$, monocalcium phosphate $Ca(H_2PO_4)_2 \times 2H_2O$ or $Ca(H_2PO_4)_2$. TCP can be formed as a result of thermal conversion of Ca-deficient HAP or amorphous CP $3Ca_3(PO_4)_2 \times xH_2O$ which were synthesized at Ca/P = 1.5 and $6 \leq pH \leq 8$. Pyrophosphate can be formed due to thermal conversion of brushite, monetite or amorphous CP$Ca_2P_2O_7 \times xH_2O$. Metaphosphate or polyphosphate can be formed from monocalcium phosphate during thermal conversion. TCP and CPP can be formed due to a solid-state reaction of two calcium phosphates with Ca/P ratio different (above and below) from 1.5 for TCP or from 1 for CPP. It is possible to use the following pairs of powder reagents, *e.g.* "hydroxyapatite/ pyrophosphate" for TCP or "$Ca(PO_3)_2$/TCP" for CPP.

All phase transformations in compacts made from active calcium phosphate powders can affect the microstructure formation of CPs ceramic materials in $CaO-P_2O_5$system.

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ADVANCED CERAMICS

A1

THE QUEST FOR DIRECT GAS-PHASE SYNTHESIS OF p-TYPE TCOs

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Some metal oxides such as ZnO, despite their large band gap, easily show n-type semiconducting behavior [1]. Additional doping to increase their conductivity and high optical transparency makes them ideal materials for photovoltaic cells and flat-panel displays where light needs to enter or leave through a transparent electrode. On the other hand, only very few transparent oxides exist that either intrinsically exhibit p-type behavior or can be doped to become a p-type semiconductor [2]. Their availability would enable the fabrication of transparent electronic devices, such as diodes or transistors, for optoelectronic applications.

"Using" chemical vapor synthesis (CVS) [3] and precursor flash evaporation [4] as precursor delivery method, nanoparticles are prepared from solid Cu and Al precursors to form $CuAlO_2$. X-ray diffraction and Rietveld refinement are used to structurally characterize the material.

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A2

SYNTHESIS OF PEROVSKITE NANOSTRUCTURE VIA SIMPLE HYDROTHERMAL PROCESS

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The hydrothermal technique is becoming one of the most important tools for advanced materials synthesis [1]. $Bi_4Ti_3O_{12}$ is a ferroelectric material which has relatively low coercive field, low dielectric constant, high Curie temperature (675°C) and exhibits potential applications in nonvolatile memory, optical memory. On the other hand Strontium titanate ($SrTiO_3$) is an inorganic conductor with a perovskite structure (space group: Pm3m, cubic structure with a = 3.905 Å). Properties of nanomaterial is not only depends on their chemical composition but also on their size and shape. Henceforth it is worth to produce different shape of nanomaterial. Many efforts have been made in synthesizing perovskite nanostructure with controlled shapes such as $Bi_4Ti_3O_{12}$ nanorods, nanosheets [2]. Up to now, there is challenge to synthesize $Bi_4Ti_3O_{12}$ nanostructures at low temperature.

Here we are summarizing the hydrothermal synthesis of 1D, 2D and 3D nanostructure of $Bi_4Ti_3O_{12}$ and $SrTiO_3$ by simply altering the Ti^{+4} precursors. In all case we used Polyethylene glycol as organic additives and NaOH as base at 200°C with different time. In fig a), a typical SEM image shows the growth of thin transparent lamellar nanoplates of $Bi_4Ti_3O_{12}$. The diameter of these nanoplates ranges from 5 to 10 nm. The phase composition of these nano materials was confirmed by XRD and EDS analysis. Here Bi_2O_3 is used as bismuth source in all case. In case of $SrTiO_3$, we obtained different shape of final material with different Ti^{+4} precursors by keeping same reaction parameter. In fig b, a typical SEM image shows well isolated nanowires of $SrTiO_3$ which is made up of well aligned nanocubes on the surface of template. In case of $SrTiO_3$ nanowires we used as prepared Anatase TiO_2 nanaowires as self scarifying templates (chemical template). Fig a) $Bi_4Ti_3O_{12}$ nanoplates using $Ti(0C_4H_9)_4$, b) $SrTiO_3$ nanowire using Anatase TiO_2 nanowire template.

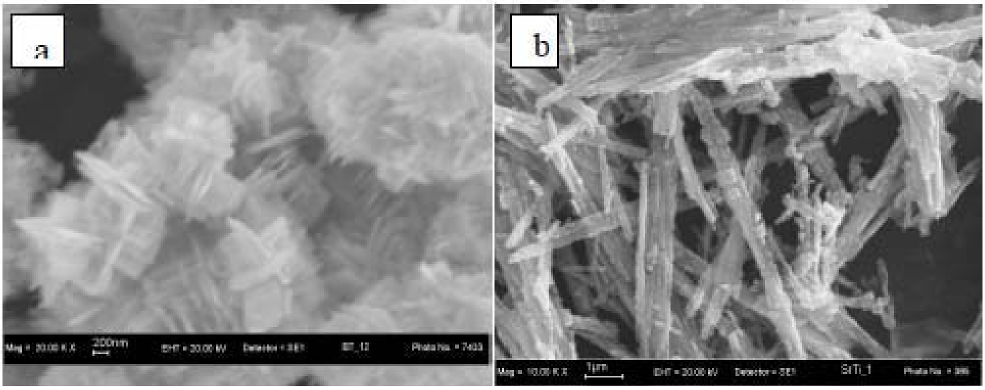

Figure 1. a) Synthesis of $Bi_4Ti_3O_{12}$ nanoplates using $Ti(0C_4H_9)_4$, b) $SrTiO_3$ nanowire using Anatase TiO_2 nanowire template.

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A3

SYNTHESIS AND CHARACTERIZATION OF ONE-DIMENSIONAL CERIUM-PHOSPHATE NANOSTRUCTURES

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In recent years much attention has been focused on the preparation of rare-earth phosphate nanomaterials. Their unique electronic configuration makes them promising candidates for the application in optoelectronics and biological fluorescent labeling.

Our research is focused on the preparation of cerium-phosphate 1D nanostructures by two different methods. Cerium-phosphate nanowires have been successfully synthesized by rapid hydrothermal and sonochemical process, respectively. For hydrothermal synthesis cerium-nitrate was dissolved in distilled water, than an appropriate amount of orthophosphoric acid was added to the solution. A white precipitate appeared immediately. The suspension was stirred for 20 minutes and transferred into a Teflon lined stainless steel autoclave and was kept at 200°C for 4 hours. The product was collected by centrifugation and washed with distilled water several times. For the sonochemical method we prepared the same suspension as mentioned before then it was irradiated with ultrasound using an ultrasonic generator for 20 minutes. The product was purified as described earlier.

The as synthesized nanowires and nanorods were examined by transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The crystalline nature of the nanowires/nanorods were determined by X-ray diffraction (XRD) and electron diffraction (ED) measurement.

A4

SYNTHESIS OF NANOCRYSTALLINE TUNGSTEN CARBIDE

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Nano-sized tungsten carbide (WC) was synthesized by thermal treatment of mixture of tungsten powder and carbon under an argon atmosphere. Different reaction condition, temperature, time of heating and C/W ratio, were applied. It was found that longer retention time, higher temperature and higher W/C ratio contribute to disappearance of elementary W and production of different W_xC mixtures. It was possible to obtain pure WC after eight-hour heat treatment at temperature of 1000°C with C/W ratio 3. WC powders were characterized by X-ray diffraction, BET method and SEM. Very fine WC particles below 50 nm can be produced. Specific surface area obtained powder is 17 m^2/g.

A5

RAPID MIXING: A NEW ROUTE TO SYNTHESIZED MAGNETITE NANOPARTICLES WITH HIGH MOMENT

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Magnetite (Fe_3O_4) nanoparticles in the size range 3 ~ 200 nm, belong to a versatile class of materials of interest for a wide range of applications from catalysis, ferro-fluidic systems, to magnetic recording and memory devices. Because these oxides are biocompatible on appropriate functionalization, superparamagnetic magnetite nanoparticles are particularly attractive for biomedical applications like cellular therapy, tissue repair, targeted drug delivery, magnetic resonance imaging (MRI), hyperthermia, to name a few. For effective therapeutic treatment, each application requires a specific particle size, with a rather narrow size distribution, and a high magnetic moment. Hitherto, Iron oxide nanoparticles have been prepared by a variety of techniques, the most popular of which is co-precipitation from solutions. The different methods of processing these particles often result in inadequate oxidation state of the final state and complexities of the consequences of nucleation and growth, and we believe the rate of mixing of the precursors as well.

In this work we demonstrate the role of rapid mixing (RM) of the reactants and its impact on the reaction speed and the magnetic properties of the obtained Fe_3O_4. The mixing is accomplished with a custom made rapid-mixer. The iron ion solution and the base flow in two jets with diameters of 0.19 mm and impinge onto each other and subsequently flow in a Teflon tube 0.5 mm dia. A pneumatic cylinder actuates two syringes arranged in tandem to acquire equal volume flow of the precursor solutions. Considering the small dimensions and the high jet velocity, the mixing time is estimated to be of order milliseconds. The mixed volume is further directed into an AC magnetic susceptometer in order to monitor the magnetic response from the growing particles in real-time. The susceptibility measurements show that the reaction is mostly completed within a minute, which is a much shorter time scale than for conventional co-precipitation. With this novel approach the obtained superparamagnetic nanoparticles are found to have a narrower size distribution with a much larger value of up to 87 Am^2kg^{-1} for the magnetization that is much closer to the expected theoretical value of 96.43 Am^2kg^{-1} for the fully oxidized magnetite. A TEM investigation suggests better crystallinity and a more uniform morphology of the nano particles produced by rapid mixing.

A6

HYDROTHERMAL SYNTHESIS OF NANOCRYSTALLINE ZIRCONIA POWDERS

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ZrO_2-based ceramics are perspective for orthopedic applications such as femoral heads for total hip replacement owing to high strength behavior and appropriate compatibility.

Although pure ZrO_2 exhibits three polymorphs: monoclinic (M-ZrO_2), tetragonal and cubic, only ZrO_2 of tetragonal modification allows to produce high-performance ceramics with satisfactory strength behavior. The high temperature tetragonal phase of ZrO_2 can be retained at lower temperatures by doping with other metal oxide such as Y_2O_3, CeO_2, etc.

The aim of this research is to investigate the properties of hydrothermal nanocrystalline powder in the ZrO_2-Y_2O_3-CeO_2-CoO-Al_2O_3 system.

Starting nanocrystalline zirconia powders of complex composition were produced by the hydrothermal decomposition of the hydroxides blend in an alkali medium (pH>9). Nanocrystalline zirconia powders based on ZrO_2 were heat treated at temperatures of 500, 700, 900 and 1200°C.

It was found, that the low temperature metastable solid solution of F-ZrO_2 was formed after hydrothermal treatment at 210°C for 3 h. The phase composition was not varied after heat treatment at 500°C for 1.5 h. The phase transformation from the ZrO_2 metastable cubic solid solution to the ZrO_2 tetragonal solid solution began at $T \leq 700$°C and finished at $T \geq 1200$°C. The formation of M-ZrO_2 was not identified.

The average size of synthesized zirconia based nanoparticles increased from 7 to 20 nm as temperature rised to 1200°C. The color of powders varied from white to bright blue when the powders were calcinated at temperature 1200°C that suggested about the formation of $CoAl_2O_4$ spinel.

Produced nanocrystalline powders will be used for designing of femoral heads.

A7

ASSEMBLING AND SPHEROIDATION OF COLLOIDAL TiO_2 NANOPARTICLES VIA AEROSOL ROUTE

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Spherical, submicronic assemblage of TiO_2 nanoparticles were processed through spray drying process from initial colloidal solution. Ultrasonically generated aerosol droplets containing 5nm dia colloidal nanoparticles were transported to tubular flow reactor with N_2 as carrier gas. Low temperature heat treatment (≤300°C) was applied for solvent removal. After droplet to particle conversion, the particles were collected by electric field precipitator. The selection of temperature profile was a key step in process optimization.

The obtained TiO_2 particles were modified with different type of ligands (dopamine, 1,2-dihydroxybenzene, 2,3-dihydroxynaphthalene and 1,2,10-anthratriol). Binding of the modifier molecules results in formation of charge transpher complexes and leads to the visible light sensitisation of modified TiO_2 particles.

X-ray powder diffraction (XRPD), field emission and scanning electron microscopy (FESEM/SEM), energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM) were employed for phase indentification, structural and morphological characterization of obtained particles. Optical properties of modified TiO_2 particles were studied in details using reflection spectroscopy. Adsorption of ligands was investigated by using FTIR spectroscopy. Laser particle size analysis (LPS) is used for particle size distribution (PSD).

A8

EFFECT OF CALCINATION TEMPERATURE ON MICROSTRUCTURE AND MECHANICAL BEHAVIOUR OF ALUMINOSILICATE INORGANIC POLYMERS

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Aluminosilicate inorganic polymers (AIP), often called "geopolymers", are ceramic-like materials characterised by amorphous three-dimensional aluminosilicate network. AIPs exhibit attractive mechanical properties, especially high compressive strength, which makes them a prospective construction material. AIPs are prepared by the "alkali activation" of materials based on dehydroxylated aluminosilicates with disordered structure like calcinated kaoline. In this study, the effect of calcination temperature of kaoline on AIP's properties was investigated. Two kaoline clays from different source areas, differing in particle size, were calcinated at 550, 650 or 750°C, respectively, and then geopolymerized. The compressive strength of geopolymers increased with increasing calcination temperature, reaching 30 MPa for the highest calcination temperature. At the same time, the smaller partical size led to higher values of the strength. The microstructure of raw materials and products was invectigated using X-ray diffraction analysis (XRD), magic angle spinning - nuclear magnetic resonance (^{29}Si and ^{27}Al MAS-NMR) and scanning electron microscopy (SEM). In addition, the Chappelle test of calcinated kaoline was provided to explore effects of calcium hydroxide, which is present within kaoline, on hardening process. The results revealed that "state of disorder" of the structure varied with calcination temperature. Kaoline treated at 750°C contained higher amount of disordered phase then the kaoline treated at lower temperature. It has been shown, that the increase in disordered phase proportion of starting materials promotes better mechanical properties of AIPs.

The contribution summarizes the interrelations between calcination temperature, structure and resulting mechanical behaviour of aluminosilicate inorganic polymers.

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A9

PREPARATION AND CHARACTERIZATION OF SILVER DOPED HYDROXYAPATITE SCAFFOLDS

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Synthetic hydroxyapatite (HAp) is used in bone tissue engineering due to its bioactivity, osteoconductivity and biocompatibility. Silver ions Ag^+ can be used in medicine as antimicrobial agents. At low concentrations silver ions are not toxic to human body but high concentrations can cause cytotoxicity. Silver ions in the Hap scaffold should act as antimicrobial agents [1,2]. The aim of this work was to synthesize silver doped hydroxyapatite and to determine its antimicrobial and drug delivery properties. To obtain scaffolds with antimicrobial properties silver ions should be attached on the surface of scaffolds. Modifications with lidocaine hydrochloride were made to evaluate the prepared scaffold suitability as drug delivery system. Lidocaine hydrochloride is commonly used local anesthetic [3].

Modified wet chemical method was used to synthesize silver doped hydroxyapatite scaffolds with various silver amounts. The synthesis contains precipitation from an aqueous medium by slow addition of silver nitrate and orthophosphoric acid solutions to a calcium hydroxide suspension. The dried precipitate was milled to obtain a fine powder which was mixed with an organic additive (polyol) to obtain a highly viscous/ plastic mixture, ammonium carbonate was added as pore foaming agent, and green bodies were sintered at 1000°C and 1150°C to obtain porous ceramic scaffolds. HAp/Ag complexes were evaluated using X-ray Diffraction (XRD) and X-ray Fluorescence spectrometry (XRF) methods. Scanning electron microscopy (SEM) was used to evaluate the surface morphology and inner structure of prepared scaffolds. Vacuum infiltration was used for the drug incorporation in porous scaffolds. Release profile of lidocaine hydrochloride was determined using high performance liquid chromatography.

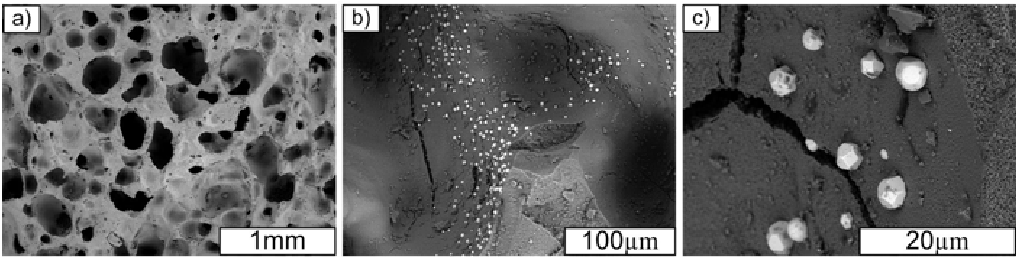

Figure 1. SEM images of: a)porous HAp/Ag scaffold; b) and c) HAp/Ag scaffold surface after sintering for 2 h in 1000°C.

Results showed that after sintering of porous silver doped hydroxyapatite scaffolds the content of silver is from 0.1% up to 1.2 %, depending of the sintering temperature used and silver particles are dispersed on the surface of porous HAp/Ag scaffolds (see Fig. 1).

It was discovered that during 5 h of dissolution, 80% of lidocaine from HAp scaffolds was transferred in simulated body fluid. Silver doped HAp scaffolds have the same drug delivery properties.

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A10

THEORETICAL APPROACH OF OXIDE HARDNESS

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Hardness is an important mechanical property of materials. It is defined as the resistance of a material to localized deformation. The ionicity of chemical bond plays a significant role in mechanical properties such as hardness, bulk modulus.

Recent studies have shown that hardness, a complex property, can be calculated using very simple approaches or even analytical formulae. In these models, the theoretical hardness is described as a function of the bond density and bond strength. The bond strength may be characterized by energy gap, reference potential, electron–holding energy or Gibbs free energy, and different expressions of bond strength may lead to different hardness models. In particular, the hardness model based on the chemical bond theory of complex crystals is used.

The chemical bond parameters and hardness values of oxide crystals had been calculated based on the P-V-L dielectric theory in connection with our own model of ionicity. The correlation between bond length, unit cell parameters, ionicity, bond order and hardness of the various oxides was established and discussed.

Comparison with results provided by other theories is performed.

A11

SOLID STATE NMR SPECTROSCOPY OF ALUMINOSILICATE INORGANIC POLYMER

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Aluminosilicate inorganic polymers (alternatively called as geopolymers), are materials prepared from calcinated kaolin and alkali solution (NaOH, KOH, Na_2SiO_3). The reaction usually proceeds at atmospheric pressure and ambient temperature. During the reaction the decomposition of calcinated alumino-silcate is initiated and subsequent rearrangement of aluminum and siloxane tetrahedra leads to the formation of a three-dimensional amorphous network, like ceramics. Sometime, can be transform amorphous matrix to crystalline zeolitic structure, during long time period. These transformations are accompanied by loss of water and mechanical properties.

In our case, we used advanced solid state NMR techniques for describing of differences between amorphous and crystalline phases of alumino-silicate inorganic matrix. To gain structural information provided by solid-sate NMR we used not only simple one-dimensional experiments on various nuclei like ^{1}H, ^{23}Na, ^{27}Al, ^{29}Si but we also used two-dimensional multiple-quantum experiments. We tested large scale of NMR techniques involving also cross-polarization transfer, REDOR experiments to as well as MQ/MAS NMR experiments. The attention is especially focused on 2D MQ/MAS NMR experiments because they offer better spectral resolution compared to "traditional" 1D experiments (single pulse and/or cross polarization experiments etc.). The relatively new approach of the processing of MQ/MAS NMR spectra consists of biaxial shearing of these 2D spectra. This processing can provide further cosmetic modifications of the spectra which may aid interpretation by helping to identify and characterize independent chemical sites.

A12

MECHANICAL PROPERTY CHARACTERIZATION OF TRANSPARENT $MgAl_2O_4$

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The mechanical properties and failure mechanisms of transparent $MgAl_2O_4$ are reported. In particular the elastic modulus of the material has been investigated using depth sensitive indentation, impulse excitation and ring-on-ring testing. The limits of the methods are illustrated. The importance of mathematical analysis is emphasized.

The fracture toughness has been determined from the length of indentation cracks using different analysis methodologies. Furthermore, the macroscopic fracture stress was derived from ring-on-ring biaxial bending tests whereas a local strength was evaluated by means of Brinnel indentation testing. The macroscopic fracture strengths were compared for different $MgAl_2O_4$ batches using Weibull statistics to permit an assessment of failure probabilities. Fracture mechanism and failure origins have been investigated using light, and scanning electron microscopy.

For lifetime prediction of transparent ceramic the subcritical crack growth (SCG) behavior was assessed by the often used constant stress-rate testing.

Novel experimental methods were implemented for a detailed analysis of fracture behavior and stress field. An in-house developed indentation testing device permitted in-situ through thickness fracture observation. Particular attention was directed to the effect of defects and associated local stress fields as assessed using polarized light in photoelastic measurements.

A13

CHEMICAL AND MECHANICAL STABILITY OF NANOENGINEERED TITANIA THIN FILMS

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TiO_2 and related semiconducting materials have been widely used as photocatalysts in order to decompose environmental pollutants. For number practical applications, the photocatalyst, usually immobilized in the form of a thin film on an inert substrate, has to show high stability over long time periods, including resistance to corrosion as well as to mechanical stresses.

This work describes the synthesis of efficient titania pastes and nanostructured films and is focused on the investigation of their chemical and mechanical stability under specific conditions. For the paste preparation, commercially available TiO_2 P25 AEROXIDE (Evonik) was used and both viscosity and rheology were thoroughly controlled [1]. In addition, the effect of essential parameters, such as paste composition, addition of binder molecules and dispersion temperature were examined. The titania thin films were deposited by doctor-blade technique onto microscope glass surfaces, air dried in air and calcined at 450°C and The resulting materials were subsequently characterized using X-Ray Diffraction (XRD), Atomic Force Microscopy (AFM), SEM, Raman spectroscopy and IR.

The films chemical stability was investigated as a function of calcination temperature, PH and immersion time in aqueous solutions. The relationship between film morphology and thickness has been elucidated. A critical film thickness below which films were crack-free was determined. Existing models for the mechanical stability of the films were also examined [2].

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A14

THE EFFECT OF DEPOSITION CONDITIONS OF DIFFERENT MAGNETRON SPUTTERING MODE ON NANOHARDNESS OF WC/C COATINGS

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Keywords: WC/C coatings, bias voltage, DC/RF magnetron sputtering, micro/nano-hardness

The aim of the present contribution is to study the influence of pressure and bias voltage during DC and RF magnetron sputtering on nanohardness of nanocomposite WC/C coatings. The data were used for the optimization of the deposition conditions of WC/C coatings.

WC_x/C ($x \approx 0.8$) coatings were deposited on a polished steel substrates (C45) using an unbalanced direct current (DC) and radiofrequency (RF) magnetron sputtering from stoichiometric WC target. Sputtering pressure was in the range from 0.1 Pa to 1 Pa for DC sputtering and from 0.8 to 2 Pa for RF sputtering. Negative bias in the range -170 V ÷ -500 V was applied to the substrate. Indentation hardness (H_{IT}) and indentation modulus (E_{IT}) were investigated by nanoindenter with Berkovich diamond indenter using sinusoidal loading with maximum load of 10 mN. Coating topography was observed by Atomic Force Microscope and contact profilometry.

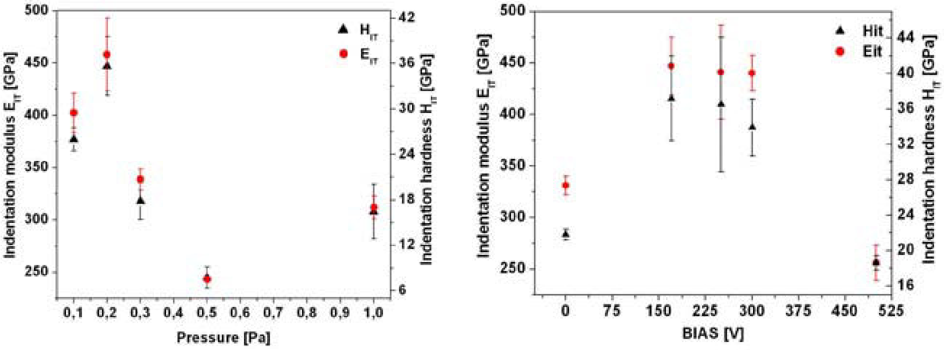

Figure 1. Comparison of the influence of pressure and bias for DC sputtered WC/C coatings

Nanohardness values of DC sputtered coatings were in the range from 7 to 37 GPa, their $E_{IT} \approx 250–470$ GPa. RF coatings nanohardness values were $H_{IT} \approx 8–22$ GPa and $E_{IT} \approx 135–270$ GPa. The results indicate that mechanical properties of DC sputtered WC/C coatings are higher than those from RF sputtered coatings at this stage of optimization. Additional optimization of deposition conditions during RF magnetron sputtering is necessary to determine the limits of the technique.

A15

STRUCTURAL ROLE OF Cu IN SOIL ACTIVE GLASSES

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For the proper growth of plants is required optimal amount of copper and its corresponding proportion in relation to other elements. Both deficiency and excess copper inhibits the life processes of various plants. Copper also has fungicidal properties, so based on copper, copper fungicides are produced to protect the vegetables and fruit trees.

Active soil glasses of the SiO_2-P_2O_5-K_2O-MgO-CaO-CuO system, which can act as a slowly dissolving fertilizer supplying plants macroelements (P, K, Mg, Ca) and microelements (Cu), have been subject of the present investigations. The aim of this study is to determine structural role of copper in these glasses.

Copper can be introduced into the glass structure at three different oxidation states Cu^{2+}, Cu^{+} and Cu^{o}. During melting, when the conditions are reducing, this element can dominate in the form of Cu^{2+} ions, giving the glass a blue-green color. In soils, copper occurs most often in the form of Cu^{2+} ions. Copper in the structure of studied glasses acts as a modifier, and so will break some part of the chemical bonds present in the type of oxygen bridges Si-O-Si, P-O-P, Si-O-P.

The influence of copper content on the structure of glasses was examined by Raman spectroscopy, and ^{29}Si, and ^{31}P MAS NMR. The chemical activity of the glasses in the 2 wt % citric acid solution, which simulates physico-chemical state similar to the natural environment between plants and surrounding soil, was measured by the ICP-AES method.

It was found that biological activity of silicate-phosphate glasses modified by copper addition is connected with the changes in the structure of domains which are formed in the structure of glasses. Along with the copper content increase in the glasses structure, the amount of domains showing similar structure to phosphates with P-O-Cu bonds increases. Simultaneously, that of formation of chemically stable P-O-Cu bonds decreases glass solubility in conditions simulating the soil environment.

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A16

INTERACTION CERIUM OXIDE WITH ERBIA AT 1100°C

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Systems with ceria and erbia are perspective for the development of alternative ceramics for the thermal barrier coatings (TBCs) and intermediate temperature solid oxide fuel cells (SOFCs). Doped ceria is a promising materials for applications as a solid electrolyte for its use in oxygen concentration cells and in solid oxide fuel cells. It is also a candidate material for application in controlling air-to-fuel ratio in automobile exhaust. The high ionic conductivity coupled with the low activation energy for ionic conduction makes the doped CeO_2, a superior material in comparison yttria-stabilized zirconia (YSZ), for use at temperatures below 800°C, which would allow greater flexibility in design of the electrode and interconnectors. Moreover, this system can be widely used in atomic power engineering for creation of the safe disposal techniques for the nuclear waste and development of advanced and new generation of reactors. Phase diagram based on CeO_2 and Er_2O_3 is interesting from both fundamental and practical viewpoints.

The objective of this work is to investigate the phase relations in the binary system CeO_2-Er_2O_3 at 1100°C in air in the whole concentration range. Powders of $CeO(NO_3)_2$ and Er_2O_3 (99.99 %) were used as raw materials. The samples were prepared in steps of 1 and 5 mol % from nitrate solutions with their subsequent evaporation and decomposition at 1000°C for 2 h. Thermal treatment was carried out in the furnaces with heating elements based on H23U5T (1100°C for 16800 h) in air. The phase compositions were investigated by X-ray diffraction, microstructural phase and electron microprobe X-ray analyses.

The study of solid state reaction of CeO_2 (fluorite-type, F) and Er_2O_3 (cubic modification of rare-earth oxides, type C) at 1500°C showed that two types of solid solutions based on cubic modifications of F-CeO_2 and C-Er_2O_3 in the CeO_2-Er_2O_3 system. These solid solution regimes were separated from end to end with the two-phase field: (F+C).

The boundaries of the homogeneity fields for the solid solutions based on F-CeO_2 and C-Er_2O_3, as well as lattice parameters of the unit cells for solid solutions F-CeO_2 and C-Er_2O_3 were determined. The solubility of Er_2O_3 in F- modification of CeO_2 is about 20 mol% at 1100°C (16800 h).The lattice parameter of the unit cell decreased from a = 0.5409 nm in pure CeO_2 to a = 0.5376 nm for the solid solution of boundary composition. The solubility of CeO_2 in cubic C- erbium oxide attain ~15 mol% at 1100°C. The lattice parameters of the unit cell C phase varies from a = 1.0531 nm in pure Er_2O_3 to a = 1.0537 nm in the sample containing 85 mol% Er_2O_3.

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A17

THE EFFECT OF Mn AND La SUBSTITUTIONS ON FUNCTIONAL PROPERTIES OF BiFeO$_3$ MULTIFERROIC CERAMICS

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A large number of publications was dedicated in the last years to BiFeO$_3$ multiferroic (ferroelectric with T_C = 830°C and incommensurate antiferromagnetic with Néel temperature T_N = 370°C) [1,2]. Doping with small amounts of La demonstrated to improve the dielectric and ferroelectric properties [3]. On the other hand, substituting Fe with Mn in BiFeO$_3$-based compounds is supposed cause better properties in terms of leakage current density and also to induce changes in the magnetic order of the system. In the present paper, the effect of Mn substitution on the magnetic properties of the Bi$_{0.9}$La$_{0.1}$Fe$_{1-x}$Mn$_x$O$_3$ and on the dielelectric and magnetic properties of the BiFe$_{1-x}$Mn$_x$O$_3$ ceramics prepared by solid state reaction was investigated.

In the case of BiFe$_{1-x}$Mn$_x$O$_3$ ($0 \le x \le 0.40$) ceramics, all the compositions exhibit a rhombohedral perovskite *R*3c structure. The lattice parameters, the unit cell volume and the crystallite average size decrease with the increasing of the manganese content. SEM investigation performed on the surface of the undoped BiFeO$_3$ ceramic sample pointed out a microstructure consisting of large, well-interlinked and non-uniform (as shape and size) grains and a certain intergranular porosity, which affects the sample density. Very high values of permittivity in the range of 1000–30000 are observed at low frequency, followed by a monotonous decrease with frequency at room temperature, for all the compositions losses above unity are characteristic for these ceramics, particularly at low frequency, for which tanδ is about a few tenths. The low-frequency is dominated by dc-conductivity contributions, but improving of the dielectric characteristics was achieved at higher frequencies (10^6–10^9) Hz. The magnetization at room temperature slightly increases with the Mn substitution, while antiferromagnetic behavior determined by the BiFeO$_3$ remains prevalent.

In the case of Bi$_{0.9}$La$_{0.1}$Fe$_{1-x}$Mn$_x$O$_3$ ($0 \le x \le 0.50$) samples, the ceramics with lower Mn content ($x < 0.40$) tend to a single phase composition even after calcination at 650°C/2h. Unlike the porous Bi$_{0.9}$La$_{0.1}$FeO$_3$ ceramic sample, homogeneous and dense microstructures were obtained for the manganese-modified compositions. While the 10% La addition does not change the antiferromagnetism typical to the BiFeO$_3$ system, the presence of Mn has a non-monotonous influence on the magnetic properties. A weak ferromagnetism seems to be induced around $x = 0.20$, while for $x = 0.40$ and 0.50, the antiferromagnetic order is recovered. The composition $x = 0.20$ is a candidate as multiferroic magnetoelectric system and it will be further investigated in detail.

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A18

SYNTHESIS OF FERRITES FROM THE SYSTEM MeO - Fe_2O_3 AND STUDY THEIR SOLUBILITY IN SULFURIC ACID SULUTIONS AFTER PRELIMENARY TREATMENT

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Ferrites from the MeO-Fe_2O_3 system, where Me - Zn, Cd, were synthesized using a ceramic technology. Their composition, structure and properties were studied by chemical and X-ray diffraction analysis and Mössbauer spectroscopy. They are mono phase samples and have properties very close to the ones obtaining during zinc sulfide concentrates roasting in fluidized bed furnace.

The solubility of the ferrites in diluted sulfuric acid solutions (7, 10.5 и 14 vol.%) was studied using microwave heating. The obtained results were compared with the results from the experiments using conventional heating.

The experimental results show that the Zn and Cd degrees of solubility using microwave heating are higher than the ones obtained by other conditions of leaching. On the basis of the data obtained a conclusion can be made that $CdFe_2O_4$ is lower stable than $ZnFe_2O_4$ using the both ways of heating.

The solubility of ferrite of the $Zn_{0,5}Cd_{0,5}Fe_2O_4$ type as well as of samples of zinc cake where the zinc is mainly as $ZnFe_2O_4$ is investigated using a method of mechanical activation and following acid and high-temperature leaching. For this purpose the energy of mechanical milling techniques was used. It was established that crystal structure changes have occurred in the studied samples. The results obtained show that the degree of Zn and Cd solubility from ferrites and zinc cake after mechanical treatment increase significantly comparing with the obtained data for the initial non-treated samples.

A19

MAGNETIC PROPERTIES OF Fe^{2+}/Fe^{3+} DOPED CeO_2 NANOCRYSTALS

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We report magnetization and Raman measurements of iron doped ceria, $Ce_{0.88}Fe^{2+/3+}_{0.12}O_{2-\delta}$ nanocrystals. Compared to undoped CeO_2 samples, the Fe doped samples show strong magnetic properties. The Fe^{3+} doped sample has a higher saturation magnetization then the Fe^{2+} doped one, showing a strong influence of Fe valence state. The Raman spectra of Fe^{3+} doped sample shows significantly broader F_{2g} mode compared to Fe^{2+} doped and undoped samples. These effects can be attributed to strong delocalization of electrons onto Ce-O(V_O)-Ce orbitals.

A20

MAGNETIC AND FERROELECTRIC PHASE COEXISTENCE AND MAGNETOELECTRIC PROPERTIES IN MULTIFERROICS PFW-PT CERAMIC SOLID SOLUTION

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A material is generally considered functional if it possesses a physical property that is usable in applications. In recent years, much attention has been paid to multiferroics-materials which possessing two or more switchable states such as polarization, magnetization or strain, due to their projected novel applications and interesting physics behind their behavior. The coexistence of electric and magnetic orders in multiferroic opens new possibilities of the cross-controlling of electric (magnetic) parameters by magnetic (electric) field. These new possibilities appear due the coupling of magnetic and electric subsystems in matter which named magnetoelectric (ME) coupling. For other hand, in multiferroics, strong spontaneous ME effects manifest themselves near the phase-transition temperatures, were the indirect interaction between electric and magnetic subsystems occurs through an elastic subsystem. The presence of an elastic subsystem in the crystal leads to the emergence of electrostriction which accompanies the FE ordering and changes the crystal size. Thus, in turn, changes the magnetic state due to magnetostriction, and vice versa. In most approaches to the modeling or determining the magnetoelectric coefficient in single-phase multiferroic materials have not been considered the elastic contribution, so that, this contribution has never been quantified. PFW is one of the classical relaxors with lower sintering temperature (about 830°C). However, the lower Curie temperature T_C at 180 K is a disadvantage. The lattice structure, the dielectric and magnetic properties of the pure PFW can be easily changed by adding $PbTiO_3$, a typical ferroelectric with a sharp maximum of dielectric constant at 763 K. It is possible to move the Curie and Neél temperature and to modify the relaxation behavior by choosing an appropriate amount of PT in the system. From the point of view of synthesis, a part of this work was to look for a "tuning" the coexistence of ferroelectric and magnetic orderings in the PFW-PT as close to room temperature (~230 K for 12% PT) in order to obtain materials with magnetoelectric response, with an optimization properties of electric ($\sim 10^8$ Ω·m), ferroelectric (18 $\mu C/cm^2$) and magnetic subsystems (1emu/g). Since fundamental aspect, we systematically investigate the properties of PFW-PT multiferroic single phase solid solution: characterization of magnetoelectric coupling and influence of strain and external electric and magnetic fields (up to 3 KV and 2 T) on this coefficient.

A21

SYNTHESIS AND CHARACTERIZATION OF SOL-GEL PREPARED TiO_2 MESOPOROUS FILMS FOR APPLICATION IN DYE-SENSITIZED SOLAR CELLS

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Modified sol-gel method combined with spin-coating and doctor blade techniques were used for preparation of TiO_2 thin films on FTO glass substrates. Chemistry of the process involves reactions of complexation, hydrolysis and polycondensation, starting from titanium isopropoxide, and TEA or EDTA as complexing agents. Polyethylene glycol with high molecular weight (6000 and 8000) was added for optimal film porosity. Morphology of films was characterized by SEM, crystal structure was investigated by XRD, and film thickness was measured by profilometer. Precursor gels were subjected to viscosity measurements. I-V characteristic of prepared films was used for solar efficiency calculations. Although the XRD analysis confirmed the presence of dominant anatase phase, and the optimal porosity of the films was achieved, the issues of film cracking and poor adhesion should be solved in order to get higher solar conversion efficiencies.

A22

SYNTHESIS OF ZEOLITE-BASED CATALYSTS BY CHEMICAL VAPOR INFILTRATION FOR NITROGEN OXIDE CONVERSION

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Zeolite-based catalysts such as Fe-ZSM-5 or Cu-ZSM-5 are well known candidates for catalytic nitrogen oxide conversion [1,2]. Infiltration of the microporous zeolites and ion exchange is commonly performed by wet impregnation routes. However, homogeneous infiltration and precursor distribution are often still a challenge due to precursor solvent viscosity or electrostatic shielding and may be inhibited by clogging of pores. Infiltration of precursor vapors into the zeolite matrix (chemical vapor infiltration) can solve viscosity issues and may contribute to homogeneous infiltration.

New nitrogen oxide-storage catalysts based on commercial zeolite substrates were synthesized by chemical vapor infiltration. The primary zeolite cations were exchanged for iron, copper and manganese cations.

The resulting materials were investigated by scanning electron microscopy and energy-dispersive X-ray spectroscopy. They exhibited a comparable homogeneity of the precursor distribution infiltrated through the gas phase compared to the wet impregnation technique. Crystallite sizes below 2 nm and deposited species were estimated from X-ray diffraction. Nitrogen adsorption (BET) revealed a moderately reduced inner surface of transition metal-exchanged zeolite catalysts in comparison to the original matrix.

Gas phase infiltrated ZSM-5-zeolites are compared to the same matrix material infiltrated by wet chemistry at similar precursor-substrate ratios regarding their activity in NO_2 conversion. Catalytic activity is studied in a recycle-reactor by time-resolved mass spectrometry in the temperature range of 303 to 573 K. Oxide species and precursor-substrate-mass ratios are studied in relation to NO_2-conversion.

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A23

FABRICATION OF MULTILAYERED CELLULAR CERAMICS VIA RADIATION CURABLE DISPERSIONS

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Cellular ceramic articles find applications in high temperature thermal insulation, light weight structures, separation and catalysis, smart materials and devices and they become more important for these engineering applications due to their unique combination of properties.

A general and versatile method for the production of cellular ceramic materials from radiation curable solvent-free colloidal ceramic dispersions containing pore formers has been developed. By this technique cellular ceramic articles with a precisely controlled porosity, cell size and shape are obtained for compositions containing solid pore formers. Upon curing these compositions solidify fully. Cell sizes ranging from 5 up to 150 µm, porosities up to 80% and cell walls in the submicrometer range have been demonstrated.

The different aspects of this method are demonstrated here for alumina as a model material. However, the general method is demonstrated also for other functional materials such as titania, hematite and hydroxyapatite. Depending on the desired form of the resulting articles, different curing strategies can be applied. Larger monolithic bodies are advantageously produced by thermal curing whereas thin films by UV curing.

The method and compositions described above are ideal for homogeneous and heterogeneous layer-by-layer processing. High quality articles having either a gradient in pore size or porosity, or consisting of alternating layer structures are demonstrated. Examples for such multilayered structures and their thermal and mechanical properties will be presented.

A24

POROUS ALUMINA CERAMICS MODIFIED WITH SiC NANOPOWDER

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The aim of this study was to clarify how addition of plasma synthesized SiC nanopowder (up to 5 wt.%) affects some properties (e.g., shrinkage, bending strength and thermal shock resistance) of porous alumina ceramics produced by slip casting method. Pores in these materials are formed in a result of chemical reaction of aluminium with water in alkaline medium ($pH \geq 9$). Samples were fired at three different temperatures – 1650, 1700 and 1750°C. SiC particles during sintering oxidize into SiO_2 and form mullite in the reaction with alumina. Addition of SiC nanopowder slightly reduces shrinkage of the material in comparison with unmodified material. It was found that the addition of SiC nanopowder considerably improves bending strength and thermal shock resistance of porous alumina ceramics. Resistance to deformation under load at elevated temperatures of the samples modified with SiC nanopowder is higher than that of unmodified samples. The porosity of investigated materials is within the range of 57% and 65%, and the bulk density is within the range of 1.0 and 1.3 g/cm^3.

A25

POROUS CERAMICS BY ICE TEMPLATING: FREEZING CHARACTERISTICS, RHEOLOGICAL AND MICROSTRUCTURE PROPERTIES

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The present work aims at fabricating porous ceramics exhibiting an unidirectional channels structure. Different processing routes for slurry freeze casting are studied in order to control the microstructure orientation.

Various cellular ceramic samples are fabricated by ice templating technique. It consists of freezing an aqueous liquid suspension, followed by the sublimation of the ice

under reduced pressure. Subsequent sintering treatment are used to consolidate and densify the channel walls. A suitable anisotropic structure is favored by a perfectly controlled directional solidification step.

Porous zirconia and alumina ceramics with different particles size were investigated. Namely, the influence of processing parameters on the channels size, shape and organization of the obtained samples are assessed as well as the effect of binder addition (PEG) and seeding. The microstructure is observed by micro tomography and scanning electron microscope.

A26

DEVELOPMENT OF PERMIABLE HIGH POROUS CELLULAR GLASSCERAMICS FOR SORPTION-REDUCTION Fe-CONTAINING MATERIALS FOR WATER REMEDIATION

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The main parameters that determine the suitability of the material as a carrier of the active reactive coating for remediation of large volumes of polluted water is a compromise between its functional and value characteristics. It must have high specific surface reactivity, adsorption (or ion exchange) capacity, coefficient of filtration, wear resistance, crumble resistance, compressive strength, durability and low cost. That is why the use of high porous crystalline glass-ceramic materials as carriers of sorption-reduction coating in permeable reactive barriers is a promising task.

At the present time many materials are used as carriers of iron containing coatings from the most common and cheapest coarse-grained sand to high porous cellular materials obtained by duplicating of polymer matrix method. They have or low permeability and, correspondingly, low durability in real-time use, or complex and expensive production technology.

The foam glass combines all advantages of existing materials. It can be fabricated by use simple and inexpensive equipment. Moreover, cheap and available raw materials such as broken glass and various active admixtures can be used for it production. Porosity and permeability of the material prepared from foam glass can be controlled in wide range using different types of foaming agents and their combinations in various amounts.

In the present study broken glass of different composition, calcium and magnesium carbonates, magnomass, as well as such minerals as dolomite, chalk, limestone, magnesite were used.

The matter of the study was the following. The thermograms of decomposition of carbonate admixtures were recorded and thus determined the temperature range of it decomposition. For known chemical composition of broken glass the temperature dependence of viscosity were calculated by Okhotin’s method. For known range of viscosity the composition of foaming agent was selected. It temperature range of active decomposition corresponded to the calculated value of the viscosity of glass at the present temperature.

In addition, total porosity, open porosity and compressive strength were tested. It was established the possibility preparation of high porous glassceramic material based on broken glass and magnomass with open porosity up to 67%, compressive strength up to 10 MPa.

A27

POROUS MULLITE CERAMICS FORMATION AND MODIFICATION WITH SOME ADDITIVES

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Keywords: mullite, kaolin, alumina, silica, porosity, refractory ceramics

Development of modern technologies requires the production of materials with perfect and sometimes universal properties. Favorable refractoriness at temperature above 1580°C can be achieved when raw materials such as kaolin, alumina and silica were mixed and sintered about at 1700°C temperature, when mullite phase was formed as result of sintering. Mullite ceramics display excellent resistance to chemical corrosion [1,2]. Therefore mullite ceramics have been widely used as constructional materials and also its high porosity ceramics serve as a heat insulator in the thermal protection systems.

The aim of this work was to investigate the influence of different ratio of kaolin, silica and alumina with different grain size as raw materials on mullite formation and its quantity in our ceramic materials. The method of preparation and sintering temperature as well as additives influence on such properties of mullite ceramics as the bending strength, thermal shock resistance, coefficient of thermal expansion, load resistance at high temperatures, the volume and shape stability at the high temperatures [1,2].

The porous mullite materials were prepared by slip casting of suspension of raw materials where the aluminium paste (0.18 wt.%) was used as a pore former. Pore formation occurred as a result of hydrogen formation in a chemical reaction between aluminium paste and raw materials, when water suspension is with pH > 7, because it is prerequisite condition for gas elimination reaction ability of metallic aluminium. This method was used, because it is more ecological than the method of additive of combustible matter [1,2].

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A28

APPLICATION OF VARIOUS PORE-FORMERS FOR CERAMIC MATERIALS

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Porous ceramics are a subject of intensive research for their wide applications for instance as catalyst carrier, ceramic filter, sensor, porous electrode, biomaterials, thermal barrier, and so on [1,2]. There is a long list of various methods use to fabricate ceramics with porous structure include, among others, pore-foaming technique, infiltration of ceramic sol into template structures, gel-casting, starch consolidation, microwave processing, electrophoretic deposition, freeze casting [3–6].

Porous ceramic is defined as an inorganic material obtained by heat treatment, whose characteristic feature is porosity. Porosity is a measure of void spaces in material and it is a fraction of the volume of voids over the total volume of the specimen. Such porous materials have less mechanical strength than solid ones. In most cases void spaces are obtained by removal of the filling agent so it is important that the base material has created stable structure, which preserve appropriate relationship between porosity and mechanical properties.

Therefore a several ceramic pellets were fabricated using various pore-formers. Density and porosity of pellets was measured by Archimedes method and the properties of microstructure were determined by scanning electron microscope. As an example ceramic material strontium titanate was used.

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A29

Bi_2WO_6 PHOTOCATALYST NANOPOWDER SYNTHESIS AND PHOTODEGRADATION OF MB

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Photocatalysis and ceramic based photocatalysts are being studied more extensively and semiconductor photocatalysts are the most popular for different applications for example photo-degradation of organic compounds and water splitting technologies under ultraviolet and visible light irradiation. Therefore it is important to study these kinds of materials. Bismuth tungstate Bi_2WO_6 shows that it is photo-active in both ways - under visible and UV light which is very convenient and expands its usage.

There are several synthetic methods which can be used for nanopowder synthesis - solid state synthesis at high temperatures, molten salts or hydrothermal synthesis. More attractive method is sol-gel combustion method which is attractive as low temperature method. Bismuth tungstate is prepared with modified sol-gel combustion method from the appropriate bismuth nitrate salt solution in water and tungstic acid in metal molar ratio Bi:W = 2:1, mixed with organic fuel (ethanol, glycine, ethylene glycol) and nitric acid. As-prepared crystalline powder, besides Bi_2WO_6 depending on reaction conditions and used organic fuel contained some extra phases - WO_3, $Bi_2W_2O_9$, and $Bi_{14}WO_{24}$. Additional calcination at 650–800°C high temperature range leaded to formation of pure crystalline Bi_2WO_6. The calcinated bismuth tungstate nanopowders have high crystallinity and specific surface area (BET) in range of 12–22 m^2/g depending on used fuel and calcinations temperature.

The photocatalytic activity experiments of Bi_2WO_6 nanoparticles were done by degradation of methylene blue (MB) solution under ultraviolet illumination. The photocatalytic activity and the degradation of MB strongly depended on specific surface area. The Bi_2WO_6 nanoparticles with specific surface area 23.4 m^2/g and 13.3 m^2/g ensured 93 % and 74 % degradation of MB during 3 hours respectively.

A30

SPHENE BASED CERAMIC

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Sphene ($CaTiSiO_5$) has been prepared from a powder mixture of $CaCO_3$, TiO_2 and SiO_2 using vibro-miling for homogenization and activation precursors. The mechanochemical process initially yielded amorphous powders, which on further calcination, crystallized to yield sphene ceramic. The evolution of the phase composition with thermal treatment was investigated by X-ray powder diffraction (XRPD) and thermal analyses (DTA-TGA). Powder morphology and particle size distribution were analyzed by scanning electron microscopy (SEM) and laser diffraction, respectively. The Rietveld refinement was employed to get the structural information of the synthesized powder. Densification and microstructure evolution was determined by means of density and scanning electron microscopy (SEM) measurement.

A31

CHARACTERIZATION OF DOPING INFLUENCE ON BARIUM CERATE PROPERTIES

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Perovskite ceramics characterized by the ABO_3 configuration can be solid protonic conductors. These materials may be widely used in a number of technological applications [1,2]. Pure barium cerate, $BaCeO_3$, however, exhibits rather low electronic conductivity. On the other hand, it was observed, that substitution of B-site by trivalent ions in these perovskite oxide results in formation of oxygen vacancies that facilitate proton conductivity (hydrogen ion transport) upon exposure to water vapour at work temperature, usually 800°C and below [3,4]. Thanks to high proton conductivity many types of rare-earth-doped barium cerate are considered as materials useful as electrolytes for solid oxide fuel cells (SOFC). On account of this application barium cerate should meet requirements such as: high ionic conductivity, low contribution of inter-grain

resistivity, suitable chemical and mechanical stability and high density without open porosity [5,6]. As another point of view, some of difficulty emerges due to the fact, that this material proves to be chemically unstable at high temperature in the presence of CO_2 and steam [7,8]. In CO_2 or humid atmospheres, barium cerate easily reacts and decomposes into barium carbonate (or barium hydroxide) and cerium oxide [9,10]. Notwithstanding, barium cerate may be modified by substitution of cerium ions with a number of other elements, which may have different influence on the properties of this material. Some of them affect proton conductivity, while other change chemical stability, density or required sintering temperature.

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A32

INVESTIGATION OF La-DOPED $BaTiO_3$ CERAMICS PREPARED BY ALTERNATIVE METHODS

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Electrical properties of doped-$BaTiO_3$ ceramics are strongly influenced by the type and concentration of the dopants and by the processing conditions. Undoped $BaTiO_3$ is a highly insulator material, but doped with donors it turns in semiconductor at room temperature and shows an anomalous electrical resistivity jump of several orders of magnitude at the Curie temperature, known as Positive Temperature Coefficient of Resistivity (PTCR) [1]. This effect is due to the ferroelectric behaviour correlated with the semiconducting-insulating properties of the grain cores and grain boundaries, respectively.

In the present work, the electrical properties of the sol-gel and solid state La-doped $BaTiO_3$ ceramics with different amounts of dopant in the range ($0 \leq x \leq 0.025$) were reported. Impedance spectroscopy (IS) measurements were used in order to estimate the charge compensation mechanisms. The experimental results were compared with the Double Barrier Schottky theory for the description of the PTCR effect. D.C. resistance results and SEM analyses were used to discuss the influence of the dopant concentration on the PTCR behaviour and microstructure. IS measurements were performed at various temperature over the whole transition range and grain boundary and bulk contributions were extracted by a non linear least squares fitting procedure. Dielectric constant and resistivity values were obtained and used to derive model parameters for different samples. Temperature dependence of the total resistivity was calculated and compared to the D.C. curves. A good agreement in a large range of temperature, including the ferro-para phase transition, was observed.

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A33

NIOBIUM DOPED BARIUM BISMUTH-TITANATE CERAMICS

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Ferroelectric materials with diffuse phase transition (DPT) chractreristics and/or relaxor properties have been exstensively studied in the last few decades mainly due to their very interesting and still not completely explained physical properties. According to that, the aim of our work is to investigate how niobium (Nb^{5+}) as donor dopant influences on microstructure and electrical properties in relaxor BBT ceramics.

Dense pure and doped $BaBi_4Ti_{4-5/4x}Nb_xO_{15}$ (BBNT) ceramics (x = 0, 0.05, 0.15, 0.30 mol) ceramics were prepared by conventional solid state reaction from appropriate oxide mixture. Dielectric properties were investigated in a wide range of temperatures and frequencies (Fig. 1). It is indicated that the temperature of dielectric constant maximum (T_m) of BBNT specimens significantly decreases with the increase of niobium content.

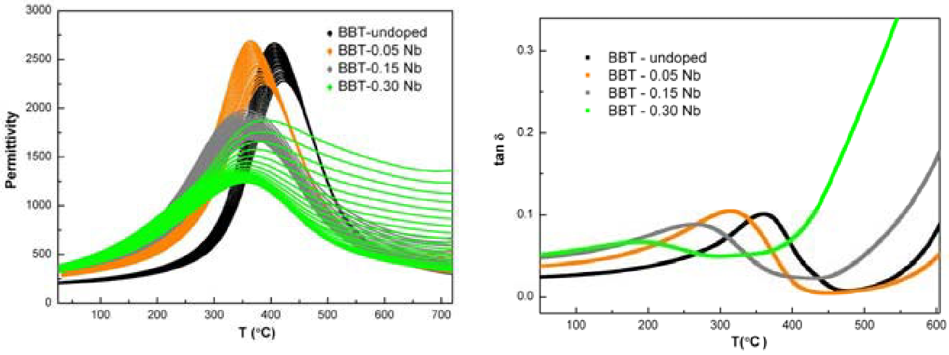

Figure 1. The temperature dependence of dielectric constant of pure and doped BBT at frequency range from 1 kHz to 1MHz and dielectric losses at 100 kHz

The dielectric relaxation rate follows the Vogel-Fulcher relation and fitting parameters which are measured at 100 kHz (T_{VF}, Ea, and f_o) are given in the table below.

Composition, x	ε_{RT}	T_m [K]	ε_m	Ea [eV]	T_{VF} [K]	f_o [Hz]
0	205	688	2430	0.023	660	$2.14\cdot10^9$
0.05	292	651	2424	0.204	555	$5.61\cdot10^{15}$
0.15	341	632	1770	0.040	594	$1.31\cdot10^{10}$
0.30	345	624	1300	0.001	621	$6.43\cdot10^6$

A34

STUDY OF THE FORMATION REACTION OF LEAD-FREE (1-x)BNT–xBKT CERAMIC

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The promising alternative lead-free system Bismuth sodium titanate-bismuth potassium titanate solid solutions [(1-x)BNT–xBKT] offers good piezoelectric properties which can be easily modified by various doping materials. A necessary condition for successful production of high performing materials lies in a better knowledge of the formation reaction.

To improve the understanding with regard of further doping attempts various compositions of (1-x)BNT–xBKT (x=10, 25, 50, 75, 90) were prepared by the commonly used mixed oxide route. The calcination process was identified as the most critical step during the solid solution synthesis with respect to stoichiometry, defect chemistry and formation of secondary phases.

The behavior during thermal processing was studied by thermo-gravimetric analysis and dilatometry. Further solid state characterization methods as X-ray-diffraction, scanning electron microscopy with EDX and electrical measurements were carried out on the resulting powders and sintered ceramics. Microstructure of sintered samples was investigated using channeling contrast imaging revealing the decrease of the average grain size exhibiting a minimum at x = 75. Distribution of elements was mapped by electron probe micro analysis.

A35

FINE GRAINED VARISTORS PREPARED FROM ZnO NANOPARTICLES

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Main problem of varistors' application in telecommunication systems is their low resistivity in a high frequency region. In our study ZnO nanoparticles were used for preparation of varistors with smaller grains than common varistors in order to increase resistivity. Doped ZnO nanopowder was synthesized by pyrolytical method starting from $Zn(COO)_2 \times 2H_2O$. ZnO phase was doped with Co and Mn during synthesis by addition

of $Mn(CH_3COO)_3 \times 2H_2O$ and $Co(CH_3COO)_2 \times 4H_2O$ in such amounts to obtain 0.1 mol% and 0.3 mol% of metal relatively to Zn. After synthesis, the powder was washed with distilled water and dried at 80°C for 2 hours. Powder was characterized by SEM and XRD methods. Varistor mixture was prepared by mixing ZnO powder with previously prepared spinel ($Zn_{1.971}Ni_{0.090}Co_{0.030}Cr_{0.247}Mn_{0.090}Sb_{0.545}O_4$) and γ-Bi_2O_3 (6 $Bi_2O_3 \cdot MnO_2$) phase in ethanol. Stearic acid was added to achieve uniform phase distribution. Varistor pellets with different mass ratios of ZnO, spinel and γ-Bi_2O_3 were uniaxially pressed and sintered at 1000°C/1h. Microstructural properties of obtained varistors were analyzed by SEM on polished and etched surfaces. Characteristic parameters (α, J_l, K_c) were obtained from *U*-*I* curves.

A36

CONTRIBUTIONS TO THE STUDY OF NON-LINEAR DIELECTRIC PROPERTIES OF $BaTi_{1-x}Sn_xO_3$ CERAMICS

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Pure perovskite phase of barium stannate titanate ($BaTi_{1-x}Sn_xO_3$ with composition $x = 0$; 0.05; 0.10; 0.15 and 0.20) ceramics were prepared by solid state procedure (Fig. 1) and sintered at 1400°C for 4 h [1]. As the Sn content (x) increases, the tetragonal distortion c/a decreases and the symmetry changes from tetragonal (specific for pure $BaTiO_3$) to cubic. Therefore, a ferroelectric-relaxor crossover is induced when increasing the Sn concentration.

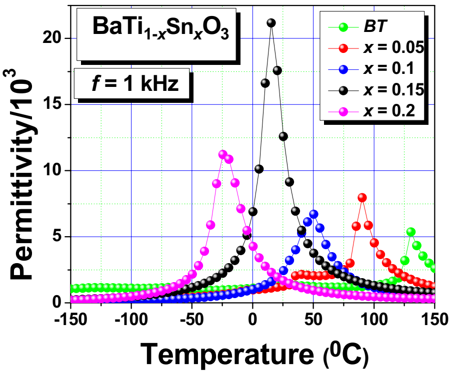

Figure 1 Permittivity vs. temperature

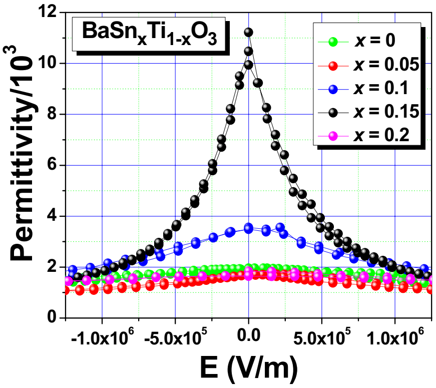

Figure 2 Permittivity vs. applied field

Dielectric constant of $BaTi_{1-x}Sn_xO_3$ is field-dependent: relative tunabilities between 0.18–0.85 were found for all the compositions at the applied field of E = 12 kV/cm (Fig. 2). The dielectric and ferroelectric properties of the $Ba(Ti,Sn)O_3$ ceramics are strongly dependent on the Sn addition, grain size and on temperature. The non-linear dielectric properties were described by multi-polar mechanism of polarisation with composition and temperature-dependent parameters [2].

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A37

MULTIFERROICS APPLICATION – MAGNETIC CONTROLLED PIEZOELECTRIC TRANSFORMER

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Modern electronic devices development steady trends toward multifunctionality. Multiferroics have been defined as materials that exhibit more than one primary ferroic order parameter, however many researchers consider multiferroic state only if they exhibit coupling between the mentioned parameters. The application of such advanced devices is realised by superimposing two different effects. In recent years, there has been great interest shown in mulitferroic materials in form laminated heterostructures taking advantage of mentioned superposition.

In spite of progress in lead free materials technology the lead zirconate titanate (PZT) - ceramic posses currently still the highest electromechanical coefficients as for piezoelectric applications, so that this material was chosen to our experiment. The magnetoelectric laminate Piezoelectric Transformer from investigated material and magnetostrictor was fabricated. This construction exhibits many interesting and unusual piezoelectric and magnetic properties. A demonstration of magnetic polarization reversal via the application of magnetic field across a multiferroic heterostructure is presented.

A38

BNT-BASED MULTILAYER DEVICE WITH LARGE AND TEMPERATURE INDEPENDENT STRAIN

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Among the many proposed systems to replace PZT, BNT (Bismuth sodium titanate) -derived materials seem to be the prime candidate for replacing lead based ceramics in high strain actuators. In addition to the as of yet unknown mechanism for achieving its very high strain, the processing presents unique challenges regarding the inclusion of alkaline metals which usually force an ethanol based production route. We have successfully prepared a prototype multilayer stack utilizing a multilayer production route based on the mixed metal oxide technique and industry-standard silver palladium electrodes, which would make the process compatible to existing production machinery. The displacement of 0.19 % is temperature independent within 25–150°C, and the design principles for both the processing route as well as the T- independent strain will be discussed. Due to the low thickness (35 μm) of the active layers compared to disc samples we are able to bypass the high voltage requirements of this system and this enables us to make field dependent Raman measurements, in addition to T-dependent ones.

A39

CONDUCTIVITY, DIELECTRIC LOSES AND DIELECTRIC PERMITTIVITY DEPENDING ON THE TEMPERATURE OF BISMUTH TITANATE CERAMICS AND GLASS-CERAMICS, CONTAINING SiO_2 AND Nd_2O_3 AS ADDITIVES

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Keywords: bismuth titanate, melt quenching, electrical characteristics, microstructure

Bismuth-titanate ceramics are synthesized by two methods - solid state reaction and melt quenching. Applying these techniques polycrystalline samples and glass-ceramics with different morphology and amount of crystalline phases are obtained in the systems Bi_2O_3-TiO_2-SiO_2, Bi_2O_3-TiO_2-Nd_2O_3 and Bi_2O_3-TiO_2-SiO_2-Nd_2O_3.

The phase composition of the produced materials is indentified by X-ray diffraction analysis and electron probe microanalysis. The phase $Bi_4Ti_3O_{12}$ is registered as main crystalline one. The texturing of the crystals upon the axe C after appropriate thermal treatment is established.

The measurement of the conductivity, dielectric loses and dielectric permittivity depending on the temperature are performed by DC resistible bridge using two-terminal method and AC capacity meter. The polarization (*P-E*) is presented for selected samples. It is found that all investigated samples are dielectric materials with conductivity between 10^{-5} and 10^{-13} $(\Omega \text{ cm})^{-1}$, capacitance - between 10^{-8} and 10^{-12} F and dielectric losses tgδ between 0.0002 and 0.1.

The experimental results show that the applied techniques of synthesis are promising basis for modification of the electrical properties.

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A40

$SrTiO_3$ + La THIN FILMS AND ITS POTENTIAL APPLICATION

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$SrTiO_3$ thin films with a La (SLTO) nominal concentration of 3%, 5%, 8%, 12% were grown by Chemical Solution Deposition (CSD) on the silicon substrate with Pt texture. The films had the thickness of 30-80nm. The XPS and TOF-SIMS showed they have correct stoichiometry and homogenous distribution of La. Resistive switching behaviour was studied with using a Local-Conductivity AFM (LC-AFM). La concentration was optimized to influence the character of switching. The scanned areas with the conductive AFM tip (according with applied DC voltage) showed high electrical inhomogeneities.

The study of the electronic structure of SLTO thin films shows that additional electrons in the host matrix are accommodated by a change of the valence of Ti ions from Ti^{+4} to Ti^{+3} and Ti^{+2}. Similarly, electrical characterisation of the material revealed semiconducting and metallic behaviour. This stands in accordance to the general idea of a macroscopic insulator to metal transition in SLTO at a doping level of a few percent of La. By means of conducting AFM we found evidence of an inhomogeneity of the in-plane conductivity at the nanoscale and the possibility of locally manipulating the electric resistivity by means of an electrical stimuli, i.e. resistive switching. The observed inhomogeneity is attributed to a nonuniform distribution of La doping (in the nanoscale) and a high density of extended defects.

SLTO pretends to be a new class of resistance memories (RRAM) emerging as serious candidate for further high density memory application replacement.

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A41

DENSIFICATION AND STRENGTH OF POROUS Y-TZP CERAMICS

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The purpose of modern ceramic processing is to better control both the particle packing during consolidation as well as the microstructure during sintering. One of the possible ways to improve particle packing is by blending together powders with different particle size distributions. In order to avoid particle segregation, which can occur by combining inappropriate proportions and size ratios of coarse and fine particles, the so-called core-shell concept was proposed for the preparation of the starting materials. This concept exploits homo-aggregation, which results in a uniform distribution of fine particles attached to the surface of the coarse particles in the slurry.

In our work the sintering behaviours of nano-sized particles, sub-micron-sized particles and their powder blend in core-shell configuration during non-isothermal and isothermal regimes were evaluated. Note that after sintering powder blend is termed as a nanostructured material since an appropriate combination and distribution of nano-sized and submicron-sized particles enables the preparation of biscuit-sintered Y-TZP ceramics with improved mechanical properties.

The results revealed that during non-isothermal treatment the compacts of nano-sized particles started to densify at 600°C and reached almost theoretical density at 1200°C. Furthermore, the densification of the nanostructured (powder blend) and submicron-sized materials started at 800°C and 900°C, respectively, and reached the final density at 1400°C and 1450°C, respectively.

By introducing the isothermal stage into the sintering schedule, the densification of nanostructured ceramics is retarded in comparison with the dry-pressed, sub-micron-sized Y-TZP powders and the phenomenon was explained using an in-situ-heating TEM study.

As a consequence of retarded densification, the flexural strength of the biscuit-sintered, nanostructured material rapidly increases with the fractional density, starting from 80 MPa at 55 %TD and reaching a plateau of 670 MPa at 70 %TD. The highest increase, from 200 MPa to 450 MPa, was observed for a minimal increase in the densification from 58 %TD to 60 %TD. This remarkable increase in strength is related to the larger area of the interparticle contacts. The addition of the nanoparticles enhances the formation of the necks between the nano-sized and/or the submicron-sized particles. At 70 %TD the flexural strength almost doubles with respect to the conventionally used, dry-pressed, submicron-sized Y-TZP powder. The occurrence of a plateau at 70 %TD is explained by an SEM-based model of pore growth, which shows the extensive growth of pores/flaws by increasing the end temperature.

A42

MASTER SINTERING CURVE APPLIED ON TWO STEP SINTERING

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The Two-Step Sintering (TSS) as well as Single Step Sintering (SSS) heating profiles were used for sintering of tetragonal zirconia, cubic zirconia and hexagonal alumina powder compacts. The sintering behaviour was examined via high-temperature dilatometry using Master Sintering Curve (MSC) concept and the activation energy of sintering was determined from SSS experiments performed with different heating rates. On the other hand the significant deflection of MSC after applying second step of TSS was observed. This deflection was denoted to change of sintering mechanism acting at higher temperatures. This sintering mechanism was described by lower activation energy.

A43

SINTERING KINETICS OF NANOCRYSTALLINE CERAMICS USING MICROWAVE ASSISTED SINTERING

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Microwave and Spark Plasma Sintering (SPS) are the newly developed rapid sintering techniques that exploit fast heating rates and cause minimal grain growth during processing of ceramics [1,2]. Both methods have been successfully applied for consolidation of various metals, alloys and metal-ceramic composites showing high densification rates while having mechanical properties superior in comparison to those prepared by means of conventional sintering. However, in depth-understanding of these two processes is yet to be achieved [3–5]. Several phenomena observed during sintering such as non-thermal "microwave effects" [3], specific electrical field [4] and pressure [5] effects. Those are shown to contribute to enhanced densification and to minimal grain coarsening for the vast majority of materials during MWS [6] and SPS [7], respectively.

In present study the direct comparison of the spark plasma sintering (SPS) and the microwave sintering method for producing fine-grained fully dense nanoceramics has been used. The experiments include processing of both as-received powder and pressed samples. The *same heating rates* for both methods and sintering conditions were used (atmosphere, temperatures and dwell times). The densification process for both methods used follows mechanism of grain-boundary diffusion for the high temperature region (900–1200°C) with an apparent activation energies of 260 and 275 kJ/mol for the microwave sintering and SPS, respectively. The later is lower then usually found in case of conventional sintering of nanocrystalline zirconia (292 and 309 kJ/mol) [8,9]. The main difference between two

processes is for the low-temperatures region and the grain growth stage. An explanation and analysis of this phenomenon has been attempted.

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A44

CHEMICAL VAPOUR SYNTHESIS: A NOVEL ROUTE FOR THE SYNTHESIS OF $ZnFe_2O_4$ NANOPARTICLES

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Magnetic nanoparticles have received much attention recently because of their attractive properties which could lead to applications in catalysis, biomedicine, magnetic resonance imaging, data storage, environmental remediation and many more. Currently there are a number of reliable routes for the synthesis of magnetic nanoparticles, including $ZnFe_2O_4$ nanoparticles. However, the synthesis of magnetic nanoparticles with improved properties is always desired and exploring novel synthesis routes is still worthwhile. Chemical vapour synthesis was already proven to be an attractive route for the synthesis of simpler ceramic compounds [1,2]. In this work, we have shown that it can also be used for the synthesis of more complex, spinel, structures such as $ZnFe_2O_4$. The synthesised nanopowders were characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and low temperature nitrogen adsorption. Nanocrystalline zinc-ferrite synthesised by the CVS method was then compared to the powders obtained by wet chemical synthesis.

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A45

SPECIFICS OF THE RESULTS OF BAUXITE THERMAL ANALYSIS

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Keywords: Bauxite, Thermal analysis, Aluminum oxides

This paper presents results of thermal analysis of bauxite. As is known, bauxite is the basic raw material in the technological process of alumina production. Respect to its complex mineral composition, one of the basic methods to the analysis of bauxite, is thermal TG-DTG-DTA analysis. This thermal analysis provides essential results in terms of determining the quality bauxite and its application. Thermal analysis can be used for qualitative analysis of bauxite in respect of mineral basic form as well as partial quantitative analysis, particularly in terms de-hydroxylation of hydrated mineral forms of aluminum, iron and other hydrated mineral forms.

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A46

STRENGTH OF WICK-DEBINDED CERAMIC PARTS PREPARED BY LOW PRESSURE INJECTION MOLDING

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Wick-debinding is an effective process for removing binder from injection-molded ceramic parts. However, a drawback of this procedure is the delicate operation of removing debinded pieces from the embedment after the process has been completed, cleaning and then loading them into a sintering furnace. It is important that high enough strength after the debinding is achieved with only a small amount of residual binder in order to avoid the formation of defects.

High solid content suspension for injection molding was prepared from Al_2O_3 powder and paraffin-based binder. The binder was thermally removed in the wicking

embedment in air and in nitrogen atmospheres. After the process bending strengths of samples has been determined.

We found, that strength depends on the residual paraffin-wax binder content and on the chemical nature of the binder, which changes drastically if the wick-debinding takes place in air at a temperature above 190°C. Under these conditions the paraffin binder undergoes a transformation, as a result of complex exothermic chemical reactions with oxygen. Part of it forms volatile products, while the remaining part cures into a non-volatile, brown-colored, solid substance, which resides in the wick-debinded part and bonds the powder particles firmly together. The curing can be beneficial, since strong wick-debinded parts with bending strengths up to 14 MPa, containing less than 2 % of the binder, can be obtained without flaws. The strength of the partially debinded parts increases with the dwelling time at 200°C, whereas the binder content reaches a minimum value of about 1.6 % and then remains constant with the dwell time. The residual binder is finally removed in the sintering cycle, where a very rapid heating can be applied without the risk of introducing flaws, because parts contain only little amount of residual binder.

A47

INFLUENCE OF HYDROXYAPATITE PORE GEOMETRY ON DRUG RELEASE KINETICS

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Release kinetics of antibiotic tigecycline from calcium hydroxyapatite (CHA), as one of the most used ceramic materials in bone tissue engineering, was investigated in this study. Tigecycline, in solid state, was mixed with CHA powder and the obtained mixture was compressed into tablets. The release of tigecycline from these tablets was measured in a pH 7.4 phosphate-buffered saline solution at 37°C by UV-VIS spectrophotometer. The total amount of tigecycline was released within 5 or 28 days, depending on the pressure applied during tablet production. Since the equations, commonly used for description of drug release kinetics, are confined to the first 60% of the release curve, a new drug release mechanism is suggested here. It determines the relationship between pore sizes and drug release rate, quantifying the drug release kinetics based on pore sizes and pore size distribution.

A48

SYNTHESIS AND CHARACTERIZATION OF BIO-CALCIUM PHOSPHATES

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Calcium phosphates (CaP) such as octacalcium phosphate (OCP, $Ca_8H_2(PO_4)_6{\cdot}5H_2O$), hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(HO)_2$), brushite ($CaHPO_4{\cdot}2H_2O$) and monetite $CaHPO_4$ are of great scientific interest in the field of biomaterials. HAP is the main constituent of bone, dental calculi and enamel. OCP is structurally similar to HAP, and is a precursor for formation of HAP in bone, also it has very good biodegradabile and osteoconductive characteristics. Brushite is a biological mineral that occurs in bones and teeth and as a transitional phase in the crystallization of HAP. It is also used as a CaP cement, and as an abrasive in toothpaste. When bushite loses its structural water it transforms to monetite.

The preliminary objective of this study is synthesis and characterization of these CaP phases. Materials were obtained by titration of the solution $(CH_3COO)_2Ca{\cdot}H_2O$ with the solution $NaH_2PO_4{\cdot}H_2O$ in different molar ratios, under constant stirring, and temperature around 60°C. Brushite forms at pH around 5, and at pH around 7 OCP and HAP are formed. This was confirmed by XRD and Raman spectroscopy.

Pure brushite nanomaterial was obtained, and crystallite size was modified by grinding in vibrating mill. The sample was ground five times for 2.5 minutes and analyzed by XRD between grinding. Particle sizes were determined from the XRD patterns using Scherer equation. After about 5 min of grinding an increase in temperature causes a phase transformation from brushite to monetite. BET method indicates that synthesized brushite have micro porosity. After 5 minutes of milling have mezzo porosity, these results were confirmed by SEM images.

Using this method can be very difficult to synthesize pure HAP or OCP. Ritveld refinement showed that we did not get pure OCP. Synthesized OCP was examined also by SEM method.

A49

SYNTHESIS AND CHARACTERIZATION OF MULLITE

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Mullite is important material for ceramic applications due to excellent mechanical and thermal properties. In this study, synthesis of mullite ($3Al_2O_3·2SiO_2$) powder by sol-gel combustion procedure was investigated. Synthesis was performed from ethanol-water solution of aluminium nitrate, TEOS and glycine as the fuel. The molar ratio of TEOS : Ethanol : Water = 1 : 20 : 40 was kept the same during the synthesis while the ratio of glycine:nitrate ions was varied (0.56 and 1). Besides, HNO_3 and NH_4NO_3 were used as the combustion aids. In all reaction mixtures amorphous products were obtained during combustion process and mullite was formed by their complete reaction at 1200°C for 4 hours. The phase identification and the characterization of crystal structure were performed by X-ray diffraction. According to the values of the mullite lattice parameters, the chemical composition corresponds to the 3/2 mullite with 62 mol.% of Al_2O_3. The average crystallite size of mullite powders was 40 nm.

A50

APPLICATION OF MTT VIABILITY TEST FOR BIOCOMPATIBILITY EXAMINATION OF N-CP/DLPLG NANOPARTICLES ON CELL CULTURES

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Keywords: nanoparticles, N-CP/DLPLG , HeLa, MDCK, viability, MTT

MTT test is a widely used method for examination of material biocompatibility *in vitro*. Nanomaterials, because of their specific structure, usually require a special approach in this kind of examination. The aim of this study was to examine the effect of calcium phosphate/poly-(DL-lactide-co-glycolide) (N-CP/DLPLG) nanoparticles suspension on viability of morphologically different cells from the two cell lines, HeLa and MDCK, respectively, by performing standard MTT viability/cytotoxicity test. Nanoparticles N-CP/DLPLG were suspended in the culture media DMEM (*Dulbecco's Modified Eagle Medium*) and examined in the following effective concentrations: 1000 mcg/ml, 200 mcg/ml, 40 mcg/ml, 8 mcg/ml, 1.6 mcg/ml and. The cells were seeded into

the 96 well plates and cultivated for 24 h in *Dulbecco's Modified Eagle Medium* (DMEM) at 37°C in humidified 5% CO_2 atmosphere. Cells were incubated with suspension of nanoparticles for 24 h. Cells only with medium were used as a control. After an incubation period MTT test was performed. Final absorbance value was calculated according to level of MTT reduction made only by nanoparticles under the same conditions. The minimum concentration of the nanoparticles suspension is not cytotoxic for HeLa cells and cytotoxic effect can be noticed at higher concentrations beginning from 8 mcg/ml. Cytotoxic effect of the nanoparticles suspension on MDCK cells is less pronounced and has been manifested by the two highest concentrations. Considering the morphological characteristics of HeLa and MDCK cells and the differences in their size and formation of monolayer, it can be assumed that an important factor in the effect on the viability is a cell surface area which is exposed to nanoparticles.

A51

NANOCRYSTALLINE SOLID SOLUTION CeO_2-Bi_2O_3

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Nanocrystalline powders of solid solution CeO_2 - Bi_2O_3 were synthesized by self-propagating room temperature reaction (SPRT) procedure with composition ($Ce_{1-x}Bi_xO_{2-\delta}$ where the $x = 0.1–1$). X-ray diffraction analyses show that for $x < 0.50$ a solid solution with fluorite structure is formed. Rietveld's structure refinement method was applied to characterize prepared powder and its microstructure (size-strain). The lattice parameters increase according to Vegard's law with increasing of Bi concentration. The average crystal size is about 2–3 nm. Spectroscopic ellipsometry and Raman scattering measurements were used to characterize the samples at room temperature. The Raman measurements demonstrated electron molecular vibrational coupling and increase of oxygen vacancy concentration whereas doping provokes a small decrease of optical absorption edge in comparison with pure ceria. Specific surface area of obtained powder was measured by Brunauer-Emmet-Teller (BET) method.

A52

Sr AND Mg DOPED LaGaO$_3$ PREPARED BY NON-CONVETIONAL METHODS

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Doped LaGaO$_3$ is an excellent ions conductor oxide, with potential application in oxide solid fuel cells. Sr^{2+} and Mg^{2+} simultaneous doped lanthanum gallate (LSGM) powders, prepared by a modified Pechini route, were dandified using an activated microwave technique at 2.45 GHz to develop a dense stable electrolyte layer for application in intermediate temperature - solid oxide fuel cells (IT-SOFC).

The evolution of secondary phases, such as LaSrGa$_3$O$_7$ has been identified to be a problem in the preparation of LSGM. Actually, no mater what technical preparation route was adopted (wet-based or solid state-based method) the presence of the secondary phases is related accompanying the main phase.

The present paper deals with investigations regarding the preparation and characterization of doped La(Sr)Ga(Mg)O$_3$ powders and sintered bodies. Thermal analysis (DTA/TG) on the stoichiometric raw mixture was realized. Morphological and structural aspects were investigated by mean of X-ray diffractometry, FT-IR spectrometry, scanning electron microscopy and dilatometry measurements. The electrical behaviour of the obtained ceramic was characterized by impedance spectroscopy.

Good densities (90–93%TD) and TEC constant ($\alpha \sim 9.5\times10^{-6}$ K^{-1}) were obtained. The LSGM electrolyte prepared through this technique has higher quality than the solid-state route and is suitable for thick films preparation.

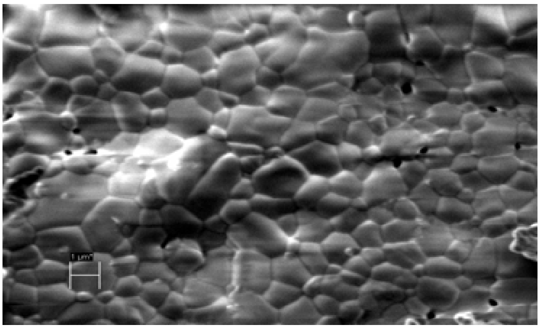

Figure 1. SEM micrographs of sintered pellets at activated microwave field 1400°C/10min

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A53

NANOSTRUCTURED THIN FILM $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ SYNTHESIZED VIA SPRAY PYROLYSIS FOR MICRO-SOFC APPLICATION

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Micro-solid oxide fuel cells (micro-SOFCs) are anticipated for battery replacement due to their increased energy capacity for applications such as portable electronic devices, mobile phones, laptops and personal digital assistants [1,2]. A micro-SOFC consists of a free standing stack of thin film electrodes and electrolyte which is formed on a substrate that can be microstructured. The reduction of performance in micro-SOFCs is attributed mainly to the ohmic resistance of the electrolyte and the over-potential losses at the cathode [3]. Therefore, in order to increase the performance and reduce the operating temperature of SOFCs, thinner electrolytes and alternative cathode materials with mixed ionic-electronic conductivity (MIEC) have been studied [4].

In this work, $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ (LSC) a potential cathode material with MIEC was synthesized via spray pyrolysis. The LSC powder was characterized by X-ray diffraction, nitrogen adsorption (BET), and high-resolution electron microscopy. The XRD results show the formation of a nanocrystalline single phase perovskite structure. According to the BET results, the specific surface area of the nanopowders is between 25–30 m^2/g. LSC nanopowders were dispersed in water-based solution via ultrasonic processing. Thin films consisting of LSC nanoparticles with thickness less than 1 μm were prepared via single step spin coating on yttria doped zirconia (8YSZ) substrates. Gadolinium doped ceria (GDC) film was applied to YSZ substrate to avoid the chemical reaction between cathode and electrolyte. The performance of the thin film cathodes was evaluated by high temperature impedance spectroscopy on symmetrical samples.

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A54

STRUCTURAL AND ELECTRICAL PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$ THIN FILMS DEPOSITED BY PLD AND RF-ASSISTED PLD

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$YBa_2Cu_3O_{7-\delta}$ (YBCO) superconducting films were grown on (001) oriented $SrTiO_3$ single substrates using a PLD and RF-PLD.

Control of the YBCO thin film epitaxial growth and microstructure quality are important factors for superconducting electronic device applications. In this study, it was observed the influence of the deposition parameters on the properties: oxygen pressure during the growth, the distance between target-substrate, and substrate temperature during the deposition.

For studying the structural properties, morphology, and optical properties of thin films, there were used specific analysis (X-ray diffraction, atomic force microscopy and spectroscopic ellipsometry). The electrical properties of thin films were measured by four probe method in liquid helium, for temperature range of 4.2–300K.

A55

PHOTOSENSITIZATION OF TITANATE NANOWIRES WITH CORE-SHELL QUANTUM DOTS

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Quantum Dots (QDs) are semiconductor nanocrystals. They have a great interest because of their unique, size-dependent optical and electronical properties due to the quantum confinement effect. Since they have extraordinary optical properties there are many efforts to use them as light-emitting diodes, lasers, biological imaging and recently as sensitizers in TiO_2 based photovoltaics and photocatalytic systems.

In order to sensitize the anatase TiO_2 nanowires which have a relatively large 3.2 eV band gap, different sized CdSe nanocrystals were anchored on them via linker molecules. In the case of photosensitized TiO_2 nanowires the visible light can excite the electrons from the valence band of the CdSe quantum dots which can get through to the conduction band of the anatase nanowires.

The main method to produce CdSe quantum dots with different size was a simple organometallic synthesis where the diameter of the nanocrystals depends on the growing time of the nanocrystals. The synthesis of the TiO_2 nanowires was based on the hydrothermal method in autoclave and the trititanate nanowires was washed with HCl and calcinated for 5 h at 600°C to get anatase form TiO_2 nanowires. The anchoring of the CdSe QDs to the surface of the nanowires was accomplished in acetonitrile solution with thioglycolic acid.

We studied the properties of sensitized TiO_2 nanowires with Transmission Electron Microscopy, Energy Dispersive Spectroscopy, Reflactance spectroscopy and X-ray Diffraction. We were able to get photosensitized titanium-dioxide nanowires decorated with three different sized CdSe nanocrystals.

A56

SEMICODUCTING PROPERTIES OF TITANIUM OXIDE CONTAINING CERAMICS

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Nonstoichiometric rutile TiO_{2-x} is advanced material for many applications among which the electrodes in photoelectrical cells for contaminated water purification are the most important from the point of view of this work.

Cylindrical TiO_2 (anatase) samples (10 mm in diameter) were formed using uniaxial pressing by dry extrusion technology and cut into the specimens with 18 mm height. Thermal treatment was carried out in two stages: sintering in air at 1000 and 1200°C (transition from anatase to rutile) and annealing in high-vacuum conditions at 1150 and 950°C (forming of nonstoichiometric rutile TiO_{2-x}).

Electrical measurements were carried out in temperature range from room temperature up to 400°C with simultaneous thermopower and resistance measurements.

The electrical properties of TiO_{2-x} depend on phase transitions, which in turn depend on thermal treatment regime in air and vacuum. Grain size of ceramics affect grain boundary, which may absorb charge carriers. There are different charge transport mechanisms. All obtained specimens have *n*-conductivity type.

A57

BISMUTH TITANATE THIN FILMS PREPARED BY WET-CHEMICAL TECHNIQUE

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In recent years, considerable attention has been devoted to ferroelectric thin films, because of their possibilities in device applications to sensors, micro-electro-mechanical system, and nonvolatile random access memories

In this work, layered perovskite bismuth titanate ($Bi_4Ti_3O_{12}$) thin films were deposited on α-alumina substrate by spin coating. Bismuth titanate precursor sol, used to prepare thin films, was prepared by sol-gel process, using bismuth nitrate and titanium butoxide as starting materials, and glacial acetic acid and diethanolamine as solvent and stabilizing agent, respectively. Precursor sol was examined during the aging period of 20 days, while microstructure and characteristics of obtained thin films were followed in respect to the aging time of precursor sol used for the fabrication of the film. Prepared thin films are crack-free, with the thickness of ~1 μm, uniform surface texture and rounded grains having grain size in nanometer range.

A58

GAS SENSORS BASED ON WO_3 NANOPARTICLES, WO_3 AND WO_3/TiO_2 NANOWIRES

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Gas sensing is an integral part of many industrial processes and also of everyday life. The most important factors of gas sensors are sensitivity, detection limit, response time, reliability, long-term stability and price. Metal oxide semiconductors, owing to

their high sensitivity, are widely used as gas sensing materials. In our research, we have concentrated on various WO_3 nanostructures and nanocomposites.

On the one hand, we prepared WO_3 nanoparticles with controlled composition (oxidized/yellow or partially reduced/blue) and crystal structure (hexagonal or monoclinic) by heating hexagonal-ammonium-oxide bronze, $(NH_4)_xWO_{3-y}$. Our aim was to study the effect of the composition and structure of WO_3 on its gas sensing properties. On the other hand, we obtained WO_3 and WO_3/TiO_2 nanowires by electrospinning and atomic layer deposition (ALD). The core WO_3 nanowires were manufactured by electrospinning aqueous solutions of ammonium-metatungstate and polyvinyl-pyrrolidone. Then the WO_3 nanowires were covered with a 1.5 nm TiO_2 layer by ALD using $TiCl_4$ and H_2O as precursors at 250°C. The structure, composition, morphology and optical properties of the samples were examined with XRD, Raman, diffuse reflectance UV-VIS, XPS, SEM and TEM-ED. Their gas sensing was tested by sensing NH_3 at three temperatures (120°C, 200°C, 300°C) and seven concentrations (250, 500, 1000, 2000, 3000, 4000, 5000 ppm).

Among the studied WO_3 nanoparticles, the monoclinic WO_3 samples had the best gas sensing properties at 300°C. In contrast, the hexagonal WO_3 samples exhibited the highest sensitivity at 120°C. Therefore, the crystal structure of WO_3 determined the proper sensing temperature. Additionally, if WO_3 samples were a bit reduced, the gas sensing response changed its direction between 120–200°C, just as if a *p-n* change would have occured in the samples. Thus, the composition of the samples also affected the sensing properties .

The pure WO_3 nanowires, due to the higher specific surface, were more sensitive than WO_3 nanoparticles. The sensitivity of the WO_3/TiO_2 composite nanowires was similar to the pure WO_3 nanowires, but higher resistance values were detected owing to the interface between WO_3 and TiO_2.

A59

PHONON-PHONON AND ELECTRON-PHONON INTERACTION IN PURE AND DOPED $FeSb_2$ SINGLE CRYSTALS

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Iron diantimonide $FeSb_2$ is a narrow-gap semiconductor representative of the TM_2 pnictide family with a number of interesting and unusual behaviors. The lattice dynamics of $FeSb_2$ is investigated by the first-principles DFT calculations and Raman spectroscopy. All Raman active phonon modes, predicted by factor-group analysis, are properly assigned. We also present polarized low temperature Raman scattering spectra of the $Fe_{1-x}Co_xSb_2$ and $Fe_{1-x}Cr_xSb_2$ single crystals. The linewidths and energies of the Raman modes were analyzed as a function of Co and Cr concentration and temperature. Strong electron-phonon interaction has been observed for the B^2_{1g} mode of pure $FeSb_2$. With increasing concentration of Co and Cr in $Fe_{1-x}(Co,Cr)_xSb_2$ alloys electron-phonon interaction contribution is drastically reduced. We have observed strong mixing of the Ag symmetry modes, with the intensity exchange in the temperature range between 210 K and 260 K. The Ag mode repulsion increases by Co doping of $FeSb_2$ with no signatures of the electron-phonon interaction.

A60

THE INFLUENCE OF THE NATURE OF MATRICES AND EMBEDDED SPECIES UPON THE PHYSICOCHEMICAL PROPERTIES OF FINAL SOL-GEL DERIVED MATERIALS

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Inorganic-organic hybrid materials have attracted considerable attention with the expansion of soft inorganic chemistry processes. The sol-gel process has particular interest for designing active materials with specific properties and improving performance. The flexibility involved in this process allows the entrapment of active

species in a porous matrix, with the production of the designed microstructures at low synthesis temperature [1,2]. These hybrid materials, in general, retain porosity depending on the size of the structural unit of the network [3–5]. The sol-gel technique was used to obtain powdery materials with different structures and morphology. The aim of this work is to investigate the influence of the synthesis parameters such as silica precursors nature, type of catalyst, addition of some additives (Polyvinyl alcohol or ionic liquids) effect on the properties of obtained silica matrices and of final immobilized materials. As embedded species are rare-earths (Eu^{3+} and Ce^{3+} ions), macrocyclic compounds (different porphyrins) and enzymes were selected. The physical properties of obtained doped hybrid silica materials were characterized by thermal analysis, N_2 adsorption/desorption, electron microscopy techniques, FT-IR, UV-visible and photoluminescence spectroscopy. Depending on the synthesis parameters mesoporous silica materials with pore diameter of 2–6 nm, specific surface area of 100–1200 m^2/g and improved activity of the embedded species were obtained.

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A61

EFFECT OF SURFACE FUNCTIONALIYATION ON SZNTHESIS OF MESOPOROUS SILICA CORE/SHELL PARTICLES

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A pure and simple procedure for preparation of silica core/shell particles is reported. Core/shell particles were formed by assembling the primary silica nanoparticles synthesized by neutralization of highly basic sodium silicate solution, on the surface of monodispersed silica core (average size ~0.4 µm). The core particles were prepared by hydrolysis and condensation of tetraethylortosilicate. To improve assembling of primary silica particles on the surface of silica core particles, the latter were functionalized with 3-aminopropyltriethoxysilane (APTES) or poly(diallyl-dimethylammonium chloride) (PDDA). The obtained particles have mesoporous, uniform and continuous silica shell and were prepared without pore-forming agents. The pore size and volume are easily adjustable by controlling the formation of primary silica nanoparticles during synthesis of core/shell particles.

CERAMIC COMPOSITES

C1

LANTHANUM ORTHO-NIOBATE BASED COMPOSITE MATERIAL FOR IT-SOFC ANODE SUPPORTS

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The problem with producing the proper material for intermediate temperature solid oxide fuel cells seems crucial nowadays. The development of electrolyte materials leads to strong need of suitable electrodes. The lanthanum ortho-niobates doped both on lanthanum and niobium site are strongly investigated nowadays [1,2]. The problem of finding the suitable electrodes for this material has been considered previously both for cathodes [3] and anodes [4]. In this work the proposed anode materials was composite of strontium doped lanthanum ortho-niobate and nickel. The electrolyte matrix was prepared from powders prepared by solid state method with usage of pore-former.The green bodies were sintered to obtained the bulk samples with desired density. The composite material was prepared by impregnating the electrolyte matrix by $Ni(NO_3)_2$ and then after drying reduced in hydrogen in order to obtain desired material. The samples phase content was determined by X-ray diffraction method. Sample microstructure was obtained by scanning electron microscopy and the composite electrical conductivity was measured by Van Der Pauw method.

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C2

THE INFLUENCE OF SILICA PARTICLES AND LAYERED NANOSILICATES ON PROPERTIES OF ORGANIC-INORGANIC NANOCOMPOSITES

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The study of the effect of colloidal silica, layered nanofillers (LN), either of natural character or with alcohol- and amino-functional compatibilizers in organic-inorganic (O–I) nanocomposites, prepared in the form of coatings is presented. The alcohol-functional compatibilizers are shown to have a marked influence on morphology, surface- and mechanical properties of the coatings prepared, in spite of the very small amount of both filler and compatibilizer (together 0.5 wt.%). On the other hand, silica nanoparticles influence end-use nanocomposite properties at higher concentration, 5 to 20 wt.%. Two O–I matrices were synthesized, both are formed by polyepoxy-functional polysiloxanes and organic diamines. Two commercial clays were used as LN, mainly in the concentration of 0.5 wt.%: natural montmorillonite (Cloisite Na) and Bentonite for water systems. These clays were used either untreated or they were compatibilized by three hydroxylcontaining substances: Polyethylene glycol 4000 (PEG), Polysorbate 80 (TWEEN), and tris(hydroxymethyl)aminomethane (TRIS). It was found that all compatibilizers improve the dispersion of the clay fillers, while TRIS always supports full exfoliation. They also cause an increase in the microroughness of O-I coatings and strongly modify their large-scale surface patterns. Mechanical properties are also strongly affected by the compatibilizers: toughness is increased as well as stress at break. Tensile and shear moduli are reduced by the compatibilizers, while the glass transition temperature is specifically shifted to higher or lower values.

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C3

GLASS-CERAMICS PREPARED FROM $Li_2O{\cdot}Nb_2O_5{\cdot}SiO_2$ GLASS CONTAINING TiO_2

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Keywords: glass-ceramics, crystallization, crystal growth.

This paper deals with the crystallization of glass $30Li_2O{\cdot}15Nb_2O_5{\cdot}50SiO_2{\cdot}5TiO_2$ (mol%). The parent glass was obtained by conventional melt quenching technique and its crystallization behavior was studied by non-isothermal method using differential thermal analysis (DTA). Also, the additional experiment were performed under isothermal condition by heating the bulk glass samples in the temperature range 650–830°C for different times from t = 15 min to t = 100 h. XRD and SEM methods were employed for determination of phase composition and microstructure of crystallized glass. It was detected that this glass crystallises by the surface crystallization mechanism. SEM micrographs of the crystallized samples revealed that the crystals grow in the form of dendrites. The glass-ceramics with complex phase composition was obtained. Three crystalline phases were detected where $LiNbO_3$ grown as primary phase and a secondary ones $Li_2{\cdot}Si_2O_5$ and SiO_2 appeared. From the non-isothermal experimental data an activation energy of crystals growth is calculated $E_a = 275 \pm 10$ KJ/mol.

C4

STUDY ON LOW-TEMPERATURE PRINTABLE FERROELECTRIC CAPACITORS BASED ON $BaTiO_3$ – POLYMER NANOCOMPOSITES

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Ferroelectric barium titanate - polymer nanocomposites have been great attention of printable low cost non-volatile memory technology. Ticketing and game cards as possible applications require lightweight and flexible functional components. Through the use of the printing roll-to-roll technologies, such as screen, flexo or gravure printing, large areas containing storage elements based on a plate capacitor layout with the metal /

ferroelectric / metal (MFM) layers can be manufactured with high productivity. Ceramic - polymer nanocomposite films are attractive as a functional layer in such capacitors, since the composite films are expected to synergize the advantages of both components: those are the good ferroelectric properties of ceramic materials as well as the good mechanic properties and easiness of film deposition of polymers.

In this work we studied the preparation of stable $BaTiO_3$ - polymer dispersions assisted by a three-roll mill process. The influence of different parameters on the dispersibility and film morphology, for instance particle size, ceramic - polymer ratio, viscosity and surface energy, were investigated. For the morphological (AFM, SEM) and electrical (dielectric constant, ferroelectric polarisation) characterization the coatings on ITO glass substrates were provided by using an applicator frame and dried at < 180°C for several minutes. Due to the low curing temperatures and rapid drying, flexible substrates such as polyethylene terephthalate (PET) or polyimide (PI) can be coated by low-cost roll-to-roll printing process.

C5

PYROELECTRIC RESPONSE OF POLYMER-CERAMICS COMPOSITES OF 0-3 CONNECTIVITY

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The 0-3 connectivity composites can be prepared by simple fabrication routes in a cost-effective manner. Composites based on pyroelectric ceramic particles embedded in polymer combine the properties derived from individual components: large pyroelectric coefficient of ceramics and the excellent mechanical strength, formability and robustness

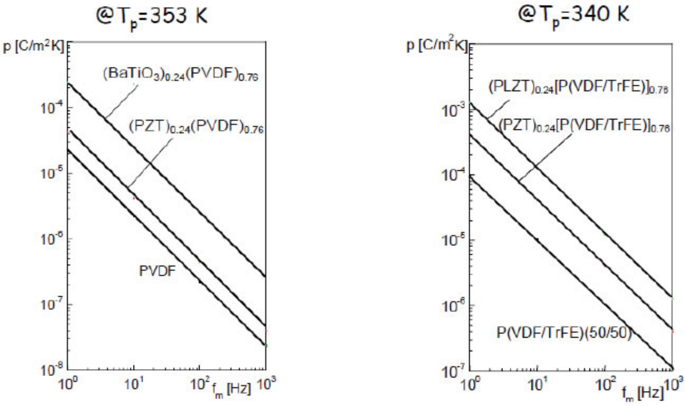

Figure 1. Pyroelectric coefficients p of PVDF, $P(VDF_{0.5}TrFE_{0.5})$ and the composites vs. modulation frequency

of the polymer. We studied the pyroelectric responses of PVDF polymer and $P(VDF_{0.5}TrFE_{0.5})$ copolymer matrices as well as the responses of the composites: $(BaTiO_3)_{0.24}PVDF_{0.76}$, $(PZT)_{0.24}PVDF_{0.76}$, $(PZT)_{0.24}[P(VDF/TrFE)]_{0.76}$, $(PLZT)_{0.24}[P(VDF/TrFE)]_{0.76}$ polarized in E_p = 25 MV/m during t_p = 10 minutes. Figures of merit: $FOM_I = p/\varepsilon'$, important for sensors used with high impedance amplifier, and $FOM_{II} = p/(\varepsilon'\cdot \mathrm{tg}\delta)^{1/2}$ important in the case when the noises are mainly due to the pyroelectric element, were calculated.

Composites $(PZT)_{0.24}[P(VDF/TrFE)]_{0.76}$, $(PLZT)_{0.24}[P(VDF/TrFE)]_{0.76}$ show the highest values of pyroelectric coefficients p, FOM_I and FOM_{II} due to better pyroelectric activity of the copolymer $P(VDF_{0.5}TrFE_{0.5})$ in comparison to that of the PVDF polymer.

C6

MORPHOLOGICAL CHARACTERIZATION OF INORGANIC NANOCOMPOSITES

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Nowadays, several material science studies are focused on nanoscaled materials due to the unique properties (electronic, thermal, mechanical, magnetic etc.) related to their tiny size which makes these materials different from their bulk counterparts.

Titanate nanotubes are one of the recently discovered and widely researched nanomaterials. They represent a beneficial mixture of the positive chemical features of titanium-dioxide and the morphological advantages related to their one dimensional nanostructure. They measure 6–10 nm in diameter and up to 200 nm in length. They are open-ended and resemble miniscule rolled-up carpets consisting of layers no more than a few atoms thick.

High specific surface area due to the tiny size, high aspect ratio and special crystal structure are endowing these materials with high mechanical strength and ability for the usage as a filler material for polymers, ceramics, glasses to enhance tensile strength, wear resistance, hardness and other mechanical properties. In our work, such positive properties of the titanate nanotubes are exploited for low-temperature ceramic-type nanocomposite synthesis.

Several composites of titanate nanotubes, vanadium-dioxide and borone-trioxide of systematically varied composition were made by co-grinding and high energy ball milling process. The resulted materials were thermally treated at different temperatures.

The resulted materials were characterized by Transmission electron microscopy (TEM), Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS) X-ray as well as electron diffractometry (XRD, ED). In this contribution we will discuss the effects of the composition on the morphology.

C7

THE INFLUENCE OF SOME ADDITIVES CONTAINING SiO_2 ON PHYSICO-MECHANICAL PROPERTIES OF DRY MORTARS

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The range of commercial additives used to improve the properties of dry mortars is constantly diversifying. The effects of these additives on the physico-mechanical properties of dry mortars are complex and far from being completely known.

This paper shows the influence of three commecial additives A1, A2 and A3, on physico-mechanical properties of a mortar composed of Portand cement CEM I 42.5 R, limestone filler and quartz sand. The A1 additive is a modified sepiolite clay, the A2 additive is a metakaolin and the A3 additive is a modified bentonite.

Consistency, apparent density, mechanical strengths at different periods of hydration, durability through mechanical strength after freeze-thaw cycles and water absorption coefficient due to capilary action of hardened mortar were determinated using 0.12% A1, 2% A2 respectively 2% A3.

The influence of studied additives on the hydration processes of portland cement was followed through thermal analysis and phasal analysis through XR diffraction, on samples hydrated 7 respectively 28 days.

It was noted that these additives increase the water necessary and initial and final setting time of mortars. Regarding mechanical strength, mortars containing additives A1 respectively A3, have lower mechanical strength than the etalon mortar (without additives), while mortars containing additive A2 have higher mechanical strength than the etalon mortar. The water absorption coefficient due to capilary action is higher for mortars containing additives A1 respectively A3 than etalon mortar and lower for mortars containing the A2 additive.

The obtained results show that A2 can be used to increase the mechanical strength of mortars and to lower their water absorption coefficient, while A1 and A3 can be used with cellulose ethers or other water retainers in order to improve their thickening effect.

C8

ALUMINA ZIRCONIA DENSE COMPOSITE CERAMICS

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Alumina ceramics exhibits good mechanical properties for a relative low production cost. They have been studied intensively because of their above average physical and chemical properties which give them a very wide range of applications: cutting tools, ballistic ceramics, hard implants and others.

Present study deals with alumina ceramics and ceramic composites (ZTA type with 15% or 20% zirconia) with or without Cr_2O_3, used in different proportions (0.3 - 0.5%). The presence of Cr_2O_3 allows the obtaining of a high densification degree (relative densities are 98% and higher) and improves mechanical behaviour.

Another factor of influence is the oxide used for zirconia stabilization – MgO (5%) or a mix between MgO and CaO (4% and 2%).

The sintered bodies were ceramic and mechanically characterized by determining the relative density, shrinkage, open porosity and compressive strength and calculating the Young modulus. Also, the microstructure of the ceramic bodies was observed using scanning electron microscopy. The influence of microstructure upon mechanical properties is discussed.

C9

ROLE OF SINTERING ADDITIVES ON SiC FORMATION IN Si_3N_4 CERAMICS

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Keywords: Si_3N_4/SiC composites, microstructure, gas pressure sintering

The role of Y_2O_3-SiO_2-C addition on sinterability of Si_3N_4 ceramics was studied. The aim of this study was to find out the conditions for *in situ* formation of SiC inclusions in Si_3N_4 microstructure via carbothermic reduction of SiO_2 during gas pressure sintering. Series of samples were prepared with 15 wt.% addition of sintering additives Y_2O_3 and SiO_2. Their molar ratio was changed from 0.39 to 0.51 mol%. The thermodynamical analysis of possible reactions was done. This analysis showed the importance of powder bed composition on formation of SiC inclusions in the samples. Phase composition, microstructure, mechanical properties of studied samples were

investigated. This way prepared samples were compared with Si_3N_4/SiC reference composites, which were prepared by direct addition of polycrystalline SiC submicrometre powder to the starting powder mixture of the same composition as mentioned above. Samples were sintered by the identical sintering process.

C10

LASER CLADDING OF METAL/CERAMIC COMPOSITES FOR WEAR APPLICATIONS

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To increase the lifetime of mechanical parts submitted to severe abrasive environments, a common strategy is to reinforce their top surface by depositing a more resistant layer. This approach is particularly interesting for metallic parts exhibiting a poor wear resistance at high temperature. The incorporation of a dispersed ceramic phase within the reinforcing layer is known to dramatically enhance the resistance and the hardness of the top surface layer.

In the present work, laser cladding was used to fabricate thick metal/ceramic coatings on steel substrates. Laser cladding is a melting process in which the laser beam is used to fuse an alloy and/or a composite directly onto a substrate. This technique can be processed in one or two-step. In the one-step method, powders are fed directly into the melt pool whereas in the two-step method, powders are pre-placed onto the substrate and then laser treated. In this work a one step method was considered with a 1 kW Nd:YAG laser source operating in a continuous mode and equipped with a Precitec co-axial nozzle.

Metal matrix composite (MMC) coatings composed of Ni-, Co- or Fe-based matrix alloy containing hard tungsten carbides have been considered to improve the wear resistance of steel (S235) parts. The secondary phase amount, its morphology (spherical or not), its dimension and its chemistry (presence or not of a cement Co binder in the WC phase) have been studied. In order to limit the formation of cracks during the layer cooling down subsequent to laser cladding, the influence of a pre- and post- heating of the sample was assessed. Similarly, the effect of a bond-coat to improve adherence and limit the thermal mismatch between the layer and the substrate was carefully investigated.

It is shown that a net improvement of the wear properties can be obtained although the suitable range of process parameters is very narrow.

C11

INFLUENCE OF CARBON ALLOTROPES ON THE PREPARATION AND MECHANICAL PROPERTIES OF Al_2O_3-C COMPOSITES

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Alumina ceramics, one of extensively utilised structural ceramics, have great potential to be used in many special applications where low density, high hardness, chemical inertness and good high temperature properties are required. However, alumina is a brittle material with poor fracture toughness and thermal shock resistance. Carbon allotropes, such as carbon nanotubes (CNTs) and diamond have excellent mechanical properties. Therefore, they are attractive candidates for reinforcement of various materials. Except their reinforcing effect, they can improve also functional properties, such as thermal and electrical conductivity. In this work alumina ceramics containing 2.5, 5, 7.5 and 10 vol.% addition of various carbon allotropes amorphous carbon, multi-walled carbon nanotubes (MWCNTs) and/or diamond powder are densified by hot-press method and studied. This work is focused on the study of influence of amorphous carbon, CNTs and diamond powder addition to microstructural evolution, change of mechanical, functional properties, chemical resistance and thermal shock behaviour of ceramic materials. In this study we try to optimize content of various additions and conditions of heating in order to obtain material usable in harsh environments.

C12

SYNTHESIS AND PROPERTIES OF TITANIA/ALUMINA CERAMIC FOAM FORMED BY CATALYTIC HYDROGEN RELEASE

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There are a lot of applications for porous ceramic materials, such as water filtration, filtration of exhaust gases and molten metals, as catalyst carrier, refractory applications etc. However most of the commercially available porous ceramic materials are produced using different pyrolysis techniques which have some considerable disadvantages - as toxic fumes, leftover residues of organic precursors etc.

Lightweight ceramic is produced by foaming the slurry of raw materials, using a chemical hydrogen gas formation technique. This technique is quite similar to the one used in aerated concrete production as it uses fine grade aluminium powder as foaming

agent. Hydrogen is an end product in the reaction between water and aluminium in the catalytic presence of two of the base materials – γ-Al_2O_3 and kaolin

This study shows that even without using polymer fillers and sponges it is possible to achieve porosity over 50%. Viscosity of the slurry and thus the porosity could be regulated by changing the content of water in the slurry In the first part of this study, dependency of pore size, geometry and porosity on the properties of slurry are analysed. In second part, after sintering and mechanical preparation, composition, thermal shock resistance, mechanical strenght as well as filtering properties etc., are evaluated.

C13

MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF $SrTi_{0.98}Nb_{0.02}O_{3-\delta}$-BASED COMPOSITES APPLIED AS POROUS LAYERS

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Solid oxide fuel cell (SOFC) is a promising device that directly converts the chemical energy of various gaseous fuels into electrical energy and heat. The traditionally used anode for SOFC is Ni-YSZ cermet that suffers from many limitations. Hence, it is necessary to investigate some alternative materials, as perovskite-related structures. Among them $SrTiO_3$ is one the most promising compositions, but in a pure form it is a dielectric material. Its electronic conductivity can be simply increased by high temperature reduction and substituting with some donor dopants [1,2]. However, its ionic conductivity and electrocatalytic activity are still too low for commercial application of this material [3]. Moreover, there is a mechanical misfit between pure strontium titanate and yttria-stabilized zirconia (YSZ) electrolyte. This indicates the need of composites materials use to make the operating fuel cell more efficient and mechanically stronger [4,5]. The most popular are the composites consisting of the electrolyte and anode material. The former is and ionic conductor, whereas the later is an electronic conductor. Material prepared in this way prevents the process of layers delamination and increases the triple phase boundary (TPB), what should result in the fuel cell performance improvement. Also composites with some catalysts can be prepared in order to increase the electrocatalytic activity of the material.

The composites of $SrTi_{0.98}Nb_{0.02}O_{3-\delta}$ with CeO_2 and YSZ will be discussed. In order to evaluate the possibility of materials' application both bulk samples and composite porous layers have been investigated. The electrical conductivity of bulk samples was measured using the four terminal DC method, whereas the conductivity of the porous layers was determined using the DC Van der Pauw method in the temperature range of 400–850°C in humidified hydrogen. Morphology of samples was characterized by scanning electron microscopy (SEM) technique.

Although a significant advantage of bulk Sr(Ti,Nb)$O_{3-\delta}$-YSZ over Sr(Ti,Nb)$O_{3-\delta}$-CeO_2 composite was noticed in the electrical conductivity plots, a reverse tendency has been observed in the case of porous layers.

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C14

DIELECTRIC RELAXATION SPECTROSCOPY OF TITANATE NANOSTRUCTURES

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Keywords: dielectric relaxation spectroscopy, titanate, relative humidity, water content

Dielectric relaxation spectroscopy measures the dielectric properties of a medium as a function of frequency, by applying alternating electric field in the frequency range of 10^{-6}–10^{11} Hz.

When a medium is exposed to an electric field, some charges are bound, yet these positive and negative charges can move locally relative to each other, which results in a polarized medium. The polarizability of the medium is represented by its permittivity. In other words, permittivity is a measure of the extent to which the electrical charge distributed in a material can be polarized by the application of an electric field. The spectral response of permittivity with frequency captures the various polarization mechanisms.

In recent years, solid state humidity sensors have been widely used for the measurement and control of humidity in an industrial or household environment, so there was the need to investigate different materials for making more sophisticated sensors. Various titanium oxides have attracted much attention in the past decades because of their unique properties and the several opportunities of environmental applications. Water sensing properties of 1D titanate nanocrystals have also been studied recently.

The principle of humidity sensing is the change in the dielectric properties of the used material due to the varying water content. In this study water sensing mechanism of titanate nanostructures was investigated in the relative humidity range from 6% to 100%.

C15

EFFECT OF THE GRAIN SIZE OF CONDUCTIVE PARTICLES ON MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF Si_3N_4-ZrC CERAMICS COMPOSITES

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Ceramics composites are widely used as heaters. Generally they consist of an insulator layer (Si_3N_4, Al_2O_3, AlN etc.) and an active resistive layer (matrix insulator with conductive additives, such as ZrC, TaN, TiB_2, TiN, $MoSi_2$ etc.). In the work we have investigated Si_3N_4-ZrC ceramics because the silicon nitride ceramics are usually known as strongly refractory and enduring materials and addition of electroconductive particles, such as ZrC to the insulating Si_3N_4 matrix not only provides high electron conductivity but also toughen the material by mechanisms, including residual stresses and crack deflection [1].

The aim of this work was to study the influence of the size and morphology of ZrC grains on microstructure and electrical properties of hot-pressed Si_3N_4-ZrC composites.

We have used powder of $(Si_3N_4)_{0.93} + (Al_2O_3)_{0.07}$ as feedstock for insulator layers. Active resistive layers were obtained by the adding of 13.5 vol.% of ZrC with different grain size (d = 5–46 µm) to starting dielectric. All samples "insulator – conductor – insulator" were hot pressed in graphite furnace with the same conditions: mechanical pressure of 20 MPa, sintering time 20 min and temperature 1680°C.

It was found that the grain size had no influence on the void formation because the total porosity of all of the samples did not exceed 4 %.

Electrical resistance at room temperature rose with increasing of conductor grain size. It can be explained by the augmentation of grain boundaries contribution and reduction of contact area between the grains.

Analysis of temperature dependence of electrical conductivity and temperature coefficient of resistance (TCR) revealed that only if $d < 10$ µm electrical resistance linearly increases with the temperature across the examined range (20°C to 400–800°C) and its TCR is positive. It shows metallic conductivity. And if $d > 30$ µm, there appears an area with Arrhenius-low temperature dependence of conductivity and positive TCR in the middle of temperature range. It is caused by activation of carriers localized at grain boundaries. Activation temperature decreases with increasing of the grain size.

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C16

DETERMINATION OF THE COMPACT BONE MINERAL VOLUME FRACTION BY DIELECTRIC INVESTIGATION

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Compact Bone is a complex, highly organized and specialized connective tissue. The complex bone material can be simplified as a di-phase composite formed by an inorganic part, consisting from carbonated hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ layers and an organic part, manly consisting of collagen protein matrix [1].

The main objective of this work is to determine the value of volume filling factor of mineral phase from a compact bone tissue using Maxwell-Garnet and Bruggeman Effective Field Models [2]. We determined the room temperature dielectric properties of the compact bone and of the collagen components, while the dielectric constant of the HydroxyApatite was considered from literature data [3,4]. The volume filling fraction of the HydroxyApatite is better described by the Bruggeman model. Our results were confirmed by the mineral phase calculation from SEM microstructure (Figs. 1 and 2). The proposed method can be used to develop a non-invasive dielectric technique for in-vivo monitoring of the changes in the mineral composition of bones.

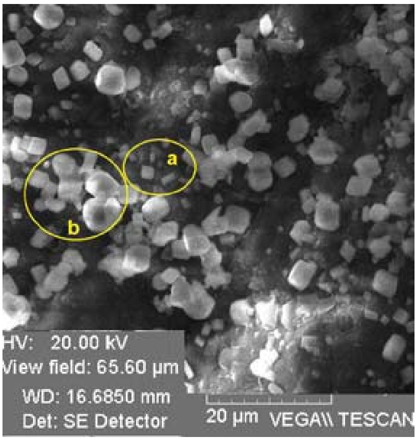

Figure 1. SEM micrograph of bone sample presents small crystals (in area market with a) and large crystals (in area market with b)

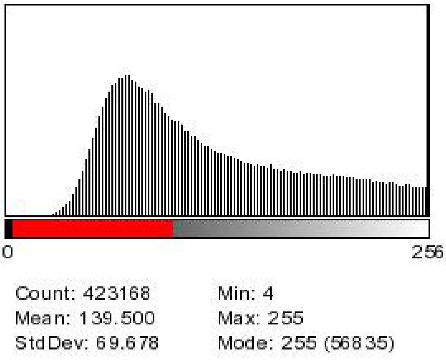

Figure 2. Histogram from SEM image (horizontal: pixels from 0 to 256; red color: collagen, gray color: mineral phase), vertical: pixel counts

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C17

MECHANISMS OF OPTICAL LIMITING IN A COANP SOLUTION DOPED WITH FULLERENES C_{70}: APPLICABILITY FOR THE OPTOELECTRONICS DEVICES

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Fullerenes and carbon nanotubes are widely studying due to their unique properties [1,2]. Carbon nanoobjects-doped materials can be used to limit a powerful laser radiation in the visible and near-infrared spectral ranges and to protect eyes and photosensitive equipments. In this paper we are discussing optical limiting properties of COANP solution doped with fullerenes C_{70} and carbon nanoobjects. There are several mechanisms explaining the properties: reverse saturable absorption (RSA), photoinduced change in the refractive index, Förster mechanism, complex formation, etc.

We have investigated the nonlinear-optical properties of a 1% solution of COANP in tetrachloretane sensitized with fullerenes C_{70} of 0.5 wt.%, 1.0 wt.%, 10 wt.% and 20 wt.%. Experiments have demonstrated the limiting of the laser irradiation with energy density from 0.1 J/cm^2 to 0.5 J/cm^2. We have obtained the maximum attenuation of the radiation with density energy of 0.35 J/cm^2 for the solution COANP-C_{70} with fullerene content of 20 wt.%. The energy value passed through solution has been decreased up to 18 times.

We are considering RSA and the complex formation as the mechanisms responsible for the optical limiting of COANP-C_{70} system. The RSA mechanism is based on a difference in cross section for excited and unexcited fullerene molecule. The cross section of singlet-triplet state in the fullerene is larger than one of the unexcited molecule. The population of excitation levels increases with laser energy so absorption increases. The intermolecular charge transfer complex with large cross section absorption can be formed in π-conjucted organic system doped with C_{70} due to the large electron affinity in fullerene (2.65 eV) [3]. This parameter for acceptor fragment in COANP is 0.45 eV. In comparison, some evidence of optical limiting properties of different materials doped with nanotubes has been done. The results can be used to protect optoelectronic devices including ceramics from high laser irradiation.

Acknowledgement: The results have been supported by RFBR grant #10-03-00916 and by Russian Federal Program, Project "Modulator with SEW" (2011).

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C18

EFFECT OF La_2O_3 ON STRUCTURE AND PROPERTIES OF HYDROXYAPATITE/GLASS BIOCOMPOSITES

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Composites based on hydroxyapatite (HA) and glass have been successfully used in medicine. Rare earth element, lanthanum, has the similar property to calcium. It was reported that osteoblasts, bone-forming cells, adhered and differentiated at earlier time points in response to the HA doped with La^{3+} than the HA doped with divalent cations [1].

In this study we investigated the effect of lanthanum oxide La_2O_3 on structure and properties of BHA/glass biocomposites. The BHA/glass composites were prepared via two-step sintering process according the method followed in the previous study [2]. La_2O_3 powder (5, 10 and 15 wt.%) was introduced into BHA/glass composites before the final sintering. The porous structure of composites was analyzed using a scanning electron microscope JEOL JSM 6490 LV. The density of materials was measured by displacement (Archimedes) method using toluene. The compression strength was measured by testing machine PSU-50. Evaluation of the solubility *in vitro* of composite samples was carried out by technique reported in our previous work [2]. In addition, the effect of La_2O_3 on volume shrinkage, porosity change and mass loss in the composite samples upon the secondary sintering was investigated.

It was established that mass loss of composites increase over incorporation and increasing La_2O_3 content from 0.3% up to 2.3%. Moreover, La_2O_3 influence on the volume change upon the sintering. The incorporation of 5% La_2O_3 leads to volume growth (31 %) and significant vitrification of composite samples. Volume growth of materials decrease with increasing of La_2O_3 content and 15% La_2O_3 leads to volume shrinkage (4.5 %). The volume shrinkage for non-doped BHA/glass composites is 10.2 [2]. An analogous dependence is observed for the porosity of composite with different La_2O_3 content that decrease with increasing of La_2O_3 content from 48 % down to 34 %. However, the porosity of non-doped BHA/glass composites is less than doped 1.5 times. The compression strength of composites correlates with porosity and decreases with La_2O_3 incorporation from 219 MPa down to 26 MPa.

Though, La_2O_3 addition three times increases the solubility of materials and stabilizes the pH of solution. The effect of La_2O_3 on the cell culture studies of doped HA/glass composites will be an interesting topic for future investigation.

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C19

SYNTHESIS OF BIOACTIVE CALCIUM PHOSPHATE COATINGS ON ZrO_2 (Y-TZP) CERAMICS FOR APPLICATIONS IN MEDICINE

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Zirconia implants are becoming increasingly important in the field of dental medicine because of their good mechanical properties, biocompatibility, and for aesthetic reasons. However, zirconia is bioinert and this can lead to poor fixation of the ceramic implant in the bone. A promising approach to circumvent this problem is to coat the implants with thin layers of calcium phosphates (Ca-P), which are known to be bioactive, i.e., they show a good bone-bonding ability. A promising method for the preparation of Ca-P coatings is biomimetic synthesis, which includes the immersion of the implant into a solution with a composition similar to human blood plasma and under physiological conditions. This method has several benefits, such as a good surface coverage of materials with complex shapes, as well as good control over the coating thickness, crystallinity and phase composition. However, the conventional biomimetic method has some drawbacks, connected with the long time of the synthesis, the complicated preparation of the solutions, low reproducibility and relatively low adhesion of the coatings to the substrate. The motivation for our study was to develop a simple biomimetic procedure that would enable fast and reproducible preparation of the Ca-P coatings with good adhesion to the substrate.

In our research work we have synthesized octacalcium phosphate (OCP) coatings on zirconia (Y-TZP) ceramics by utilizing a two-step biomimetic method. Firstly, cleaned substrates were immersed into a supersaturated Ca-P solution with a pH = 7.4. After 1 hour of soaking a thin nanocrystalline Ca-P layer was precipitated on the substrate. Secondly, substrates were transferred from the first solution to another Ca-P solution with pH = 7.0. After a short time of immersion in this solution, a thick coating consisting of large OCP lamellae was obtained. The thin Ca-P layer, formed in the first step, served as a seeding layer for the growth of the OCP crystals and allowed for the rapid deposition of a coating during the second step. The adhesive strength of the coating, measured with pull-off test, was 1.8 MPa. Some of the samples were subjected to a heat treatment at 800°C for 1 hour, resulting in a transformation of the OCP crystals to the β-TCP phase. Moreover, the adhesion of the coatings was strongly improved and reached the value of 29 MPa. The main benefits of the method are a relatively quick synthesis, simplicity and a good reproducibility. In addition, β-TCP coatings with firm attachment to the substrate can be obtained by firing the samples at 800°C.

C20

CROSS-SECTION HARDNESS MAPING OF HUMAN TEETH

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Keywords: enamel, nanoindentation, AFM

As the hardest and one of the most durable load-bearing tissues of the body, enamel has attracted considerable interest from both material scientists and clinical practitioners due to its excellent mechanical properties. Instrumented indentation was used with the aim to investigate the hardness, elastic modulus and indentation fatigue of enamel (E), dentin (D) and dentin-enamel juction (DEJ) of human teeth.

The used samples were fresh extracted human teeth by orthodnontic way. Before the test these were cut to halfs, polish and stored in salt solution to protect dehydratation.

Single and Continuous Multi Cycle indentation was applied using instrumented Nano Hardness Tester with Berkovich diamond indenter. The indents have been applied to the area of enamel, dentin and dentin-enamel juction. The indentation hardness, H_{IT}, and elastic modulus, E_{IT}, values were calculated. The influence of the applied load on the hardness was investigated, too. Indentation fatigue experiments were performed up to the maximum loads of 200 mN, then unloaded to 50% and repeatedly re-loaded 25 times with a time to maximum load/unloadload 1 second. The indents have been studied using Atomic Force Microscopy.

The nanohardness and elastic modulus of the enamel from top to bottom region are significantly different. Both nanohardness and elastic modulus gradually decreased from enamel to dentin (Fig. 1). The indentation size effect was significantly higher in enamel in comparison to dentin.

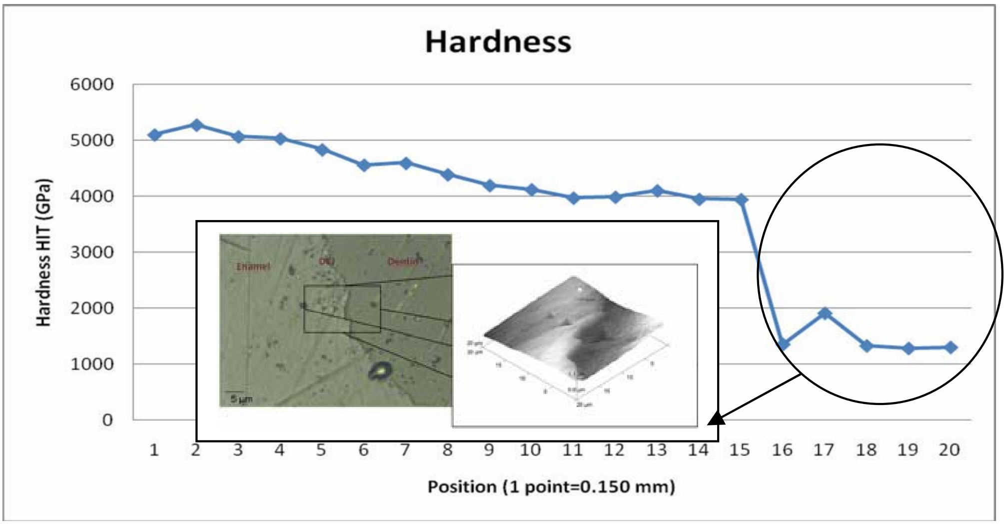

Figure 1. Nanoindentation mapping of tooth - incisor.

C21

ANTIMICROBIAL PROPERTIES OF CERAMIC BASED NANOCOMPOSITE BIOMATERIALS

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Keywords: Ca/Co-HAp, HAp/PLGA, S. aureus, E. coli, antimicrobial activity

Introduction: The loss of bone tissue in oral implantology presents a great problem in treatment of these patients. Synthetic biomaterials, with properties that resemble natural bone, are used in order to successfully treat these bone defects. Particular attention is paid to the possibility of infection after their implementation and therefore the antimicrobial hydroxyapatite-based materials are very attractive for use in implantology.

Aim of study: The objective of this paper is to examine antimicrobial activity of the cobalt-substituted calcium hydroxyapatite nanopowders (Ca/Co-HAp) and hydroxyapatite/ poli-lactide-coglycolide (HAp/PLGA).

Material and methods: The antimicrobial activity of samples was tested on two strains of bacteria: E. coli and S. aureus, by means of disc diffusion method on Muller-Hinton agar. The inoculums of microorganisms were prepared from fresh overnight broth cultures that were incubated at 37°C. The results of antimicrobial tests were obtained by measuring the width of the bacterial cells inhibition zone, which were exposed to the samples.

Results: Biomaterial HAp/PLGA did not show any antibacterial effect on E. coli and S. aureus. The number of the grown bacteria colonies close to biomaterial Ca/Co-HAp was equal to zero. It was noted that the inhibition zone of the bacterial cells S. aureus around the sample of the Ca/Co-HAp, was a lot bigger compared to the inhibition zone of bacterial cells E. coli around the sample of the mentioned biomaterial.

Conclusion: Hap/PLGA does not show any signs of antimicrobial activity on E. coli and S. aureus. Ca/Co-HAp has stronger antimicrobial activity on S. aureus compared to E. coli. It can be concluded that the prepared cobalt-substituted calcium hydroxyapatite nanopowders, show satisfactory antimicrobial activity and may be used as an antimicrobial material for different purposes, most importantly for implants in bone defect reconstruction.

TRADITIONAL CERAMICS

T1

AGEING OF LOW-FIRING PREHISTORIC CERAMICS IN HYDROTHERMAL CONDITIONS

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Ceramics is a material, which is possible to subject under certain condition to the rehydration process of unstable non-crystalline products arising during firing below 1000°C. This work deals with the observation of a hydrothermal ageing of the archaeological ceramics (moon-shaped idol) and of the calcined samples of raw materials. It was found the kaolinite was present only in the border part of the low firing archaeological object having trapezoidal shape (50 cm × 10 cm).

The goal of this work was to determine, whether the kaolinite in archaeological ceramics comes from the raw material or it could originate as a product of possible rehydration during the object storage under the ground. Analyses were performed on samples from central and border part as well as on samples prepared from model compounds. The samples were calcined at 600°C with time endurance 10–120 minutes. The samples after the calcination were subjected to the hydrothermal ageing by modified French standard (NF P 13-302). The XRF, XRD, IR and DTA analyses were used for the product characterization before and after hydrothermal ageing. Kaolinite, which was detected in the original sample, was not identified after the calcination in the archaeological ceramics. The model samples contained different amounts of the kaolinite and of unstable non-crystalline products after the calcination. The resulting content of kaolinite depended on the time endurance during the calcination. No kaolinite, was identified neither in the sample of the archaeological ceramic nor in the model samples after the normalized hydrothermal ageing. It was proved by IR spectroscopy the presence of kaolinite occurred at higher temperature of 230°C. It was shown, that the time and the temperature of the hydrothermal ageing had effect to the reverse rehydration of unstable metakaolinite to the structures close to the kaolinite. The highest ability of rehydration had both types of samples during the hydrothermal ageing at 10 h 230°C. The content of the kaolinite in low-firing ceramics (500–700°C) is given not only by the temperature but also by the time of endurance during the calcination. The evaluation of the measured data showed that moon-shaped idol had been burned irregularly. We can assume the firing of the ceramic body occurred probably due to the exposition near to an open fireplace where was placed for a longtime. Regarding obtained information we can suppose kaolinite identified in the prehistoric ceramic body was not a product of the rehydration process, but originated in raw materials.

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T2

IMPROVEMENT OF RESISTANCE AT WEATHERING OF GLASSES WHICH REPRODUCED HISTORICAL ITEMS BY PROTECTIVE COATING

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The ancient and middle age historical glasses can be classified as lead containing glasses or soda lime silicate glasses. In the last years the interest to maintain objects in good conditions has increased. With the aim to preserve historical glasses against future weathering, several reference glasses that reproduce the composition of the ancient or middle age ones were designed and prepared. The glass samples were prepared by traditional route. The melting was performed at 1300°C for 2 h and after that the glasses were thermally treated at 300°C for 1 h. The structure of the obtained glasses was characterized by XRD and FT-IR spectroscopy. A part of the characterized glasses were coated with sol-gel hybrid films by deep coating. The behavior of the materials in different acid and aqueous media were studied. The chemical durability in water determined by ISO 719/ 1996 test on the initial and coated samples has shown the improvement of chemical stability by coating. The morphology of initial glasses and coated glasses before and after chemical stability tests were studies with scanning electron microscopy. The improvement of resistance at aqueous and acid media was shown.

T3

DIFFERENT METHODS IN THE EXAMINATION OF THE MOVABLE CULTURAL HERITAGE IN THE ACTIVITIES OF THE OF THE CONSERVATION DEPARTMENT OF THE GALLERY OF THE MATICA SRPSKA

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Keywords: conservation, restoration, examination of cultural heritage

Conservation department of the Gallery of the Matica srpska is founded in 1960 and is actively engaged in the protection of movable cultural heritage from the collection of the Gallery of the Matica srpska, as well as participates in different conservation projects as a partner with other institutions. The work is prevalently oriented towards the conservation of easel paintings on panel and canvas support and works of art on paper,

covering the period from 16th century and traditional post-byzantine art to the modern art of the 20th century.

The conservation work requires multidisciplinary approach in order to have good results. In the last ten years, the collaboration with chemists, technologists and physics in the field of characterization of materials, artistic technologies and degradation processes has been intensified, leading to significant results.

This presentation will show the practical examples of non destructive analysis using imaging methods in visible, IR and UV range, as well as X-ray examination. In the field of micro-destructive examination on samples, the results of optical microscopy for stratigraphic examination, as well as analysis of pigment using FTIR and Raman spectrometry will be presented.

T4

CHARACTERIZATION AND EVOLUTION OF THE SYSTEM CERAMIC BODY - GLAZE

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Glazes and engobes have not only aesthetic function but mainly improve ceramic body stability and its weathering resistance. The glaze closes the opened pores on the surface of the ceramic body and so increases the resistance of the ceramic material. The lack of compatibility between the glaze surface and the ceramic substrate is one of the major causes of defect. Selection of the suitable glaze should be in accordance with the properties of whole system.

The mass transfer of components between body and glaze is the important process during the firing of glazed products. In addition, chemical reactions at the interface body and glaze take place. The glaze melt penetrates into the pores of the body, partial dissolution of silica takes place resulting in enrichment of the melt by both SiO_2 and Al_2O_3 as well as by alkalis. When the firing process is long enough, the interlayer is formed between the glaze and the body having the thickness 10–50 microns. This interlayer is different in the chemical composition and in the content of crystalline phases from the body and glaze. The stress resulting from the different values of the coefficient of linear thermal expansion (α) of the glaze and the body may be partially compensated. Two situations can occure during the equilibrium state. Firstly the linear thermal expansion coefficient (α) of the glaze is bigger than α of the body and so causes the compressive stress in the glaze layer. On the other hand when α of the glaze is smaller then α of the body the tension stress occures in the glaze. No stress arrises neither in the glaze non in the body only when the thermal expansion coefficients (α) are the same.

The aim of this work was to introduce methods used for testing of glazes and to demonstrate laboratory tests on different types of glazes for two types of ceramic porous body.

Acknowledgement: This work was supported by Grant MSM 6046137302 and by the programme Specific University Research (MSMT No. 21/2011).

T5

INVESTIGATION OF NEW OPAQUE GLAZES FOR FLOOR TILES

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Keywords: floor tile, opaque glazes, reducing cost

Opaque, white floor tile glazes are the most preferable one in terms of the appearance of the product by the consumers. These glazes contain high amount of zirconium oxide in their recipies which increase the cost of products. Aim of present study was cost reduction of this expensive component by making alternative, low cost frit compositions. To achieve this, new recipes were prepared with in the K_2O-MgO-CaO-ZnO-B_2O_3-Al_2O_3-SiO_2 system to obtain alternative glass- ceramic phase(s). Industrially fired specimens pointed out promising surface properties. To identify the phases formed on the glaze surface, X-ray diffraction equipment was employed. Additionally, a scanning electron microscope was also used to get information about the shape, size and distribution of the obtained phase(s). Sintering behaviour of the glazes were studies by using a hot stage microscope. Consequently, decreasing zircon oxide from glazes give a chance to produce approximately 20 % cheaper floor tile products which have similar appearance and surface properties with commercial ones.

T6

ENCAPSULATING SLUDGES IN BRICK STRUCTURE

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Taking into account the sustainable development requirements in the production of heavy clay bricks, the possibility of using waste in composites based on clay has been recently increasingly explored. Traditional building materials on the basis of clay raw materials allow combining with different wastes or secondary materials without significant modification of the process of production or use of such products. The introduction of industrial waste in brick products becomes common practice.

The purpose of this study is to test utilization possibilities of industrial sludges as additives in the production of heavy clay bricks. These arise as waste sludges during the neutralization of waste water in the process of hot dip galvanizing.

Research included the study of the composition, structure and properties of laboratory samples prepared from pure heavy clay and a mixture of clay and waste sludge. Change in the structure and properties of samples are determined, depending on the composition of the sludge, their share in the mixture and firing temperature. During the experimental work the following methods of characterization were applied: X-ray analysis (XRD), simultaneous differential and thermal analysis (DTA/TG), scanning electron microscopy (SEM) and mercury porosimetry. The chemical composition was determined by energy-dispersive X-ray fluorescence spectrometry (EDXRF) and heavy metals content after leaching in distilled water is measured using inductively coupled plasma (ICP). Physical and mechanical properties of samples were also observed, such as water absorption, porosity and pore distribution, volume mass and compressive strength.

The Second Early Stage Researchers Workshop
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INVITED TALKS
FERROICS & MULTIFERROICS

IM1

SIZE AND INTERFACE EFFECTS IN NANOSTRUCTURED DENSE FERROELECTRIC CERAMICS

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The present endeavour in microelectronic industry is to reduce the layer thickness and the grain size in submicron range of $BaTiO_3$-based dielectric materials for applications in passive components like multilayer ceramic capacitors and embedded capacitance in printed circuit boards, in order to increase the volume capacitance. The maximum dielectric constant of $BaTiO_3$ ceramics was observed for a grain size around 1 μm. The further reduction of grain size below this limit results in a diminishing of ferroelectric polarisation, permittivity and of other material constants and gives rise to a fundamental question about the stability of the ferroelectric state below a ceratin size limit, *i.e.* about the existence of the *critical size* of ferroelectricity [1]. Preparation of dense, bulk nanocrystalline ceramics requires ultra-fine, non-agglomerated powders with a narrow particle size distribution and a suitable densification technique (*e.g.* spark plasma sintering) to minimize the grain growth [1].

When reducing grain sizes at nanoscale, an important role is given by the interfaces (grain boundaries) and by defects and it is important to understand the interplay of the intrinsic/extrinsic effects on the macroscopic properties. For applications as high volume capacity, it is needed to reduce as much as possible the grain size in ceramics by maintaining as high as possible the dielectric permittivity to above 1000, while for tunability applications the permittivity should be lowered at a few hundreds, while keeping a high variation of permittivity with the applied field [2]. Therefore, the grain size reduction is an additional factor besides the material choice to tune these properties towards the desired range needed for specific applications. This concept is illustrated with a few results obtained for $BaTiO_3$ and $BaZr,TiO_3$ nanostructured dense ceramics.

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IM2

INTEGRATION OF FERROELECTRIC MATERIALS: A LONG-TERM VIEW

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From the initial idea to the actual use of ferroelectric materials the time scale is several decades and the evolution is never continuous. In this talk, I will illustrate this long term trend, taking two examples: ferroelectric thin films and piezoelectric ceramics.

In the former case, it is right after the discovery of transistors (which always include a dielectric film), that researchers in academic laboratories thought of using the permanent polarization to increase the functionalities of such devices [1]. While the modeling of such coupling between the semi-conductor band structure and the ferroelectric polarization was improving along the years, no decisive step further was taken until the first ferroelectric hysteresis loop was recorded at the end of the eighties [2]. This discovery boosted the academic and industry research. Some of the present remaining issues will be given focusing on agile devices and multiferroic multilayers.

In the second case, the story also started in the middle of twentieth century, when perovskites ceramics were shown to display piezoelectric efficiency much higher than well known Quartz crystals. While the solid solution $PbTiO_3$-$PbZrO_3$ (PZT) became soon the most used material [3], the basic understanding of its large piezoelectric coefficients is still a matter of debate. Whatever the tendency, the close coexistence of several symmetries in a restricted compositional range seems to be a necessary condition. This is what is driving the nowadays research effort towards the replacement of lead in piezoceramics.

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IM3

SOLUTION BASED SYNTHESIS OF ELECTROCERAMIC NANOSTRUCTURED MATERIALS

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Encouraged by the need for miniaturization, enhanced functionality, reduced cost as well as ecological aspects, the research for efficient and improved synthesis methods for nanostructured electroceramics is currently a very active field in academia as well as in industry. Strong control over the composition, phase, morphology and nanostructure is required for optimal performance, and in the case of thin films requirements with respect to texture or compatibility with the underlaying material (substrate or other functional layers) impose further constraints. Also, fundamental questions related to composition-morphology-property relations at the nanoscale need to be addressed in order to investigate new materials for new functionalities. For this, it is required that nanostructured oxides are synthesized with the desired purity and in the specific appearance in which their functional properties need to be established. We will present synthesis strategies for functional metal oxides by means of soft (mostly water based) solution based routes as an alternative to vacuum deposition or ceramic methods. Different approaches lead to different forms of the metal oxides obtained: powders, nanospheres, nanorods, films with thicknesses from a few 100 nm down to a few nm, substrate supported nanoislands either disordered or in ordered patterns and conformal coatings. Current challenges including nanostructuring, screening of new unexplored compositions, using non-toxic solvents and lowering thermal budget will be discussed based on a few case studies.

IM4

BROADBAND DIELECTRIC SPECTROSCOPY OF FERROELECTRICS AND RELATED MATERIALS

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The dielectric response of materials provides information about the orientational adjustment of dipoles and the translational adjustment of mobile charges present in a dielectric medium in response to an applied electric field. Microwave dielectric spectroscopy of ferroelectrics and related materials enables the independent determination of the dielectric permittivity and loss in the dispersion region, as well as the parameters of the soft modes related to phase transitions.

Besides scientific purposes, microwave dielectric measurements are of increasing importance in telecommunications related applications and the design of microwave circuit components. These applications include imaging radars, guidance systems, surveillance and secure communications. The magnetic properties are also of crucial importance. Dielectric and magnetic parameters fully characterize the manner in which electromagnetic waves propagate within the medium. The difficulties of making measurements on a wide range of materials over a wide frequency (and temperature) range have led to the development of various direct and indirect methods.

At microwave frequencies, the direct single-frequency methods were enriched in the recent years with more convenient broad band frequency domain dielectric spectroscopy (FDDS), time – domain spectroscopy (TDS), Fourier transform spectroscopy (FTS).

Computer controlled spectrometers are now the norm in dielectric spectroscopy. Computers allow the computation of electromagnetic fields in entirely new measurement geometries and the use of numerical analysis in the direct measurement process. The use of such spectrometers is now one of the most fruitful factors in new approaches to microwave dielectric spectroscopy. Each investigator employs the method adequate for the size and shape of a sample. The most important problem now is the rigorous mathematical solution of the microwave interaction with the samples in various geometries.

Although there is now complete overlap and coverage of the radio frequency to the infrared band, the different experimental methods based on coaxial, waveguide, resonator and free-space technique is still divided and will be presented.

After the measurement next step is the evaluation of the obtained results. Different models are used starting from Debye model through Cole-Cole, Havriliak-Negami, Davidson-Cole and so on. Different models will be presented and compared in terms of the distribution of the relaxation times.

FERROICS & MULTIFERROICS

M1

DIELECTRIC AND NONLINEAR PROPERTIES OF $BaTi_{1-x}Sn_xO_3$ CERAMICS PREPARED BY SOLID STATE METHOD

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A wide range of technological applications such as: piezoelectric actuators and electro-optic modulators uses the ferroelectric materials. $BaTi_{1-x}Sn_xO_3$ is a valuable candidate for high permittivity applications. The properties of $BaTi_{1-x}Sn_xO_3$ are expected to be tuned by the Sn composition (x) and by controlling their microstructural characteristics (grain size, secondary phases, porosity level, core–shell structures).

A pure single phase of barium stannate titanate ($BaTi_{1-x}Sn_xO_3$ with composition x = 0; 0.05; 0.10; 0.15 and 0.20) ceramics were prepared by solid state procedure and sintered at 1400°C for 4 h. With Sn addition (x), the tetragonal distortion c/a decreases and the symmetry changes from tetragonal (specific for pure $BaTiO_3$) to cubic. Hence, a ferroelectric-relaxor crossover is induced when increasing the Sn concentration.

The dielectric and ferroelectric properties of the $Ba(Ti,Sn)O_3$ ceramics depend on microstructural characteristics (grain size, secondary phases, porosity level, etc.). Dielectric constant of $BaTi_{1-x}Sn_xO_3$ is field-dependent: relative tunabilities between 0.08 – 0.85 were found for all the compositions at the applied field of E = 12 kV/cm (Fig. 1).

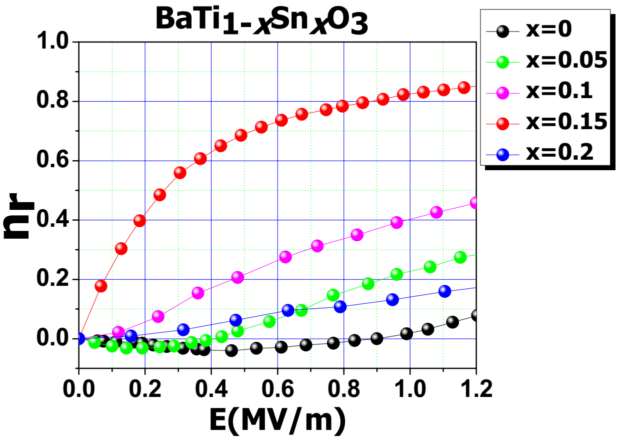

Figure 1. Relative tunabilities vs. the applied field.

Acknowledgments: Financial support from Grant POSDRU/88/1.5/S/47646 (ESF Romania) and COST MP0904 Action is highly acknowledged.

M2

TRANSPARENT OXIDE THIN FILMS FROM SOLUTIONS PROCESSED AT LOW TEMPERATURES

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Transparent electronics is a new and fast developing industry. The interest in this field has been commercially and scientifically accelerated during the past years. Its aim is the realization of fully transparent devices, which could be used in applications such as flat panel displays, thin-film transistors, solar cells, organic light emitting diodes (OLEDs), etc. This requires low cost deposition of thin films with suitable properties onto substrates requiring low temperature processing, such as glass and recently even on flexible substrates.

Transparent electronic devices employ both dielectrics and transparent conductive oxides. Among dielectric materials, tantalum oxide is considered one of the most promising candidates, due to its high refractive index, high dielectric constant and good thermal and chemical stability [1].

In this work, Ta_2O_5-based thin films were deposited by Chemical Solution Deposition (CSD). After deposition the samples were heatedin the range of temperature: 300°C–450°C. In this study pure Ta_2O_5 and a ternary composition Ta_2O_5-Al_2O_3-SiO_2 with a Ta : Al : Si = 8 : 1 : 1 atomic ratio were investigated.

The high-k dielectric Ta_2O_5 based thin films processed at low temperatures were characterized from the structural, optical and electrical point of view. Regardless their composition and the substrate used, the films are amorphous and show very flat surfaces. All samples exhibit high optical transparency in the visible range. The dielectric permittivity of the films ranges from about 20 to about 28 depending on the thermal budget, values which are comparable to the ones reported for amorphous Ta_2O_5 films [2].

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M3

CONTROL OF THE MICROWAVE DIELECTRIC PROPERTIES OF $Ca_{(1-x)}Nd_{2x/3}TiO_3$

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The structures and microwave dielectric properties of $Ca_{(1-x)}Nd_{2x/3}TiO_3$ has been investigated in detail using electron microscopy, Raman spectroscopy and X-ray diffraction. Room temperature studies revealed an unreported phase transition from orthorhombic Pbnm to monoclinic P2/m. Scanning electron microscopy revealed the presence of twin domains with a series of complex morphologies. Further investigation using high temperature Raman spectroscopy and X-ray diffraction revealed that the dominant domain morphology is controlled by the phase transition temperatures on cooling to ambient conditions. It is proposed that control of twin domain morphology can have an influence on the microwave dielectric properties of ceramics, especially the $Q \times f$ factor which is shown to vary in a grossly non-systematic way in the $Ca_{(1-x)}Nd_{2x/3}TiO_3$ system.

M4

PROCESSING OF BULK $Sr_{0,3}Ba_{0,7}TiO_3$ CERAMIC

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In the last decades the importance of ferroelectric (energetically independent FeRAM memories, capacitors, sensors, etc.) and ferromagnetic (artificial magnets, hard drives, etc.) materials in electronic applications increased significantly. Barium strontium titanate $Ba_xSr_{1-x}TiO_3$ solid solution is a classical prototypic ferroelectric system [1,2].

The main objective of this research was the synthesis of phase-pure $Sr_{0.3}Ba_{0.7}TiO_3$ ceramic powder with nanometric particle size by synthesis in liquid media. Titanium butoxide, barium and strontium chloride solutions were used as precursors for this type of reaction in alkaline environment. After drying and calcination at 400°C the product was single phase $BaSrTiO_3$ solid solution.

Pressureless sintering of isostatically pressed bodies in air was used to reach bulk ceramic. Phase pure bulk samples reach relative densities higher than 94% of theoretical

density at 1250°C for 1 hour. Higher relative density is expected in case of samples prepared by some of advanced sintering technique for example by spark plasma sintering.

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M5

DIELECTRIC SPECTROSCOPY OF NANOGRAIN PSN CERAMICS

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Ferroelectric materials are of high interest for both researchers and engineers due to their remarkable properties – high dielectric permittivity, piezoelectric, pyroelectric effects, which makes them great targets of fundamental research for scientists and materials of choice for a variety of devices [1]. In particular the so-called "relaxor" compounds exhibit a strong, anomalous dependency of their dielectric response with frequency, making them fundamentally intriguing and with lot of potential technological applications. However, there is a strong need for integration of these materials at the nanoscale, in the form of thin films or in the form of nanostructured ceramics. Therefore one may wonder what happens to properties of low-dimensional relaxors, and how their sizes affect their ground states. In particular it is of uttermost importance to separate size and internal strain effects, what we have performed in lead based perovskite $PbSc_{1/2}Nb_{1/2}O_3$ (PSN) ceramics [2].

In the present study 6 samples of disordered PSN with mean grain sizes of 20, 40, 60, 70, 80 and 85 nm were studied. Dielectric measurements were performed in 50–500 K temperature and 100 Hz – 36 GHz frequency range. During every measurement samples were heated up to 500 K to achieve similar conditions for all experiments and avoid memory effects.

Size has clear influence on dielectric properties of the ceramics. The most obvious being absence of a spontaneous phase transition typical for bulk ceramics [3]. Futhermore, dielectric permittivity diminishes with size in a way, which cannot be attributed to grain-boundary dilution.

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M6

BARIUM TITANATE PROPERTIES ENHANCED BY ATTRITION MILLING

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Barium titanate powder was prepared by soft chemical process from polymeric precursors (modified Pechini process). The obtained BT powder was nanosized with primary particles ~ 74 nm, but factor of agglomeration (F_{agg}) pointed on existence of agglomerates ~ 6 μm. It is well known that agglomerates could influence on setback of materials structure and properties. In order to de-agglomerate nanopowder and to improve BT properties, attrition milling was performed.

Barium titanate powder was treated in attrition mill with zirconia media for 1h in 2% polyacrylic acid. Milling induced reduction of agglomerates in size and number. Characterization of both starting (BT) and milled (BTA) powders was performed. The comparison of obtained results showed enhancement of powders properties generated by attrition milling. To investigate the effect of milling on electrical properties of ceramics, both BT powders were uniaxially pressed and sintered at 1300°C for 8 h in air. The density of BTA ceramics was 95 % of theoretical value and 90 % of BT. Temperature dependence of relative permittivity showed three structural transitions characteristic for ferroelectric BT ceramics. The temperature transition from ferroelectric to paraelectric was found to be at 120°C for BT and 122°C for BTA. Dielectric constant value was around 6700 for BTA which is much higher value in comparison with non-treated BT where permittivity was 1340. Dielectric losses were below 0.03 for both BT ceramics.

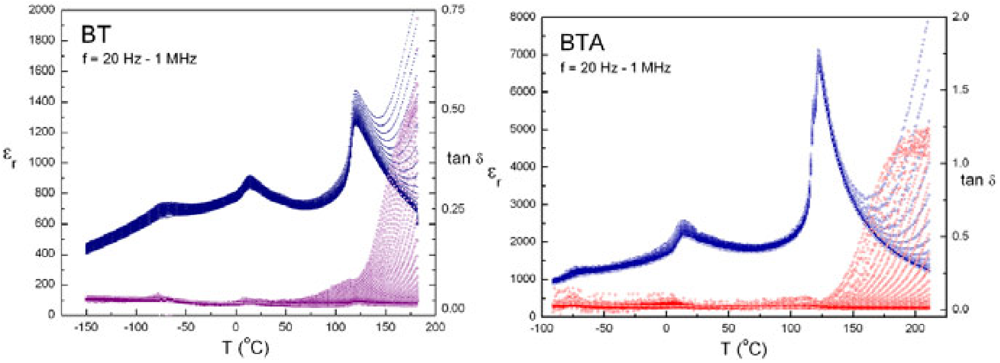

Figure 1 Dielectric properties of BT and BTA ceramics

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M7

FUNCTIONAL PROPERTIES OF PZT-NiFe$_2$O$_4$ MAGNETOELECTRIC CERAMIC COMPOSITES DESCRIBED BY EFFECTIVE FIELD MODELS

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Magnetoelectric composites of xNiFe$_2$O$_4$-(1-x)Pb$_{0.988}$(Zr$_{0.52}$Ti$_{0.48}$)$_{0.976}$Nb$_{0.024}$O$_3$ (NFO-PZTNb) with x = 2, 5, 10, 20, 30% were prepared by citrate-nitrate combustion using PZTNb-based template powders. To ensure a better connectivity of dissimilar phases, chemical methods for preparation of *in-situ* composites, followed by adequate sintering procedure was employed.

The dielectric constant of the composites decreases with the increasing the addition of x, as a consequence of the sum property. The dielectric responses show a Debye relaxation in the range of 10^2–10^4 Hz and a Maxwell-Wagner relaxation for frequencies below 10 Hz and the corresponding maximum is shifted with increasing of ferrite content to higher frequency. The magnetic properties were investigated. The initial permeability increased with increasing NFO content, which indicates that the magnetization ability became higher. The dielectric and magnetic properties of the individual phases of Ni ferrite and PZTNb, their relative fractional volumes and the shapes of the inclusions affect the macroscopic properties of the composite.

The dielectric and magnetic data obtained experimentally were compared with the calculated results in the frame of Maxwell–Garnett (corresponding to small filling concentration of isolated spherical inclusions embedded into continuous matrix) and Bruggeman approximations [1,2]. The Bruggeman formula gives a good approximation for all the filling concentrations and considers the properties of matrix or host the same as the effective field medium itself and predict a percolation value of 1/3 volume filling fraction [3]. With addition of Ni ferrite magnetic phase in the neighborhood of the percolation threshold, a higher dielectric constant is obtained by increasing the conductivity of the ferrite phase. The conductivity of ferrites is tunable by adjusting the value of filling factor of spinel structure. Taking into consideration the dielectric and magnetic properties of the phases, the volume fraction inclusions, geometrical shapes and arrangements, we investigated the dielectric and magnetic properties of the composites as a sum property of the phases. The effective field models give just a rough approximation of the composite property. The obtained differences and the usefulness of a model for this composite microstructure are discussed in the paper.

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M8

P-TYPE $Na_xCo_2O_4$-BASED THERMOELECTRIC CERAMICS FOR ENERGY GENERATION FROM WASTE HEAT

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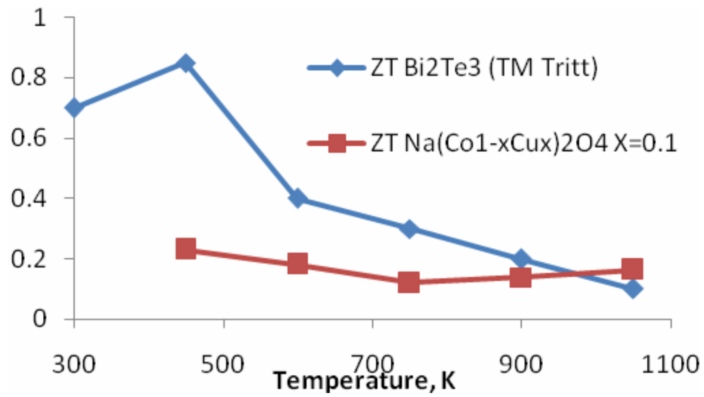
A large amount of energy is wasted as unused heat whenever fossil fuels are burnt, such as in coal power plants or in internal combustion engines in automobiles. Due to diminishing fossil fuel reserves and an increasing desire for green technology to help reduce harmful greenhouse gases into the atmosphere, thermoelectric materials have become a highly researched topic. Thermoelectric devices consisting of both p-type and n-type semiconductor components can be used to scavenge energy from waste heat and convert it into useful electricity. Currently the highest performing thermoelectric materials are based on metallic semiconducting alloys, such as n-type Bismuth Telluride, however these are unstable at temperatures experienced in environments such as found in automobile exhausts, therefore research has intensified in the metal oxide thermoelectric materials based on the sodium ($Na_xCo_2O_4$) and calcium ($Ca_3Co_4O_9$) cobaltites. The key property of a thermoelectric is the Thermoelectric Figure of Merit (Z), a goal of this research is to investigate ways of maximising this value. Z = Thermoelectric Figure of Merit, S = Seebeck Coefficient, k = Thermal Conductivity, ρ = Electrical Resistivity

To maximise Z, thermal conductivity and electrical resistivity need to be minimised while maximising the Seebeck coefficient. Density plays an important role in these properties, so manipulation of the density of the material has been an important aspect of this research. The Sodium Cobaltite system was explored through doping with Copper on the Cobalt site. This included using pseudo-Hot Pressing in a standard laboratory furnace in an atmosphere of air with a pressure of ~60 kPa which used to produce high quality dense materials. Pseudo-HP also gave the unexpected benefit of requiring a lower sintering temperature. The ZT for $Na(Co_{0.9}Cu_{0.1})_2O_4$ at temperatures of ~ 1000 K and above was predicted to match traditional TEG materials such as Bi_2Te_3.



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M9

INK-JET PRINTING ON FLEXIBLE SUBSTRATES AND SENSORS FABRICATION

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Ink jet printing machine enables direct deposition of fluids (organic or inorganic inks) on flexible substrates in order to offer easier, faster and less expensive product and material development for electronics, optics, life science and medicine. Electronic components printed on flexible substrate offer new possibilities and open new application areas.

In this paper, we describe technique how to deposit silver conductive ink in different shape (geometry) on Kapton film in order to obtain capacitor or inductor in the plane and after that to roll them. Rolled capacitor or inductor can be used as capacitive sensors for detecting various chemicals or as inductive position sensors. In order to determine optimal fabrication parameters, we have also investigated different conductive line width and printing resolution.

M10

TTB CRYSTAL GROWTH BY THE VERTICAL OPTICAL FLOATING ZONE METHOD: OPTIMIZATION OF ROD SINTERING AND GROWTH PARAMETERS

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The tetragonal tungsten bronze structure (TTB) has been first reported in 1949, by [1]. They observed that such materials have both ferroelectric and ferromagnetic properties at room temperature. This recently came back through the investigation of $Ba_2LnFeNb_4O_{15}$ ceramics and crystals, which revealed their composite multiferroic nature [2].

Following the recent studies on ceramics from this family [3], we investigate crystal growth by 2 different methods: the flux and the optical vertical floating zone. This last technique, carried on by a double-ellipsoid image furnace, has several benefits like checking the melt congruency, getting centimeter single-crystals and short-time experiments. Moreover, this allows growing high melt temperature crystals (~1500°C). By using this technique, we are heading to grow good single-crystal sections to investigate more precisely the structure by XRD and neutron diffraction and carry on the dielectric and magnetic measurements. Nevertheless, the floating zone method depends on several parameters, like the growth speed, the rotation rate, the atmosphere [4]. We first investigated the synthesis and sintering effect on the feed rod used for the growth. Indeed, using lower temperature and shorter sintering time always lead to a better growth and a well-faceted crystal. We have then focused on the growth atmosphere, in which different oxygen pressure were tested.

Figure 1. TTB lanthanum rod obtained by the floating zone method

The first results using lanthanum and neodymium confirm the appearance of secondary phases directly linked to the lanthanide inserted, including the ferromagnetic iron-rich phase, and so the composite state.

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M11

COBALT NANOSTRUCTURES BY ELECTROLESS REDUCTION: THE ROLE OF SURFACTANTS AND COMPLEXING ANIONS

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In the last few years, the synthesis of metal nanoparticles or nanostructures by wet-chemical reduction processes assumed a growing interest [1-4]. In particular, electroless methods prove to be highly efficient as they allow to obtain metal nanoparticles with narrow distributions and high stability in time. However, such reduction processes often require drastic thermal conditions and/or toxic solvents as embedding media that actually limit their use on a scale higher than the laboratory one.

To overcome these drawbacks, a cementation technique may represent a valid strategy for the preparation of metal nanostructures with easily reproducible operating conditions and environmentally sound reagents.

In this context, we present a technique aiming at obtaining cobalt nanostructures by reduction of a cobalt salt in mixed solvents using zinc spheres as sacrificial element with proper complexing anions and different surfactants at temperatures below 100°C to tune the shape, aggregation and distribution of metal nanoparticles. We show that the synergetic choice of a complexing molecule, the surfactant and their relevant concentrations are the most sensitive parameters governing the process. In particular, the carbonyl functional group in the complexing anion proves to be highly efficient in cobalt cementation and this result is in good agreement with other studies in literature concerning electroless thermal reduction of cobalt salts [5].

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M12

DC-ELECTRIC-FIELD DEPENDENCE OF DIELECTRIC CONSTANT IN FERROELECTRIC SYSTEMS

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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 paper the theoretical models that described dc electric field dependence are reviewed and the limits of their applicability are determinated. In case of a dielectric with a single polarisation mechanism the nonlinearity can be described by the Landau-Ginzburg-Devonshire theory and its' approximations.

The Johnson relation is a reasonable approximation for describing $\varepsilon(E)$ for the system is in its ferroelectric state [1]. By using calculation curves and experimental data it is found the limit of LGD theory for the ferroelectric systems.

Many ferroelectric systems exhibit more than one polarization mechanism contributing to the total dielectric constant. The dielectric response for such dielectrics can be described by the Johnson model completed with a Langevin term that describes "extrinsic" contribution. The "multipolar mechanism model" was used for explaining the nonlinear ε(E) dependences in case of ferroelectrics systems in paraelectric state and for relaxors [2].

The electric field dependence in nanostructured ceramics can be explained by considering them as a composite formed by ferroelectric grains whose non-linearity is reducing and by low-permittivity non-ferroelectric grain boundaries whose volume fraction increases, when reducing grain size. The grain size effect on the $\varepsilon(E)$ dependence in ferroelectric systems can be predicted using the effective field model and Johnson approach [3].

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M13

AQUEOUS CHEMICAL SOLUTION DEPOSITION OF $LuFeO_3$ THIN FILMS IN VIEW OF ENERGY STORAGE APPLICATIONS

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In recent years, research for energy storage applications has attracted a lot of attention. The miniaturization of devices and the need of high energy density systems are leading to the exploration of novel materials. These are indispensable to overcome the challenge of storing electrical energy in a more efficient way. For the fabrication of thin-film capacitors, the use of materials with an extremely high dielectric constant (k>10000) is very promising. Such high k-values have been reported for $LuFeO_3$ (LFO) ceramics, for which a dielectric constant of 10000 can be obtained at room temperature [1]. Literature on the deposition of LFO thin films, indispensable for the thin film applications envisaged, is only scarcely available, especially when it comes to wet chemical deposition. In this work, an aqueous low-cost chemical solution deposition route was investigated – to our knowledge for the first time. A stable Lu/Fe multimetal ion precursor was prepared by mixing a citrato complex-based Fe and Lu solution in the desired ratio. From this precursor solution, films were deposited on SiO_2/Si substrates via spin coating, followed by a thermal treatment on hot plates (steps determined by TGA) and a post deposition anneal (PDA). Characterization by means of AFM, SEM, GATR-FTIR, XRD and *C-V*/*I-V* measurements was applied to collect information about topography, microstructure, film purity, phase formation and the dielectric constant of deposited layers as a function of various deposition parameters. It was shown that the obtained layers were smooth and the thickness could be largely controlled. Nevertheless, it seems to be very challenging to obtain crystalline LFO films in the sub 100 nm thickness range on silicon. It was confirmed by TGA/DTA that very high temperatures are necessary for the material to crystallize, and consequently to acquire the orthorhombic phase.

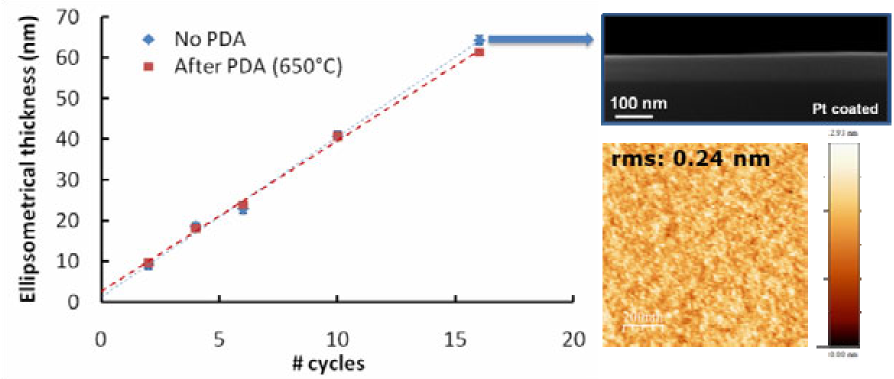

Figure 1. Plot of the ellipsometrical film thickness as a function of the amount of deposition cycles, and SEM/AFM images of the film obtained after 16 cycles (no PDA)

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M14

MODELING METHODS FOR COMPOSITES ELECTROMAGNETIC PROPERTIES

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Multiferroic composite systems and novel metamaterials have attracted great attention in recent years due to their very high applied potential. This leads to an increased concern in domain of modeling and simulation of electromagnetic properties of these mixtures. The response of a composite to an external stimulus like an electric or magnetic field or the internal deformation behavior to a mechanical stimulus like elastic or torsion deformation can be anticipate.

As a result of processing methods, any ceramic dielectric is actually a diphase or a multiphase composite. Electric and magnetic properties of composites depend on each phase properties, on shape and size of phases and on the degree of mixing and interconnectivity between them. Any presence in ceramic's volume of secondary phases or presence of structural and compositional inhomogeneities also leads to variations in electric and magnetic properties of composites compared with pure composites.

Since 19th century have been proposed several analytical and numerical models to predict composite's characteristics and their electric and magnetic behaviors [1]. Models are usually mathematical approximations that use simple functional forms for functions or get approximate solutions to differential equations. The approach in analytical mixing formulas is to idealize the geometry and model the microstructure using simple forms for inclusions, spheres, ellipsoids, needles [2]. The effective permittivity of a mixture can be calculated as a function of the constituent permittivities, their fractional volumes, and possibly some other parameters characterizing the microstructure of the mixture.

Quantum mechanics offer a mathematical description of the wave-particle duality of matter and energy. Many macroscopic properties of composite systems are fully understood and explained starting from time-dependent ab-initio Schrödinger equations [3]. Density Functional Theory methods permit to determine the molecular electronic structure using parameters derived from empirical data. Also, Molecular Mechanics set up a simple algebraic expression for the total energy of a compound. Energy expression consists of simple classical equations, such as the harmonic oscillator equation in order to describe the energy associated with bond stretching, bending, rotation and intermolecular forces, such as Van der Waals interactions and hydrogen bonding. All constants used in these equations must be obtained from experimental data or an ab-initio calculation.

Our paper presents some of methods used for describing, characterizing and anticipating composite electromagnetic properties.

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M15

ORIGINAL DIELECTRIC BEHAVIORS RELATED TO THE TETRAGONAL TUNGSTEN BRONZE STRUCTURE

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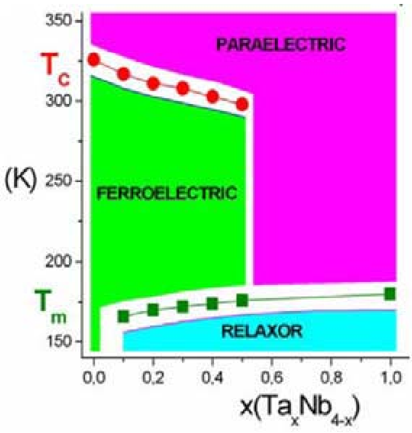
Forty five years before the “revival of the magnetoelectric effect” [1] – Fang & Roth reported the existence of several niobate-based materials with Tetragonal Tungsten Bronze (TTB) structure which may exhibit both ferroelectric and ferrimagnetic behaviour at room temperature [2]. In a recent reinvestigation of the $Ba_2LnFeNb_4O_{15}$ (Ln=La, Pr, Nd, Sm, Eu, Gd) system, we emphasized its composite multiferroic nature [3] and discovered unexpected ferroelectric to relaxor crossover in several related systems [4,5].



Following a recent study focused on tantalum substituted $Ba_2LnFeNb_{4-x}Ta_xO_{15}$ TTBs [6], we investigated a series of compositions with small substitution ratios. First results confirmed a ferroelectric to relaxor crossover, characterized by the same relaxor-ferroelectric-paraelectric sequence of phase transitions on heating (Fig 1), previously observed in the $Ba_2Pr_{1-x}Nd_xFeNb_4O_{15}$ system [4].

Aiming at understanding the chemical and physical properties of these complexes phases, we focused our attention on the crystal growth of $Ba_2LnFeNb_4O_{15}$ relaxors [3,6] from high temperature solutions. X-Ray diffraction validated the TTB structure as well as dielectric measurements confirmed the relaxor behaviour of grown crystals (Fig. 2).

The occurrence of similar ferroelectric to relaxor crossovers in solid solutions based on different cationic substitutions, along with the availability of large single crystals, give us the opportunity for insightful studies on the mechanism involved in TTB relaxors, which are probably different from the mechanisms involved in perovskite relaxors.

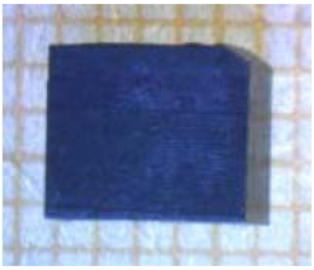


Figure 2. $Ba_2LaFeNb_4O_{15}$ single crystal oriented along the (001) direction

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M16

ELECTRICAL CONDUCTIVITY AND MAGNETIC PROPRETIES OF NANOCRYSTALLINE In-DOPED ZINC FERRITES

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This paper presents results concerning on dielectric behavior, conductivity and some magnetic properties of the nanosized $Zn_{1-x}xIn_xFe_2O_4$ powders (x = 0, 0.15, 0.2, and 0.3), obtained by coprecipitation method. The frequency dependance of the dielectric permittivity and the conductivity of the samples are determined in the frequency range (1–10^5) Hz, at temperatures from 300 to 350 K. The AC conductivity was found to follow universal dielectric response. Analyzing the variation of the parameter n with the temperatures we discuss the possible conduction mechanism in investigated samples. The obtained hysteresis curves can be attributed to the ferrimagnetic ordering of the investigated samples.

Keywords*:* nanoparticles; coprecipitation; X-ray diffraction; specific surface area; TEM; AC conductivity; correlated barrier hopping; nonoveralpping small polaron; superparamagnetics

M17

SYNTHESIS, SINTERING AND CHARACTERISATION OF STRONTIUM DOPED $K_{0.5}Na_{0.5}NbO_3$ CERAMICS

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Solid solution of sodium potassium niobate with the composition $K_{0.5}Na_{0.5}NbO_3$ (KNN) is one of the most studied lead-free piezoelectric materials [1]. Despite a throughout research done on the synthesis and characterization of this lead-free ceramics the problems of compositional fluctuations of alkaline niobate-based ceramics are still an important issue and may become even more significant in chemically modified KNN [2]. Another challenge about KNNis thatthe ceramics is extremely difficult to consolidate, therefore various wayshave been proposed to overcome this problem [1]. One way to improve the sinterability of KNN is the creation of vacancies on the A site of the perovskite unit cell by introducing alkaline earth ions [3]. Malič *et al.* found, that calcium and strontium doping promotes densification, while magnesium inhibits it and barium doping results in secondary phases. Doping with 0.5 at.% Sr resulted in 96% of TD of KNN (TD = 4.51 g/cm^3). Godnjavec [4] studied the influence of addition of strontium on structure, microstructure and electrical properties of $K_{0.5}Na_{0.5}NbO_3$ and achieved the highest density at $K_{0.5}Na_{0.5}NbO_3$ doped with 2 at.%Sr (approaching the TD).

The aim of this study was to explore and define how the addition of strontium influences the synthesis, sintering, phase and crystal structure of KNN. The$Sr_{0.02}K_{0.49}Na_{0.49}NbO_3$was prepared by solid state synthesis from alkaline carbonates, niobium oxide and strontium nitrate with two times calcination at 800°C, 4 h. Sr-doped KNN ceramics was sintered at 1120°C, 2 h in air. Different analytical methods, such as granulometric analysis, XRD, heating stage microscope and microscopy techniques (SEM, TEM) were used to optimize the synthesis and sintering conditions to obtain dense, one phase ceramics. In order to define the influence of strontium on the crystal structure of KNN at nanolevel the results of the detailed analysis using TEM microscopy techniques (EDXS, SAED, HRTEM) will be presented.

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M18

INVESTIGATION OF FUNCTIONAL PROPERTIES OF $BiFeO_3$ CERAMICS PREPARED BY ONE-STEP SINTERING METHOD

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Only few reports show relevant data concerning the properties of pure $BiFeO_3$ ceramics, due to the high difficulties in preparing $BiFeO_3$ ceramics free of secondary phases and rarely their functional properties could be discussed and compared with ones reported for single crystals or films, due to the major role of the extrinsic contributions (defects, secondary phases, porosity).

In the present study, the electrical and magnetical properties of pure $BiFeO_3$ ceramics prepared by a single-step solid-state sintering method were investigated. A very interesting feature is observed on the conductivity in the low-frequency range (dc-conductivity). The ac-conductivity vs. temperature presented in the Fig. 1b shows that,

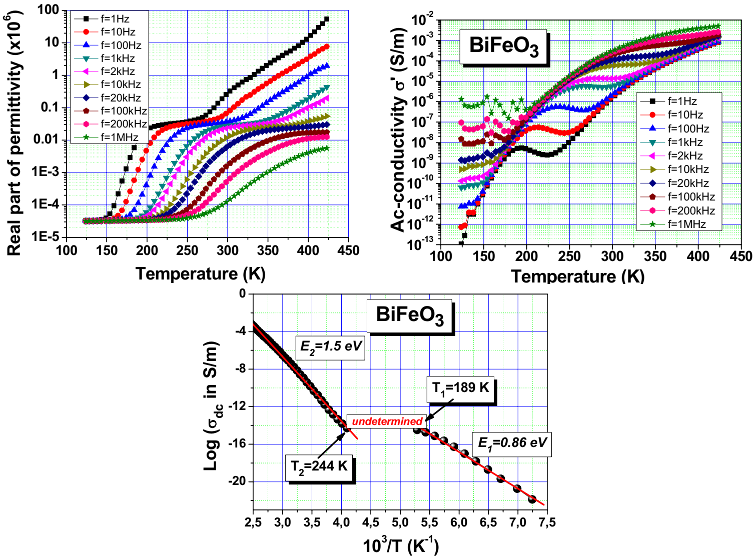

Figure 1. Temperature dependence of the: (a) real part of permittivity, (b) dc-conductivity for the $BiFeO_3$ ceramic at a few selected frequencies. (c) Frequency-dependence at a few selected temperatures in the range of 124 – 425 K of the Arrhenius plot of the dc-conductivity.

except the low-temperature range, all the values of conductivity at any frequencies lay between the two "master-curves" corresponding to the lowest and highest frequency, respectively, giving rise to the much higher extreme activation energies. The Arrhenius plot of the dc-conductivity determined at the lowest frequency *vs.* $1/T$ (Fig. 1c) shows two distinct linear regions separated by the mentioned temperature range of 189–244 K, for which the dc conductivity could not be determined from the present impedance spectroscopy data only. It is clear, out of any doubt that in the mentioned temperature range, a conduction anomaly takes place. Further detailed studies to confirm the presence of such anomaly in ceramics prepared by various routes in order to understand the origin of this conductivity anomaly are under way.

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M19

PREPARATION AND MULTIFUNCTIONAL PROPERTIES OF $Ba_{12}Fe_{28}Ti_{15}O_{84}$ NATURALLY SELF-ASSEMBLED LAYERED CERAMICS

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The $BaO-Fe_2O_3-TiO_2$ system has a rich crystal chemistry and the existence of at least 16 different quaternary compounds has been reported [1,2]. The quaternary phases are expected to have a high dielectric constant and some of them show a magnetic activity, although a systematic investigation is still lacking.

In this study we report for the first time the preparation and multifunctional properties (magnetic and dielectric properties) of the quaternary layered ferrite $Ba_{12}Fe_{28}Ti_{15}O_{84}$. Dense ferrite ceramics were prepared by conventional sintering using powders obtained by solid-state reaction. A minor amount of secondary phases were observed in the material obtained by the solid-state route. According to the HRTEM investigation, the ferrite lattice is originated by the intergrowth of perovskite-like and spinel-like slabs, in agreement with previous XRD crystal structure investigations, and can be considered as a natural magnetic superlattice. A ferrimagnetic order with saturation magnetization of $\approx$13 emu/g and coercivity of ~20 Oe was determined at room temperature, while the thermomagnetic data indicate a Curie temperature of $\approx$420K. Ceramics obtained by the solid-state route show constricted magnetization loops owing to the presence of a minor, not identified hard magnetic phase and an additional magnetic transition at ~700 K. An intrinsic relative dielectric constant of the order of 23–50 at room temperature was measured at 10^9 Hz. At lower frequency the dielectric behaviour is dominated by extrinsic effects related to the heterogeneous electrical nature of the ceramics corresponding to semiconducting grains separated by more insulating grain boundary regions. The dielectric losses are rather high, often >1, indicating an overall semiconducting character of the material.

Acknowledgements: The support of the COST Action MP0904 SIMUFER and Erasmus placement Programme are acknowledged.

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M20

$BiFeO_3$ THIN FILMS: BETWEEN DECOMPOSITION AND PHASE STABILITY

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Bismuth ferrite, $BiFeO_3$, is considered as one of the potential materials for novel device applications due to its electric and magnetic ordering in a single phase. Although $BiFeO_3$ is extensively studied, there are still some different opinions in literature about phase formation, phase stability and decomposition of this multiferroic material. Reasons for the appearance of the most common secondary phases, e.g. $Bi_{25}FeO_{39}$ and $Bi_2Fe_4O_9$, vary from a low peritectic decomposition temperature of bismuth ferrite to thorough control of processing parameters [1–4].

To study process parameters for the deposition of phase pure $BiFeO_3$, films were deposited on $Si/SiO_2/TiO_2/Pt$ substrates by spin coating a mixture of stable aqueous Bi (III) and Fe (III) precursor solutions [5], followed by a thermal treatment. X-ray diffraction (XRD) and microstructural analysis (SEM and AFM) showed that drastic changes in phase composition and morphology occur after annealing at 700°C. As indicated by XRD and EDX, an iron rich $Bi_2Fe_4O_9$ phase appears as the major phase probably due to bismuth loss during the thermal treatment. It is shown that an increase of the film thickness or conducting the anneal step in a different atmosphere (air, N_2 and O_2) does not improve the $BiFeO_3$ phase stability. Doping with Ti^{4+} ions on the other hand, significantly decreases the amount of iron rich phase in the obtained films, as confirmed by comprehensive structural and microstructural analysis.

An Hardy is a postdoctoral fellow of the Research foundation-Flanders (FWO-Vlaanderen).

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M21

FERROELECTRIC-RELAXOR CROSSOVER OF $BaTiO_3$-BASED SOLID SOLUTIONS AND GRAIN SIZE EFFECT IN $BaTiO_3$ DESCRIBED BY MONTE CARLO SIMULATIONS

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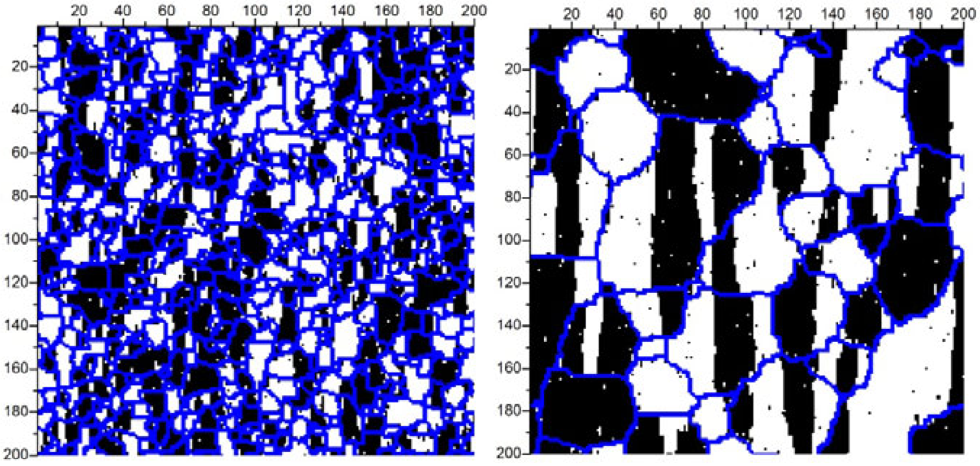
In the present work we investigated the influence of the non-ferroelectric cells on the properties of the ferroelectric systems based on $BaTiO_3$ using a Monte Carlo model.

The properties induced by the homovalent addition M^{4+} (M = Zr, Sn, Hf) in $BaM_xTi_{1-x}O_3$ solid solutions were described on the basis of a Monte Carlo model, in which non-ferroelectric $BaMO_3$ randomly distributed unit cells were considered. The $P(T)$ dependence shows a continuous reduction of Pr and TC and a modification from a first-order to a second-order phase transition, when increasing the M^{4+} concentration. The system's tendency to reduce the polar clusters size while increasing their stability above the Curie range by increasing the substitution (x) is also described.

Using the same model the dimensional effect in polycrystalline $BaTiO_3$ ceramics has been qualitatively investigated. The polycrystalline $BaTiO_3$ structures were obtained by a Potts model. In this case the non-ferroelectric cells are not randomly distributed, they are located on grains boundaries (blue lines in Fig. 1). It was observed that for small



(a) (b)

Figure 1. The domains structure simulations for two different grain sizes: (a) small grains; (b) large grains (white: upwards polarization, black: downwards polarization, blue: grain boundary with non-ferroelectric cells)

grains the domains are larger than the grain size (Fig. 1a) while for coarse-grain ceramics the most of the grains are polydomains.

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BROADBAND DIELECTRIC INVESTIGATION OF BISMUTH MODIFIED LEAD-FREE STRONTIUM TITANATE CERAMIC

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Lead containing piezoelectric ceramics and single crystals as $Pb(Zr_xTi_{1-x})O_3$ (PZT) or $(1-x)[Pb(Mg_{1/3}Nb_{2/3})O_3]-x[PbTiO_3]$ (PMN-PT) has been widely used as actuator, transducer, and sensor materials. However, the toxicity of lead has led to a demand for alternative materials that are more human and environment friendly.$(Sr_{1-1.5x}Bi_x)TiO_3$ - strontium bismuth titanate (SBT for short) is the one of such compounds.

It is well known that in pure incipient ferroelectrics, like $KTaO_3$ or $SrTiO_3$ polar phonons are responsible for the dielectric permittivity and no pronounced dielectric dispersion is observed below the soft-mode response [1]. The ferroelectricity in such materials can be induced by application of strong enough electric field, uniaxial stress or substitution of A-site ions by isovalent or heterovalent ions. The broadband dielectric studies on SBT ceramic (up to $x \leq 0.17$) [2,3] showed that Bi doping induces formation of local antiferrodistortive and polar regions. This causes a complex relaxational dynamics below the phonon frequency range. Because no broadband dielectric studies (on our knowledge) have been carried out for SBT with bigger bismuth content, the aim of this work was to extend the investigations of dielectric dispersion in SBT toward x=0.25, 0.2 and 0.15.

The dielectric properties of SBT ceramic have been investigated from 20 Hz to GHz range. Obtained results showed the pronounced dielectric dispersion which looks similar to the one typically observed in disordered materials. In contrast to the proposals in [2], which claimed that the dispersion in SBT ceramic looks relaxor-like, we have to point its close similarity to the dispersion typically found in dipolar glasses. The calculated distribution of relaxation times $f(\tau)$ confirms our findings because no splitting into two parts of $f(\tau)$ can be seen.

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FUNCTIONAL PROPERTIES OF $Mg_xNi_{1-x}Fe_2O_4$ FERRITE

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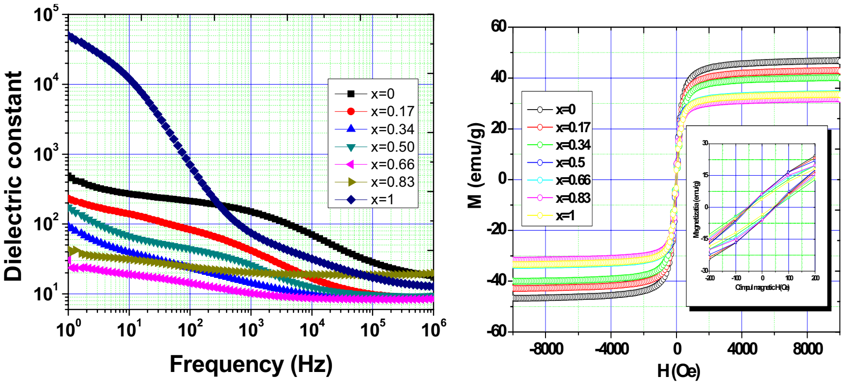
Synthesis and functional characterization of ferrite has increased of attraction in the last years, due to their applications in telecommunication devices, computer memories, heat transfer and transmitting microwaves. Large-scale applications of ferrite have promoted the development of various chemical methods for preparation as alternative solid state reaction, which result in large agglomerate particles, with limited degree of homogeneity [1]. Among different ferrites, magnesium ferrite $MgFe_2O_4$ enjoys a special attention because of its vast applications in high-density recording media, heterogeneous catalysis, adsorption, sensors and magnetic technologies. Nanoparticles of $MgFe_2O_4$ have good photoelectrical properties. Nickel ferrites $NiFe_2O_4$ are one of the most important ferrites with reversed spinel structure having ferrimagnetic properties.



(a) *(b)*

Figure 1 (a) Dielectric properties and (b) M(H) loops for (Mg,Ni)Fe_2O_4 at room temperature

In the present paper the structural and functional properties of magnesium-substituted nickel ferrite having the general formula $Ni_{1-x}Mg_xFe_2O_4$ (x = 0; 0.17; 0.34; 0.5; 0.64; 0.83; 1) has been studied as a function of magnesium ion concentration.

Study of the electrical and dielectric properties give valuable information about the behaviour of electric charge carriers which leads to good understanding and explanation of conduction mechanism in ferrite. The dielectric properties have been investigated as a function of frequency and temperature and were interpreted in terms of Maxwell-Wagner phenomena and conductivity mechanism.

The magnetic properties show a typical ferrimagnetic character, with a strong non-linearity, very small coercitive field around 50 Oe and saturation field of ~ 1 kOe. A decrease of magnetic moment and saturation magnetization takes place with increasing Mg content combined with a constant coercitiv field.

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PREPARATION OF MULTIFERROIC TITANATE/FERRITE COMPOSITE CERAMICS WITH CORE/SHELL STRUCTURE

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Growing research interest in multiferroic composite ceramics, in the last few years, is mostly stimulated by the fact that niether of the known one-phase multiferroic materials exhibit satisfactiory properties at the room temperature. Untill today, various combinations of ferroelectric and ferromagnetic materials were used for preparation of multiferroic composite ceramic.This work presents a possible approach for obtaining multiferroic composite ceramics form core/shell particles, consisting of titanate core and ferrite shell. Core/shell particles are sinthesized by sol-gel and co-precipitaion rutes, and further pressed into pallets and sintered at various temeperatures. Parameters affecting the morphology of the particles (pH, core-shell mass ratio) were examined, as well as the optimal processing conditions for obtaining homogenious dense ceramics. Prepared and pressed powder was densified by the means of conventional sintering in air or Spark plasma sintering.

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