

**13th CONFERENCE for
YOUNG SCIENTISTS in CERAMICS**

**PROGRAMME
and
BOOK OF ABSTRACTS**

**October 16-19, 2019
Novi Sad, Serbia**

Programme and Book of Abstracts of The 13th Conference for Young Scientists in Ceramics (CYSC-2019) publishes abstracts from the field of ceramics, which are presented at traditional international Conference for Young Scientists in Ceramics.

Editors-in-Chief

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Publisher

Faculty of Technology, University of Novi Sad
Bul. cara Lazara 1, 21000 Novi Sad, Serbia

For Publisher

Prof. Dr. Biljana Pajin

Printing layout

Vladimir V. Srdić, Marija Milanović, Ivan Stijepović

Press

SLUŽBENI GLASNIK, Beograd

CIP – Каталогизacija у публикацији
Библиотека Матице српске, Нови Сад

666.3/.7(048.3)

CONFERENCE for Young Scientists in Ceramics (13 ; 2019 ; Novi Sad)

Programme and book of abstracts / 13th Conference for Young Scientists in Ceramics (CYSC-2017), October 16-19, 2019, Novi Sad ; [editor-in-chief Vladimir V. Srdić]. - Novi Sad : Faculty of Technology, 2019 (Beograd : Službeni glasnik). - XX, 152 str. : ilustr. ; 24 cm

Tiraž 180. - Registar.

ISBN 978-86-6253-104-9

a) Керамика - Технологија - Апстракти
COBISS.SR-ID 331006727



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Preface

Dear conference participants and readers we have the pleasure to once again welcome you all to Novi Sad, Serbia as the venue for the 13th Conference for Young Scientists in Ceramics. This year again the event is jointly organized by the Faculty of Technology Novi Sad, University of Novi Sad and the Young Ceramists Network (YCN) of the European Ceramic Society.

The Conference for Young Scientists in Ceramics, previously known as the Students' Meeting, is the conference with more than twenty years of tradition. It grew from the Serbian only conference in 1998 to the truly international event with participants coming from 31 different countries from all over the world. The one important thing that did not change from the beginning is the basic concept which has always been the promotion of young stage researchers and their achievements. Every two years the Conference becomes the place where young MSc and PhD students and young doctors meet to exchange their ideas, make new networks and share their knowledge of the topics covering ever expanding field of ceramics. Beside 134 oral presentations given by their peers, the young scientists will have the opportunity to hear 14 invited talks and 1 plenary lecture of the more experienced scientists and experts. The presented topics include many important scientific issues and cutting edge results in ceramics ranging from the theoretical and modelling results over the experimental structural and functional characterizations all the way to the applicative examples and industrial scale production of ceramic materials. In this way, all participants will have the chance to expand their knowledge and strengthen their basic understanding of the various branches of ceramics science covering advance materials, ceramic composites and traditional ceramics. It is important to mention that this year, for the first time, we will have the student competition for the best oral presentation of a young researcher. There will be three awards which are dedicated to the late Prof. Dr. Paolo Nanni.

We want to use this opportunity to thank our sponsors and co-organizers for helping us to successfully prepare the Conference. First of all, we want to thank the JECS Trust Fund of the European Ceramic Society for their strong financial support. Also, we want to mention that the Serbian Ministry of education, science and technological development recognized our conference as an important event and gave their financial endorsement. The financial part of the awards for three best presentations dedicated to Prof. Nanni was sponsored by Prof. Liliana Mitoseriu from University of Iasi, Romania. At the end, we would like to thank to all the people in the local organizing committee and colleagues from YCN who participated in the preparations of the Conference.

Editor

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Novi Sad European youth capital - OPENS



Tourist organization city of Novi Sad

“European youth Capital – Novi Sad 2019” – adding colors to the city

Novi Sad is a small, but compact city in the heart of the Vojvodina region. This vivid city with over 80,000 young people will surprise you with it's transformative energy, multi-nationality and cultural diversity. For centuries, Novi Sad has been a pioneer and symbol of youth activism, making significant social changes in the history and systematic youth care. But, the 2019 is the year when Novi Sad is European Youth Capital. With OPENS Programme Novi Sad is creating more opportunities for youth by youth, empowering them to become pro-active initiators of positive changes, introducing innovative ideas not only in Novi Sad, but also at national and international level.

We have addressed the needs aiming to improve young people's lives, to increase participation in the social-political process with a strong feeling of ownership of the development of the city. Young people are directly engaged in programs creation when it comes to youth activities, as well as in their implementation.

There is no fixed pattern previously established based on which one could dance through the whole project of the European Youth Capital. Each city tells its own story. At the same time, this is where the excitement of the challenge lies. At the moment, OPENS, within the existing system, is breaking new grounds towards the construction of a new system in order to make a radical change in innovative ideas.

Perhaps you would not say if led only by first impressions, history or architecture that Novi Sad is a city of young people. As a perfect blend of modern and traditional, openness and tolerance, this city has inspired more than 400,000 people to express themselves through different activities. Each month Novi Sad puts on a new color and becomes over and over again the host of a number of festivals, fairs and celebrations, aimed first of all, at young people. And now, at every corner, you can find the traces of history and blend of cultures. The city whose rhythm you must hear to be able to feel it. The city which celebrates the diversity, energy and activism of young people! This is the city led by young people, students, artists. The city for curious, dynamic and motivating people. This is the city you wish to experience! And this is the European youth capital

At the moment, Novi Sad is on the doorstep of becoming the center in which young people have the main say. If young people are to inherit the Earth, our role and our final goal is not only to be led by this saying, but to make it happen as well.



Photo by Vladimir Velickovic, Omladinska prestonica Evrope Novi Sad 2019

Novi Sad European youth capital - OPENS

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Content

PROGRAMME

Wednesday, October 16, 2019	2
Thursday, October 17, 2019	5
Friday, October 18, 2019	9
Saturday, October 19, 2019	13

PLENARY and INVITED LETURES

J.-O. Durand MESOPOROUS SILICA, PERIODIC MESOPOROUS ORGANOSILICA, AND MESOPOROUS SILICON NANOPARTICLES FOR DRUG DELIVERY AND TWO- PHOTON PHOTODYNAMIC THERAPY	18
M. Winterer LEARNING ABOUT NANOPARTICLES BY X-RAY ABSORPTION SPECTROSCOPY	19
Á. Kukovecz KEY ELEMENTS OF A SUCCESSFUL H2020 MARIA-SKODOWSKA CURIE – INDIVIDUAL FELLOWSHIP APPLICATION	20
L. Kozielski MECHANO-LUMINESCENT MATERIALS FOR DIRECT CONVERSION OF MECHANICAL ENERGY INTO LIGHT	20
J. Binner DESIGNING THE PROCESSING OF ADVANCED CERAMICS AND COMPOSITES TO YIELD THE REQUIRED PROPERTIES	22
P.M. Vilarinho ALTERATIVE SINTERING ROUTES OF ELECTROCERAMICS: THE CASE OF SODIUM POTASSIUM NIOBATE, A LEAD FREE PIEZOELECTRICS	22
D. Bossini ULTRAFast OPTICAL CONTROL OF MAGNETISM IN SOLIDS	23

I. Djerdj, S. Urban, P. Dolcet, L. Chen, M. Möller, O. Khalid, H. Camuka, R. Ellinghaus, C. Li, S. Gross, P.J. Klar, B. Smarsly, H. Over THE APPLICATION OF Ce-Zr-O COMPOUNDS IN HCl OXIDATION AND INTHREE-WAY CATALYSIS	24
V. Buscaglia, M.T. Buscaglia, G. Canu SIZE AND SCALLING EFFECTS IN FERROELECTRIC CERAMICS	25
J.U. Nandhini, A. Sarkar, P. Arivazhagan, L. Velasco, H. Hahn, S.S. Bhattacharya PHASE-PURE MULTICOMPONENT EQUIMOLAR OXIDES AS A NOVEL CLASS OF FUNCTIONAL MATERIALS: SYNTHESIS, CHARACTERIZATION AND PROPERTIES	26
L. Silvestroni THE IMPORTANCE OF TEM ANALYSES FOR THE DEVELOPMENT OF UHTCs	27
N. Knežević CORE/SHELL MESOPOROUS SILICA AND ORGANOSILICA NANOPARTICLES: SYNTHESIS, CHARACTERIZATION AND APPLICATION IN TARGETED CANCER TREATMENT	28
P.M. Vilarinho HOW TO TALK SCIENCE ... clearly	28
C. Leonelli THE MAGIC OF HIGH POWER MICROWAVE PROCESSING OF CERAMIC MATERIALS	29
N. Kanas, M.-A. Einarsrud, K. Wiik OXIDE THERMOELECTRICS – CURRENT STATUS AND FUTURE PROSPECTS	30

ORAL PRESENTATIONS

A. Levish, M. Winterer IRON(III)ACETYLACETONATE VAPOR STUDIED BY X-RAY ABSORPTION ...	34
T.S.R.C. Murthy, V. Venkatachalam, J. Zou, J. Binner SOL-GEL-BASED INTERFACIAL COATINGS OF RARE EARTH OXIDES ON 2.5D CARBON FIBRE PREFORMS	35
R. Nicola, C. Ianăși, M. Picioruș, S. Ilieș, A.-M. Putz, R. Lazău, A. Ercuța MAGNETIC NANOCOMPOSITES CONTAINING IRON OXIDE@SiO ₂ OBTAINED VIA CONVENTIONAL OR ULTRASONIC METHOD	36
J. Aleksić, T. Barudžija, D. Jugović, M. Mitrić, M. Bošković, Z. Jagličić, S. Gyergyek, Lj. Kostić SYNTHESIS, STRUCTURAL AND MAGNETIC PROPERTIES OF Y _{1-x} Yb _x F ₃ SOLID SOLUTION	37

J. Schulte, J. Geiss, M. Winterer LASER-FLASH-EVAPORATION OF SOLID METAL ORGANIC PRECURSORS	38
I. Milenković, K. Radotić, B. Matović, M. Prekajski, L. Živković, V. Beškosi COATING OF CERIUM OXIDE NANOPARTICLES WITH DIFFERENT CARBOHYDRATES AND THEIR APPLICATION ON PLANTS	39
J. Zygmuntowicz, P. Piotrkiewicz, W. Kaszuwara PROCESSING AND CHARACTERIZATION OF Al_2O_3 -Cu-Ni COMPOSITES	39
M. Wachowski, L. Śniezek, W. Kaszuwara, K. Konopka, J. Zygmuntowicz EFFECT OF METAL PARTICLES SIZE ON MICROSTRUCTURE OF ZrO_2 -Ni COMPOSITE FABRICATED BY CSC IN MAGNETIC FIELD	40
K. Dudek, M. Dulski, A. Nowak FABRICATION AND CHARACTERIZATION OF MULTI-FUNCTIONAL HYBRID LAYERS ON NiTi SHAPE MEMORY ALLOY	41
M. Vukšić, I. Žmak, L. Čurković INFLUENC OF THE ADDITION OF WASTE ALUMINA POWDER ON MECHANICAL PROPERTIES OF ALUMINA CERAMICS	42
I. Nomel, O. Durand-Panteix, L. Boyer, P. Marchet PREPARATION OF ZnO DISPERSIONS SUITABLE FOR SENSORS FABRICATION USING INKJET PRINTING	42
J. Stanojev, B. Bajac, J. Matovic, V.V. Srdic PROCESSING OF NANOSTRUCTURED CNT-BASED THIN FILM ELECTRODE	43
E. Eray, G. Magnacca, V. Boffa, V. Candelario NEW GENERATION SILICON CARBIDE MEMBRANES FOR WATER PURIFICATION	44
A. Policicchio, A.-M. Putz, S. Stelitano, C. Ianăși, G. Conte, R.G. Agostino ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA SYNTHESIZED FOR HYDROGEN STORAGE APPLICATION	45
P.F. Großmann, C. Troll, B. Rieger DIRECT INK WRITING AS AN ADDITIVE MANUFACTURING TECHNIQUE FOR SHAPING HETEROGENEOUS CATALYSTS	46
Á. Szamosvölgyi, A. Sápi, Á. Kukovecz, Z. Kónya EXAMINATION OF THE ROLE AND BEHAVIOUR OF MESOPOROUS SUPPORT MATERIALS IN CO_2 HYDROGENATION	46
G. Conte, A. Policicchio, A.-M. Putz, C. Ianăși, S. Stelitano, R.G. Agostino THE HYDROGEN STORAGE CAPACITY OF ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA	47
A.-M. Putz, A. Policicchio, R.G. Agostino, S. Stelitano, G. Conte, C. Ianăși ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA WITH GAS STORAGE PROPERTIES: SYNTHESIS AND CHARACTERIZATION	48

J. Mrówka, J. Partyka, M. Hasik THERMAL PROPERTIES AND CERAMIC YIELD OF POROUS POLY(METHYL- VINYLSILOXANE) CROSS-LINKED WITH 1,3,5,7-TETRAMETHYLCYCLO- TETRASILOXANE BEFORE AND AFTER DEPOSITION OF PALLADIUM NANOPARTICLES	49
E. İşlek, H.B. Poyraz, M. Polat, İ.Ö. Özer NANO-SIZED MAGNESIUM SPINEL POWDERS SYNTHESIZED VIA FLAME SPRAY PYROLYSIS	50
V. Mackert, M. Winterer PROBING SONICATION OF COLLOIDAL SnO ₂ BY <i>IN SITU</i> SMALL-ANGLE X- RAY SCATTERING	51
B. Özdemir, N. Canikoğlu SYNTHESIS OF TITANIUM NITRIDE POWDER FROM RUTILE AND ANATASE	51
M. Temnikova, A. Glukharev, O. Kurapova, V. Konakov SYNTHESIS, PHASE COMPOSITION AND MICROSTRUCTURE OF ZrO ₂ -Y ₂ O ₃ - rGO COMPOSITE PRECURSORS OBTAINED BY SOL-GEL SYNTHESIS	52
D.A. Rayan, M.A. Zayed, G. Mahmoud COMPARATIVE STUDY OF La AND Zn IONS CO-DOPED MAGNETITE NANOSTRUCTURES SYNTHESIZED IN OXYGEN AND INERT ATMOSPHERE	53
J. Geiss, M. Winterer STRUCTURAL CHARACTERIZATION OF LaFeO ₃ PEROVSKITE NANOPARTICLES	54
I. Dinic, M. Vukovic, P. Vulic, M. Nikolic, L. Mancic, O. Milosevic EFFECTS OF CITRIC ION ON HEXAGONAL NaYF ₄ : Yb/Er PHASE FORMATION DURING SOLVOTHERMAL SYNTHESIS	54
S. Hříbalová, W. Pabst LIGHT SCATTERING IN TRANSPARENT CERAMICS: USE AND VALIDITY OF MIE THEORY APPROXIMATIONS	55
Y.H. Elbashar, D.A. Rayan COLOR AND OPTICAL SPECTROSCOPIC ANALYSIS OF Cu ₂ O-K ₂ O-ZnO-P ₂ O ₅ GLASS MATRIX	56
D. Danilović, D.K. Božanić, R. Dojčilović, V. Vodnik, A. Milosavljević, C. Nicolas², L. Nahon, G.G. Macias, V. Djoković PHOTOEMISSION SPECTROSCOPY ON ISOLATED SILVER-BASED HYBRID NANOSTRUCTURES: A NOVEL APPROACH TO UNDERSTAND FUNDAMENTAL PROPERTIES OF SOLAR CELL ABSORBER MATERIAL	57
D.A. Rayan, A.M. Abdelghany, Abdallah Elshourbgy, S.H. Moustafa REMARKABLE IMPACT FOR OPTICAL PROPERTIES OF SAMARIUM DOPED ALUMINUM SODIUM PHOSPHATE GLASS FOR VISIBLE DEVICES	58
A. Alzahrani EFFECT OF SINTERING TEMPERATURE ON CRYSTALLIZATION OF NEPHELINE-LEUCITE GLASS-CERAMICS	59

P. Staciwa, U. Narkiewicz, D. Sibera PREPARATION OF IRON/CARBON COMPOSITES	60
A. Zavjalov, P. Nikiforov, E. Goncharov, D. Kosyanov, K. Talskikh, O. Shichalin, E. Papynov SPARK PLASMA SINTERING OF Hf-C-N CERAMICS	61
M. Kosiorek, A. Żurawska, M. Blesznowski RHEOLOGICAL PROPERTIES OF GLASS-CERAMIC SEALS FOR SOC APPLICATION FABRICATED BY TAPE CASTING	62
Ł. Rakoczy, R. Cygan, M. Grudzień-Rakoczy APPLICATION OF SHELL MOLDS STRENGTHENED BY METAL POWDERS AND GLASS FIBRES IN THE FABRICATION OF JET ENGINE COMPONENTS ..	63
D. Tovar-Vargas, M. Turón-Viñas, M. Anglada, E. Jiménez-Pique PROCESSING, CHARACTERIZATION AND MECHANICAL PROPERTIES OF CERIA CALCIA STABILIZED ZIRCONIA CERAMICS WITH α -ALUMINA ADDITION	64
L. Vászárhelyi, I. Szenti, D. Sebők, Á. Kukovecz, Z. Kónya IN SITU INVESTIGATION OF CRACK PROPAGATION IN CERAMICS BY MICRO-CT TECHNIQUE	64
A. Sedegov, V. Tsybulin, D. Pankratova, A. Taranova, K. Kuskov, S. Vorotylo, D. Moscovskikh, A. Rogachev, A. Mukasyan SELFPROPAGATING HIGH TEMPERATURE SYNTHESIS AND STUDY OF CARBIDE AND DIBORIDE BASED ON REFRACTORY HIGH-ENTROPY ALLOY Hf-Ta-Ti-Nb-Zr	65
A. Qadir, J. Dusza PROCESSING AND PROPERTIES OF SILICON NITRIDE + MWCNTs COMPOSITES PREPARED FROM OXIDIZED α -Si ₃ N ₄ STARTING POWDER	67
V. Nečina, T. Uhlířová, W. Pabst, P. Diblíková THE EFFECT OF HEATING RATE ON THE GRAIN GROWTH OF ALUMINA PREPARED VIA ELECTRIC CURRENT ASSISTED SINTERING (ECAS)	68
T. Boteva, P. Petkov, T. Petkova DIAMOND ELECTRODES FOR ELECTROCHEMICAL APPLICATIONS	68
C. Gorynski, L. Engelke, D.E. Wolf, U. Anselmi-Tamburini, M. Winterer INFLUENCE OF ELECTRIC CURRENT ON MICROSTRUCTURE OF Al DOPED ZnO	69
R. Guillén, L. Gil, M.D. Salvador, A. Borrell, F. Penaranda-Foix, C. González PROPERTIES OF Nb ₂ O ₅ COMPOSITE DOPED WITH ZrO ₂ SINTERED BY MICROWAVE	70
N. Fakrالمobasheri, A. Nourmohammadi EFFECT OF TWO-STEP SINTERING ON MICROSTRUCTURE OF BARIUM TITANATE CERAMICS	71

M. Abedi, D. Moskovskikh¹, A. Mukasyan FABRICATION OF SILICON NITRIDE REINFORCED BY SILICON CARBIDE BY FLASH SPARK PLASMA SINTERING	73
M. Čebela, P. Šenjug, F. Torić, Ž. Skoko, T. Klaser, D. Zagorac, D. Pajić STRUCTURE AND MAGNETIC PROPERTIES OF MULTIFERROIC BiFeO ₃ AND Bi _{1-x} Ho _x FeO ₃	74
D. Szalbot, M. Adamczyk-Habrajska, J.A. Bartkowska, M. Bara, K. Feliksik MAGNETOELECTRIC PROPERTIES OF MULTIFERROIC AURIVILLIUS TYPE Bi ₇ Fe ₃ Ti ₃ O ₂₁ CERAMICS MODIFIED BY STRONTIUM	75
P. Czaja, J. Suchanicz, M. Karolus, M. Adamczyk-Habrajska, D. Bochenek INFLUENCE OF BALL MILLING TIME ON PREPARATION AND DIELECTRIC PROPERTIES OF LEAD FREE K _{0.5} Bi _{0.5} TiO ₃ CERAMICS	76
D. Blaskova-Kochnitcharova, L. Fachikov, T. Petkova, E. Lefterova, D. Vladikova, E. Mladenova, B. Burdin STRUCTURE AND ELECTRICAL PROPERTIES OF TiO ₂ -V ₂ O ₅ -P ₂ O ₅ OXIDES	76
I. Turcan, L. Curecheriu, L. Padurariu, L. Mitoseriu Ag-BaTiO ₃ COMPOSITE CERAMICS WITH MULTIPLE PERCOLATIVE BEHAVIOR	77
A. Nesterovic, J. Vukmirovic, I. Stijepovic, J. Bobic, M. Milanovic, V.V. Srdic PREPARATION AND CHARACTERIZATION OF Bi _{0.5} Na _{0.5} TiO ₃ BASED PIEZOELECTRIC CERAMICS	78
V.A. Lukacs, L.P. Curecheriu, J.L. Jones, L. Mitoseriu SCALE DEPENDENT PHENOMENA IN BaTiO ₃ -BASED CERAMICS	79
J. Dzik, T. Pikula, D. Szalbot, Ł. Cieply, M. Adamczyk-Habrajska INFLUENCE OF RARE-EARTH IONS ON THE PROPERTIES OF BiFeO ₃ CERAMICS	80
V.K.Veerapandiyan, M. Kratzer, M. Popov, P.B. Groszewicz, J. Spitaler, C. Teichert, G. Canu, V. Buscaglia, M. Deluca ROLE OF POLAR DEFECTS IN THE ORIGIN OF RELAXOR BEHAVIOUR IN NIOBIUM MODIFIED BARIUM TITANATE POLYCRYSTALS	81
D. Brzezińska, R. Skulski, D. Bochenek, P. Niemiec THE PROPERTIES OF (1-x)(0.5PZT-0.5PFW)-xPFN CERAMICS DOPED BY Li....	82
L. Eglite, E. Birks, Maija Antonova, M. Livinsh EFFECT OF SINTERING PARAMETERS AND NON-STOICHIOMETRY ON THE MICROSTRUCTURE OF Na _{0.5} Bi _{0.5} TiO ₃	83
M. Bara, B. Wodecka-Duś, T. Goryczka, M. Adamczyk-Habrajska DIELECTRIC AND ELECTRICAL PROPERTIES OF BLT CERAMICS MODIFIED BY Fe IONS	84
J. Lelièvre, P. Marchet STRUCTURE AND PROPERTIES OF THE LEAD-FREE PERVSKITE COMPOUNDS (Na _{1/2} Bi _{1/2})ZrO ₃ (NBZ) AND (K _{1/2} Bi _{1/2})ZrO ₃ (KBZ)	84

M. Stan, R. Lach, K. Wojciechowski, Ł. Łańcucki, M.M. Bucko SINTERING AND PREPARATION OF POLYCRYSTALS GADOLINIUM-IRON GARNET ($Gd_3Fe_5O_{12}$) BY THE SOLID-STATE REACTION	85
A. Ur Rehman, L. Tingting ,M.U. Salamci, F. Pitir, G. Küçüktürk, Z. Kai, L. Wenhe, Z. Changdong ADDITIVE MANUFACTURING OF MAGNESIA AND ALUMINA EUTECTIC COMPOSITES	86
C. Aciksari, S. Pelin Erden, I. Gozde Tuncolu, Umut Savaci, S. Turan, E. Ozel, E. Suvaci SYNTHESIS OF ZINC TIN OXIDE (Zn_2SnO_4) PARTICLES BY VARIOUS METHODS USED AS CERAMIC TARGET FOR SPUTTER TECHNIQUE	87
S. Behara, T. Thomas THEORETICAL INVESTIGATION OF CRYSTAL STRUCTURE PREDICTION USING BOND-VALENCE (BV) MODELLING	88
H. My Bui, O. Hinrichsen CHALLENGES FOR BINDER JETTING OF PACKINGS AND MONOLITHS FOR HETEROGENEOUS CATALYSIS	89
N. Ilić, J. Bobić, M. Vijatović Petrović, A. Džunuzović, V. Veerapandiyan, M. Deluca, B. Stojanović PHOTOCATALYTIC PROPERTIES OF $BiFeO_3$ AND $Bi_5Ti_3FeO_{15}$ BASED POWDERS	90
M. Miklós, T. Korim IMMOBILIZATION OF SIMULATED RADIOISOTOPES IN ALKALI ACTIVATED INORGANIC POLYMERS	90
A. Sápi CATALYSIS REVOLUTION: WITH NANOTECHNOLOGY AND MOLECULAR LEVEL UNDERSTANDING TOWARDS A GREEN FUTURE	91
M. Kocijan, L. Čurković, D. Ljubas, K. Mužina SYNTHESIS, CHARACTERIZATION AND PHOTOCATALYTIC PROPERTIES OF TiO_2 -rGO NANOPARTICLES	92
A. Efremova, A. Sápi, M. Orosz-Ábel Pt NANOPARTICLES – SUPPORTED AND PRISTINE MESOPOROUS METAL OXIDES AS EFFICIENT CATALYSTS FOR CO_2 ACTIVATION	93
M.M. Uzelac, A.G. Šoškić, B.F. Abramović OPTIMIZATION OF THE PHOTOCATALYTIC DEGRADATION OF THIOTRI- AZINONE STABLE HYDROLYSIS PRODUCT OF ANTIBIOTIC CEFTRIAXONE	94
E.-M. Picioruș, C. Ianăși, R. Nicola, M. Ciopec, A. Negrea, A. Len, L. Almásy, A.-M. Putz ADSORPTION STUDIES OF CADMIUM (II) FROM AQUEOUS SOLUTIONS USING INORGANIC POROUS NANOCOMPOSITES	95
A. Boros, T. Korim PHOTOCHEMICAL ACTIVITY OF METAKAOLIN BASED GEOPOLYMER FOAMS	96

A. Kovács, É. Makó PREPARATION OF KAOLINITE-METHANOL AND KAOLINITE- CETYLTRIMETHYLAMMONIUM CHLORIDE COMPLEXES	97
F.V. Simão, H. Chambart, L. Vandemeulebroeke, V. Cappuyns SUSTAINABLE USE OF SULFIDIC TAILING RESIDUES IN THE PRODUCTION OF CERAMIC ROOF TILES	97
A. Skawińska, P. Pichaniarczyk, Ł. Kotwica THE EFFECT OF LIGHTWEIGHT AGGREGATES FROM SEWAGE SLUDGE ON TOBERMORITE FORMATION IN CaO-SiO ₂ -H ₂ O SYSTEM UNDER HYDROTHERMAL CONDITIONS	98
L.J. Jaramillo Nieves, F. Elyseu, E. Zabet, M. Souza, L. Zilli, M. Damazio, S. Martins, G.C. Bellettini, S. Goulart, A.M. Bernardin SYNTHESIS OF COAL ASH BASED GEOPOLYMERS: INFLUENCE OF NaOH CONCENTRATION AND SOLID FRACTION	99
J. Ramult, K. Tyrała, R. Prorok, D. Madej THE EFFECT OF TEMPERATURE ON THE PROPERTIES OF NON-CEMENT ALUMINA-SPINEL CASTABLE	100
J. Rakić, Z. Baščarević, R. Petrović, S. Kovač POSSIBILITY TO USE SPENT FLUID CATALYTIC CRACKING CATALYST AS COMPONENT OF PORTLAND CEMENT BINDERS	101
R. Kusiorowski EFFECT OF TITANIUM OXIDE ADDITION INTO MAGNESIA REFRACTORIES ..	101
H. Ünsal, I. Shepa, O. Hanzel, E. Múdra, J. Dusza, P. Tatarko THE EFFECT OF FIELD ASSISTED SINTERING PARAMETERS ON PROCESSING OF IN-SITU FORMED B ₄ C-TiB ₂ CERAMICS	102
G. Can Tatlisu, C. Aciksari, Y. Teke, C. Karakaya, O. Akarcay, E. Keles, S. Celebi, S. Turan FABRICATION OF POWDER BASED CERAMIC INSULATION MATERIAL FOR HIGH TEMPERATURE APPLICATIONS	103
N. Gilli, A. Migliori, J. Watts, W.G. Fahrenholtz, G.E. Hilmas, L. Silvestroni EFFECT OF ANNEALING ON THE STRENGTH RETENTION AT ULTRA-HIGH TEMPERATURE	104
A. Martiz, A.M. Keszler DEVELOPMENT OF ZrC-BASED CERAMIC NANOCOMPOSITES	105
J. Zou, M. Porter, X.-Y. Lu, Y.-L. Chiu, J. Binner CHARACTERIZATION OF SiC _p /SiC COMPOSITES DENSIFIED BY MICROWAVE ASSISTED CHEMICAL VAPOUR INFILTRATION	105
Ö. Ulaş Kudu, T. Famprakis, E. Suard, M.-D. Braidă, T.L. Mercier, B. Fleutot, C. Masquelier INORGANIC Li-ION CONDUCTORS FOR ALL SOLID STATE BATERIES: CRYSTAL CHEMISTRY AND TRANSPORT PROPERTIES IN THE Li ₂ S-P ₂ S ₅ SYSTEM	106

I. Podunavac, M. Radović, J. Vidić, G. Kitić, V. Radonić ULTRA-SENSITIVE ELECTROCHEMICAL BIOSENSOR REALIZED USING LTCC TECHNOLOGY	108
A. Robles, A. Orera, R.I. Merino, P.R. Slater SUITABILITY OF Sr AND Co-FREE LANTHANUM PEROVSKITE MATERIALS AS CATHODES FOR IT-SOFC WITH A LANTHANUM SILICATE APATITE-TYPE ELECTRODE	109
A.G. Glukharev, O.Y. Kurapova, V.G. Konakov THE DEVELOPMENT OF THE NOVEL TERNARY Y_2O_3 - CeO_2 - ZrO_2 AND TiO_2 - CeO_2 - ZrO_2 SOLID ELECTROLYTES VIA CRYOCHEMICAL ROUTE	110
L. Ajdys, A. Żurawska, Y. Naumovich PARTICLE SIZE INFLUENCE ON THE FORMING OF THIN SPINEL LAYERS ON A STEEL SUBSTRATE FOR SOFC APPLICATION	111
I. Polishko, Y. Brodnikovskiy, N. McDonald, D. Brodnikovskiy, I. Brodni- kovska, M. Brychevskiy, N. Lysunenko, L. Kovalenko, O. Vasylyev, A. Belous, R. Steinberger-Wilckens PROPERTIES OF ADVANCED YTTRIA- AND SCANDIA-STABILIZED ZIRCONIA COMPOSITE FOR SOLID OXIDE FUEL CELL APPLICATION	112
A. Novokhatska, G. Akimov, L. Kovalenko THE STUDY OF CERAMIC COMPOSITES BASED ON ZIRCONIA AND MANGANITE WITH EXCESS MANGANESE FOR CATHODE OF SOFCs	113
F. Elyseu, L.J. Jaramillo Nieves, B. Nascimento de Souza, A.M. Bernardin, A.G. Dal Bó ELECTRICAL PROPERTIES OF SiO_2 - Na_2O - LiO_2 - Bi_2O_3 CONDUCTIVE GLASSES	114
A. Ressler, M. Antunović, M. Cvetnić, I. Marijanović, M. Ivanković, H. Ivanković BIOMIMETIC ZINC AND MAGNESIUM SUBSTITUTED CALCIUM PHOSPHATES DERIVED FROM BIOGENIC SOURCE	115
M. Rashwan, N. Karpukhina, R.G. Hill THE EFFECT OF THE PARTICLE SIZE ON SYNTHESIS AND CRYSTALLIZA- TION OF POTASSIUM FLUORMICA GLASS-CERAMICS	116
T. Matić, M. Ležaja Zebić, Miletić, R. Petrović, Đ. Janačković, Đ. Veljović THE COMPARISON OF THE BONDING ABILITY OF DENTAL INSERTS BASED ON STRONTIUM AND MAGNESIUM DOPED HYDROXYAPATITE WITH RESTORATIVE MATERILAS	117
C. Ianasi, A.-M. Putz EVALUATION OF THE MORPHO-TEXTURAL AND MAGNETIC PARAMETERS OF THE SiO_2 - Fe_2O_3 SYSTEM	118
M. Mladenović, N. Ilić, G. Kaluderović, N. Knežević pH-RESPONSIVE DELIVERY OF ANTICANCER METAL COMPLEXES BY FUNCTIONALIZED MESOPOROUS SILICA-BASED NANOCARRIERS	119

V.S. Buinevich, A.A. Nepapushev, G.V. Trusov, D.O. Moskovskikh, A.S. Rogachev, A.S. Mukasyan SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS AND MECHANOCHEMICAL SYNTHESIS OF ULTRA-HIGH TEMPERATURE Hf-C-N CERAMICS FOR EXPLOITATION IN EXTREME CONDITIONS	120
D. Kozień, M.M. Bućko SINTERING OF BORON-RICH CARBIDE POWDERS	121
V.K. Manisa, T. Williams, L. Naicker, M. J. Whiting, R.A. Dorey TAILORING A NEAR ZERO TEMPERATURE COEFFICIENT OF RESISTANCE IN PRESSURELESS SINTERED MWCNT/Al ₂ O ₃ AND GPO/Al ₂ O ₃ COMPOSITES ..	122
V. Pavkov, G. Bakić, V. Maksimović, M. Mirković, J. Luković¹, A. Maslarević, B. Matović SYNTHESIS AND CHARACTERIZATION OF METAL-GLASS COMPOSITE MATERIAL	122
Y. Tabak, A. Kılıç, Ş. Polat, B.K. Vatansever, H. Ünsal PRODUCTION OF Si ₃ N ₄ CERAMIC TAPES BY TAPE CASTING METHOD FOR ARTIFICIAL BONE APPLICATION	123
P. Piotrkiewicz, J. Zygmuntowicz, A. Miazga, M. Wachowski, W. Kaszuwara THE Al ₂ O ₃ -Cu-Cr COMPOSITES – MICROSTRUCTURE AND SELECTED PROPERTIES	124
S. Bakan, H. Boussebha, B. Özdemir, A.O. Kurt SYNTHESIS OF B ₄ C POWDER VIA DYNAMIC THERMOCHEMICAL METHOD	125
T. Lomakina, O. Kurapova, V. Konakov HYDROLYSIS PROCESSES AND PHASE EQUILIBRIA IN ZrO ₂ AND 4Y ₂ O ₃ - 96ZrO ₂ PRECURSORS (mol%), OBTAINED BY REVERSE CO-PRECIPIATION	126
M.M. Ismail, D.A. Rayan STRUCTURE, MAGNETIC PROPERTIES AND INDUCTION HEATING ABILITY STUDIES FOR HYPERTHERMIA TREATMENT OF TUMORS BY Mn SUBSTITUTED CuFe ₂ O ₄ NANOPARTICLES	127
S. Laketić, M. Rakin, M. Momčilović, J. Ciganović, Đ. Veljović, I. Cvijović-Alagić LASER SURFACE MODIFICATION OF CP-Ti IN DIFFERENT GAS ATMOSPHERES	128
A. Pahomi, V. Chiriac, G. Vlase, T. Vlase, P. Albu, G. Ilia STRUCTURAL AND THERMIC INVESTIGATION ON A NOVEL METHOD OF SYNTHESIS FOR HYDROXYAPATITE	129
M. Mirković, M. Stojmenović, S. Erić, A. Rosić, S. Nenadović, L. Kljajević, B. Matović SYNTHESIS, STRUCTURE AND THE POSSIBILITY OF USING DIFFERENT MATERIALS FROM THE CALCIUM PHOSPHATE GROUP	130

M. Pantović Pavlović, M. Pavlović, S. Eraković, T. Barudžija, J. Stevanović, N. Ignjatović, V. Panić <i>IN SITU</i> SYNTHESIS AND CHARACTERIZATION OF HYDROXYAPATITE/ TITANIUM OXIDE COATINGS DERIVED BY ANODIZATION AND ANAPHORETIC DEPOSITION	131
J. Vujančević, A. Bjelajac, G. Provatas, Z. Siketić, M. Jakšić, V. Pavlović, Đ. Janačković ToF-ERDA/RBS ANALYSIS OF ANNEALED TiO ₂ NANOTUBES GROWN ONTO FTO GLASS	132
Z.Z. Vasiljevic, M. Dojcinovic, I. Jankovic-Castvan, J. Vujancevic, N. Tadic, M.V. Nikolic STRUCTURE AND PHOTOCATALYTIC PROPERTIES OF SOL-GEL SYNTHESIZED PSEUDOBROOKITE	133
C. Belkessam, M. Mechouet, J. Ghilane, N. Djelali, N. Idiri PRECURSORS EFFECT ON Ni _{0.3} Co _{2.7} O ₄ OXIDE ELECTROCATALYTIC ACTIVITY	133
P. Szoldra, W. Pichór TiO ₂ -BASED PHOTOCATALYTIC COATING FOR CERAMIC MATERILAS	134
P. Svera, C. Ianasi STUDY ON THE PHOTOCATALYTIC BEHAVIOR OF Zn BASED SEMICONDUCTOR IN THE PRESENCE OF ORGANIC STABILIZATOR AND CO-CATALYST ..	135
M. Dojcinovic, Z. Z. Vasiljevic, J. Vujancevic, V.P. Pavlovic, S. Markovic, N.B. Tadic, M.V. Nikolic VISIBLE LIGHT PHOTOCATALYTIC ACTIVITY OF NANOCRYSTALLINE Co _x Mg _{1-x} Fe ₂ O ₄ (x = 0-1)	136
S. Ilies, C. Ianasi, F. Manea, A.Baciu, C. Delcioiu HUMIC ACID REMOVAL FROM WATER USING A-Fe ₂ O ₃ NANOMATERIAL BY SORPTION AND PHOTOCATALYTIC UNDER VIS IRRADIATION	136
A. Džunuzović, M. Vijatović Petrović, J. Bobić, N. Ilić, B. Stojanović INFLUENCE OF FERRITES PHASE ON PROPERTIES OF THE BARIUM ZIRCONIUM TITANATE BASED MULTIFERROIC COMPOSITES	137
S.R. Balauca, L. Padurariu, L. Mitoseriu FINITE DIFFERENCE METHOD FOR MODELLING THE DIELECTRIC PROPERTIES OF CERAMIC COMPOSITES	138
P. Zachariasz, P. Pęczkowski, P. Konieczny, K. Kluczevska-Chmielarz, P. Czaja, D. Bochenek, S. Baran MAGNETIC AND FERROELECTRIC PROPERTIES OF SCTO-(C,M)FO MULTIFERROICS	139

P. Pęczkowski, P. Zachariasz, C. Jastrzębski, B.C. Camargo, J. Szczytko, M. Muzyk CHARACTERIZATION OF SUPERCONDUCTOR ($\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$) – MULTI-FERROIC (YMnO_3) HETEROSTRUCTURES	140
M. Jahanara Mohammad, H. Ramachandran, P. Swaminathan ELECTRICAL CHARACTERIZATION OF METAL OXIDE COMPOSITES PREPARED THROUGH PHYSICAL AND CHEMICAL ROUTES	141
M. Parfenova, V. Lutsyk CERAMICS MICROSTRUCTURE SIMULATION ON THE ISOTHERMAL SECTIONS OF T-x-y DIAGRAM BY MEANS OF EXCEL SHEETS AND AutoCAD SOFTWARE	142
D. Kostić, D. Balaban, M. Perušić, B. Pejović PROCESS PARAMETERS OPTIMIZATION OF ALUMINIUM-TRIHIDRATE SYNTHESIS	143
K. Kaczmarczyk, J. Michalek, K. Pasiut, J. Partyka EFFECT OF ZrSiO_4 ADDITION ON SINTERING OF GLASS-CERAMIC MATERIALS FROM THE $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O-K}_2\text{O-CaO-MgO-BaO}$ SYSTEM	144
A. Duša, I. Podunavac, S. Kojić, V. Radonić MICROFLUIDIC SENSORS REALIZED USING LOW-TEMPERATURE CO-FIRED CERAMIC TECHNOLOGY	144
C. Belkessam, M. Mechouet, J. Ghilane, N. Djelali, N. Idiri $\text{Ni}_{0.3}\text{Co}_{2.7}\text{O}_4$ SPINEL OXIDE IMMOBILIZED IN TEFLON CAVITY ELECTRODE FOR ENVIRONMENTAL APPLICATIONS	145
J. Michalek, K. Kaczmarczyk, K. Pasiut, J. Partyka PRIMARY RESEARCH ON PORCELAIN WASTE UTILISATION	146
Đ. Kerkez, D. Radenović, D. Tomašević Pilipović, N. Slijepčević, M. Bečelić-Tomin, D. Krčmar, B. Dalmacija MICROSTRUCTURAL CHARACTERIZATION OF SOLIDIFIED/STABILIZED MATRICES OF POLLUTED SEDIMENT AND CLAY IN LONG-TERM TREATMENT	146
K. Rajczyk, A. Kaliciak, G. Janus, D. Brukhanska CEMENTLESS CONCRETE COMPOSITE FROM KAOLINITE AND ALUMINO-SILICATE WASTE MATERIALS	147
T. Labbilita, M.A. Hareh STUDY OF THE EFFECT OF CALCIUM SUBSTITUTION BY MAGNESIUM IN A GLASS-CERAMIC FERTILIZER	148
J. Gonciarzyk, K. Stec, A. Bobrowski, A. Królicka APPLICATION OF ICP AES METHOD AND MICROWAVE DIGESTION TO ROUTINE ANALYSIS OF CORUNDUM MATERIALS	149
Y.V.Yurchenko O.A. Korniienko, O.I. Bykov, A.V. Sameljuk, E.R. Andrievskaya PHASE RELATIONS AND ADVANCED MATERIALS IN THE $\text{ZrO}_2\text{-La}_2\text{O}_3\text{-Gd}_2\text{O}_3$ SYSTEM	150

A. Oufakir, M. Afqir, N. Tahiri, M. Elaatmani CONTRIBUTION TO THE IMPROVEMENT OF THE PROPERTIES OF SiO ₂ - BASED POLYMER COMPOSITE MATERIALS	151
E.S. Karaimchuk, V.A. Maslyuk, V.T. Varchenko THE EFFECT OF TITANIUM BORIDE ADDITIVES ON THE MECHANICAL AND TRIBOLOGICAL PROPERTIES OF MATERIALS IN Fe-Cr-C SYSTEM	152

INDEX OF AUTHORS

13th Conference for Young Scientists in Ceramics, CYSC-2019
Novi Sad, Serbia, October 16-19, 2019

<p>PROGRAMME</p>

WEDNESDAY, OCTOBER 16, 2019.

09.00 – 11.00 h – Registration (Conference desk)

11.00 – 11.30 h – Oppening (Rectorate-Amphitheater)

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

Jean-Oliver Durand, *France*, Mesoporous silica, periodic mesoporous organosilica, and mesoporous silicon nanoparticles for drug delivery and two-photon Photodynamic Therapy

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

13.15 – 14.00 h – IL-2 Invited lecture (Rectorate-Amphitheater)

Markus Winterer, *Germany*, Learning about nanoparticles by X-ray absorption spectroscopy

14.00 – 14.45 h – IL-3 Invited lecture (Rectorate-Amphitheater)

Ákos Kukovecz, *Hungary*, Key elements of a successful H2020 Marie-Sklodowska Curie individual fellowship application

14.45 – 15.15 h – Coffe Break (Blue Hall & Class Room, R12)

15.15 – 16.45 h – Section 1

Synthesis of ceramic powders - 1 (Blue Hall)

15.15 – 15.30 h **OA-1 A. Levish, *et al.*, Germany**

Iron(III)acetylacetonate vapor studied by X-ray absorption

15.30 – 15.45 h **OC-2 T.S.R.C. Murthy, *et al.*, UK/India**

Sol-gel-based interfacial coatings of rare earth oxides on 2.5D carbon fibre preforms

15.45 – 16.00 h **OA-3 R. Nicola, *et al.*, Romania**

Magnetic nanocomposites containing iron oxide@SiO₂ obtained via conventional or ultrasonic method

16.00 – 16.15 h **OA-4 J. Aleksić, *et al.*, Serbia**

Synthesis, structural and magnetic properties of Y_{1-x}Yb_xF₃ solid solution

16.15 – 16.30 h **OA-5 J. Schulte, *et al.*, Germany**

Laser-Flash-Evaporation of solid metal organic precursors

16.30 – 16.45 h **OC-6 I. Milenković, *et al.*, Serbia**

Coating of cerium oxide nanoparticles with different carbohydrates and their application on plants

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

Ceramic processing - 1 (Class Room, R12)

- 15.15 – 15.30 h **CC-7 J. Zygmuntowicz, *et al.*, Poland**
Processing and characterization of Al₂O₃-Cu-Ni composites
- 15.30 – 15.45 h **CC-8 M. Wachowski, *et al.*, Poland**
Effect of metal particles size on microstructure of ZrO₂-Ni composite fabricated by CSC in magnetic field
- 15.45 – 16.00 h **CC-9 K. Dudek, *et al.*, Poland**
Fabrication and characterization of multifunctional hybrid layers on NiTi shape memory alloy
- 16.00 – 16.15 h **OA-10 M. Vukšić, *et al.*, Croatia**
Influence of the addition of waste alumina powder on mechanical properties of alumina ceramics
- 16.15 – 16.30 h **OA-11 I. Nomel, *et al.*, France**
Elaboration of lead-free piezoelectric materials for thick films coating by aerosol deposition
- 16.30 – 16.45 h **OA-12 J. Stanojev, *et al.*, Serbia**
Processing of nanostructured CNT-based thin film electrode

16.45 – 17.00 h – Coffe Break (Blue Hall & Class Room, R12)

17.00 – 18.45 h – Section 2

Porous ceramic (Blue Hall)

- 17.00 – 17.15 h **OA-13 E. Eray, *et al.*, Denmark**
New generation silicon carbide membranes for water purification
- 17.15 – 17.30 h **OA-14 A. Policicchio, *et al.*, Italy**
Organically functionalised mesoporous silica synthetized for hydrogen storage application
- 17.30 – 17.45 h **OA-15 P.F. Großmann, *et al.*, Germany**
Direct Ink Writing as an additive manufacturing technique for shaping heterogeneous catalysts
- 17.45 – 18.00 h **OA-16 Á. Szamosvölgyi, *et al.*, Hungary**
Examination of the role and behaviour of mesoporous support materials in CO₂ hydrogenation
- 18.00 – 18.15 h **OA-17 G. Conte, *et al.*, Italy**
The hydrogen storage capacity of organically functionalised mesoporous silica
- 18.15 – 18.30 h **OA-18 A.-M. Putz, *et al.*, Romania**
Organically functionalised mesoporous silica with gas storage properties synthesis and characterization
- 18.30 – 18.45 h **OA-19 J. Mrówka, *et al.*, Poland**
Thermal properties and ceramic yield of porous poly(methyl-vinylsiloxane) cross-linked with 1,3,5,7-tetramethylcyclotetra-siloxane before and after deposition of palladium nanoparticles

Synthesis of ceramic powders - 2 (Class Room, R12)

- 17.00 – 17.15 h **OA-20 E. İşlek, *et al.*, Turkey**
Nano-sized magnesium spinel powders synthesized via flame spray pyrolysis
- 17.15 – 17.30 h **OA-21 V. Mackert, *et al.*, Germany**
Probing sonication of colloidal SnO₂ by *in situ* Small-Angle X-ray Scattering
- 17.30 – 17.45 h **OA-22 B. Özdemir, *et al.*, Turkey**
Synthesis of titanium nitride powder from rutile and anatase
- 17.45 – 18.00 h **OA-23 M. Temnikova, *et al.*, Russia**
Synthesis, phase composition and microstructure of ZrO₂-Y₂O₃-rGO composite precursors obtained by sol-gel synthesis
- 18.00 – 18.15 h **OA-24 D.A. Rayan, *et al.*, Egypt**
Comparative study of La and Zn ions co-doped magnetite nanostructures synthesized in oxygen and inert atmosphere
- 18.15 – 18.30 h **OA-25 J. Geiss, *et al.*, Germany**
Structural characterization of LaFeO₃ perovskite nanoparticles
- 18.30 – 18.45 h **OA-26 I. Dinic, *et al.*, Serbia**
Effects of citric ion on hexagonal NaYF₄: Yb/Er phase formation during solvothermal synthesis

THURSDAY, OCTOBER 17, 2019.

09.15 – 11.15 h – Section 3

Optics (Blue Hall)

09.15 – 10.00 h **IL-4 Invited lecture - Lucjan Kozielski, Poland,**
Mechano-luminescent materials for direct conversion of
mechanical energy into light

Optics (Blue Hall)

10.00 – 10.15 h **OA-27 S. Hříbalová, et al., Czech Republic**
Light scattering in transparent ceramics: Use and validity of
Mie theory approximations

10.15 – 10.30 h **OA-28 Y.H. Elbasha, et al., Egypt**
Color and optical spectroscopic analysis of Cu₂O-K₂O-ZnO-
P₂O₅ glass matrix

10.30 – 10.45 h **OC-29 D. Danilović, et al., Serbia**
Photoemission spectroscopy on isolated silver-based hybrid
nanostructures: A novel approach to understand fundamental
properties of solar cell absorber material

10.45 – 11.00 h **OC-30 D.A. Rayan, et al., Egypt**
Remarkable impact for optical properties of samarium doped
aluminum sodium phosphate glass for visible devices

11.00 – 11.15 h **OC-31 A. Alzahrani, Saudi Arabia**
Effect of sintering temperature on crystallisation of nepheline-
leucite glass-ceramics

Engineering and refractory ceramics - I (Class Room, R12)

09.15 – 09.30 h **OC-32 P. Staciwa, et al., Poland**
Preparation of iron/carbon composites

09.30 – 09.45 h **OA-33 A. Zavjalov, et al., Russia**
Spark plasma sintering of Hf-C-N ceramics

09.45 – 10.00 h **OC-34 M. Kosiorek, et al., Poland**
Rheological properties of glass-ceramic seals for SOC
application fabricated by tape casting

10.00 – 10.15 h **OA-35 L. Rakoczy, et al., Poland**
Application of shell molds strengthened by metal powders and
glass fibres in the fabrication of jet engines components

10.15 – 10.30 h **OA-36 D. Tovar-Vargas, et al., Spain**
Processing, characterization and mechanical properties of ce-
ria-calcia stabilized zirconia ceramics with α -alumina addition

10.30 – 10.45 h **OA-37 L. Vásárhelyi, et al., Hungary**
In situ investigation of crack propagation in ceramics by
micro-CT technique

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

- 10.45 – 11.00 h **OA-38 A. Sedegov, *et al.*, Russia**
Selfpropagating high temperature synthesis and study of
carbide and diboride based on refractory high-entropy alloy
Hf-Ta-Ti-Nb-Zr
- 11.00 – 11.15 h **OC-39 A. Qadir, *et al.*, Hungary**
Processing and properties of silicon nitride + MWCNTs
composites prepared from oxidized α -Si₃N₄ starting powder

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

11.45 – 12.30 h – PL-1 Plenary lecture (Rectorate-Amphitheater)

Jon Binner, *United Kingdom*, Designing the processing of
advanced ceramics and composites to yield the required properties

12.30 – 13.15 h – IL-5 Invited lecture (Rectorate-Amphitheater)

Paula Vilarinho, *Portugal*, Alternative sintering routes of
electroceramics: The case of sodium potassium niobate, a lead free
piezoelectrics

13.15 – 14.00 h – IL-6 Invited lecture (Rectorate-Amphitheater)

Davide Bossini, *Germany*, Ultrafast optical control of magnetism in
solids

14.00 – 15.15 h – Lunch (Faculty of Technology-Ceremonial Hall)

15.15 – 16.45 h – Section 4

Ceramic processing - 2 (Blue Hall)

- 15.15 – 15.30 h **OA-40 V. Nečina, *et al.*, Czech Republic**
The effect of heating rate on the grain growth of alumina
prepared via electric current assisted sintering (ECAS)
- 15.30 – 15.45 h **OA-41 T. Boteva, *et al.*, Bulgaria**
Diamond electrodes for electrochemical applications
- 15.45 – 16.00 h **OC-42 C. Gorynski, *et al.*, Germany**
Influence of electric current on microstructure of Al doped
ZnO
- 16.00 – 16.15 h **OC-43 R.M. Guillén Pineda, *et al.*, Spain**
Properties of Nb₂O₅ composite doped with ZrO₂ sintered by
microwave
- 16.15 – 16.30 h **OA-44 N. Fakhralmobasheri, *et al.*, Iran**
Effect of two-step sintering on microstructure of barium
titanate ceramics
- 16.30 – 16.45 h **OC-45 M. Abedi, *et al.*, Russia**
Fabrication of silicon nitride reinforced by silicon carbide by
flash spark plasma sintering

Electroceramics - 1 (Class Room, R12)

- 15.15 – 15.30 h **OA-46 M. Čebela, *et al.*, Serbia/Croatia**
Structural and magnetic properties of multiferroic BiFeO₃ and Bi_{1-x}Ho_xFeO₃
- 15.30 – 15.45 h **OA-47 D. Szalbot, *et al.*, Poland**
Magnetoelectric properties of multiferroic Aurivillius type Bi₇Fe₃Ti₃O₂₁ ceramics modified by strontium
- 15.45 – 16.00 h **OA-48 P. Czaja, *et al.*, Poland**
Influence of ball milling time on preparation and dielectric properties of lead free K_{0.5}Bi_{0.5}TiO₃ ceramics
- 16.00 – 16.15 h **OC-49 D. Blaskova-Kochntcharova, *et al.*, Bulgaria**
Structural and electrical properties of TiO₂-V₂O₅-P₂O₅ oxides
- 16.15 – 16.30 h **OC-50 I. Turcan, *et al.*, Romania**
Ag-BaTiO₃ composite ceramics with multiple percolative behavior
- 16.30 – 16.45 h **OA-51 A. Nesterovic, *et al.*, Serbia**
Preparation and characterization of Bi_{0.5}Na_{0.5}TiO₃ based piezoelectric ceramics

16.45 – 17.00 h – Coffe Break (Blue Hall & Class Room, R12)

17.00 – 18.45 h – Section 5

Electroceramics - 2 (Blue Hall)

- 17.00 – 17.15 h **OA-52 V.A. Lukacs, *et al.*, Romania**
Scale dependent phenomena in BaTiO₃-based ceramics
- 17.15 – 17.30 h **OA-53 J. Dzik, *et al.*, Poland**
Influence of rare earth ions on the properties of bifeo₃ ceramics
- 17.30 – 17.45 h **OA-54 V.K. Veerapandiyam, *et al.*, Austria**
Role of polar defects in the origin of relaxor behaviour in niobium modified barium titanate polycrystals
- 17.45 – 18.00 h **OA-55 D. Brzezińska, *et al.*, Poland**
The properties of (1-x)(0.5PZT-0.5PFW)-xPFN ceramics doped by Li
- 18.00 – 18.15 h **OA-56 L. Eglite, *et al.*, Latvia**
Effect of sintering parameters and non-stoichiometry on the microstructure of Na_{0.5}Bi_{0.5}TiO₃
- 18.15 – 18.30 h **OA-57 M. Bara, *et al.*, Poland**
Dielectric and electrical properties BLT ceramics modified by Fe ions
- 18.30 – 18.45 **OA-58 J. Lelièvre, *et al.*, France**
Structure and properties of the lead-free perovskite compounds (Na_{1/2}Bi_{1/2})ZrO₃ (NBZ) and (K_{1/2}Bi_{1/2})ZrO₃ (KBZ)

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

Ceramic processing - 3 (Class Room, R12)

- 17.00 – 17.15 h **OA-59 M. Stan, *et al.*, Poland**
Sintering and preparation of polycrystals gadolinium- iron garnet ($\text{Gd}_3\text{Fe}_5\text{O}_{12}$) by the solid-state reaction
- 17.15 – 17.30 h **OA-60 A.U. Rehman, *et al.*, China/Turkey**
Additive manufacturing of magnesia and alumina eutectic composites
- 17.30 – 17.45 h **OA-61 C. Aciksari, *et al.*, Turkey**
Synthesis of zinc tin oxide (Zn_2SnO_4) particles by various methods used as ceramic target for sputter technique
- 17.45 – 18.00 h **OA-62 S.K. Behara, *et al.*, India**
Theoretical investigation of crystal structure prediction using Bond-valence (BV) modelling

Catalists - 1 (Class Room, R12)

- 18.00 – 18.15 h **OA-63 H.M. Bui, *et al.*, Germany**
Challenges for binder jetting of packings and monoliths for heterogeneous catalysis
- 18.15 – 18.30 h **OA-64 N. Ilić, *et al.*, Serbia**
Photocatalytic properties of BiFeO_3 and $\text{Bi}_5\text{Ti}_3\text{FeO}_{15}$ based powders
- 18.30 – 18.45 h **OA-65 M. Miklos, *et al.*, Hungary**
Immobilization of simulated radioisotopes in alkali activated inorganic polymers

20.00 h – Social Event

FRIDAY, OCTOBER 18, 2019.

09.15 – 11.15 h – Section 6

Catalysts - 2 (Blue Hall)

09.15 – 10.00 h **IL-7 Invited lecture – Igor Djerdj, Croatia**, The application of Ce-Zr-O compounds in HCl oxidation and in three-way catalysis

Catalysts - 2 (Blue Hall)

10.00 – 10.15 h **OA-66 A. Sápi, Hungary**
Catalysis revolution: with nanotechnology and molecular level understanding towards a green

10.15 – 10.30 h **OA-67 M. Kocijan, et al., Croatia**
Synthesis, characterization and photocatalytic properties of TiO₂-rGO nanoparticles

10.30 – 10.45 h **OC-68 A. Efremova, et al., Hungary**
Pt nanoparticles-supported and pristine mesoporous metal oxides as efficient catalysts for CO₂ activation

10.45 – 11.00 h **OA-69 M.M. Uzelac, et al., Serbia**
Optimization of the photocatalytic degradation of thiotriazinone stable hydrolysis product of antibiotic ceftriaxone

11.00 – 11.15 h **OC-70 E.-M. Picioruș, et al., Romania**
Adsorption studies of cadmium (II) from aqueous solutions using inorganic porous nanocomposites

Traditional ceramics - 1 (Class Room, R12)

09.15 – 09.30 h **OT-71 A. Boros, et al., Hungary**
Photochemical activity of metakaolin based geopolymer foam

09.30 – 09.45 h **OT-72 A. Kovács, et al., Hungary**
Preparation of kaolinite-methanol and kaolinite-cetyltrimethylammonium chloride complexes

09.45 – 10.00 h **OT -73 F.V. Simão, et al., Belgium**
Sustainable use of sulfidic tailing residues in the production of ceramic roof tiles

10.00 – 10.15 h **OT -74 A. Skawińska, et al., Poland**
The effect of lightweight aggregates from sewage sludge on tobermorite formation in CaO-SiO₂-H₂O system under hydrothermal conditions

10.15 – 10.30 h **OT -75 L.J. Jaramillo Nieves, et al., Brazil**
Synthesis of coal ash based geopolymers: Influence of NaOH concentration and solid fraction

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

- 10.30 – 10.45 h **OT -76 J. Ramult, *et al.*, Poland**
The effect of temperature on the properties of non-cement alumina-spinel castables
- 10.45 – 11.00 h **OT -77 J. Rakić, *et al.*, Serbia**
Possibility to use spent fluid catalytic cracking catalyst as component of Portland cement binders
- 11.00 – 11.15 h **OT -78 R. Kusiorowski, Poland**
Effect of titanium oxide addition into magnesia refractories

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

11.45 – 12.30 h – IL-8 Invited lecture (Rectorate-Amphitheater)

Vincenzo Buscaglia, *Italy*, Size and scaling effects in ferroelectric ceramics

12.30 – 13.15 h – IL-9 Invited lecture (Rectorate-Amphitheater)

Subramshu S. Bhattacharya, *India*, Phase-pure multicomponent equimolar oxides as a novel class of functional materials: synthesis, characterisation and properties

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

14.30 – 16.30 h – Section 7

Engineering and refractory ceramics - 2 (Blue Hall)

- 14.30 – 15.15 h **IL-10 Invited lecture - Laura Silvestroni, *Italy***, The importance of TEM analyses for the development of UHTCs

Engineering and refractory ceramics - 2 (Blue Hall)

- 15.15 – 15.30 h **OC-79 H. Ünsal, *et al.*, Slovakia**
The effect of field assisted sintering parameters on processing of in-situ formed B₄C-TiB₂ ceramics
- 15.30 – 15.45 h **OC-80 G. Can Tatlisu, *et al.*, Turkey**
Fabrication of powder based ceramic insulation material for high temperature applications
- 15.45 – 16.00 h **OC-81 N. Gilli, *et al.*, Italy**
Effect of annealing on the strength retention at ultra-high temperature
- 16.00 – 16.15 h **OC-82 A. Martiz, *et al.*, Hungary**
Development of ZrC-based ceramic nanocomposites
- 16.15 – 16.30 h **OC-83 J. Zou, *et al.*, United Kingdom**
Characterisation of SiC_f/SiC composites densified by microwave assisted chemical vapour infiltration

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

Electroceramics - 3 (Class Room, R12)

- 14.30 – 14.45 h **OA-84 Ö. Ulaş Kudu, *et al.*, France**
Inorganic Li-ion conductors for all solid state batteries:
Crystal chemistry and transport properties in the $\text{Li}_2\text{S}-\text{P}_2\text{S}_5$ system
- 14.45 – 15.00 h **OA-85 I. Podunavac, *et al.*, Serbia**
Ultra-sensitive electrochemical biosensor realized using LTCC technology
- 15.00 – 15.15 h **OA-86 A. Robles, *et al.*, Spain**
Suitability of Sr and Co-free lanthanum perovskite materials as cathodes for IT-SOFC with a lanthanum silicate apatite-type electrolyte
- 15.15 – 15.30 h **OA-87 A.G. Glukharev, *et al.*, Russia**
The development of the novel ternary $\text{Y}_2\text{O}_3\text{-CeO}_2\text{-ZrO}_2$ and $\text{TiO}_2\text{-CeO}_2\text{-ZrO}_2$ solid electrolytes via cryochemical route
- 15.30 – 15.45 h **OC-88 L. Ajdys, *et al.*, Poland**
Particle size influence on the forming of thin spinel layers on a steel substrate for SOFC application
- 15.45 – 16.00 h **OC-89 I. Polishko, *et al.*, Ukraine**
Properties of advanced yttria- and scandia-stabilized zirconia composite for solid oxide fuel cell application
- 16.00 – 16.15 h **OC-90 A. Novokhatska, *et al.*, Ukraine**
The study of ceramic composites based on zirconia and manganite with excess manganese for cathode of SOFCs
- 16.15 – 16.30 h **OA-91 F. Elyseu, *et al.*, Brazil**
Electrical properties of $\text{SiO}_2\cdot\text{Na}_2\text{O}\cdot\text{LiO}_2\cdot\text{Bi}_2\text{O}_3$ conductive glasses

16.30 – 16.45 h – Coffe Break (Blue Hall & Class Room, R12)

16.45 – 18.45 h – Section 8

Bioceramics - 1 (Blue Hall)

- 16.45 – 17.30 h **IL-11 Invited lecture - Nikola Knežević, *Serbia*,**
Core/shell mesoporous silica and organosilica nano-particles:
Synthesis, characterization and application in targeted cancer treatment

Bioceramics - 1 (Blue Hall)

- 17.30 – 15.30 h **OA-92 A. Ressler, *et al.*, Croatia**
Biomimetic zinc and magnesium substituted calcium phosphates derived from biogenic source
- 17.45 – 18.00 h **OA-93 M. Rashwan, *et al.*, United Kingdom**
The effect of the particle size on synthesis and crystallization of potassium fluormica glass-ceramics

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

- 18.00 – 18.15 h **OC-94 T. Matić, *et al.*, Serbia**
The comparison of the bonding ability of dental inserts based on strontium and magnesium doped hydroxyapatite with restorative materials
- 18.15 – 18.30 h **OA-95 C. Ianasi, *et al.*, Romania**
Evaluation of the morpho-textural and magnetic parameters of the SiO₂-Fe₂O₃ system
- 18.30 – 18.45 h **OC-96 M. Mladenović, *et al.*, Serbia**
pH-responsive delivery of anticancer metal complexes by functionalized mesoporous silica-based nanocarriers

Engineering and refractory ceramics - 3 (Class Room, R12)

- 16.45 – 17.00 h **OC-97 V.S. Buinevich, *et al.*, Russia**
Self-propagating high-temperature synthesis and mechanochemical synthesis of ultra-high temperature Hf-C-N ceramics for exploitation in extreme conditions
- 17.00 – 17.15 h **OA-98 D. Kozień, *et al.*, Poland**
Sintering of boron-rich boron carbide powder
- 17.15 – 17.30 h **OC-99 V.K. Manisa, *et al.*, United Kingdom**
Tailoring a near zero temperature coefficient of resistance in pressureless sintered MWCNT/Al₂O₃ and GPO/Al₂O₃ composites
- 17.30 – 17.45 h **OC-100 V. Pavkov, *et al.*, Serbia**
Synthesis and characterization of metal-glass composite material
- 17.45 – 18.00 h **OA-101 Y. Tabak, *et al.*, Turkey**
Production of Si₃N₄ ceramic tapes by tape casting method for artificial bone application
- 18.00 – 18.15 h **OC-102 P. Piotrkiewicz, *et al.*, Poland**
The Al₂O₃-Cu-Cr composites – Microstructure and selected properties
- 18.15 – 18.30 h **OA-103 H. Boussebha, *et al.*, Turkey**
Synthesis of B₄C powder via dynamic thermochemical method
- 18.30 – 18.45 h **OA-104 T. Lomakina, *et al.*, Russia**
Hydrolysis processes and phase equilibria in ZrO₂ and 4Y₂O₃-96ZrO₂ precursors (mol%), obtained by reversed co-precipitation

20.00 h – Conference dinner - Restaurant “Fontana”

Meeting of Young Ceramists Network of the European Ceramic Society -

Chairs: Maria Canillas Perez, *Spain* and Laura Taiainen, *Finland*

Invited lecture IL-12: Paula Vilarinho, *Portugal*, HOW TO TALK SCIENCE ... clearly

SATURDAY, OCTOBER 19, 2019.

09.00 – 10.30 h – Section 9

Bioceramics - 2 (Blue Hall)

- 09.00 – 09.15 h **OA-105 M.M. Ismail, *et al.*, Egypt**
Structure, magnetic properties and induction heating ability studies for hyperthermia treatment of tumours by Mn substitution CuFe₂O₄ nanoparticles
- 09.15 – 09.30 h **OA-106 S. Laketić, *et al.*, Serbia**
Laser surface modification of CP-Ti in different gas atmospheres
- 09.30 – 09.45 h **OA-107 A. Pahomi, *et al.*, Romania**
Structural and thermic investigation on a novel method of synthesis for hydroxyapatite
- 09.45 – 10.00 h **OC-108 M. Mirković, *et al.*, Serbia**
Synthesis, structure and the possibility of using different materials from the calcium phosphate group
- 10.00 – 10.15 h **OC-109 M. Pantović Pavlović, *et al.*, Serbia**
In situ synthesis and characterization of hydroxyapatite/titanium oxide coatings derived by anodization and anaphoretic deposition
- 10.15 – 10.30 h **OA-110 J. Vujančević, *et al.*, Serbia**
ToF-ERDA/RBS analysis of annealed TiO₂ nanotubes grown onto FTO glass

Catalysts - 3 (Class Room, R12)

- 09.00 – 09.15 h **OA-111 Z.Z. Vasiljevic, *et al.*, Serbia**
Structural and photocatalytic properties of sol-gel synthesized pseudobrookite
- 09.15 – 09.30 h **OA-112 C. Belkessam, *et al.*, Algeria**
Precursors effect on Ni_{0.3}Co_{2.7}O₄ oxide electrocatalytic activity
- 09.30 – 09.45 h **OA-113 P. Szoldra, *et al.*, Poland**
TiO₂-based photocatalytic coating for ceramic materials
- 09.45 – 10.00 h **OC-114 P. Svera, *et al.*, Romania**
Study on the photocatalytic behavior of Zn based semiconductor in the presence of organic stabilizator and co-catalyst
- 10.00 – 10.15 h **OA-115 M. Dojcinovic, *et al.*, Serbia**
Visible light photocatalytic activity of nanocrystalline Co_xMg_{1-x}Fe₂O₄ ($x = 0-1$)
- 10.15 – 10.30 h **OC-116 S. Ilies, *et al.*, România**
Humic acid removal from water using A-Fe₂O₃ nanomaterial by sorption and photocatalysis under Vis irradiatio

10.30 – 11.15 h – IL-13 Invited lecture (Blue Hall)

Cristina Leonelli, Italy, The magic of high power microwave processing of ceramic materials

11.15 – 11.30 h – Coffe Break (Blue Hall)

11.30 – 12.15 h – IL-14 Invited lecture (Blue Hall)

Nikola Kanas, Serbia, Oxide thermoelectrics - Current status and future prospects

12.15 – 13.30 h – Section 10

Electroceramics - 4 (Blue Hall)

- 12.15 – 12.30 h **OC-117 A. Džunuzović, et al., Serbia**
Influence of ferrites phase on properties of the barium zirconium titanate based multiferroic composites
- 12.30 – 12.45 h **OC-118 S.R. Balauca, et al., Romania**
Finite difference method for modeling the dielectric properties of ceramic composites
- 12.45 – 13.00 h **OC-119 P. Zachariasz, et al., Poland**
Magnetic and ferroelectric properties of SCTO-(C,M)FO multiferroics
- 13.00 – 13.15 h **OC-120 P. Pęczkowski, et al., Poland**
Characterization of superconductor (YBa₂Cu₃O_{7-δ}) - multiferroic (YMnO₃) heterostructures
- 13.15 – 13.30 h **OC-121 M. Jahanara Mohammad, et al., India**
Electrical characterization of metal oxide composites prepared through physical and chemical routes

Ceramics processing - 4 (Class Room, R12)

- 12.15 – 12.30 h **OA-122 M. Parfenova, et al., Russia**
Ceramics microstructure simulation on the isothermal sections of T-x-y diagram by means of Excel sheets and AutoCAD software
- 12.30 – 12.45 h **OA-123 D. Kostić, et al., Bosnia & Herzegovina**
Process parameters optimization of aluminium-trihydrate synthesis
- 12.45 – 13.00 h **OC-124 K. Kaczmarczyk, et al., Poland**
Effect of ZrSiO₄ addition on sintering of glass-ceramic materials form the SiO₂-Al₂O₃-Na₂O-K₂O-CaO-MgO-BaO system
- 13.00 – 13.15 h **OC-125 A. Duša, et al., Serbia**
Microfluidic sensors realized using low temperature Co-fired ceramic technology

13th Conference for Young Scientists in Ceramics, CYSC-2019

Novi Sad, Serbia, October 16-19, 2019

13.15 – 13.30 h **OC-126 M. Mechouet, *et al.*, Algeria**
Ni_{0.3}Co_{2.7}O₄ spinel oxide immobilized in teflon cavity
electrode for environmental applications

13.30 – 13.45 h – Coffe Break (Blue Hall & Class Room, R12)

13.45 – 15.00 h – Section 11

Traditional ceramics - 2 (Blue Hall)

13.45 – 14.00 h **OT-127 J. Michalek, *et al.*, Poland**
Primary research on porcelain waste utilisation

14.00 – 14.15 h **OT-128 Đ. Kerkez, *et al.*, Serbia**
Microstructural characterization of solidified/stabilized
matrices of polluted sediment and clay in long-term treatment

14.15 – 14.30 h **OT-129 K. Rajczyk, *et al.*, Poland**
Cementless concrete composite from kaolinite and alumino-
silicate waste materials

14.30 – 14.45 h **OT-130 T. Labbilita, *et al.*, Morocco**
Study of the effect of calcium substitution by magnesium in a
glass-ceramic fertilizer

Ceramics processing - 5 (Class Room, R12)

13.45 – 14.00 h **OA-131 J. Gonciarczyk, *et al.*, Poland**
Study of the effect of calcium substitution by magnesium in a
glass-ceramic fertilizer

14.00 – 14.15 h **OC-132 O.A. Korniienko, *et al.*, Ukraine**
Application of ICP AES method and microwave digestion to
routine analysis of corundum materials

14.15 – 14.30 h **OC-133 A. Oufakir, *et al.*, Morocco**
Contribution to the improvement of the properties of SiO₂-
based polymer composite materials

14.30 – 14.45 h **OC-134 E.S. Karaimchuk, *et al.*, Ukraine**
The effect of titanium boride additives on the mechanical and
tribological properties of materials in Fe-Cr-C system

14.45 – 15.00 h – Closing (Blue Hall)

13th Conference for Young Scientists in Ceramics, CYSC-2019
Novi Sad, Serbia, October 16-19, 2019

INVITED TALKS

Invited Lecture, IL-1

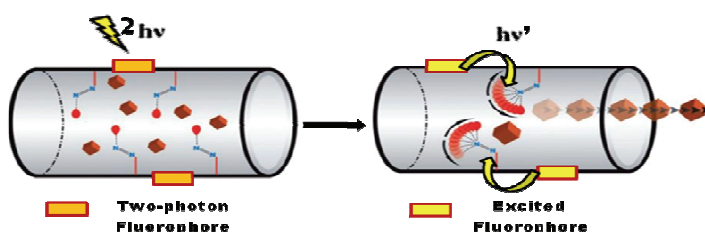
MESOPOROUS SILICA, PERIODIC MESOPOROUS ORGANOSILICA, AND MESOPOROUS SILICON NANOPARTICLES FOR DRUG DELIVERY AND TWO-PHOTON PHOTODYNAMIC THERAPY

Jean-Olivier Durand

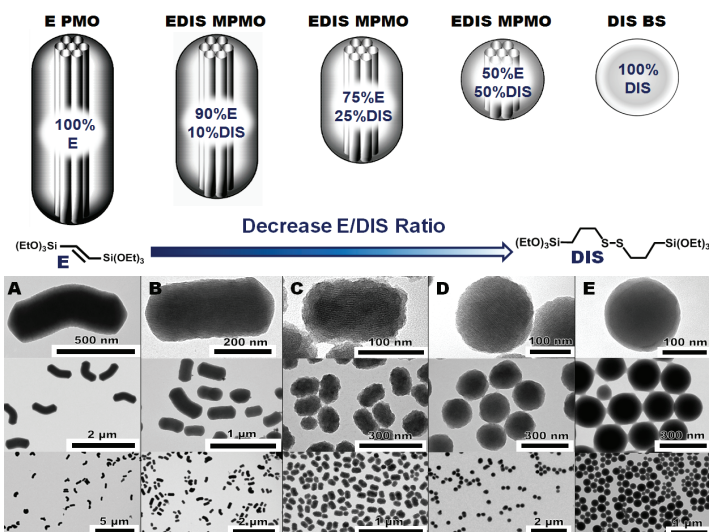
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Mesoporous silica nanoparticles (MSN) have attracted much attention the last decade for nanomedicine applications due to their biocompatibility, flexible functionalisation, tunable pore size and diameter. We describe here MSN engineered for two-photon triggered drug delivery or photodynamic therapy, in MCF-7 breast cancer cells. The two-photon triggered drug delivery system was based on a FRET mechanism from a two-photon dye in the walls of the MSN to an azobenzene moiety in the pores of the MSN (nanoimpellers, scheme 1). Concerning photodynamic therapy, a two-photon



Scheme 1. Two-photon nanoimpeller



Scheme 2. Biodegradable nanoPMOs

photosensitizer was encapsulated in the walls of the MSN. Two-photon photodynamic therapy was performed *in vitro* and *in vivo* on mice bearing colon xenografted tumors. We also studied the use of porous silicon nanoparticles (pSiNP) functionalized with both a photosensitizer and a targeting agent. Porous silicon is a biocompatible and biodegradable material which can generate $^1\text{O}_2$ when excited by visible light due to quantum-confinement effect. pSiNP had been shown to be degraded into non-toxic silicic acid byproducts *in vivo*. The multi-functionalized pSiNP studied here were able to target, image and kill cancer cells *in vitro* by photodynamic therapy mechanisms both with 1-photon and 2-photon excitation. Alternatively, the synthesis of disulfide-based biodegradable Periodic Mesoporous Organosilica Nanoparticles (nanoPMOs, scheme 2) was realized and the nanoparticles were efficient in delivering doxorubicin in cancer cells

References

- [1] O. Vaillant, J.-O. Durand, *et al.*, Mannose-6-phosphate receptor, A target for theranostics of prostate cancer, *Angew. Chem. Int. Ed.*, **54** (2015) 5952-5956.
- [2] E. Secret, J.-O. Durand, *et al.*, Two-photon excitation of porphyrin-functionalized porous silicon nanoparticles for photodynamic therapy, *Adv. Mater.*, **26** (2014) 7643–7648.
- [3] J. Croissant, J.-O. Durand, *et al.*, Biodegradable ethylene-bis(propyl) disulfide-based periodic mesoporous organosilica nanorods and nanospheres for efficient in-vitro drug delivery, *Adv. Mater.*, **26** (2014) 6174-6180.
- [4] J. Croissant, J.-O. Durand, *et al.*, Two-photon-triggered drug delivery in cancer cells using nanoimpellers, *Angew. Chem. Int. Ed.*, **52** (2013) 13813-13817.

Invited Lecture, IL-2

LEARNING ABOUT NANOPARTICLES BY X-RAY ABSORPTION SPECTROSCOPY

Markus Winterer

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Nanoparticles are heterogeneously disordered due to their large surface to volume ratio. Therefore, it is a challenge to obtain detailed structural. Whereas diffraction provides (average) information regarding long range order, spectroscopic methods such as X-ray absorption spectroscopy contain information about the local structure. We will discuss how to determine the valence of cations in nanoparticles from X-ray absorption near edge structure (XANES) spectra and the location of dopants in nanoparticles by Reverse Monte Carlo Analysis of extended X-ray absorption fine structure (EXAFS) spectra.

- [1] A. Kompch, M. Winterer, *et al.*, Localization of Ag dopant atoms in CdSe nanocrystals by Reverse Monte Carlo Analysis of EXAFS spectra, *J. Phys. Chem.*, **119** (2015) 18762-18772.

Invited Lecture, IL-3

**KEY ELEMENTS OF A SUCCESSFUL H2020 MARIA-SKODOWSKA
CURIE – INDIVIDUAL FELLOWSHIP APPLICATION**

Ákos Kukovecz

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Every year, the individual Marie-Sklodowska Curie fellowship (MSCA IF) call is the single largest H2020 venture with close to ten thousand applications. The action is aimed at young researchers at the early/senior postdoc level. Being an MSCA grant holder is widely recognized as a European symbol of excellence and is considered highly beneficial for one's academic career. Considering the huge overlap between the audience of this conference and the main target group of the MSCA IF call, it makes sense to review the key elements of a potentially successful application to this highly competitive call.

In this talk, we will first discuss the basics of proposal writing in general including the differences between proposal and project, milestones and deliverables, dissemination and public outreach etc. Then we will move on to the specificities of the MSCA IF call, introduce the different actors involved in the call and understand their motivations. Finally, I will give hands-on advice about the do's and don'ts you should respect when preparing your own MSCA IF proposal.

Invited Lecture, IL-4

**MECHANO-LUMINESCENT MATERIALS FOR DIRECT
CONVERSION OF MECHANICAL ENERGY INTO LIGHT**

Lucjan Kozielski

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The phenomenon of piezoluminescence was discovered relatively long ago, but in the first years after the discovery it did not arouse much interest in the world of science. Only the development of the technology of intelligent materials has contributed to the interest in piezoluminescence materials, i.e. materials with correlated piezoelectric and luminescent properties.

In the world's literature on the subject, there are references to compounds exhibiting this type of properties. These are, inter alia, ceramic materials CaZnOS and ZNs [1], but they are semiconductors with poor piezoelectric properties, and hence low

light emission. While studying scientific reports, one can find information on fluorides exhibiting the phenomenon of piezoluminescence [2]. However, their high toxicity as well as chemical and physical instability make them unattractive both from the point of view of cognitive and application research. Finally the literature reports and also our preliminary experiments indicates calcium barium titanate (BaCaTiO_3) (BCT) as an prospective material in terms of piezoluminescence. There is especially attractive BCT compositions from the area of so-called morphotropic border additionally modified with rare earth elements [3].

These reports, however extremely interesting, are niche reports that do not fully shed light on the mechanisms governing the phenomenon of luminescence in piezoelectric materials with a perovskite structure. It is a peculiar niche that should undoubtedly be filled. The detailed analysis of the results will significantly expand and organize knowledge on the influence of admixtures of rare earth elements on the piezoluminescence properties of the tested compounds. They fill the gap existing in the world literature and will undoubtedly contribute to the development of knowledge about the mechanisms governing the piezoluminescence phenomenon in ceramics.



References

- [1] J. Zhang, *et al.*, An intense elastico-mechanoluminescence material CaZnOS:Mn^{2+} for sensing and imaging multiple mechanical stresses, *Optics Express*, **21** (2013) 12976.
- [2] G.T. Reynolds, Piezoluminescence from a ferroelectric polymer and quartz, *J. Luminescence*, **75** (1997) 295-299.
- [3] G.N. Bhargavi, A. Khare, Luminescence studies of perovskite structured titanates: A Review, *Optics Spectrosc.*, **118** (2015) 902-917.

Plenary Lecture, PL-1

DESIGNING THE PROCESSING OF ADVANCED CERAMICS AND COMPOSITES TO YIELD THE REQUIRED PROPERTIES

Jon Binner

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Advanced ceramics and composites find widespread uses across a very wide range of industries, including aerospace, electronics, health, defence, energy and many others. Their global market is predicted to reach ~US\$80B by 2022 [1] as a result of constant developments driven by end-users, the manufacturing sector and researchers. Currently, however, when new materials and components are needed they are developed by an iterative process based largely on empirical research. This is slow, time consuming, uncertain and hence costly; hence there is a tremendous need to develop a much smarter approach. This paper will discuss the first steps towards achieving a highly ambitious vision to develop the required understanding of how the microstructures, processing and properties interact to the point whereby a very wide range of ceramics materials with the required performance can be designed, manufactured and used for a wide range of end applications.

- [1] The Global Advanced Ceramics Market (MCP 1001), Global Analysts Industry Inc, 2016, http://www.strategyr.com/MarketResearch/Advanced_Ceramics_Market_Trends.asp.

Invited Lecture, IL-5

ALTERNATIVE SINTERING ROUTES OF ELECTROCERAMICS: THE CASE OF SODIUM POTASSIUM NIOBATE, A LEAD FREE PIEZOELECTRICS

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This talk is about alternative sintering routes of electroceramics, with a particular focus on the case of a lead free piezoelectric niobate, as $K_{1-x}Na_xNbO_3$ (KNN).

Producing monophasic stoichiometric dense KNN is still challenging. Dense KNN ceramics require relative high sintering temperatures, not compatible with the high partial pressure of alkaline elements and alternative processing techniques are required. In our work we are exploiting different alternative sintering approaches that includes: 1. electric current activated / assisted sintering (ECAS), as Spark Plasma Sintering (SPS),

that combines the application of pressure and electric current directly on the sample, enabling high heating rates due to Joule heating of conductive dies and Spark Plasma Texturing (SPT), defined as an edge-free SPS; 2. FLASH sintering, in which the ceramics under direct electric field, densify in a few seconds (≤ 60 s) during a limited current controlled stage and also at a low temperature and 3. Cold Sintering and hydrothermal-assisted sintering conditions, in which liquid phase-enhanced creep under external pressure, in the presence of a liquid at the interparticle contacts and concomitant dissolution-precipitation at liquid/solid interfaces, allows to decrease the temperature of the process to below 300 °C. HRTEM combined with impedance spectroscopy allowed to establish the defect concentration profile and its relation to defect chemistry, dependence on the grain growth and electrical performance for the difference KNN ceramics.

The relevance of the talk is related to: i) the Piezoelectric Devices Market worth 31.33 Billion USD by 2022, ii) the need of “connectivity” and “digitalization”, fundamental for a more equative society, that puts a demand on systems and technological solutions, as Internet of Things, that requires more and efficient sensors and actuators, iii) the sustainable manufacturing where limiting emissions of carbon dioxide during the manufacturing processes and increased resource and energy-efficiency are still key issues and iv) ultimately to a sustainable development, to promote prosperity while protecting the planet, one of the current challenges of our society.

Keywords: sintering, alternative sintering, SPS, SPT, FLASH sintering, Cold sintering, piezoelectrics, lead free electroceramics

Invited Lecture, IL-6

ULTRAFAST OPTICAL CONTROL OF MAGNETISM IN SOLIDS

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The ultrafast magnetism research area is driven by the goal of manipulating the magnetic order in solids on the shortest possible timescale, while minimising the energy dissipations [1]. A series of spectacular phenomena has already been observed, such as the ultrafast demagnetisation [2] and the picosecond-deterministic all-optical switching of the magnetisation [3] of metallic magnets. Despite the breakthrough nature of these observations, it was realised that the presence of free electrons in metals not only complicates the physical description of the processes, but it also induces huge energy dissipations driven by laser heating. For this reason, an alternative trend investigating dielectric materials emerged [1]. In my lecture I will introduce the entire research field and then I will report on the most relevant results reported in this class of compounds, such as the impulsive excitation of coherent magnons [4], even in the absence of lattice and electronic heating [5], the picosecond cold optomagnetic recording [6], the coherent

manipulation of highenergy spin excitations [7] and even the femtosecond activation of the magnetoelectricity [8].

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Invited Lecture, IL-7

THE APPLICATION OF Ce-Zr-O COMPOUNDS IN HCl OXIDATION AND INTREE-WAY CATALYSIS

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The temporary storage of oxygen in a solid catalyst is imperative for many important industrial oxidation reactions in the gas phase, for instance the post-treatment of automotive exhaust gas. CeO₂ is a promising catalyst for the HCl oxidation (Deacon process) in order to recover Cl₂. Employing shape-controlled CeO₂ nanoparticles (cubes, octahedrons, rods) with facets of preferential orientations ((100), (111), (110)), we studied the activity and stability under two reaction conditions (harsh: Ar:HCl:O₂ = 6:2:2 and mild: Ar:HCl:O₂ = 7:1:2). It turns out that both activity and stability are structure sensitive. In terms of space time yield (STY), the rods are the most active particles, followed by the cubes and finally the octahedrons [1].

Ceria also possesses high OSC (oxygen storage capacity) which is a measure of the oxygen quantity that material can store and release. This makes ceria suitable for redox reactions which usually follow Mars-van Krevelen mechanism (surface oxygen atoms directly involved in reactions). A peculiar mixed Ce-Zr (1:1) oxide, the ordered k-

Ce₂Zr₂O₈ phase, is a promising catalytic material exhibiting an extraordinarily high oxygen storage capacity (OSC) and high thermal and chemical stability. We elucidate the temperature-dependent transformation between the pyrochlore *pyr*-Ce₂Zr₂O_{7.3} and κ -Ce₂Zr₂O₈ phase upon oxygen uptake by in-situ X-ray diffraction, X-ray absorption and *in-situ* Raman spectroscopy, providing insights into the electronic and structural changes on the atomic level, which are at the heart of the extraordinarily high OSC. We demonstrate that the Ce³⁺ concentration can be followed during transformation *in-situ* by Raman spectroscopy of the electronic spin flip in the f-shell of Ce³⁺. The catalytic activity of the κ -Ce₂Zr₂O₈ phase has been investigated without an additional active component such as Pt: While the high OSC of the kappa phase is beneficial for the oxidation of CO, the oxidation of HCl turns out to be not affected by the high OSC [2].

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Invited Lecture, IL-8

SIZE AND SCALLING EFFECTS IN FERROELECTRIC CERAMICS

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The change in properties with decreasing the physical dimensions of the system is commonly referred to as “size effect”. Ferroelectrics are particularly prone to show strong size effects as ferroelectricity arises from a long-range ordering phenomenon. Barium titanate (BaTiO₃), the prototype ferroelectric perovskite, exhibits long-range ordering of the electrical dipoles related to the cooperative off-center displacement of the Ti⁴⁺ ions in the TiO₆ octahedra, determining the appearance of a spontaneous polarization and lattice strain. The confinement of the system in a small volume and the presence of interfaces and extended defect (dislocations, grain boundaries, etc.) which disrupt the long-range ferroelectric order will have an impact on ferroelectricity-related properties, such as polarization, dielectric permittivity, tunability and piezoelectric coefficients. The modification of the domain structure, domain wall density and domain wall mobility with decreasing grain size greatly contributes to the alteration of properties in comparison to the bulk reference values. In the case of BaTiO₃ ceramics, the dielectric permittivity and the piezoelectric coefficients are maximized for a grain size of ≈ 1 micron. Below this value, a progressive suppression of the properties is observed.

Size and scaling effects in ferroelectric materials have received significant interest over the last decades from a fundamental point of view, i.e. the determination of the critical size below which ferroelectricity eventually disappears, and the practical

implications related to the continuous miniaturization of active and passive devices utilizing ferroelectric materials. For example, the thickness of the dielectric layers in BaTiO₃-based multilayer ceramic capacitors (MLCCs) has decreased in twenty years from about 10 micron to half a micron with grains smaller than 100 nm while the number of layers has increased to ≈ 1000 .

After a general introduction on ferroelectricity, hystorical development and microstructural aspects, an overview on the current state of understanding of size and scaling effects in ferroelectric ceramics with particular emphasis on barium titanate ceramics, will be presented.

Invited Lecture, IL-9

**PHASE-PURE MULTICOMPONENT EQUIMOLAR OXIDES AS A
NOVEL CLASS OF FUNCTIONAL MATERIALS: SYNTHESIS,
CHARACTERIZATION AND PROPERTIES**

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The recent interest in high entropy materials has opened up a large number of opportunities for the development of different classes of multicomponent equimolar oxide (MEO) ceramics with phase-pure crystal structures. The fundamental premise appears to point to the fact that the presence of a large number of cations in a random fashion in the crystal sublattice results in its stabilization and that the overall properties of the compound is superior to those exhibited by the individual unary oxides. Three different cases of nanocrystalline MEOs have been considered here - one in which 4–5 transition metal cations stabilise into a phase-pure cubic rocksalt structure (multicomponent equimolar – transition metal oxides, ME-TMO), another wherein 3–7 rare earth cations stabilise into a phase-pure fluorite structure (multicomponent equimolar - rare earth oxides, ME-REO) and a third where up to 6 transition metal cations stabilise into a phase-pure spinel ferrite structure (multicomponent equimolar – spinel ferrites, ME-SPF). The selection of the cations in all the cases was made on the basis of Hume-Rothery and Pauling's rules.

Nanocrystalline ME-TMO, ME-REO and ME-SPF powders were synthesised by a wet chemical, reverse co-precipitation (RCP) process as well as by aerosol processes (flame spray pyrolysis, FSP or nebular spray pyrolysis, NSP) using nitrate precursors. Standard characterization techniques were used to ascertain the phases and phase composition, and the crystallite size (X-ray diffraction, XRD), particle size and

morphology (high resolution transmission and scanning electron microscopy, HRTEM and HRSEM), phase stability at different temperatures (thermogravimetry and differential scanning calorimetry, TG-DSC), bond structure (Fourier transform infra-red and Raman spectroscopy, FT-IR and FT Raman), oxidation states (X-ray photoemission spectroscopy, XPS) and bandgap energy (photoluminescence and diffuse reflectance spectroscopy, PL and DRS). The rocksalt type ME-TMO exhibited structural stabilisation as a single phase through the high configurational entropy achieved due to the presence of multiple cations in equiatomic amounts. These ME-TMO show high room temperature lithium ion conductivity with superior capacity retention ability when used as an electrode material for secondary Li-ion batteries. On the other hand, factors other than entropy were found to stabilize the single phase in case of the fluorite type ME-REO. Both the ME-REOs and ME-SPFs exhibited a narrow bandgap, lower than the constituent unary oxides. These studies show that ME-TMO, ME-REO and ME-SPF form a novel class of materials that can be used in a variety of practical applications along with a wide range of tuning possibilities.

Keywords: Multicomponent equimolar oxides (MEO); Entropy stabilization; Rocksalt type TMO; Fluorite type REO; Spinel ferrites

Invited Lecture, IL-10

THE IMPORTANCE OF TEM ANALYSES FOR THE DEVELOPMENT OF UHTCs

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Sintering is a crucial step for structural ceramics as it leads to the elimination of porosity and hence to the achievement of high thermo-mechanical performances. The class of materials known as ultra-high temperature ceramics (UHTCs) requires a combination of severe consolidation process, such high temperature above 2000 °C and the application of pressure, which leads to coarse microstructure with poor mechanical behavior. UHTCs include borides and carbides of transition metals and are characterized by melting point above 3000 °C and physic-chemical properties suitable for application in the aero-space field. Efforts have been concentrated on the mitigation of the sintering conditions and the addition of secondary phases forming eutectics or transient liquid phase is the most successful approach. Drawbacks of multiphase system can be related to the precipitation of reaction phases at the triple points or formation of grain boundary glass that may hamper the mechanical behavior at high temperature. Transmission electron microscopy is the most suitable method to investigate the microstructure at nano-scale length and obtain a set of information not achievable with other analytical techniques. A series of UHTC systems is presented and the process-microstructure-properties relationships are discussed.

Invited Lecture, IL-11

**CORE/SHELL MESOPOROUS SILICA AND ORGANOSILICA
NANOPARTICLES: SYNTHESIS, CHARACTERIZATION AND
APPLICATION IN TARGETED CANCER TREATMENT**

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Magnetic analogues of mesoporous silica nanoparticles (MMSN) and periodic mesoporous organosilica (MPMO) exhibit high potential for construction cancer-targeting nanotherapeutics. The materials typically contain superparamagnetic iron-oxide nanoparticles (SPION) as the core and mesoporous silica and organosilica shell, with the material retaining superparamagnetic features of the core SPION. The silica shells retain their characteristics as well, and the final materials exhibit high surface areas, structured porosity, high biocompatibility and various possibilities for surface functionalization. Hence, different cancer-targeting nanomaterials can be constructed, having stimuli-responsive cancer treatment capabilities and functionalized surface for cancer targeting, supported by the capabilities for magnetic field-guided therapy. This talk details on construction and characterization of different composition and morphologies of core/shell mesoporous silica and organosilica nanostructures. SPION-containing MMSNs and MPMOs, as well as core/shell PMO nanoparticles containing core nanodiamond nanoparticles will be presented, evidencing their applicability for targeted cancer therapy.

Invited Lecture, IL-12

HOW TO TALK SCIENCE ... clearly

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Why is it important to Speak Science?

Why giving a scientific talk can be so challenging?

What should we do to give a brilliant scientific talk?

This talk is about how to prepare and deliver excellent scientific talks. During this talk I'll share with the audience my own perspective on how to speak science, considering that scientific talks are currently one of the most important communication tools for the scientific community. In conjunction with scientific papers, scientific talks are part of the scientific communication process. As scientists our research reputation

depends on the content of our science but also on the way we transmit it to the community. In a general sense, the quality of our speech has a direct effect on how the audience will recognize us as scientists, i.e. our research reputation will be enhanced or diminished. Effective communication of scientific knowledge is indeed an essential component of any career in science, but also a responsibility of scientists.

I have recognized very early in my career as a scientist, the importance of a good scientific talk. Listening to scientists talking (excellent and terrible) I started to identify common aspects among excellent speakers. Since that time, keeping my own identity, I have been using these skills that I'll share with you in this talk.

If I have to identify the key ingredients for an excellent scientific talk, I would make the analogy to the *Theory of Rhetoric*, by Aristoteles, for a good speech: *Logos* for a clever argument, *Ethos* for convincing presentation and *Pathos*, to enthuse the audience. There is an entire process that we all need to consider if we indeed intend to give a remarkable talk.

Are you interested in improving how you clearly talk your science? If yes, come and join!

Keywords: scientific talks, scientific process, importance, performance

Invited Lecture, IL-13

THE MAGIC OF HIGH POWER MICROWAVE PROCESSING OF CERAMIC MATERIALS

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Microwave/matter interactions are the phenomena on which the volumetric heating could be explained. The seminar will summarize dielectric heating mechanism and correlate these with materials' dielectric properties at the frequency of microwaves (MW). The ceramic processes supported by MW irradiation will be distinguished accordingly to the temperature. Geometries of microwave applicators will also be presented jointly with a series of examples and case studies in the field of solid state synthesis, hydrothermal nanoparticle preparation and materials sintering and crystallization. A completion of the presentation will be the evaluation of the energetic of microwave-assisted heating processes and safety regulation.

Invited Lecture, IL-14

OXIDE THERMOELECTRICS – CURRENT STATUS AND FUTURE PROSPECTS

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Thermoelectric generators (TEGs) convert heat directly into electric energy and represent a promising technology for sustainable energy conversion. The principle is illustrated in Fig. 1 and shows the connection between two different materials with n- and p-type electrical conductivity, respectively. The efficiency of a TEG relies on the figure-of-merit of both p- and n-type active components

$$zT = \frac{S^2 \sigma T}{\kappa}$$

Where S is the Seebeck coefficient, σ is the electrical conductivity and κ is the thermal conductivity. In applications for waste heat harvesting from infinite heat sources the power factor, $P = S^2 \sigma$, is considered equally important [1]. Hence, materials with high σ and S combined with a low κ will in general enhance the efficiency of a given TEG. The state-of-the-art materials are heavy, toxic and expensive metals and alloys, which are hampered by severe limitations due to oxidation and melting even at moderate temperatures. This paves the way for oxide-based materials and TEGs due to their chemical and thermal stability at high temperatures in air combined with low-cost and being environmentally benign. So far, the efficiencies of oxide based TEGs are still less than devices based on metallic components and alloys and calls for the development of oxides with improved thermoelectric properties.

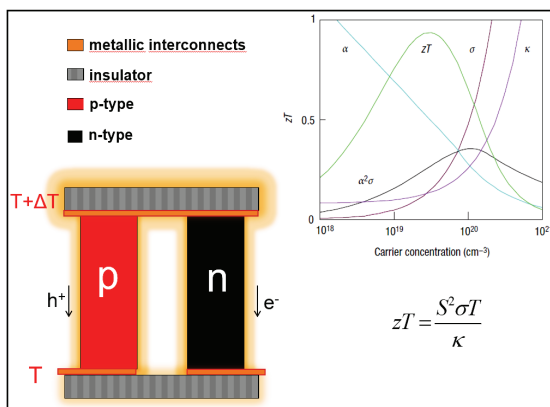


Figure 1. A single p-n pair of a conventional TEG and the variation in thermoelectric properties (S , σ , κ , zT) as function of carrier concentration

In this presentation we will review the state-of-the-art of oxide-based thermoelectric devices and discuss the challenges and benefits of TEGs based on oxides. We will also present our recent results related to improved material properties and a promising novel design of all-oxide TEGs for operation at high temperatures in ambient atmosphere [2].

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13th Conference for Young Scientists in Ceramics, CYSC-2019
Novi Sad, Serbia, October 16-19, 2019

ORAL PRESENTATIONS

OA-1

IRON(III)ACETYLACETONATE VAPOR STUDIED BY X-RAY ABSORPTION

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Due to a large variety of chemical (e.g. redox potential) and physical properties iron containing nanoparticles exhibit a broad spectrum of applications. Those are highly depending on the oxidation state and crystal structure. Different phases and morphologies can be achieved by changing the nanoparticle synthesis parameters [1]. Chemical vapor synthesis (CVS) is a technique which enables reproducible synthesis of nanoparticles with reliable control of synthesis parameters. Organometallic compounds are used to introduce metals into reactor. Solid precursors offer easier handling, mostly lower toxicity and are advantageous for the gas redox potential adjustment through a high carbon content.

Iron(III)acetylacetonate ($\text{Fe}(\text{acac})_3$, $\text{Fe}(\text{C}_5\text{H}_7\text{O}_2)_3$) is such solid precursor used for CVD (chemical vapor deposition) and CVS to generate iron containing nanoparticles. However, due to a higher molecular mass and intermolecular interaction, the volatility of $\text{Fe}(\text{acac})_3$ is relatively low, still it needs to be evaporated to deliver it as vapor to CVD and CVS processes. In fact, only little is known about the local and electronic structure of $\text{Fe}(\text{acac})_3$ vapor.

We designed a novel cell for measuring gas phase species by XAS (X-ray Absorption Spectroscopy). We investigate $\text{Fe}(\text{acac})_3$ vapor in-situ at the Fe-K edge using this vapor cell at conditions close to those at the CVS reactor inlet at different temperatures. Using Reverse Monte Carlo analysis of EXAFS spectra we obtain information about local structure and changes in precursor molecules due to evaporation [2]. We compare those data to solid $\text{Fe}(\text{acac})_3$ and $\text{Fe}(\text{acac})_3$ solutions (water, isopropanol and acetone). This enables a better understanding of processes occurring in the CVS system using iron as a probe.

Acknowledgment: DFG (German Research Foundation) DLS (Diamond Light Source), DESY (German Electron Synchrotron).

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OC-2

SOL-GEL-BASED INTERFACIAL COATINGS OF RARE EARTH OXIDES ON 2.5D CARBON FIBRE PREFORMSTammana S.R.C. Murthy^{1,2}, Vinothini Venkatachalam¹, Ji Zou¹, Jon Binner¹¹*School of Metallurgy and Materials, University of Birmingham, UK*²*Materials Group, Bhabha Atomic Research Centre, Mumbai 400085, India*

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The critical driving force in the development of fibre-reinforced ceramic matrix composites (CMCs) has been to provide high-temperature structural ceramic materials that display the benefits of damage tolerance and graceful failure; properties not available in unreinforced monolithic ceramics. Control of the interface between the fibre and matrix plays a significant role in achieving this important objective. CMCs with properly designed and fabricated interfacial regions can survive local damage and its associated inelastic deformation without catastrophic failure, thereby providing greater strength and strain capability than monolithic matrices alone. Historically, the direct introduction of a ceramic fibre into a ceramic matrix has yielded a brittle composite since the strong chemical bond or frictional resistance between the fibre and matrix allows matrix cracks to penetrate directly into the fibres. The predominant approach to developing the desired weak bond at the interface has been to deposit a thin, typically 0.1 to 0.5 μm , 'weak or compliant' coating layer around the ceramic fibres. The coating acts as a debonding layer and a "low-friction" sliding interface. In addition, interface coatings can also act as reaction and diffusion barriers for the underlying ceramic fibres, preventing chemical attack and fibre degradation during processing and service conditions. In the present study, sol-gel processing was used to deposit yttria and ceria coatings; the use of a sol-gel approach meaning that the conversion process could be

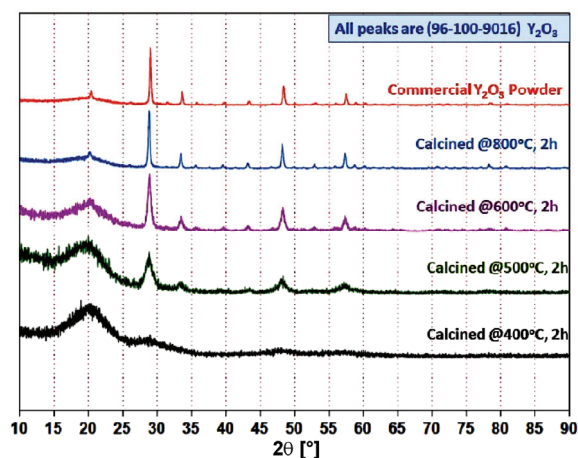


Figure 1. XRD plot showing the analysis of calcined Gel of Y₂O₃ at different temperatures

conducted at a relatively low temperature. The formation of the rare earth oxides were analysed by TGA, whilst the phase composition was confirmed by XRD and coating thickness by SEM-EDS and TEM analysis. Thicker coatings were obtained by using multiple coating steps. The XRD pattern shows the peaks of Y_2O_3 formed during calcination of gel at different temperatures and data was compared with commercially available powder.

OA-3

MAGNETIC NANOCOMPOSITES CONTAINING IRON OXIDE@SiO₂ OBTAINED VIA CONVENTIONAL OR ULTRASONIC METHOD

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Magnetic nanocomposites composed by superparamagnetic iron oxide@SiO₂ were obtained by using conventional (MS) or sonochemical method (US). As SiO₂ source, tetraethoxysilan (TEOS) was used. This study shows the influence of the used method for iron oxide nanoparticles coating with silica upon structural and morpho-textural properties of the obtained magnetic nanocomposites. FT-IR, BET, XRD and magnetic measurements were used to characterize the obtained materials. FT-IR confirmed that the superparamagnetic iron oxide nanoparticles were successfully coated by silica. BET measurement showed that higher surface areas were obtained for MS produced nanocomposites compared with US method, and the values are increasing with the amount of added TEOS for both used methods, MS and US. XRD measurements demonstrate that the magnetic core embedded in silica shell retains its original crystalline nature. Saturation magnetization decreased after silica-coating due to the presence of non-magnetic layer and higher value were obtained for US synthesized nanocomposites compared with MS method, these results could indicate that in the case of ultrasonic irradiation a better dispensability of magnetic iron oxide nanoparticles was obtained.

OA-4

**SYNTHESIS, STRUCTURAL AND MAGNETIC PROPERTIES OF
 $\text{Y}_{1-x}\text{Yb}_x\text{F}_3$ SOLID SOLUTION**

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Many works devoted to obtaining nanodispersed BaTiO_3 powder modified with different dopants for suitable properties providing. In particular, recently considerable attention has been given to obtaining modified nanopowders BaTiO_3 possessing relaxor behavior order to ensure reliable work of dielectrics. Generally, Ca,Zr,Mn, Pb and rare earth elements such as Nb,Y adds order to provide stress, inhibit grain growth and provide Pinching effect, and hence to increase dielectrics relaxor behavior.

However, there is still an issue associated with obtaining satisfactory stoichiometry of the obtained powder. From this viewpoint Ca,Zr-doped BaTiO_3 were prepared with co-precipitation method via multiligand complexes formation and influence of the precursor type on Ca,Zr-doped BaTiO_3 stoichiometry were investigated. Their stoichiometry, crystal structure was examined in order to determine preferential solubility site of Ca,Zr ions in perovskite structure. Stoichiometry Ca,Zr-modified BaTiO_3 will be evaluated considering different precursor type. X-ray, IR spectroscopy and X-ray fluorescence analysis were carried out to obtain the knowledge on the occupation site in the $\text{Ba}_{1-x}\text{Ca}_x\text{Ti}_{1-y}\text{Zr}_y\text{O}_3$ perovskite structure. These results proved influence complex formation on Ca,Zr-modified BaTiO_3 stoichiometry.

OA-5

LASER-FLASH-EVAPORATION OF SOLID METAL ORGANIC PRECURSORS

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Chemical vapor synthesis (CVS) is a gas phase process to produce nanopowders with high purity and complex compositions, e.g. Perovskites, which exhibit unique catalytic and ferroelectric properties.

A CVS set-up consists of a precursor delivery unit, a reactor and a particle collector. The delivery unit provides a mixture of precursor and carrier gas at a steady mass flow. For the formation of perovskite nanoparticles, a gas mixture with precise stoichiometry is needed. One solution for a tunable and accurate precursor delivery is the Infrared-Laser-Flash-Evaporation of solid metal organic precursors, where a solid precursor is instantly sublimed by an infrared laser beam [1].

In this presentation, a new Laser-Flash-Evaporator is introduced which allows a fast and precise scanning of a Laser beam over spatially separated precursor reservoirs. FTIR spectroscopy is conducted to determine a threshold for the decomposition of metal organic molecules by increased power densities. In addition, the influence of the power density on the evaporation rate will be evaluated.

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OC-6

COATING OF CERIUM OXIDE NANOPARTICLES WITH DIFFERENT CARBOHYDRATES AND THEIR APPLICATION ON PLANTS

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Cerium oxide nanoparticles (nCeO₂) are nanomaterial with various applications in biomedicine, electronics and cosmetics. The aim of this research was to improve their low suspension stability by coating with carbohydrates - glucose, levan, and pullulan. The coating was performed at different temperatures and coating times, by adding the carbohydrates during or after the synthesis of nCeO₂. Characterization of nanoparticles' powders was performed by X-ray diffraction analysis, Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Nanoparticles' suspension stability was estimated by measuring of zeta potential, hydrodynamic size and turbidity. The differences among coated nCeO₂ have been confirmed with FT-IR spectra. The results showed improved stability of the nCeO₂ suspension and decreased size of aggregates after carbohydrate coating. Levam- and glucose-coated nCeO₂ suspensions showed the best stability. The obtained nCeO₂ were used for the investigation of their ecotoxicity on different plant species.

OC-7

PROCESSING AND CHARACTERIZATION OF Al₂O₃-Cu-Ni COMPOSITES

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In the study the results of investigation considering the use of centrifugal slip casting method in fabrication Al₂O₃-Cu-Ni composites are presented. Suspension consisted of alumina, copper and nickel powders suspended in water with addition of a liquidiser composition. The microstructure and chemical composition of the fabricated specimens were analysed using a scanning electron microscope equipped with an EDX detector. Interface between ceramic and metal phases was characterized. The distribution

of metal particles was determined by the stereological analysis. Furthermore, the X-ray diffraction was done.

The main contribution of our work is that we proved that the centrifugal slip casting technique allows obtaining of Al₂O₃-Cu-Ni hollow tubes.

Based on macroscopic observation it was found that the centrifugal slip casting technique allows obtaining gradient distribution of metallic phases in ceramic matrix. The XRD results confirmed there phases: α -Al₂O₃, Ni and CuNi in composites. Additionally, the investigations have shown that the addition of the nickel particles improved the wetting of the ceramic with liquid metal.

The obtained results were gave new knowledge about the correlation between factors of the manufacturing process and structure or selected properties of the functionally gradient materials made of the Al₂O₃-Cu-Ni system.

Acknowledgements: The study was accomplished thanks to the funds allotted by The National Science Centre within the framework of the research project 'OPUS 13' no. 2017/25/B/ST8/02036.

OC-8

EFFECT OF METAL PARTICLES SIZE ON MICROSTRUCTURE OF ZrO₂-Ni COMPOSITE FABRICATED BY CSC IN MAGNETIC FIELD

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The aim of this paper was to determine the influence of nickel particles size on microstructure of functionally gradient material ZrO₂-Ni fabricated by centrifugal slip casting (CSC) with simultaneous action of magnetic field. To obtain composite, ZrO₂ was used as a matrix and two types of nickel powder with different size of particles were used as a metallic phase. Correlation between the ZrO₂ matrix and metallic phase was investigated by the research of microstructure and selected properties. Structure was examined by XRD analysis. Microstructure was characterized by SEM observation and EDX measurements. In addition, apparent density, relative density, open porosity and water absorption of obtained composites were determined. Linear shrinkage was also defined. Research results enable to conclude that proposed method of fabrication allows to obtain a ceramic-metal composite with a zones structure. Application of the magnetic field resulted in the characteristic distribution of the metallic phase in the ceramic matrix. Use of metallic phase with smaller size of particles results in formation of microstructure

with characteristic chains directed parallel to the outer edge of the sample. Using of bigger nickel particles didn't cause chains formation. In this case, microstructure observation revealed random location of the particles on the half of the cross section of the composite.

Acknowledgment: The research has been financially supported by the Faculty of Materials Science and Engineering Warsaw University of Technology (statute work 2018 and dean grant no. 504/04034/1090/42.000103).

OC-9

FABRICATION AND CHARACTERIZATION OF MULTI-FUNCTIONAL HYBRID LAYERS ON NiTi SHAPE MEMORY ALLOY

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The surface of the NiTi shape memory alloy was functionalized through the deposition of multifunctional ceramic hybrid layers using the electrophoretic method (EPD). Hydroxyapatite (HAp) powder was used as main component of the layer, while nanocomposite silicon dioxide/silver (nSiO₂/Ag) as an addition.

It is known that calcium phosphate-based (CaP) coatings, i.e. hydroxyapatite (HAp), facilitate the connection of a bone with a metallic implant. Silica matrix enables incorporation of metal ions (e.g. silver) in low concentrations and their gradual release into the environment, which will provide a long-lasting antibacterial effect.

In the work the coatings with different ratio of HAp:nSiO₂/Ag were obtained on passivated NiTi alloy substrate by electrophoresis. Deposition was performed under different voltage (from 5 to 50 V) at time periods (from 0.5 to 5 minutes). Subsequently, the uniform layers were subjected to a heat-treatment at 700 °C for 2 h in order to sintering and improve their adhesion strength to metallic substrate. As a result of the heat-treatment the structural changes in coatings, especially in nSiO₂/Ag nanocomposite, were observed. Moreover, due to temperature effect, the crystallization of rutile was found. The obtained composite layers were homogeneous, crack-free and able to deformation related to shape memory effect. The formed coatings were characterized for morphology, topography, structure and bioactivity.

OA-10

**INFLUENCE OF THE ADDITION OF WASTE ALUMINA POWDER ON
MECHANICAL PROPERTIES OF ALUMINA CERAMICS**

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During the green machining of the alumina (Al_2O_3) green bodies, a certain amount of waste ceramic powder is generated which remains unused. In addition, the waste alumina ceramic powder should be disposed of as non-hazardous waste in a legally compliant manner. In this study, up to 20 wt.% Al_2O_3 powder was replaced by waste Al_2O_3 powder for the preparation of alumina ceramics green bodies by slip casting. Stable 70 wt.% Al_2O_3 – Al_2O_3 (waste) aqueous suspension was prepared for forming green bodies by slip casting in gypsum mould. Stabilization of concentrated aqueous alumina suspensions was achieved with the addition of 0.1 wt.% dispersant Tiron (expressed on dry weight basis of alumina powders). Green bodies were sintered by conventional method in electrical kiln from 1400 to 1600 °C.

In this study, Response Surface Methodology (RSM) based on the Box-Behnken Design (BBD) was used to investigate the effects of addition of different amounts of waste alumina powder and sintering regime (sintering temperature and holding time) onto mechanical properties of obtained ceramic samples. Mechanical properties that were investigated are hardness and fracture toughness of sintered (Al_2O_3 – Al_2O_3 (waste)) samples.

OA-11

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

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CTTC and IRCER have developed an innovative and novel 3D printing method of thick films coatings. This process called INPACT is based on the Aerosol Deposition Method (ADM or AD), also called Vacuum Kinetic Spray (VKS) and has led to the development of a Computer-Aided Manufacturing (CAM) machine [1], in order to get complex 2D-3D-shapes.

The principle of ADM [2] is to project a gaseous aerosol of dry particles onto a substrate (which doesn't require any surface preparation) through a supersonic nozzle. The kinetic energy of particles implies fragmentation and a cohesion phenomenon coupled with local thermal losses. Although the consolidation mechanisms are not fully understood yet, ADM is a low-cost method for the fabrication of dense films without post-coating thermal treatment. A huge innovation could be the manufacturing of dense ceramics on new and multiple kinds of substrates including metals or even low melting temperature or flexible materials like plastics or polymers for electronic and microelectronic applications for instance.

This talk is focused on the deposition of lead-free piezoelectric ceramics with perovskite structure for energy harvesting applications. We have investigated the coating of bismuth - sodium titanate, barium titanate and cobalt doped barium titanate on different substrates such as glass, silicon wafer and kovar (alloy Fe, Ni, Co). Indeed, we have worked to realize our own synthesis protocol of ceramics powders, suitable for ADM process. The powder, the piezoelectric thick coating elaboration and characterisation will be presented to show all potentials of this technique.

Keywords : thick films, aerosol deposition, lead-free perovskite

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OA-12

PROCESSING OF NANOSTRUCTURED CNT-BASED THIN FILM ELECTRODE

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Carbon nanotube-based thin films were developed as IR transparent electrodes. The thin film electrodes were prepared from polyelectrolyte and multi-walled carbon nanotubes by layer-by-layer (LbL) assembly. LbL assembly is an excellent technique for the deposition of the thin films with ability to control film thickness. Positively charged polyethyleneimine (PEI) and negatively charged carboxylic multi-walled carbon nanotubes (MWCNT-COOH) were deposited alternately on glass substrates. Samples were prepared with 2, 4 and 6 bilayers (PEI + MWCNT-COOH). Thickness of the films varied between 100 and 300 nm, depending of the number of bilayers. After deposition of each bilayer, the samples were dried at 120 °C for 10 min. LbL assembled thin films were characterized by Raman spectroscopy, X-ray diffraction analysis, high-resolution

scanning and transmission electron microscopy. Transparency was measured in UV/Vis, NIR and mid-IR range. Transparency obtained in the mid-IR range was between 50 and 70 % for all samples. Relative sheet resistance was measured with four-point probe station and the values were ranging from 4 kΩ/sq up to 10 kΩ/sq, depending on the number of layers.

OA-13

NEW GENERATION SILICON CARBIDE MEMBRANES FOR WATER PURIFICATION

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With technological advances and the ever-increasing stringency of water quality criteria, membrane processes are becoming a more attractive solution to the challenge of high quality water, and water reuse. In this context, membrane processes based on ceramic membranes hold a important position in industry. Ceramic membranes made from silicon carbide (SiC) are cutting-edge technology in water purification, considering the higher flux and lower fouling, compared to traditional ceramic and polymeric membranes. SiC membranes, moreover, offer high strength, high resistance towards aggressive chemicals and further withstand high temperatures.

In this present work, we developed a new type of SiC membrane on macroporous SiC support for use in water purification. The membrane layer was formed by coating porous SiC substrate with an aqueous suspension of SiC powders and sintered in a high temperature furnace. The stabilization of SiC powder in the aqueous suspension and optimization of the processing parameters were studied. Furthermore, the microstructure of as-formed SiC membranes was characterized and their performance was evaluated by filtration tests.

OA-14

**ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA
SYNTHESIZED FOR HYDROGEN STORAGE APPLICATION**

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The functionalization improving the interaction between adsorbent and adsorbate is the breakthrough for synthesizing an optimum hydrogen storage material. Subsequently the functionalized silica materials are prepared, their properties are verified, and the most adequate materials are chosen for the envisaged application. By reason of the fact that was demonstrated that the amorphous structure of the adsorbent and the use of the shorter surfactant molecules in the synthesis increased both the storage capacity and the strength of the interaction between the surface and the adsorbate, we synthesized two series of samples by using templates with shorter chain lengths, “n” was 14 or 12. On the other hand, we also have varied the molar ratio of the functionalized precursors and tetraethoxysilane, in order to obtain different sizes and surface functionalities. In each series of samples, C12 and respectively C14, different grade of interaction with the incoming hydrogen molecules and different values of homogeneity have been obtained. The maximum H₂ uptake value for the methyl functionalised sample was 2.38 wt.% at temperature of 77 K and pressure up to 1.0 bar; the latter increase to 2.41 wt.% moving up to 80 bar, a storage capacity comparable with other materials described in literature.

OA-15

DIRECT INK WRITING AS AN ADDITIVE MANUFACTURING TECHNIQUE FOR SHAPING HETEROGENEOUS CATALYSTS

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As it is well known, additive manufacturing or so-called 3D printing can provide geometries far beyond the limitations of conventional shaping techniques, which is why it is a technology of high interest for the design of highly efficient, tailor-made heterogeneous catalysts. One suitable method for shaping heterogeneous catalysts on ceramic base is *Direct Ink Writing* or so-called *Robocasting*, which has proven to be applicable in this field and is still focus of ongoing research. Here a ceramic paste is extruded through a thin nozzle and the desired object is built up layer by layer.

However, new challenges are emerging in terms of material and process development. For this reason, a detailed investigation of the process chain is indispensable, starting with a material study and thermal post-processing to catalytic test reactions. In general, the suitable catalytic material used cannot be printed purely. This makes the implementation of certain additives mandatory to overcome the limitations from the ceramic material and optimize rheological properties. In addition, the new catalyst structure has to possess a certain crush strength and a sufficiently large active surface area. One has furthermore to consider the question of additive removal to minimize influences on the catalytic activity. Against this background, catalyst shapes will be 3D printed within this project and their applicability will be investigated.

OC-16

EXAMINATION OF THE ROLE AND BEHAVIOUR OF MESOPOROUS SUPPORT MATERIALS IN CO₂ HYDROGENATION

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While noble metals' excellent catalytic properties are undeniable, they are scarce and expensive materials, hence the research for new options is of great interest. One fairly appropriate solution is to use as little noble metals as possible while maintaining or improving catalytic performance. This approach emphasises the role of support materials greatly. In this study the role and mechanism of mesoporous support materials were examined by conducting ex situ x-ray photoelectron spectroscopy on the pretreated and the spent catalysts.

In this study our catalysts' composed of different mesoporous Co or Mn or mixed transition metal oxides and platinum nanoparticles. The catalytic reactions were carried out in the modified pre-chamber of our XPS instrument. The standard pre-chamber was expanded with a quartz tube, a thermocouple, a heating element which provided uniform heating and a programmable control unit. During the pretreatment of the sample and during the reaction continuous gas flow was provided.

The gathered and processed data helps to evaluate the role of different surfaces' in the given catalytic process. Furthermore, studying their behaviour enables the design of new catalytic systems, reducing the trial-and-error nature of these experiments.

Keywords: catalysis, mesoporous support, x-ray photoelectron spectroscopy

OA-17

THE HYDROGEN STORAGE CAPACITY OF ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA

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The hydrogen adsorption properties of prepared functionalized mesoporous silica with different compositions have been studied. The different adsorption properties can be adducted to the different interaction between the sorbent and the adsorbant species, and that can be related to the structural, morphological and chemical properties of the samples. In order to influence the pore diameter dimensions and implicitly the specific surface area, different molar ratios of organically functionalized precursors (MTES)/tetraethoxysilane were used by using the sol gel co-condensation method. In the case of the series with the shorter (C12) surfactant, the higher adsorption capacity obtained for the samples synthesized with no organically functionalized precursor can be

debited to a different and stronger sorbent-adsorbent interaction, beyond those small differences in the textural properties observed in the present synthesis conditions. In the C14 directing agent series of samples, the sample with no MTES and the samples with low amount of MTES, appears the most performing at 77 K immediately followed by the samples synthesized with increase amount of MTES.

OA-18

ORGANICALLY FUNCTIONALISED MESOPOROUS SILICA WITH GAS STORAGE PROPERTIES: SYNTHESIS AND CHARACTERIZATION

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In order to be tested for their hydrogen storage properties, mesoporous silica particles were synthesized by sol-gel method, starting from tetraethyl orthosilicate silica precursor. Besides, the following organically precursors have been added in different molar ratios: triethoxyvinylsilan, diethoxy(methyl)vinylsilane and methyltriethoxysilane. Ammonia was used as catalyst and hexadecyltrimethyl ammonium bromide as directing agent. The aim of our study is the functionalization with different organically groups, with the purpose of trying to tailor their performances as gas storage materials. The molar ratio of different functionalized precursors/TEOS is varied in order to obtain different sizes for the pore diameter as for the specific surface area. The hydrogen adsorption data show that the methyl functionalized samples were better materials from the point of view of maximum adsorption capacity for H₂, compared to the functionalized silica samples with some others organic groups. The higher adsorption capacity of the methyl-functionalized sample can be attributed to a strong sorbent-adsorbent interaction, but also to the high specific surface area and big pore volume.

OA-19

THERMAL PROPERTIES AND CERAMIC YIELD OF POROUS POLY(METHYLVINYLSILOXANE) CROSS-LINKED WITH 1,3,5,7-TETRAMETHYLCYCLOTETRASILOXANE BEFORE AND AFTER DEPOSITION OF PALLADIUM NANOPARTICLESJan Mrówka, Janusz Partyka, Magdalena Hasik*AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland*

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Polysiloxanes represented by the general formula of $(\text{Si}-\text{RR}'\text{O})_n$ are a class of organic-inorganic polymers employed in many fields of materials science. The backbone of these polymers consists repeating Si–O bonds, instead of typical C–C linkages in organic polymers which are longer and more flexible than the latter. These facts result in many interesting properties of polysiloxanes, like high gas permeability, low melting point and high thermal resistance to name just a few [1]. At high temperatures in inert atmosphere, polysiloxanes undergo many decomposition reactions to form various ceramic materials, such as amorphous Si–C–O [2-4] or SiC [4,5].

In this work, we examined the influence of palladium nanoparticles on thermal properties and ceramic yields of poly(methylvinylsiloxane) (V_3) cross-linked with cyclic hydrogenosiloxane (1,3,5,7-tetramethylcyclotetrasiloxane, D_4^{H}) at different Si-H/Si-vinyl ratios. The cross-linking process was conducted in High Internal Phase Emulsion (HIPE) conditions and yielded porous materials containing Si–H groups. It was found that they readily reduce Pd^{2+} ions in a tetrahydrofuran solution to form Pd/polymer nanocomposites. Pyrolysis of the starting and Pd-containing materials was carried out in argon atmosphere at 1000 °C. Presence of Pd nanoparticles resulted in deterioration of thermal stability and decrease in ceramic yield. Porous microstructure of all samples was preserved and silicon oxycarbide-carbon composites were formed with high ceramic yields (74–80% for starting materials and 55–78% for those containing Pd). The amount of carbon phase in the ceramic material depends on the cross-linking density of the polysiloxane the precursor.

Acknowledgement: This research was financed by the Faculty of Materials Science and Ceramics of AGH-UST, as a project 16.16.160.557. Also, Jan Mrówka has been partly supported by the EU Project POWR.03.02.00-00- I004/16.

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OA-20

NANO-SIZED MAGNESIUM SPINEL POWDERS SYNTHESIZED VIA FLAME SPRAY PYROLYSIS

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Nano-sized and agglomerate free magnesium spinel particles were produced via flame spray pyrolysis method. In this method, a combustible precursor is fed through a self-sustaining flame in the form of aerosol. Synthesis of a multi-cation compound such as MgAl_2O_4 (Mg-spinel) involves decomposition or oxidation of the precursor during combustion and subsequent solid-state reaction. While different chemical processes and precursors can be utilized to synthesize Mg-spinel via FSP, trane complexes were shown to be the most economic one. These complexes are prepared by dissolving oxides/hydroxides of magnesium and aluminium in ethylene glycol and chelating them in triethanolamine at 200 °C under N_2 flow. In this study, we examined the effects of solubility of hydroxides/oxides during magnesium-alumatrane preparation, on the final powder characteristics. Despite starting with fine commercial oxide and hydroxide powders, complete dissolution has not been achieved under different reaction conditions. Incomplete dissolution was even noticeable by visual inspection due to Tyndall effect. Remaining precursors were also detected by Scanning Electron Microscopy (SEM) analysis. Phase purity could not be achieved after FSP, due to the undissolved particles. Presence of MgO and $\gamma\text{-Al}_2\text{O}_3$ along with MgAl_2O_4 indicated a diffusion-controlled process under applied conditions. To shorten the diffusion path, aluminium and magnesium hydroxides, which were co-precipitated from their aqueous metal nitrate solutions were utilized to prepare magnesium-alumatrane complex. Obtained clear solution was fed into the FSP reactor and phase pure, fine, agglomerate free powders with high sinter-activity were synthesized. Their sintering characteristics were compared with two different commercial powders.

OA-21

PROBING SONICATION OF COLLOIDAL SnO₂ BY *IN SITU* SMALL-ANGLE X-RAY SCATTERING

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Dispersion of dry powder in liquids to produce ceramic components requires the use of high-energy input to break up agglomerates. Especially nanoparticles tend to form agglomerates due to their high surface energy and van-der-Waals forces. Sonication is promising because, through cavitation, it generates high shear forces, shock waves and microjets that break down nanoparticle agglomerates into single particles. These individual nanoparticles can then be stabilized to prevent re-agglomeration.

In situ synchrotron-based Small-Angle X-ray Scattering (SAXS) is used to monitor the mesostructure of chemical vapor synthesized (CVS) SnO₂ nanocrystals during the sonication process in aqueous media. The influence of sonication time and intensity on the fracture of nanoscaled agglomerates is investigated. Structural changes like size distribution, fractal structure and particle arrangement in the agglomerates are studied for SnO₂ nanocrystals of different size.

Acknowledgements: This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

OA-22

**SYNTHESIS OF TITANIUM NITRIDE POWDER FROM
RUTILE AND ANATASE**

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In this research titanium nitride (TiN) powder was synthesized through the reaction of titanium oxide and carbon in presence of nitrogen. TiN powders were obtained via dynamic carbothermal reduction nitridation (DCRN) at 1400 °C. Starting powders were selected from two different titanium dioxide (TiO₂) sources, containing rutile and anatase phases, with different powder sizes. The raw materials mixing was

carried out using TiO_2 and carbon black with a stoichiometric ratio (C/TiO_2) of 2. The phases and microstructures of the as-synthesized powders were characterized using XRD and SEM analyses. The grain size of the obtained TiN powders was of the submicron order for anatase phase. On the other hand, a full conversion was obtained for the rutile phase with a nearly micron size.

Keywords: Titanium nitride, Titanium dioxide, Rutile, Anatase, dynamic carbothermal reduction.

OA-23

SYNTHESIS, PHASE COMPOSITION AND MICROSTRUCTURE OF $\text{ZrO}_2\text{-Y}_2\text{O}_3\text{-rGO}$ COMPOSITE PRECURSORS OBTAINED BY SOL-GEL SYNTHESIS

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Despite zirconia based ceramics is technologically important and applied as solid electrolytes, the studies for new compositions with enhanced performance are carried out continuously. Zirconia-graphene composites are regarded as being promising alternative materials for various electrochemical devices. In particular, for advanced ceramics and metal matrix composites manufacturing graphene oxide (GO) and reduced graphene oxide (rGO) attract more and more attention. The key problem related to the synthesis of such composites is the homogeneous distribution of graphene derivative in the ceramic powder. One of the promising methods for homogeneous zirconia-graphene composite powders is the use of sol-gel synthesis. So, the aim of the work is to study the effect of the synthesis technique and the dehydration method on the phase composition and microstructure of the system $91\text{ZrO}_2\text{-}9\text{Y}_2\text{O}_3\text{-}x\text{rGO}$, $x = 0.25, 0.5 \text{ wt.}\%$

This method allows the variation of many parameters, including pH and dehydration way. Nitrates hydrates $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (Acros organics, Geel, Belgium, 99.9%) and $\text{ZrO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Acros organics, Geel, Belgium, 99.5%) were used to prepare a 0.1 M aqueous solution. A 1 M aqueous ammonia solution (LenReactiv Ltd, St Petersburg, Russia, c.p.) was used as a precipitant. A dilute salt solution was added dropwise to the ammonia solution at a rate of $\sim 1\text{--}2 \text{ ml/min}$. Reduced graphene oxide (rGO) was obtained by oxidation-reduction method from the highly-oriented pyrolytic graphite (modified Hammers technique). Graphene oxide (commercial Graphenea, Spain and obtained in this work) was introduced at the various stages of co-precipitation of $\text{Zr}(\text{OH})_4\text{-Y}(\text{OH})_3$ in ammonia solution, the initial salts solution and gel formed. The resulting composite gel was dried using two methods of dehydration: freeze-drying (Labconco, 1Lchamber, Kansas City, MO, USA; 293 K, 24 h, $P = 0.018 \text{ mm Hg}$) and drying in liquid nitrogen. The phase composition and microstructure of the powders were investigated by Raman spectroscopy (LabRam HR800), particle size distribution (PSD, Horiba partica LA-950), SEM (Zeiss Merlin).

In the present work for the first time method for introducing the modifying additive GO at the precipitation stage was developed for homogeneous composite powders synthesis. It was shown that the addition of graphene oxide leads to a significant change in the particle morphology of the resulting composite precursor powder. The introduction of graphene oxide leads to the increase in the agglomeration degree of particles during freeze-drying. It was confirmed that in case of composite powders freeze-drying is more effective way remove “unbound” water from the gel structure than the dehydration in the liquid nitrogen.

OA-24

COMPARATIVE STUDY OF La AND Zn IONS CO-DOPED MAGNETITE NANOSTRUCTURES SYNTHESIZED IN OXYGEN AND INERT ATMOSPHERE

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The present paper has been addressed to the Zn²⁺ and La³⁺ ions co-doped magnetite nanostructures prepared [(1-x)Fe₃O₄:Zn:xLa where x =0.1, 0.2 and 0.3] using co-precipitated method presence O₂ and N gas at room temperature. The characterization of the samples nanostructures were carried out by X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM) and optical properties was demonstrated by UV-visible-NIR spectrophotometers well as vibrating sample magnetometer (VSM). The crystallo-chemical aspects including crystallite size, lattice parameters, theoretical X-ray density and bulk density were robustly on Zn²⁺ ions ratio. The high absorbance of Zn_{0.5}La_xFe_{3-x}O₄ over 80% was found to be evinced in the visible wavelength range of 400–800 nm absorbance be above 97% and 99% in the visible region for prepared presenceO₂ and N gas at room temperature, respectively. Besides, the incorporation of Cu ions cation was reduced the direct band gap energy 1.89 eV prepared presenceO₂ gas 1.78 eV prepared presenceN gas. Magnetic properties indicate that the prepared Zn²⁺ and La³⁺ ions co-doped Fe₃O₄ nanostructures exhibit almost superparamagnetic behavior which saturation magnetization 32.87 emu/g and 24.79 emu/g were achieved for Fe₃O₄ sample preparedpresence O₂ and N gas at room temperature, respectively. Meanwhile, an examination of the magnetic properties revealed decrease in saturation magnetization with increasing La concentrations incorporation up to x=0.3.

Keywords: magnetite, nanostructures, co-precipitated method, N₂ and O₂ gas, Co-doped, optical properties; magnetic properties.

OA-25

STRUCTURAL CHARACTERIZATION OF LaFeO₃ PEROVSKITE NANOPARTICLES

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Under the irradiation of sunlight, colloidal nanocrystals can split water efficiently by photocatalysis. Suitable nanoparticles for this application absorb the sunlight and allow the transport of excited charge carriers to active sites on the surface where the reactions take place. Besides an acceptable performance, colloidal reactors must compose of abundant elements to compete economically.

LaFeO₃ nanoparticles are among the class of promising photocatalysts for water-splitting. However, the lack of understanding of the structure-activity relationship remains the most significant obstacle in increasing the catalytic performance. Extended X-Ray Absorption Fine Structure (EXAFS) measurements can probe the local structure of the bulk and the surface of small nanoparticles.

The presentation is focused on EXAFS measurements and data analysis by Reverse Monte Carlo (RMC) analysis of LaFeO₃ nanoparticles which provide partial pair distribution functions that contain the structural evolution of the local structure.

Acknowledgements: Funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Projektnummer 388390466 – TRR 247.

OC-26

EFFECTS OF CITRIC ION ON HEXAGONAL NaYF₄: Yb/Er PHASE FORMATION DURING SOLVOTHERMAL SYNTHESIS

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Up-converting nanoparticles (UCNPs) which have ability to emit a visible light upon excitation with NIR photons are with wide applications in optoelectronic devices, forensic, biomedicine and security. The synthesis of the most efficient hexagonal β-NaYF₄:Yb/Er phase in shape of uniformly sized spherical nanoparticles is usually

performed through thermal decomposition of complex organic precursor in high-boiling organic solvents, which rise deep concerns regarding their potential cytotoxicity. In this work, citric acid and Na-citrate are used for the stabilization of hexagonal polymorph during solvothermal processing of NaYF₄:Yb/Er phase from common inorganic precursor (rare earth nitrate salts). Additionally, effect of precipitation agent, i.e. fluorine source (NaF, NH₄F and NH₄HF₂) on the final particle morphology is deduced based on comprehensive scanning and transmission electron microscopy analyses. Detailed refinement of the X-ray powder diffraction data (XRPD) revealed simultaneous nucleation of a cubic and hexagonal phase during solvothermal reaction. The use of citric acid resulted in formation of micro- and nano- spherical particles of a hexagonal and cubic NaYF₄:Yb/Er phase, respectively, while Na-citrate provoked nucleation of well crystallized hexagonal crystals of β -NaYF₄:Yb/Er phase. Size of the crystallites is determined by the choice of the precipitant, and is smallest in the case when NH₄F is used. All synthesized powders emit bright and intense green light due to the intensification of the $^2H_{11/2}, ^4S_{3/2} \rightarrow ^4I_{15/2}$ electronic transitions upon the excitation by the infrared light ($\lambda=978$ nm).

OA-27

LIGHT SCATTERING IN TRANSPARENT CERAMICS: USE AND VALIDITY OF MIE THEORY APPROXIMATIONS

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Introduction: The main obstacle in the preparation of transparent ceramics is the elimination of residual porosity and other phases, which cause light scattering and therefore a reduction of transmittance. Mie theory is a useful tool for modeling light scattering in heterogeneous systems (such as transparent ceramics with a small content of pores or inclusions) and for the prediction of their level of transparency. However, Mie theory requires numerical calculations, and for some purposes it is more convenient to dispose of analytical tools for modeling and prediction purposes. There are popular approximations of Mie theory that can be used in specific contexts (Rayleigh, Fraunhofer), but their validity is strongly limited with respect to the range of wavelengths, scatterer sizes and refractive indices. On the other hand, a less well known approximation published by van de Hulst [1] has a much wider range of validity and seems to be a suitable approximation for at least some of the cases where the other popular approximations fail.

Aims: The main goals of this contribution are: comparison of the results obtained by Mie theory and its approximations (Rayleigh, Fraunhofer, van de Hulst) in the ranges of wavelengths, scatterer sizes and refractive indices relevant to transparent ceramics, comparison of the results of Mie theory and its approximations for suspensions with

spectrophotometric measurements and, ultimately, a general critical evaluation of the use of approximations for the prediction of transparency in heterogeneous systems.

Experimental: For Mie theory calculations a web-based application was used [2]. The results based on Mie theory approximations were calculated using formulas published in [3]. The suspensions for spectrophotometric measurements were prepared using ultrasonics to achieve sufficient deagglomeration. Calculations were made for polydisperse systems with particle size distributions based on laser diffraction measurements.

Results and conclusion: It is shown that the van de Hulst approximation, a less well known approximation to Mie theory, is a simple and powerful analytical tool for modeling and predicting the transmittance of heterogeneous systems, including suspensions with polydisperse particles and transparent ceramics with residual pores and second-phase inclusions.

Acknowledgement: This study is part of the project GA18-17899S (GAČR).

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OA-28

COLOR AND OPTICAL SPECTROSCOPIC ANALYSIS OF Cu_2O - K_2O - ZnO - P_2O_5 GLASS MATRIX

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A glass matrix with a chemical composition as $42\text{P}_2\text{O}_5$ 42ZnO $(16-x) \text{K}_2\text{O}$ $x\text{Cu}_2\text{O}$ with ($x = 0, 1, 2, 3, 4, 5$ and 6) were fabricated. A good benefit of using potassium oxide rather than any alkali metal oxides, the potassium oxide is the lower melting point of all alkali metal oxides, and this can be excellent saving energy for the glass manufacturing. The green glass bandpass filters have been prepared using conventional quenching melts technique. The effect of copper content on density has been measured by the conventional Archimedes method, using toluene as immersing liquid with a calculation

to the molar volume, the investigation found that the density increases with increasing the copper oxide contents with a vise versa to the molar volume. The investigation of the glass state has been measured by using XRD technique, the observation of that result shows no natural broadening peaks that forming crystals, which proofed the systems are completely in glass state. Color analysis, optical absorption, transmittance, refractive index and some optical properties has been measured and calculated in a wide wavelength range were carried out. Further to this, some initial theoretical investigation of the problems associated with the bandpass absorption glass filter will be carried out.

Keywords: glass technology, spectroscopic analysis, bandpass filter, photonic glass

OC-29

**PHOTOEMISSION SPECTROSCOPY ON ISOLATED SILVER-BASED
HYBRID NANOSTRUCTURES: A NOVEL APPROACH TO
UNDERSTAND FUNDMENTAL PROPERIES OF SOLAR CELL
ABSORBER MATERIAL**

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Solar cell absorbers based on organic-inorganic hybrid perovskite nanostructures such as $\text{CH}_3\text{NH}_3\text{PbX}_3$ or CsPbX_3 ($\text{X}=\text{Cl}, \text{Br}, \text{I}$) recently have gained immense popularity due to high photoconversion efficiency and low cost. However, a main disadvantage in potential large-scale application of these lead-based materials is in their toxicity and environmental impact. For these reasons, we developed a chemical synthesis procedure for preparation of silver bismuth iodide (Ag-Bi-I) hybrid nanostructures, which can be used as lead-free solar cell absorbers. In this contribution, we will present the results of the structural and spectroscopic investigation of Ag-Bi-I colloids prepared using different Ag:Bi molar ratios. Particular attention will be given to the photoemission spectroscopy studies on isolated hybrid nanostructures of this type performed at SOLEIL synchrotron facility. It will be shown that in comparison to the BiI_3 nanoparticles $\text{Ag}_x\text{Bi}_y\text{I}_{x+3y}$ nanoparticles exhibit additional low-energy absorption and valence band, leading to formation of the optical bandgap at ~ 2.25 eV and the photoionization threshold of ~ 6 eV, which suggests higher photoconversion efficiency. In addition, the results of the VUV angle-resolved photoemission spectroscopy on isolated $\text{Ag-Ag}_2\text{S}$ hybrid nanostructures will be presented and explained in terms of photoabsorption and photoelectron transport.

OC-30

**REMARKABLE IMPACT FOR OPTICAL PROPERTIES OF
SAMARIUM DOPED ALUMINUM SODIUM PHOSPHATE
GLASS FOR VISIBLE DEVICES**

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Glass composition $50\text{P}_2\text{O}_5\text{--}30\text{Na}_2\text{O--}10\text{CaO--}(10\text{-}x)\text{Al}_2\text{O}_3\text{--}(x)\text{Sm}_2\text{O}_3$, with different $\text{Sm}_2\text{O}_3(x)$ concentrations where x from 0 to 8 mol % were prepared via ordinary conventional melt annealing route. Combined structural and optical properties have been considered to explore the effect of Sm^{3+} ions in the prepared glass network. Structural and optical parameters such as molar volume, absorption coefficient, refractive index, extension coefficient, the optical energy gap were deduced. X-ray diffraction analysis (XRD) was used to approve the amorphous nature of the prepared glass samples. The density has been measured by using the conventional Archimedes method, the molar volume was calculated and found, and the density and molar volume are trended in the same direction by increasing the Sm_2O_3 contents. FTIR transmission spectra have been carried out. The most active peaks were observed in the region from 450 cm^{-1} to 1700 cm^{-1} . FT-IR spectra were analyzed to determine level the maintenance of main characteristic absorption band of the phosphate glass matrix. UV–VIS–NIR spectrometer was used to carry out the absorption spectra of the studied glass samples. The variations in the spectroscopic data are talk over in relation to the structural evolution affected by the deviations in composition and coordination state of samarium ions in the glass network.

Keywords: samarium ions; phosphate glass, aluminum sodium phosphate glasses; optical properties; optical band gap; refractive index, extinction coefficient, complex dielectric constant.

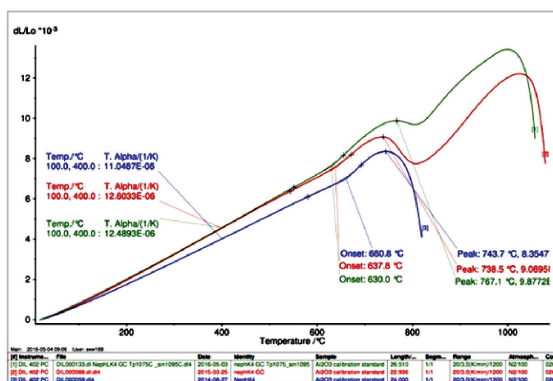
OC-31

EFFECT OF SINTERING TEMPERATURE ON CRYSTALLIZATION OF NEPHELINE-LEUCITE GLASS-CERAMICS

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Ball milling is an effective method of mechanically reducing the crystallite size after crystallisation. However, pre-crystallised glass particles tend to hinder the densification process during sintering. Introducing a green compact to a sintering temperature higher than the crystallisation one can allow for sufficient viscous flow to occur. However this can result in changes of the crystalline phase/s, size and morphology as well as the residual glassy phase composition. Therefore, the aims of this study were to investigate the effect of sintering temperature on crystallisation of alkali rich alumina silicate glasses. Wherein, two different glass compositions based on a multicomponent system were synthesised using melt-quench method. The experimental glass powders were prepared by ball milling for 1 hour and sieving below $<125\ \mu\text{m}$ (mean glass powder particles, $D_{50} = 25\ \mu\text{m}$). The prepared powders were divided into two groups one of which (group A, G1 and G2) was left as glass powders for sinter-crystallisation experiment from green compact; whereas the second (group B, GC1 and GC2) was heat-treated to produce glass-ceramics powders. The prepared glass-ceramics were ball milled for 1 hour back to find powders. Thus, sintering experiments were conducted on glass powders (G) and pre-crystallised glass powders (GC). Glasses and glass-ceramics were characterised before and after sintering using differential scanning calorimetry (DSC) Dilatometry (DIL), X-ray diffraction (XRD) and nuclear magnetic resonance (NMR). The crystalline microstructures of the glass-ceramics were analysed before and after sintering. Results illustrated a tendency towards a decrease in grain size with increasing the sintering temperature in group B (GC1 and GC2). This paper will discuss further the sintering results for both groups in relation to crystallisation, different crystalline phase developments, crystallite size and morphology.



OC-32

PREPARATION OF IRON/CARBON COMPOSITES

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In recent years carbon materials attracted a lot of attention because of their broad range of application. Carbon materials such as powders, foams, fibers, nanotubes and spheres are characterized by high surface area and low density simultaneously, thus they possess extraordinary properties such as high specific surface area, porosity, chemical and physical resistance and good electrical properties. Furthermore, in order to maximize the efficiency, carbon materials can be easily modified. Doping of the carbon matrix with other elements such as N or Fe can highly improve the adsorption and catalytic properties. One of the simplest methods to produce carbon materials is hydrothermal method, which allows to modify them through adding to the reaction pot proper compounds. Significant improvement of the hydrothermal method is applying the microwaves as a heating agent.

In presented work carbon materials doped with iron compounds have been synthesized. As a carbon precursor resorcinol-formaldehyde resin has been used. The modifiers were added into reaction pot before the polycondensation of the resin, thus high dispersion of the metal compounds in carbon matrix has been obtained. The reaction was carried out in the microwave solvothermal reactor for 15 min. The resulting product was carbonised at elevated temperature in the argon atmosphere. Finally, the physico - chemical properties of the composite materials were tested.

OA-33

SPARK PLASMA SINTERING OF Hf-C-N CERAMICS

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Thermal stability of Ta₄HfC₅ with highest known melting point (~3990 °C) was investigated by numerical modeling [1] and one was predicted that Hf_{0.53}C_{0.27}N_{0.2} melting point is ~200 °C higher. This assumption is untested yet but the first task is to obtain hafnium carbonitride.

Much earlier Hf-C-N-O materials was extensively studied using vacuum hot pressing of HfC, HfN, HfO₂, Hf and C powders mixtures [2]. The stoichiometric HfC_{0.58}N_{0.42} had extreme hardness (2400 HV) with N/C ~0.72 which is close to ~0.74 for Hf_{0.53}C_{0.27}N_{0.2} in Ref. 1. Apparently, such N/C ratio is optimal for perfect structure stability. Thus, we investigated solid solution formation and sintering behavior of Hf-C-N ceramics made from 3HfN-4HfC commercial powders mixture.

Stoichiometric solid solutions in [2] were synthesized from HfN and HfC powders at >2300 °C for 10–30 min under ~55 MPa. We used spark plasma sintering method and obtained the solid solution carbonitride phase at 1900 °C for 5 min under 60 MPa. Ceramic sample prepared at optimal conditions (2100 °C, 5 min, 60 MPa) is characterized by the average grain size of 10 μm, microhardness of 2000÷2300 HV and relative density >94%.

Acknowledgement: Russian Science Foundation (Project No. 18-73-00125) supported this work.

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OC-34

RHEOLOGICAL PROPERTIES OF GLASS-CERAMIC SEALS FOR SOC APPLICATION FABRICATED BY TAPE CASTING

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Solid oxide cells (SOC) are gaining an interest in an energy sector as an alternative way of producing energy due to their high electric efficiency and an environmentally-friendly nature. Electrochemical cell (SOFC) generates electricity while fuel and oxidant are consumed, though it can be reversed during operation and converted into solid oxide electrolyser (SOE) which produces hydrogen when energy is supplied. One of the most important obstacles to overcome while designing SOC units is the need for reliable sealing technology. The main properties of the seals are long-term stability in the high temperature SOFC/SOE environment (600–800 °C) and the value of thermal expansion coefficient of the seal material must be compatible with the steel and ceramic components of the unit. Furthermore, the glass slurry must have strictly defined viscosity so it will behave certain way while being cast on different substrates such as micanite and polyester foil.

The study was focused on defining the rheological properties of the glass slurries which enable shaping of homogenous tapes of defined thickness. The second step was to manufacture and test the glass-ceramic and hybrid (glass-ceramic-mica) seals whose main task is to prevent the air and fuel from mixing during operation in high temperature and low overpressure. The manufactured seals were finally applied in a SOFC stack designed at the Institute of Power Engineering. For this experiment, 3 anode supported cells of 100×100 mm size were used and the stack was operated in 650 °C with the supply of air and fuel. This experiment confirmed that the application of the manufactured seals ensured very high gas-tightness that allowed to reach desired effect.

OA-35

APPLICATION OF SHELL MOLDS STRENGTHENED BY METAL POWDERS AND GLASS FIBRES IN THE FABRICATION OF JET ENGINE COMPONENTS

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Fabrication of several ceramic layers around the wax pattern is a mainly time-consuming stage in the investment casting process. The ceramic shell mold should be thick enough to withstand the thermal stresses generated during the melt-pouring of the superalloy. The number of layers is designed in order to ensure sufficiently high strength but from the other side can increase the time of drying and preheating of the mold. In this work, the new approach in the manufacturing of ceramic shell mold for the aerospace industry was presented. Five Ni-based superalloy IN713C castings were fabricated in the shell molds strengthened by glass fibers and metal powders, included in the slurry and coverage respectively. Mechanical tests revealed higher green strengths of all modified molds in comparison with mold without additives. Light microscopy revealed that the modification of shell mold had a strong influence on stereological parameters of equiaxed grains. Based on the scanning electron microscopy observation the volume fraction of γ' precipitates and primary carbides were calculated. The addition of glass fibres and metal powders into the ceramic mold allowed obtaining the high-quality castings with the proper microstructure without metallurgical defects.

Acknowledgement: The authors gratefully acknowledge the funding by National Centre for Research and Development Poland under grant LIDER/227/L6/14/NCBR/2015 “New technology for investment casting manufacturing critical engine components with a new generation ceramic materials”.

OC-36

PROCESSING, CHARACTERIZATION AND MECHANICAL PROPERTIES OF CERIA CALCIA STABILIZED ZIRCONIA CERAMICS WITH α -ALUMINA ADDITION

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Zirconia ceramics stabilized using 10 mol% CeO₂ and 1 mol% CaO were tested with the addition of small amounts of α -alumina. The elaboration process of five different compositions was done by wet mixing of powders using 0, 2.5, 5, 10 and 15 wt.% α -alumina. The final powders were pressed uniaxially and isostatically. Conventional sintering was done at 1450 °C during 2 hours, obtaining full density materials. The microstructure, mechanical properties (hardness, scratch, strength and nanoindentation) and resistance to hydrothermal degradation were evaluated. Grain size reduction was achieved leading to an enhancement in the mechanical properties like hardness and strength obtaining values superior to 10 GPa and 1000 MPa respectively. The hydrothermal degradation resistance was maintained and even improved by the addition of alumina, finding only up to 6% of monoclinic volume phase after 60 hours.

OA-37

IN SITU INVESTIGATION OF CRACK PROPAGATION IN CERAMICS BY MICRO-CT TECHNIQUE

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Ceramics have wide-spread applications in many areas due to their chemical stability, electronic insulator properties and heat-resistance. Although these materials have great strength with high Young's moduli, their brittleness is a disadvantage that

needs to be improved so they could be used in a wider range of applications. Enhancing the mechanical properties of brittle materials is a difficult task, as they tend to undergo catastrophic failure events due to random crack propagation through the structure. One possible approach towards improving these materials is to understand the cracking methods in these structures. There are many well-known methods for characterizing different ceramic samples. These methods can easily investigate any occurring external cracks on the surface of the structures, but our options are limited in case of internal fault locations. One emerging technique for investigating their inner structure is the computed tomography (CT) method. CT is a non-destructive, three-dimensional imaging method, by which we can investigate the internal structure of materials. The method is based on the distinct X-ray attenuations of the various materials. Compared to traditional CT devices, micro-CT can reach a spatial resolution as high as 0.4 μm . Samples up to $20 \times 20 \times 20 \text{ cm}^3$ volume can be measured but the maximum resolution of the scan depends on the size of the sample (because of geometrical magnification). With a so-called material testing stage (MTS) it is possible to investigate the effect of well defined (vertical direction) compression or tension on samples up to 2 cm diameter, with loads up to 440 N while simultaneously monitoring the deformations caused by the different mechanical impacts. With this method we were able to identify micro-cracks inside ceramic structures, which occurred either during the production of the samples or caused by external mechanical stress. Moreover we were able to distinguish the originally present cracks from the newly formed ones during compressive tests. We were also able to follow crack formation and propagation in 4D with in situ mechanical testing. By understanding the inner processes during crack formation we can get one step closer to improve the quality of existing ceramic materials.

OA-38

**SELFPROPAGATING HIGH TEMPERATURE SYNTHESIS AND
STUDY OF CARBIDE AND DIBORIDE BASED ON REFRACTORY
HIGH-ENTROPY ALLOY Hf-Ta-Ti-Nb-Zr**

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The last few decades, special attention has been paid to research in the field of high-entropy materials. In contrast to the conventional notion of alloys, which are usually based on a single metal, which is alloyed in order to improve the properties of an

alloy - high-entropy alloys, by definition, contain five or more elements with an equimolar ratio (or close to it) of each alloy component. At the moment, more than 300 compositions of the most commonly used metals used in industrial alloys have been studied. However, interests in the study of high-entropy materials are not limited to alloys, and the class of high-entropy alloys has been expanded to create ceramics - namely, carbides and borides. However, interests in the study of high-entropy materials are not limited to alloys, and the class of high-entropy alloys has been expanded with create ceramics - namely, carbides and borides. Of particular interest is the study of ceramics based on refractory metals such as Hf, Ta, Ti, Nb, Mo, W, Zr. High-entropic alloys based on refractory metals show high strength properties at room and elevated temperatures (up to 1200 °C). The prerequisites for deeper research in the field of high-entropy ceramics are justified by the industry's need for materials whose properties exceed those of special alloys. In many respects, ceramic materials have a much higher melting point, higher strength characteristics, and together with the "high entropy effect," there are all the prerequisites for studying these systems. It is also worth noting separately that the technology for producing refractory carbides and borides has not been studied, and the available methods for producing such compounds are in most cases limited to electric arc melting in vacuum. On the contrary, self-propagating high-temperature synthesis (SHS) allows to obtain powders of refractory carbides and borides without significant energy consumption.

In this work, studies of high-entropy ceramics obtained by the SHS method are combined. As a result, the method of mechanical activation prepared reaction mixtures of powders of high-entropy ceramics of compositions $\text{Hf}_{0.2}\text{Ta}_{0.2}\text{Ti}_{0.2}\text{Nb}_{0.2}\text{Mo}_{0.2}\text{C}$, $\text{Hf}_{0.2}\text{Ta}_{0.2}\text{Ti}_{0.2}\text{Nb}_{0.2}\text{Zr}_{0.2}\text{C}$, $\text{Hf}_{0.2}\text{Ta}_{0.2}\text{Ti}_{0.2}\text{Nb}_{0.2}\text{Mo}_{0.2}\text{B}_2$ и $\text{Hf}_{0.2}\text{Ta}_{0.2}\text{Ti}_{0.2}\text{Nb}_{0.2}\text{Zr}_{0.2}\text{B}_2$. Bulk samples were obtained in two ways. The first implied a preliminary synthesis of powders and subsequent sintering in a spark plasma sintering installation. The second method is a new approach to the synthesis - carrying out the so-called reaction sintering, when the combustion reaction is initiated in the amount of powder to be poured, sparks generated by the installation. Bulk samples after sintering showed high hardness characteristics (up to 30 GPa), bending strengths. The thermal conductivity coefficient and the electrical resistivity coefficient were also measured.

OC-39

**PROCESSING AND PROPERTIES OF SILICON NITRIDE + MWCNTs
COMPOSITES PREPARED FROM OXIDIZED
 α -Si₃N₄ STARTING POWDER**

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The α -Si₃N₄ starting powders were oxidized at 1000 °C in the ambient air environment for 10 and 20 hours. HREM results showed an amorphous layer of SiO₂ on the nano-structured α -Si₃N₄ particle was formed in the result of reaction between Si₃N₄ and O₂ at higher temperature. These oxidized α -Si₃N₄ powders were subjected to mix with the 3 wt.% MWCNTs by attritor milling for 4 hours followed by dry pressing under 200 MPa pressure to produce green samples. The green samples were sintered at 1700 °C, 20 MPa in nitrogen (N₂) gas environment for 3 hours as a holding time. XRD results revealed the complete transformation of α to β phase in MWCNTs reinforced Si₃N₄ composite. X-ray diffractograph could not detect MWCNTs and Si₂N₂O in the composite but MWCNTs were observed in the SEM micrographs of fractured surfaces. More likely MWCNTs reacted with the oxygen and formed the CO/CO₂ gases at higher temperature during sintering process. At first, SiC formed in the result of reaction between CNT and Si₃N₄ and SiC is unstable above 1540 °C to Si₃N₄ CO/CO₂. The slight increase in apparent density with the increase of oxidation time was observed. The flexural strength and hardness also increased which is attributed to the increase in apparent density.

Keywords: silicon nitride, oxidation, carbon nanotubes, hot isostatic pressing (HIP).

Acknowledgement: Awais Qadir thanks the Stipendium Hungaricum Scholarship Program and Doctoral School of Materials Science & Technologies, Obuda University, Budapest for the financial support.

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OA-40

**THE EFFECT OF HEATING RATE ON THE GRAIN GROWTH OF
ALUMINA PREPARED VIA ELECTRIC CURRENT ASSISTED
SINTERING (ECAS)**

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The grain growth of alumina ceramics was investigated on samples prepared via electric current assisted sintering (ECAS), better known as spark plasma sintering (SPS), at different temperatures (900–1300 °C) and heating rates (5–100 °C/min). Ordinarily it is expected that the grain size is smaller with higher heating rate. However, in the context of alumina prepared via ECAS, the opposite case – smaller grain size with lower heating rate – is frequently reported. The aim of this work is to clarify this discrepancy in the literature about the effect of heating rate on the resulting grain size in the case of alumina ceramics. The microstructure of the prepared samples was observed via scanning electron microscopy (SEM) and the grain size was determined by means of stereology-based image analysis.

Acknowledgement: This work is part of the project “Partially and fully sintered ceramics - processing, microstructure, properties, modeling and sintering theory” (GA18-17899S), supported by the Czech Science Foundation (GAČR).

OA-41

**DIAMOND ELECTRODES FOR ELECTROCHEMICAL
APPLICATIONS**

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

One of the most efficient and promising applications of boron-doped diamond (BDD) electrodes is their use for the electro reduction of compounds that are difficult to reduce. This is the case of nitrates, which in the environment represent a health risk. The electrochemical reduction of nitrate is a multielectronic charge transfer process and the reaction mechanism is very complex. A large variety of nitrogen compounds can be formed such as NO₂, NO, N₂O, N₂, NH₂OH, N₂H₄ and NH₄⁺ (NH₃) which are dissolved in the solution or are volatile compounds. The reduction potential and the nature of the products of nitrate reduction strongly depend on experimental conditions such as pH, applied potential, and coexisting ions.

The subject of our research work is the investigation of boron doped nanocrystalline diamond layers functionalized with photosensitive molecules. The diamond thin films were deposited on silicon substrate by Hot Filament Chemical Vapour Deposition (HFCVD) technique from a mixture of methane and hydrogen. As photosensitive species were chosen manganese phthalocyanine molecules. To optimize the process of the attachment of the photosensitive molecules, the diamond layers were preliminary modified with UV/O₃ - processing, O₂-plasma and NH₃/N₂-plasma.

The modified and functionalized with phthalocyanine samples were used as working electrodes in three-electrode cell. Potassium nitrate was used as electrolyte. The electrochemical properties of the obtained layers studied by Cyclic Voltametry (CV) show the wide potential range (almost twice larger in comparison to the classical Pt electrode) and photosensitivity of the diamond films functionalized with phthalocyanine. In the present work we have studied the influence of the chemical nature of the cation, (K⁺) on the reactional mechanism at BDD electrodes and analyze the dissolved and volatile species using spectrophotometry and chromatography.

OA-42

INFLUENCE OF ELECTRIC CURRENT ON MICROSTRUCTURE OF Al DOPED ZnO

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Spark plasma sintering (SPS) utilizes an electric field or current and uniaxial pressure to consolidate powders. Compared to conventional methods, SPS enables the densification of nanostructured materials at lower temperatures in shorter times.

The reason for the improved and fast densification has been attributed to efficient diffusion processes under applied fields or currents, Joule heating, electromigration and modifications in grain boundary characteristics.

However, it remains a challenge to distinguish conventional thermal effects from nonthermal field or current effects. Therefore, new experimental methods are needed, to identify the role of the electric current or field on the microstructure.

In the present work, a new method of SPS has been designed to elucidate the effect of electric current on the microstructure. An insulating die is used to force the electric current through the sample. Microstructures are compared for different sintering conditions.

OC-43

**PROPERTIES OF Nb₂O₅ COMPOSITE DOPED WITH ZrO₂
SINTERED BY MICROWAVE**

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Niobium oxides can lead to many different and interesting properties, making it a very versatile group of materials. Specifically, niobium oxides have been showing great potentiality in many technological applications such as solid electrolytic capacitors, transparent conductive oxides, and others. Niobium oxide is a colorless insoluble solid that is unreactive. Materials of zirconia and niobium oxide could improve the mechanical properties by combining the advantages of both materials. Microwave sintering is a non-conventional technique, which allows the modification of the densification mechanisms through fast processing and a volumetric heating of material. In this case, the energy conversion of microwave electromagnetic radiation into heat by the material itself is due to the materials dielectric properties.

The aim of this work is to investigate the microstructural and mechanical properties of Nb₂O₅ doped ZrO₂ bioceramic materials. Samples partially stabilized-zirconia with 3 mol% of yttria doped with 0.5 and 1.5 mol% of Nb₂O₅ were sintered by a non-conventional technique of microwave sintering at 1200 °C for 5 min and conventional sintered at 1300 °C and 1400 °C for 2 h. The mechanical properties were evaluated through hardness, fracture toughness and Young modulus. The microstructures were characterized by FESEM.

OA-44

**EFFECT OF TWO-STEP SINTERING ON MICROSTRUCTURE OF
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High-density dielectric, piezoelectric, and ferroelectric ceramics (>95 %TD) are required for most of the applications of these ceramics. Both dielectric permittivity and piezoelectric constant usually decrease with decreasing density, because the pores or vacuum have no dielectric and piezoelectric effect [1]. Most of the Pb-free dielectric, piezoelectric, and ferroelectric ceramics require high-temperature sintering for high-density fabrication. However, high- temperature sintering may lead to abnormal grain growth. Thus, abnormally large grains form in the BTO ceramics mixed with small grains during sintering. Since grain boundaries act as sinks for porosity depletion, abnormal grain growth inhibits porosity depletion during the sintering.

Many approaches are suggested in the literature to reduce the sintering temperature of these ceramics. Some researchers have suggested two-step sintering of fine/ultrafine powders for this purpose. For example, in 2007, Karaki and coworkers suggested fabrication of high-density barium-titanate ceramics (5.91 g/cm³) by the two-step sintering approach using hydrothermally synthesized BaTiO₃ nanoparticles of 100-nm average size. After 2-stage sintering at $T_1=1320$ °C and $T_2=1150$ °C for, in-turn, $t_1=1$ minute and $t_2=15$ hours, they achieved BaTiO₃ ceramics with an average grain size of 1.6 μm and with improved dielectric and piezoelectric properties [2].

A commercially available BaTiO₃ powder, CAS No. 208108, Sigma-Aldrich GmbH, was used as the starting materials to produce suitable barium titanate granules for ceramic shaping. The starting powder was deagglomerated for 20 hours in a planetary ball mill system using 1-cm stabilized ZrO₂ balls. An appropriate solvent to powder ratio (15 g of ethanol to 6 g of BaTiO₃ powder) as well as a suitable grinding ball to powder mass ratio (ball/powder mass ratio of 6/1) was used. Then, an aqueous 30 wt.% BaTiO₃ suspension was prepared using proper amounts of ammonia, Dolapix CE 64, and Poly Vinyl Alcohol (PVA) for 67.5 wt.%, 1 wt.%, and 1.5 wt.% (of the powder's mass), respectively. The suspension was magnetically mixed at 50 °C using a magnet stirrer followed by mixing in an ultrasonic bath for 15 minutes, and in a ball mill jar for 1 hour. The final suspension was dried in a SPRAY DRY machine at 150 °C to produce BaTiO₃ granules.

The granulated powder was shaped into small pellets by a lab scale uniaxial press machine (UAP) at 200 bar and subsequently compacted by a cold isostatic press (CIP) to achieve samples with uniform green density before sintering. The compaction time was selected constant at 5 minutes during both of the shaping stages. Different two-step sintering regimes were examined to achieve near-theoretical-density (~6.025 g/ cm³) ceramics. Table 1 displays the effect of the shaping and the sintering parameters on the

green and final densities of the shaped ceramics, which both were measured based on the Archimedes principle using a density determination kit. The density measurements confirmed fabrication of near-theoretical-density (99.5 %TD) ceramics, which indicates the development of a compact and pore-free BaTiO₃ microstructure (Fig. 1). Fabrication of high-density and pure BaTiO₃ ceramics necessitates sintering about 1300 °C. However, Fig. 1 shows that dense and homogeneous microstructure is developed after sintering at 1240 °C.

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Table 1. Shaping/sintering parameters and green/final densities of the produced ceramics.

Sample	T ₁ (°C)	t ₁ (min)	T ₂ (°C)	t ₂ (min)	Green density ratio		Final density ratio
					After UAP ²	After CIP ¹	
1	1380	6	1240	20	68%	83%	93%
2	1380	6	1280	20	70%	84%	92%
3	1380	15	1240	20	73%	84%	99.5%

¹Cold Isostatic press

²Uniaxial press

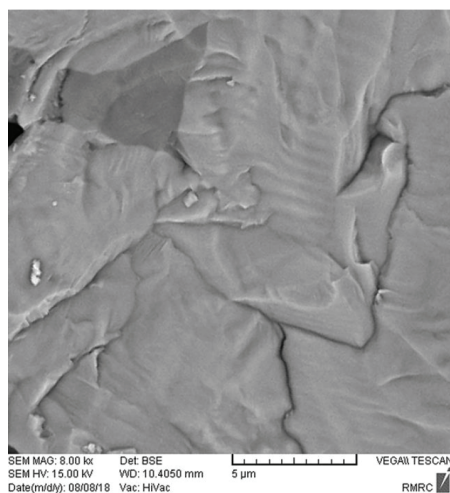


Figure 1. Backscattered electron micrograph of the sample 2 in Table 1

OC-45

FABRICATION OF SILICON NITRIDE REINFORCED BY SILICON CARBIDE BY FLASH SPARK PLASMA SINTERINGMohammad Abedi¹, Dmitry Moskovskikh¹, Alexander Mukasyan²¹*National University of Science and Technology MISiS, Moscow, 119049 Russia*²*Department of Chemical and Biomolecular Engineering, University of Notre Dame, Notre Dame, IN, 46556 USA*

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Fabrication of silicon nitride (Si_3N_4) attracted much attention due to its unique properties such as high mechanical properties at room and high temperatures, oxidation resistance, low coefficient of thermal expansion, and low density in comparison to refractory metals as well as its potential applications as electronic substrate materials [1]. Furthermore, the high-temperature strength and creep resistance can be enhanced by using silicon carbide (SiC) particles as a reinforcement in the Si_3N_4 matrix. Albeit, sintering of Si_3N_4 in the solid state sintering is very difficult due to its very strong covalent bonding and low self-diffusion constant. To overcome this obstacle, the silicon nitride consistently densifies in the liquid phase sintering regime by aiding of some additives.

Previously, the $\text{Si}_3\text{N}_4/\text{SiC}$ composites have been fabricated by hot pressing, as well as spark plasma sintering (SPS). Recently, a novel method of sintering has been developed which is known as flash sintering which is capable of the fully densifying different type of material less than one minute [2]. Recently, other methods of flash sintering were developed by inspiring of the basic model. One of these techniques is flash spark plasma sintering (FSPS), which works by a modification of tooling setups of conventional SPS [3].

In this work, we produced a $\text{Si}_3\text{N}_4/\text{SiC}$ composite with additions of Y_2O_3 by FSPS technique. The raw powder mixture was prepared by high-energy ball milling technique. The SiC phase content varies in the range 15–25 wt.% and the weight fraction of Y_2O_3 was wt.%. The microstructure of the silicon nitride reinforced by SiC was investigated by SEM microscopy. Furthermore, the mechanical properties of obtained ceramics including the fracture toughness and Vickers hardness are measured and analyzed. It is found that the hardness and the fracture toughness of obtained nanocomposites grow by increasing reinforcement content.

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OA-46

**STRUCTURE AND MAGNETIC PROPERTIES OF MULTIFERROIC
BiFeO₃ AND Bi_{1-x}Ho_xFeO₃**

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Magnetoelectric multiferroics, showing simultaneous ferroelectric and ferromagnetic ordering, have aroused wide attention in recent years, because they offer a wide range of potential applications in data storage media, spintronics and multi-state memories. The influence of Ho doping on the crystal structure and magnetic properties of bismuth ferrite (BFO) nanopowders was investigated. BiFeO₃ and Bi_{1-x}Ho_xFeO₃ ultrafine nanopowders were synthesized by the hydrothermal method. Here we use simple, low-cost and energy-saving hydrothermal method, which has advantages over the conventional methods. The diffraction pattern was recorded at room temperature and atmospheric pressure in the absence of any re-heating of the sample. A fitting refinement procedure using the Rietveld method was performed which showed the incorporation of Ho³⁺ ions in the BiFeO₃ crystal lattice, where they substitute Bi³⁺ ions. All the samples belong to *R3c* space group. In addition, theoretical investigation using bond valence calculations have been performed in order to mimic pure and Ho doped BiFeO₃ compounds produced in the experiment. Magnetic behavior of synthesized materials was investigated by SQUID magnetometer in wide temperature interval (2–800 K). Splitting between the zero-field-cooled and field-cooled magnetization curves becomes more pronounced as the Ho concentration is increased, pointing to the development of weak ferromagnetic moment, which is usually connected with uncompensated spins or spin canting. Hysteresis loops show the same fact, attaining higher magnetization with more Ho included, and becoming wider, i.e. magnetically harder.

OA-47

**MAGNETOELECTRIC PROPERTIES OF MULTIFERROIC
AURIVILLIUS TYPE $\text{Bi}_7\text{Fe}_3\text{Ti}_3\text{O}_{21}$ CERAMICS MODIFIED BY
STRONTIUM**

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Multiferroic Aurivillius type structure ceramics, with the general formula $\text{Bi}_{m+1}\text{Fe}_{m-3}\text{Ti}_3\text{O}_{3m+3}$ have been studied intensively last years. It is due to their potential applications such as ferroelectric random access memories, high temperature piezoelectric transducers etc. [1–4]. The aim of our investigation was to obtain the undoped ceramic material $\text{Bi}_7\text{Fe}_3\text{Ti}_3\text{O}_{21}$ and modified by strontium $\text{Bi}_{7-x}\text{Sr}_x\text{Fe}_3\text{Ti}_3\text{O}_{21-0.5x}$ where x changed from 0 to 1.2. The multiferroic materials were prepared by solid state reaction from simple oxides and carbonate. Influence of strontium doping on the microstructure and magnetoelectric properties was investigated. The dependence of the magnetization of the magnetic field function in several temperatures was examined. A magnetic hysteresis loop was observed at 2 K. In contrast, at higher temperature, no hysteresis loop was observed, which indicates the antiferromagnetic properties of this material. Magnetization was fitting by using theoretical formula:

$$M = NgJ\mu_B B_J(x)$$

where B_J is the Brillouin function [5]

$$B_J(x) = \frac{2J+1}{2J} \operatorname{ctgh} \frac{(2J+1)x}{2J} - \frac{1}{2J} \operatorname{ctgh} \left(\frac{x}{2J} \right)$$

The number of magnetic ions was determined by fitting eq.1 to experimental data

Keywords: multiferroics materials, Aurivillius phases, $\text{Bi}_7\text{Fe}_3\text{Ti}_3\text{O}_{21}$, magnetoelectric properties, dielectric measurements, impedance spectroscopy.

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OA-48

INFLUENCE OF BALL MILLING TIME ON PREPARATION AND DIELECTRIC PROPERTIES OF LEAD FREE $K_{0.5}Bi_{0.5}TiO_3$ CERAMICS

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The search for lead-free materials is one of the priorities of modern material science and technology. However, among a large group of lead-free materials, only a small group can replace those materials that contain toxic lead. This is due to the fact that these materials must have properties similar to or better than those containing lead in their composition. One of the most prospective materials of recent years is potassium-bismuth titanate $K_{0.5}Bi_{0.5}TiO_3$ (hereinafter referred to as KBT). The article presents technological conditions and their microstructure, electric and dielectric properties. The results of apparent density studies using the Archimedes method were presented. Microstructure investigations were performed using a high-resolution scanning electron microscope SEM. The structure of the samples was determined for using X-ray diffraction at room temperature. The dielectric measurements were carried out in the temperature range from 293 K to 873 K. The Curie-Weiss constant and activation energy for the study KBT samples were estimated.

OA-49

STRUCTURE AND ELECTRICAL PROPERTIES OF TiO_2 - V_2O_5 - P_2O_5 OXIDES

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Materials, based on phosphate and transition metals oxides provoke the scientist's interest in the last decades, due to their interesting properties and different electrochemical applications.

Oxides of the ternary system $\text{TiO}_2\text{-V}_2\text{O}_5\text{-P}_2\text{O}_5$ were prepared through a solid-state reaction, using melt quenching method in the temperature range 1000–1100 °C and air environment. The phase formation of the samples was investigated with XRD analysis. The synthesized materials were structurally characterized with IR and XPS spectroscopic techniques. DTA analysis was used for studying the thermal behavior of the samples. Electrical measurements were performed by impedance spectroscopy at different temperatures and frequencies in air environment. It has been established, that the conductivity of the oxide materials could have different nature and depends on their composition and structural features. Samples with high V_2O_5 content show predominant polaron conductivity, while oxides with high P_2O_5 concentration possess ionic or mixed conductivity.

OC-50

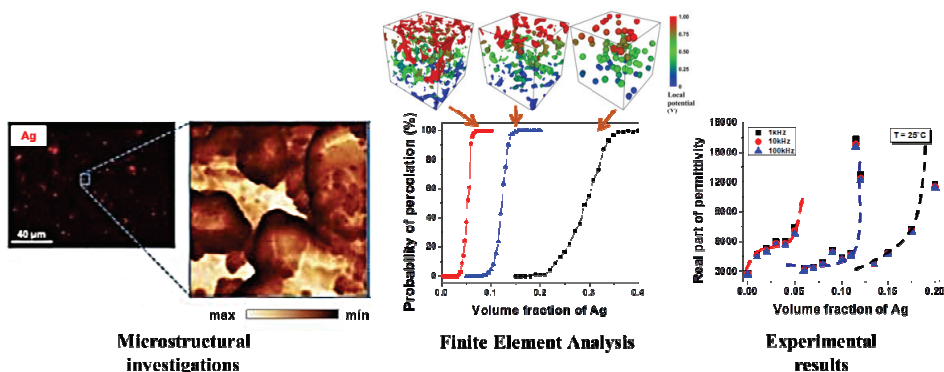
Ag-BaTiO₃ COMPOSITE CERAMICS WITH MULTIPLE PERCOLATIVE BEHAVIOR

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Achieving sub-percolative properties in metal-dielectric composites is an important tool for high permittivity applications. However, the percolation threshold is strongly dependent on filler particle size and phase interconnectivity, as well as on porosity. These parameters are very difficult to be controlled when composition is varied. In the present work, Ag-BaTiO₃ ceramic composites with multiple percolation compositions were investigated by a combined experimental and modeling approach based on finite element method in order to gain a comprehensive insight into the structure-properties relationship of sub-percolative Ag-BaTiO₃ composite ceramics.



Di-phase BaTiO₃ ceramics with (0–20) vol.% Ag addition were prepared by sintering at 1200°C/2h (above silver melting point). Dielectric investigations demonstrated the existence of three critical compositions with percolation features. Combined micro-nanoscale investigations evidenced three types of microstructures, which were used as input for FEM calculations to estimate the electrical properties and the percolation threshold for each individual case: randomly mixed phases, (0-3) Ag grains embedded into the ceramic matrix and structures with ceramic grains partially or completely covered by melted silver. The simulations explain the experimental trends of multiple percolative behavior of Ag-BaTiO₃ composite ceramics.

Acknowledgements: This work was supported by the UEFISCDI Romanian PN-III-P4-ID-PCE-2016-0817 grant.

OA-51

PREPARATION AND CHARACTERIZATION OF Bi_{0.5}Na_{0.5}TiO₃ BASED PIEZOELECTRIC CERAMICS

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Over the time, fossil fuels have been considered as the primary source of energy. Over-consumption and harmful impact on the environment and health are the main reasons why the world should seek for other energy sources. Some well-known alternative sources such as solar and wind energy are being intensively exploited due to their sustainability and environmental friendliness. For the last few decades, the piezoelectric energy harvesting technology, which converts waste mechanical energy into electricity, is in the center of research. Lead based materials such as PZT have attracted major attention. Despite their excellent piezoelectric properties, toxicity of lead limits their application in energy-related technologies. Bismuth sodium titanate (Bi_{0.5}Na_{0.5}TiO₃) based materials have been recognized as suitable candidates to replace lead-based materials due to their good piezoelectric properties and high Curie temperature.

The synthesis of (1-x)Bi_{0.5}Na_{0.5}TiO₃-xBaTiO₃ powder was performed by hydrothermal method in alkaline environment. Bismuth nitrate, barium nitrate and titanium (IV) butoxide were used as starting materials. Sodium hydroxide was used as a sodium source and mineralizer. Samples with different amount of Ba²⁺ (up to 10 at.%) were dried, pressed into pellets and sintered at different temperatures up to 1150 °C. Influence of Ba²⁺ addition as well as a sintering temperature on the structure and functional properties of Bi_{0.5}Na_{0.5}TiO₃ were examined by XRD, scanning electron microscopy and dielectric measurements.

OA-52

SCALE DEPENDENT PHENOMENA IN BaTiO₃-BASED CERAMICS

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The continuous trend of miniaturization in microelectronics attracts a broad interest concerning scale dependent phenomena in ferroelectric materials. Beside preserving the functional properties comparative to ones of bulk material when reducing the size down to nanoscale, there is also a specific interest regarding the maximization of dielectric, piezoelectric and ferroelectric properties at some specific grain size. Concerning this aspect, the researchers dedicated mainly to the critical grain size (GS) of BaTiO₃(BT) around 1 μm , for which enhanced properties have been found, irrespective of the synthesis procedure and sintering method [1,2]. However, systematic studies of critical GS in BT-based compounds have not been previously performed.

We have investigated BT and 5%Zr-BT(BZT) ceramics [3,4] with variable GSs in order to check for a similar possible critical value characterized by enhanced properties. The BZT composition is characterized at room temperature by the superposition of orthorhombic and tetragonal polymorphs, whose relative weight is expected to be affected by grain size. Dense ceramics with GS in the range of 450 nm–140 μm (BZT) and 75–2250 nm (BT) have been produced by traditional and Spark Plasma sintering.

Impedance spectroscopy has been employed for dielectric characterization and description of relaxation phenomena. The ferro-para phase transition broadens and slightly shifts towards higher temperature values, when reducing GS below 10 μm . In case of BZT ceramics, the room temperature permittivity takes values in the range of 1000 for fine ceramics (450 nm) and 2500 for coarse ceramics (above 50 μm), showing a pronounced increase in the range of 1–10 μm . The present results indicate the possibility of a critical GS for BZT compound in the range of 1–10 μm .

Acknowledgements: This work was supported by the UEFISCDI Romanian PN-III-P4-ID-PCE-2016-0817 grant.

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OA-53

INFLUENCE OF RARE-EARTH IONS ON THE PROPERTIES OF BiFeO₃ CERAMICS

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Multiferroic materials have recently attracted widely attentions, because they showing both ferromagnetic and ferroelectric ordering in a single phase and also because of potential in advance device applications and fascinating basic physics [1,2].

The bismuth ferrite is one of the rare multiferroic materials with both ferroelectric behavior (Curie temperature $T_C = 830$ °C) and antiferromagnetic ordering (Neel temperature $T_N = 380$ °C) above the room temperature. BiFeO₃ has a spin-modulated cycloidal magnetic structure with modulation period of ~62 nm. Although bismuth ferrite BiFeO₃ has a very good physical properties, still there are attempts to improve the properties of ferroelectric and ferromagnetic [4]. The doping BiFeO₃ is of interest due to the possible changes in properties of the resulting material, therefore are used the partial substitution of Bi³⁺ ions by rare earth ions resulting in improved multiferroic properties of BiFeO₃ [3].

In the present work the influence of rare earth elements concentration (0–10 at.%) on BiFeO₃:RE (RE=Gd³⁺, Dy³⁺, Nd³⁺) ceramics were studied.

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OA-54

**ROLE OF POLAR DEFECTS IN THE ORIGIN OF RELAXOR
BEHAVIOUR IN NIOBIUM MODIFIED BARIUM TITANATE
POLYCRYSTALS**

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Relaxors are a sub-class of ferroelectric polycrystals with peculiar material properties such as frequency dependent dielectric dispersion and absence of remanent macroscopic polarization. In ABO₃ perovskites, relaxor behaviour can be induced by substituting homovalent or heterovalent ions in A or B crystallographic sites. The charge, ionic size and electronic configuration of the substituent defines the substitution site and induces relaxor behaviour by different mechanisms. In this work, heterovalent (Nb⁵⁺) substituted barium titanate (BNbT) polycrystals were produced and studied to evidence the difference in origin of relaxor behaviour compared to homovalent (Zr⁴⁺) substituted barium titanate solid solutions. Experimental and theoretically modelled Raman spectra (RS) are presented to highlight the peculiarities of BNbT with reference to other systems. The extent of lattice disorder is discussed comparing RS results to Nuclear Magnetic Resonance (NMR) spectroscopy. Finally, the nature of lattice disorder is discussed in detail using time resolved Piezoresponse Force Microscopy (PFM). The multi-scale material characterization approach used here suggests that polar defects trigger the most effective mechanism to disrupt the intrinsic order in barium titanate based lead-free ferroelectric lattices.

OA-55

THE PROPERTIES OF $(1-x)(0.5\text{PZT}-0.5\text{PFW})-x\text{PFN}$ CERAMICS DOPED BY Li

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During obtaining lead containing ferroelectric ceramics with perovskite structure (for example PFN and similar) the formation of the unwanted nonperovskite phase (for example pyrochlore phase) is a big problem. Related to this is a high dielectric losses and high dielectric conductivity. In works [1,2] it has been shown that it is possible to reduce these increase of electric conductivity by doping PFN, for example using Li-ions. We suppose that also in similar materials introduction of Li should lead to decreasing of electric conductivity. In our earlier works [3,4] we obtained and investigated solid solution $(1-x)(0.5\text{PbZr}_{0.53}\text{Ti}_{0.47}\text{O}_3-0.5\text{PbFe}_{2/3}\text{W}_{1/3}\text{O}_3)-x\text{Pb}(\text{Fe}_{1/2}\text{Nb}_{1/2})\text{O}_3$ (abbreviation PZT-PFW-PFN). Now we present the results of obtaining and investigating multiferroic ceramics $(1-x)(0.5\text{PZT}-0.5\text{PFW})-x\text{PFN}$ doped by various amounts of Li (abbreviation PZT-PFW-PFN:Li) with three values of x and three amounts of Li addition for every x . For obtained ceramic samples we have investigated microstructure, dielectric properties and electric conductivity. Our results show that Li-addition influences the dielectric permittivity and dielectric loss at high temperatures. These changes are in general similar like in PFN. Investigations of d.c. electric conductivity exhibited also the changes but decrease is rather more complicated than in PFN.

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OA-56

EFFECT OF SINTERING PARAMETERS AND NON-STOICHIOMETRY ON THE MICROSTRUCTURE OF $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$

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$\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ (NBT)-based solid solutions are promising candidates for replacement of lead-based piezoelectric perovskite ceramics, such as $\text{Pb}(\text{Zr,Ti})\text{O}_3$ (PZT), which are currently dominating the global ceramic piezoelectric market [1]. Little information is currently available about the influence of sintering parameters and stoichiometric ratios on microstructural characteristics of NBT. For this reason, we demonstrate how these parameters and ratios, as well as addition of a dopant (Yb_2O_3) change average grain and pore size and distribution and secondary phase content.

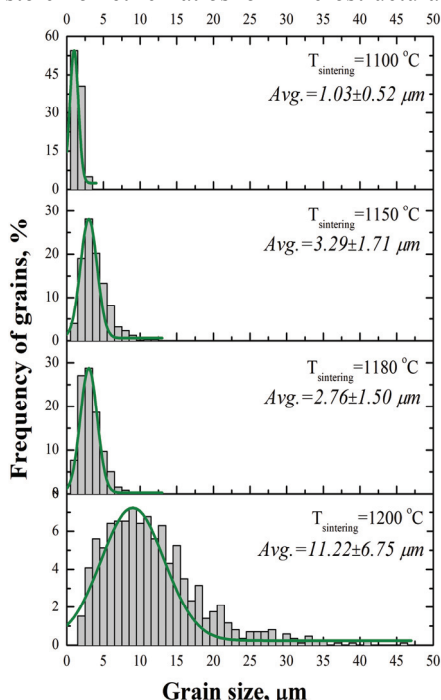


Figure 1. Grain size distribution of $\text{Na}_{0.5}\text{Bi}_{0.49}\text{Yb}_{0.01}\text{TiO}_3$ sintered for 3 hours at various temperatures

Bi over-stoichiometry suppresses grain growth and facilitates formation of unimodal grain size distribution in NBT, which can be explained by presence of liquid phase in Bi over-stoichiometric compositions. Loss of Bi during sintering has been mentioned in literature numerous times, which is proven by SEM-EDS results that show stoichiometric NBT compositions exhibiting a secondary Ti-rich phase, as opposed to $\text{Na}_{0.5}\text{Bi}_{0.52}\text{TiO}_3$. Addition of Yb promotes abnormal grain growth (AGG) and a bimodal grain size distribution, that can be reduced by adjusting suitable sintering conditions, such as sintering temperature and time. AGG along with average pore size can be effectively suppressed by decreasing both sintering temperature (Fig. 1) and time.

In addition to different sintering parameters and compositions, using sintering methods such as hot pressing, two-step and conventional sintering also leads to microstructural divergency.

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OA-57

DIELECTRIC AND ELECTRICAL PROPERTIES OF BLT CERAMICS MODIFIED BY Fe IONS

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The solid solution of the perovskite type structure $\text{Ba}_{0.996}\text{La}_{0.004}\text{Ti}_{1-y}\text{Fe}_y\text{O}_3$ (BLTF) for varying iron content ($y = 0.1 \div 0.4$ mol%) was obtained as a result of solid state reaction using the conventional method. At room temperature ($T_r < T_C$), the as-received ceramics reveals a single-phase with tetragonal structure and a $P4mm$ spatial group. An increase in the iron content causes a slight decrease in the volume of the elementary cell. In addition, this admixture significantly reduces the maximum permittivity value (ϵ_m) and the shift of the phase transition temperature (T_C) towards lower temperatures. The BLTF solid solution shows a sharp phase transition and low values of the tangent of the angle of dielectric loss ($\text{tg}\delta$), both at room temperature and in the phase transition area. The Curie-Weiss temperature (T_0) and Curie constant (C) were also determined on the basis of the dielectric measurements carried out. The analysis of temperature changes in DC conductivity revealed presence of the PTCR effect in the phase transition area. The conducted research allowed to obtain a new semiconductor ceramics material exhibiting posistor properties which will successfully find application in innovative electronic components, dedicated for application in modern mechatronic and automatic systems.

OA-58

STRUCTURE AND PROPERTIES OF THE LEAD-FREE PERVSKITE COMPOUNDS $(\text{Na}_{1/2}\text{Bi}_{1/2})\text{ZrO}_3$ (NBZ) AND $(\text{K}_{1/2}\text{Bi}_{1/2})\text{ZrO}_3$ (KBZ)

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Nowadays, most of the piezoelectric devices are using $\text{PbZr}_{1-x}\text{Ti}_x\text{O}_3$ solid solution (PZT). These compositions are the main piezo-ceramics due to their high electromechanical properties and low cost. However, since restrictions about the use of lead-compounds, due to health and environmental problems, there is a need to find new piezoelectric compounds in order to replace them. Currently, a few lead-free perovskites

are considered to be promising candidates: (i) BaTiO₃-based materials, (ii) alkaline-niobates, such as (Na,K)NbO₃, (iii) alkaline and bismuth titanates, such as ((Na, K)_{1/2}Bi_{1/2})TiO₃ (NBT and KBT), and (iv) solid solutions based on these compounds such as (Na_{1/2}Bi_{1/2})TiO₃-BaTiO₃.

This work is devoted to the study of two lead-free perovskites compounds: (Na_{1/2}Bi_{1/2})ZrO₃ and (K_{1/2}Bi_{1/2})ZrO₃ (NBZ and KBZ). These materials have first been reported by Smolenskii in 1967, who previously show the possibility to replace lead cation by an alkali/bismuth pseudo-ion (Na⁺/Bi³⁺ and K⁺/Bi³⁺) in PbTiO₃. Nevertheless, their structural and/or electrical properties have not been studied yet.

Thus, we first synthesized the two compositions and characterized it by X-ray diffraction (XRD) in order to identify the obtained phases. As expected, they led to perovskite compounds. However, secondary phases are observed, like in the previous publication for NBZ. These phases were difficult to identify: ZrO₂ is one of them but there is also some bismuth oxide with different stoichiometry due to the insertion of alkali or Zr⁴⁺ in the structure. A part of this work was to reduce the proportion of secondary phases in order to limit their impact on the structural search.

Then, we investigated the crystallographic properties of these materials using XRD and Neutron Diffraction and determined the space group of NBZ and KBZ. Finally, we studied the dielectric properties of these compounds from 80 to 450 K, to determine if they exhibit interesting properties. According to their space groups previously determined, none of these compounds are ferroelectric / piezoelectric, but they can have relaxor behaviour.

OA-59

SINTERING AND PREPARATION OF POLYCRYSTALS GADOLINIUM-IRON GARNET (Gd₃Fe₅O₁₂) BY THE SOLID-STATE REACTION

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Recently many scientists have focused on the topic of ceramic materials containing iron, due to their unique magneto-dielectric properties. The main advantage of magnetic ceramics, in comparison with their metallic counterparts, is that they are electric isolators. This property is key for maintaining low eddy currents losses, therefore allowing these materials to be applied in cases where these losses have to be minimized.

The potential applications of gadolinium-iron garnet ($\text{Gd}_3\text{Fe}_5\text{O}_{12}$) may be in various types of amplifiers in microwave devices, circulators and isolators.

The aim of this work was preparation of dense polycrystalline GdIG by sintering with solid-state reaction. Three methods of synthesis were used: sintering of gadolinium and iron oxide precursors, sintering of co-precipitated precursor of a stoichiometric mixture of gadolinium and iron nitrates precipitated with ammonium water and solid-state reaction between gadolinium- iron perovskite (GdFeO_3) and $\alpha\text{-Fe}_2\text{O}_3$. Obtained powders were pressed uniaxially and next isostatically re-pressed at 250 MPa. The compacts were sintered at temperatures from 1300 °C to 1400 °C for 2 h. The following studies were carried out: DSC-TG analysis of the precursors, XRD, dilatometry (DIL) analysis and density measurement. The microstructure analysis of obtained samples was carried out with use of SEM studies

Acknowledgement: This work was supported by NCN Project MINIATURA work no. 18.18.160.05740.

OA-60

ADDITIVE MANUFACTURING OF MAGNESIA AND ALUMINA EUTECTIC COMPOSITES

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Selective laser sintering and melting of alumina and magnesia composites has been investigated. The purpose of the work was to analyze the change in roughness and porosity with the change in material proportions, to manufacture fully dense parts. The second priority was to analyze the eutectic phase changes with the change in laser parameters during the sintering and melting process. The experiments have been conducted at various eutectic ratios of alumina and magnesia. The influence of material proportion of Magnesia and Alumina on the microstructure of composite in SLS/SLM was investigated. The sintered samples were characterised by XRD and SEM. The effects during SLS/SLM of Alumina-Magnesia Composite were studied at four different loads of Magnesia-ranging from 80, 40, 25, and up to 10. Several specimens on various laser powers ranging from 70W to 90W were fabricated. The influence of the weight

fraction of Magnesia particles on surface roughness, porosity, cracks and final microstructure of the articles were evaluated by SEM, while the phase changes during the SLS/SLM were also evaluated by the XRD. The Microstructure improves with the increase in Alumina content. The microstructure of the composites was significantly affected by Magnesia particles. Intermediate phases in eutectic composite have great influence on the microstructure and surface morphology of the printed article. This study could be valid for other eutectic composites as well. It is feasible to produce slurry ceramic parts without binders through SLM.

OA-61

SYNTHESIS OF ZINC TIN OXIDE (Zn_2SnO_4) PARTICLES BY VARIOUS METHODS USED AS CERAMIC TARGET FOR SPUTTER TECHNIQUE

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Zinc tin oxide is an important electronic material which has been widely used in an extensive range of applications such as gas sensors, transparent and amorphous thin film transistors (TFTs), transparent conductive electrodes (TCOs) and anode material in Li-ion batteries. Zn_2SnO_4 (ZTO) is n-type intrinsic semiconductor that has recently received attention because of its high electron mobility, high electrical conductivity, low visible absorption, and the better environmental stability compared with its binary compounds (ZnO and SnO_2). Indium-Tin-Oxide (ITO) is currently used material for electronic application such as TFTs and TCOs. Owing to the scarcity, toxic nature and the increasing cost of indium, other alternative oxides are needed to research. Zinc Stannate (ZTO) is one of the significant alternative compositions to replace by ITO because of its high abundancy in Earth's crust, relatively low cost, non-toxic nature. TFTs and TCOs are composed of thin films generally prepared via sputtering technique. To produce high quality film the oxide target material used in sputtering should be high purity (>3N5 grade), almost dense (>95 %TD) and having homogeneous phase and microstructure. Desired target properties are controlled by the quality of as-synthesized powder. In this study, ZTO (Zn_2SnO_4) powders were successfully synthesized via two main powder synthesis methods: solid state reaction and hydrothermal synthesis. The effect of synthesis parameters such as initial concentration of precursors, synthesis temperature and time, on physical and chemical properties of particles was studied for each technique. The particle characteristics such as particle size and distribution, particle morphology and surface area, were discussed as a function of synthesis methods. As-

synthesized particles were uniaxially pressed into a cylindrical die and then conventionally sintered at 1200 °C for 2 hours. The physical, microstructural and electrical properties of oxide ceramic target were investigated in terms of particle characteristics for each synthesis methods. Synthesis of Zn_2SnO_4 particles via hydrothermal method showed the high elemental purity (>99.95 wt.%, i.e), smaller particle size (< 100 nm, i.e), narrow particle size distribution, high surface area (> 20 m^2/g) and equiaxed particles with cubic morphology, whereas particle size of solid state synthesized ZTO particles were ranging from 200 nm to 1.0 μm (average of around 500 nm) with an irregular morphologies. Thus, hydrothermal synthesized Zn_2SnO_4 particles were sintered at relatively lower temperature for achieving desired density (>90 %TD). The as-sintered ceramic target had homogeneous microstructure and smaller grain size ($\sim 2 \mu\text{m}$) compared to the synthesis via solid state reacted particles.

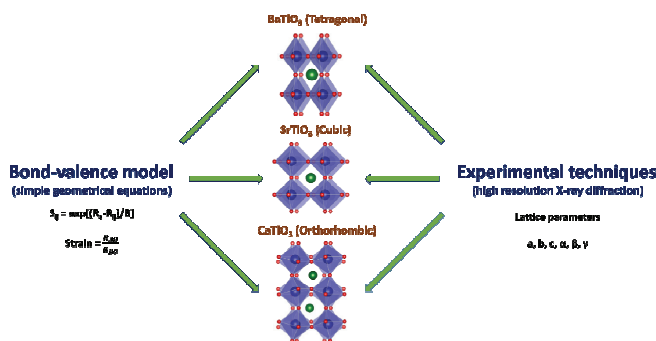
OA-62

THEORETICAL INVESTIGATION OF CRYSTAL STRUCTURE PREDICTION USING BOND-VALENCE (BV) MODELLING

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Perovskites (ABO_3 structures) are widely employed for energy harvesting, sensing, display and lighting applications due to its compositional flexibility, non-stoichiometry of the ions, and the distortion of the cation configuration. The intended applications of ABO_3 are dependent on the phase that is stable in operating conditions. It is expected that most ABO_3 systems stabilize in a cubic phase at high temperatures. As the temperature is lowered, ABO_3 shows two different types of distortions resulting in either: i) an off-centering of B-site cation (results in *tetragonal*) or/and ii) the tilting of BO_6 polyhedra (resulting in *orthorhombic/rhombohedral structures*). The exact nature of distortion observed depends on the radii of A and B-site cations.



In this work, bond valence (BV) model [1] is used for analysing the structures and properties of ABO_3 perovskites (case study by using BaTiO_3 , SrTiO_3 and CaTiO_3). The type of distortion (type I or II) and stabilized crystal structure are predicted using simple geometrical relations and expected bond length details from BV modelling. Results are correlated with the experimental reports. This BV analysis has the potential to serve as a method on the basis of which ABO_3 materials can be duly predicted.

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OA-63

CHALLENGES FOR BINDER JETTING OF PACKINGS AND MONOLITHS FOR HETEROGENEOUS CATALYSIS

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Extrusion and tableting are nowadays the most prevalent industrial methods for large-scale production of heterogeneous catalysts. Random as well as structured packings play an important role in industrial chemical process applications, but conventional fabrication methods allow for simple geometric features only. Binder jetting is an additive manufacturing method that uses powder bed-based technology and selectively applied ink to construct a three-dimensional body layer by layer.

With this relatively novel manufacturing technology it is attempted to generate ceramic bodies for use in heterogeneous catalysis. Challenges for the ink and powder development as well as the printing process are presented. Unifying knowledge from different research areas such as ceramic materials science, catalysis and additive manufacturing the research project constitutes an interdisciplinary effort for the development of a new process chain from raw materials to catalytic test reactions. For the catalyst, specific surface area, pore size distribution and side crush strength are parameters that have to be considered while ensuring an efficient printing and sintering process. Binder jetting could constitute a more elaborate way of catalyst production by enabling more complex geometries that could benefit fluid dynamic and thermodynamic aspects of chemical reactions.

OC-64

PHOTOCATALYTIC PROPERTIES OF BiFeO₃ AND Bi₅Ti₃FeO₁₅ BASED POWDERS

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Bismuth ferrite (BiFeO₃) and bismuth titanate ferrite (Bi₅Ti₃FeO₁₅) are studied a lot in recent years because of their specific structure and potential to act as single phase multiferroics. Thanks to the optical band gap energy of 2.3-2.8 eV for BiFeO₃ and 1.8-2.3 eV for Bi₅Ti₃FeO₁₅, these materials are also interesting for photocatalytic and solar energy applications, and studies of using Bi₅Ti₃FeO₁₅ for this purpose are very rare. Sol-gel methods were used to produce BiFeO₃ and BiFeO₃-modified diatomaceous earth, while Bi₅Ti₃FeO₁₅ was obtained sol-gel and solid-state methods. These powders were characterized, and their visible light photocatalytic activity for decomposition of methylene blue was tested in acidic, close-to-neutral and alkaline conditions. Fenton-like catalysis was also tested. Influence of synthesis method, microstructure, and catalyst composition on dye degradation will help in proposing the mechanism of adsorption and photocatalytic processes.

OA-65

IMMOBILIZATION OF SIMULATED RADIOISOTOPES IN ALKALI ACTIVATED INORGANIC POLYMERS

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The increasing importance of nuclear energy in the World's energy supply makes radioactive waste conditioning a constantly emerging issue. The current matrices (ordinary portland cement, bitumen) used for radioactive waste (excluding spent nuclear fuel) immobilization are far from being ideal (e.g. they have poor fire resistance and acid corrosion resistance, and have high leachability). In ordinary portland cement, radiolytic hydrogen production is a serious problem because the hydrogen bubbles induce mechanical stress in the matrix which may ultimately lead to the cracking of the material thus exposing more leachable surface to the environment.

The solution to these issues can be the utilization of alkali activated cements (AAC) which have superior chemical and thermal stability compared to ordinary portland cement and also have lower leachability by several orders of magnitude. These materials have the advantage of not containing water as a structural element and the adsorbed water content can be removed by heat treatment thus the radiolytic hydrogen yield becomes negligible in AAC radioactive waste forms.

The goal of my research is to maximize the immobilization of the matrix by optimizing its composition and to perform control experiments with ordinary portland cement. The experiments include leach tests with AAC matrices with small amounts of CsCl and SrCl embedded in them in order to simulate the most common fission products of U-235: Cs-137 and Sr-90 respectively.

Keywords: radioactive waste, geopolymer, alkali activated cement, immobilization, nuclear technology ties

OA-66

CATALYSIS REVOLUTION: WITH NANOTECHNOLOGY AND MOLECULAR LEVEL UNDERSTANDING TOWARDS A GREEN FUTURE

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Billions of dollars are investigated into information industry. Its less known, that similar budget is used for planning catalysis and industrial catalytic processes. The increasing demand for high activity and selectivity products needs new technologies and understanding of heterogeneous catalysis. The former trial-and-error method can be exchange with new techniques using in-situ methods for molecular level understanding under reaction conditions as well as controlled nanotechnology. These techniques may be exploited in CO₂ activation as well as other green methods.

In our research, mono and bimetallic nanoparticles with controlled average sizes were synthesized and anchored them on different 3D mesoporous oxide materials (SiO₂ – MCF-17, SBA-15, Co₃O₄, MnO₂, Fe₂O₃, NiO, CeO₂) prepared by the soft and hard template (replica) method. HRTEM-ED-EDX, SEM-EDX, DRIFTS, XRD, BET, H₂-TPR, NH₃-TPD etc. was used for characterizations. Catalytic tests were performed in a flow reactor in the gas phase connected to a HP GC-FID-TCD.

Tuning the size of the particles as well as the oxide/metal interfaces high activity and selectivity processes are favorable. NAP-XPS as well as DRIFTS techniques helped for molecular level understanding of the processes for future catalysts design. I will show plenty of results on the field of nanocatalysis and CO₂ activation reactions towards a new sight into green catalysis.

OA-67

SYNTHESIS, CHARACTERIZATION AND PHOTOCATALYTIC PROPERTIES OF TiO₂-rGO NANOPARTICLES

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Today, synthetic organic dyes are used in various industries. Wastewaters containing these dyes may be harmful to the environment, animals, and people. It is very important to degrade these dyes before discharging them into the environment. Heterogeneous photocatalysis could be effective in the degradation of organic dyes. A major advantage is the mineralization of organic dyes, which result in CO₂, H₂O, and corresponding mineral acids. TiO₂ as a photocatalyst has of great interest due to its high activity, photochemical inertness, non-toxicity, efficiency, and low cost. The biggest drawbacks of TiO₂ photocatalysis are the high bandgap energy (3.2 eV) and easy recombination of photoinduced electron-hole pairs. That limit its feasibility for the photocatalytic application in a visible light region.

The aim of this study was to synthesize the TiO₂-rGO (titania-reduced graphene oxide) nanoparticles as photocatalysts in the visible light region. The graphene oxide was synthesized from natural graphite powder by Hummer's method. TiO₂ sol was prepared by using titanium tetraisopropoxide as a precursor, *i*-propanol as a solvent, acetylacetone as a chelating agent and nitric acid as a catalyst. The TiO₂-rGO nanoparticles with different amounts of GO were prepared on a borosilicate glass substrate by using the sol-gel dip-coating method. They were characterized by means of powder X-ray diffraction analysis, Fourier transform infrared spectroscopy and Scanning electron microscopy. The photocatalytic activities of the TiO₂-rGO nanoparticles were evaluated by the degradation of aqueous solution of methylene blue (MB) dye as a model pollutant. The irradiation was simulated with a light-emitting diode (LED) lamp with a radiation peak at 365 nm and Solar-simulator lamp. The concentrations of MB were monitored by UV-Vis spectrophotometry. Obtained results indicated that photocatalytic properties of prepared TiO₂-rGO nanoparticles depend on the amount of rGO and on the type of the irradiation source.

OC-68

Pt NANOPARTICLES – SUPPORTED AND PRISTINE MESOPOROUS METAL OXIDES AS EFFICIENT CATALYSTS FOR CO₂ ACTIVATION

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Chemical recycling of carbon dioxide (CO₂) is of a crucial importance nowadays. In this study we proposed a solution to carbon dioxide utilization using noble metal free as well as controlled size Pt nanoparticles-loaded mesoporous metal oxides (NiO, Co₃O₄, CeO₂ and MnO₂) with high surface area and designed pore structure. Hard template method was applied for the synthesis of the catalysts. Along with carbon dioxide hydrogenation, dry reforming with methane (DRM) was also used for CO₂ activation since there are a number of feedstock systems containing both CO₂ and CH₄ together in a mixture. The physicochemical properties of catalysts were analysed by N₂ adsorption-desorption isotherm, XRD, TEM, and H₂-TPR.

Based on the activity test results, highest activity was demonstrated by Pt-free and Pt-loaded mesoporous NiO and Co₃O₄ in both CO₂ activation reactions. As a general trend, loading mesoporous metal oxide supports with Pt nanoparticles has had an enhancing effect on catalytic activity of the catalysts due to various effects, in particular, due to presence of Pt⁰ and Me⁰ species and formation of a special synergetic of Pt/Me/MeOx structures, which helps to stabilize and promotes reactivity of surface reaction intermediates. However, opposing phenomenon was recorded for NiO based systems. In this case, free-standing NiO demonstrated much better activity in CO₂ activation reactions with high CO₂ consumption rate and selectivity. Activity deteriorating effects for Pt nanoparticles were explained by the fact that the latter provides additional reduction of NiO in the interface, which degrades the dynamics of Ni/NiOx under reaction conditions. This assumption was supported by H₂-TPR-measurements.

The catalytic results emphasized the importance of the nature of metal oxide supports as well as the significance of formed interphases under reaction conditions. In the future, we would like to synthesize structures with optimal metal/metal oxide interphases in order to reach high activity and selectivity in CO₂ activation reactions.

OA-69

**OPTIMIZATION OF THE PHOTOCATALYTIC DEGRADATION OF
THIOTRIAZINONE STABLE HYDROLYSIS PRODUCT OF
ANTIBIOTIC CEFTRIAXONE**

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One of the growing concerns in recent years with pharmaceutical wastewater treatment and discharge is the health risk of emission of antibiotic residues into natural environments, since common biological treatment has limited removal efficiency on antibiotics due to their antimicrobial activity. The production output of antibiotics increases every year due to the expanding worldwide market demand. Most antibiotics do not undergo metabolic inactivation and are discharged into the environment. Since the antibiotic ceftriaxone, although unstable, appeared in wastewater, and caused many ecological, environmental, and health issues, in this work, optimization of the photocatalytic degradation of thiotriazinone, stable hydrolysis product of antibiotic ceftriaxone, was performed. Photocatalytic activity of commercial TiO_2 and ZnO catalysts using simulated solar irradiation in the photocatalytic degradation of thiotriazinone was investigated. Since thiotriazinone, at higher pH than 7, is practically not adsorbed on the surface of TiO_2 Hombikat, unlike in the case of ZnO , the loading of TiO_2 Hombikat, influence of pH and concentration of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ on the efficiency of photocatalytic degradation of thiotriazinone was examined. It was found that optimal conditions in photodegradation of thiotriazinone were 1.0 mg/cm^3 of TiO_2 Hombikat, 3 mmol/dm^3 of $(\text{NH}_4)_2\text{S}_2\text{O}_8$, and pH 7.6. Under mentioned conditions 76.0% of thiotriazinone was removed after 60 min of irradiation.

Acknowledgement: The authors acknowledge financial support of the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No. 172042).

OC-70

ADSORPTION STUDIES OF CADMIUM (II) FROM AQUEOUS SOLUTIONS USING INORGANIC POROUS NANOCOMPOSITES

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SiO₂-Fe₂O₃ nanocomposites adsorbent materials were synthesized by acidic catalyzed (HNO₃) sol-gel process using Fe(acac)₃ as precursor of iron oxide phase, TEOS as silica source, and polyvinyl alcohol (PVA, M 49000) as pore former. The molar ratio of reactants TEOS: H₂O: PVA: Fe(acac)₃: CH₃OH: HNO₃ was 1: 10: 1.89·10⁻⁵: x: 18: 0.01, where x varied from 0 to 20% Fe₂O₃. The sol-gel reactions took place at room temperature. All four synthesized xerogels were heated at 300 °C, in order to obtain the crystalline structure of iron oxide desired compounds with spinel phase confined in silica matrix. The samples have been characterized by N₂-adsorption isotherms, FT-IR, XRD, SANS and magnetization studies. These nanocomposites were tested as applications for the adsorption of Cd (II) from aqueous solutions. The results showed that the highest surface area (~ 680 m²/g) was achieved for sample with 10% Fe₂O₃ and pore size (from BJH) of 3.1 nm. The sample with the maximum 20%Fe content (20-NC) showed the best adsorption capacity (4.1 mg/g) with 30 mg Cd/L initial ion concentration. The adsorption studies were also optimized by varying several experimental parameters: pH, adsorption temperature, adsorbent dose. The optimum pH for the adsorption was found in the 4–6 pH range, being considered from literature studies [1] with the adsorbent dosage level of 0.1 g at 25 °C temperature.

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OT-71

PHOTOCHEMICAL ACTIVITY OF METAKAOLIN BASED GEOPOLYMER FOAMS

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Geopolymers or alkali activated inorganic polymers (AAIP) are not only future but today's structural materials, which are intensively investigated research area. Besides that AAIPs can be used to replace cement-based traditional binders due to their high strength, these materials can also easily be well foamed. Over the past decades AAIP foams, as eco-friendly porous materials, have been great attention due to their favorable production conditions (e.g. low energy requirements) and their promising properties (good mechanical and chemical stability, high temperature resistance etc.). These materials can be used, inter alia, in photocatalytic degradation processes as catalyst supports, if TiO₂ is immobilized on their surface. For this purpose, AAIP foams with mesoporous matrix and one-way lamellar macroporosity are required, which can be produced mainly by combined methods (gelcasting/saponification/ peroxide decomposition = GSP). With this technique, foams with up to 85% pore content (~ 70% open porosity) can be produced. Although their low compressive strength (~ 0.45 MPa) and high cracking tendency limits the application possibilities.

The aim of this work was to develop catalyst supports with optimal composition and adequate strength. Metakaolin based AAIP foams were produced by GSP combined method using sunflower oil as surfactant and H₂O₂ as foaming agent. The organic matter content of the specimens as well as their high shrinkage and the resulting cracking tendency are the most disturbing factors. By eliminating these problems, the samples can be used for photocatalytic sewage treatment processes. First the UV resistance of pure AAIP foams (without TiO₂) were investigated. Although the foams themselves have photochemical activity, the results obtained are very different. The reason for this phenomenon may be that the temperature distribution in the furnace space is not uniform during the firing of the samples. Experiments show that the pore structure formed by GSP combined method is sensitive to temperature changes. Furthermore the relationship between photochemical activity and structural characteristics was investigated, using XRD, FT-IR, SEM and NMR.

Acknowledgement: We acknowledge the financial support of GINOP-2.3.2-15-2016-00016 and Széchenyi 2020 under the EFOP-3.6.1-16-2016-00015.

OT-72

PREPARATION OF KAOLINITE-METHANOL AND KAOLINITE-CETYLTRIMETHYLAMMONIUM CHLORIDE COMPLEXES

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Nowadays, the preparation of kaolinite nanoscrolls using multi-step intercalation techniques is an important research field, because they can be applied in advanced nanocomposite and nanohybrid materials. These halloysite-like nanoscrolls can high-efficiency be produced by solvothermal technique, which involves the displacement of the primary intercalating reagent (urea) by methanol, followed by the intercalation and deintercalation of cetyltrimethylammonium chloride. After this exfoliation, kaolinite layers can be separated into individual double layers, which could curl and frequently roll up to form nanoscrolls as a consequence of the misfit between the tetrahedral and octahedral sheets.

Our efficient one- and two-pot solvothermal technique was studied in laboratory conditions using only a small amount (approximately 1 g) of primary intercalate (kaolinite-urea); therefore the increase of quantity is very important from the industrial point of view. In this work, we study the effect of quantity of primary intercalate on the formation of the kaolinite-methanol and kaolinitecetyltrimethylammonium chloride (kaolinite-CTAC) complexes using solvothermal methods. These complexes are essential to exfoliate kaolinite and to produce kaolinite nanoscrolls. The prepared kaolinite-methanol, kaolinite-CTAC complexes and kaolinite nanoscrolls are studied by X-ray diffraction (XRD) and transmission electron microscopy (TEM).

OT-73

SUSTAINABLE USE OF SULFIDIC TAILING RESIDUES IN THE PRODUCTION OF CERAMIC ROOF TILES

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Mining and quarrying waste accounts for over a quarter of all the EU-28 waste output. When poorly managed, extractive waste is considered as a significant environmental and health hazard. Sulfidic Cu-Zn, Pb-Zn and Cu-Zn-Pb tailing residues pose the largest challenge, as they can lead to acid mine drainage. Apart from the

hazardous metals, these tailing residues also contain valuable base, precious and critical metals which can be used in different applications of emergent technologies. Moreover, the mineral residue of these mine tailings can contain considerable concentrations of quartz and clay minerals which can be used in different ceramic applications.

Based on the aforementioned issue, the main goal of this study is to evaluate the potential use of sulfidic tailing residues from an inactive Pb-Zn mine waste site at Plombières (Belgium), in ceramic roof tiles taking into account production parameters (shaping, drying and firing processes), product quality (intrinsic technical and aesthetical aspects) and environmental issues (compliance with environmental legislation). After a detailed physical and chemical characterisation (grain size distribution, specific surface, chemical composition, mineralogy, loss on ignition, firing colour, moisture absorption, etc.) of the tailing material, one specific Wienerberger's blend for clay roof tiles in Belgium has been modified on a lab scale, by partly replacing the primary raw materials by the mine tailing material. The shaping, drying and firing behaviour were assessed on a lab scale and compared with the behaviour of the standard reference mix. The required technical (strength, porosity, firing shrinkage, etc.) and aesthetical (colour, efflorescence, etc.) properties for ceramic roof tiles were evaluated.

The preliminary results indicate that the mine tailing material from Plombières site, which has a very low sulphur content and high content of silicates (quartz, muscovite and feldspar), clay minerals and a few main trace elements Ba (366 mg/kg) and Zn (137 mg/kg), can be considered as a good candidate to replace partly primary raw materials using 5% of the mine tailings. Further research adding higher amounts of mine tailings to the roof tile mixture will be assessed, in order to keep evaluating the sustainable use of mining waste in ceramic roof tiles.

Keywords: ceramic roof tiles; mining waste; secondary raw materials; circular economy; Plombières

OT-74

THE EFFECT OF LIGHTWEIGHT AGGREGATES FROM SEWAGE SLUDGE ON TOBERMORITE FORMATION IN $\text{CaO-SiO}_2\text{-H}_2\text{O}$ SYSTEM UNDER HYDROTHERMAL CONDITIONS

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The effect of artificial lightweight aggregates on formation of tobermorite was examined. Tobermorite is a mineral analog of calcium silicate hydrate, due to the structural similarity. Tobermorite synthesis with constant CaO/SiO_2 molar ratio of 0.86

was studied. The artificial lightweight aggregates was used as a substitute of quartz sand at 5%, 10%, 20%, 40%, 70% and 100% by weight. Research was carried out in constant hydrothermal conditions. Synthesis time was 12 hours. The microstructural investigation showed that artificial lightweight aggregates have a positive influence on the formation of 1.1 nm tobermorite. Tobermorite and quartz peaks were present. The artificial lightweight aggregates causes increase in tobermorite content. The beneficial effect of this aggregates are related with the presence of aluminium ions in chemical composition.

OT-75

SYNTHESIS OF COAL ASH BASED GEOPOLYMERS: INFLUENCE OF NaOH CONCENTRATION AND SOLID FRACTION

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Geopolymers are being extensively studied as sustainable building materials due to their characteristics as potential substitutes for Portland cement based materials. One of their most interesting characteristics is the possibility to combine them with waste or byproducts from other processes, which is very attractive from an environmental and sustainable point of view. Geopolymer manufacturing consists of mixing an aluminosilicate source with an alkaline medium. These materials react in a low temperature setting process, approximately 40–60 °C and finally harden. The concentration of the alkaline medium must be considered in the geopolymerization process, because an excess of alkalis can lead to the formation of salts after solidification that impairs the performance of the material. Therefore, the aim of this work was to evaluate the molar concentration of NaOH, as well as the solid fraction added to the geopolymer system based on fly ash, bottom ash and coal gangue. A constrained mixture design was used for each waste. The independent variables were the amount (mass %) of NaOH solution (and therefore, its concentration), the solid fraction of each waste and the amount of sodium silicate, resulting in 7 mixtures for each waste. The mixtures were synthesized according to the design and, after demolding, the samples settled at 50 °C for 28 days. The mechanical strength was evaluated through compression tests. In addition, the samples were also characterized by X-ray diffraction (XRD), thermogravimetric analysis (TGA), bulk density, efflorescence (salt permeation) analysis and scanning electron microscopy (SEM). The results show that it is possible to obtain geopolymers using coal ashes and waste at high solid content, which shows their potential for assimilation of waste or byproducts from other processes into geopolymers matrices.

OT-76

THE EFFECT OF TEMPERATURE ON THE PROPERTIES OF NON-CEMENT ALUMINA-SPINEL CASTABLE

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Non-cement alumina-spinel castables during temperature treatment show different behaviour than traditional calcium aluminate-based castables. It may result from the fact that the synthesis and sintering processes occurring in the calcia-free castables dominate over the thermal dehydration of calcium aluminates occurring in the traditional calcium aluminate-based castables at the intermediate temperature.

The research was aimed at obtaining non-cement alumina-spinel castables with satisfactory mechanical and technological properties, as well as considering the relationship between microstructure and mechanical properties of these refractories heat-treated at different temperatures. The composition of non-cement alumina-spinel castables was designed based on the Dinger and Funk model. Reactive Al_2O_3 , MgO and SiO_2 oxides were used as hydraulic binding materials, besides tabular alumina as a aggregate. The effect of thermal treatments on the properties of castables was determined in the terms of apparent density, open porosity, compressive strength and bending strength. Moreover, all samples were also tested using X-ray powder diffraction (XRD) and scanning electron microscope (SEM) techniques.

Based on the conducted tests, a positive effect of replacing calcium aluminate cement with reactive Al_2O_3 , MgO and SiO_2 powders was found. The improved consolidation and strength properties of spinel forming corundum castables were determined. Thanks to the use of reactive micro-powders, sintering process for cement-free alumina-spinel castables can take place at intermediate temperatures. Contrary to traditional castables, non-cement spinel corundum castables show an increase in apparent density and mechanical strength at intermediate temperatures, which is associated with the high surface energy of reactive powders Al_2O_3 , MgO , SiO_2 , as well as a small amount of phases, which provides the hydraulic binding property of the material.

Keywords: non-cement alumina-spinel castables, microstructure, MgAl_2O_4 , XRD

Acknowledgements: This study was founded by The National Centre for Research and Development within the framework of LIDER VIII project No. LIDER/5/0034/L-8/16/NCBR/2017.

OT-77

POSSIBILITY TO USE SPENT FLUID CATALYTIC CRACKING CATALYST AS COMPONENT OF PORTLAND CEMENT BINDERS

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Fluid cracking catalyst residue (spent FCC) is a by-product from petrol refineries. It primarily consists of zeolite (in used sample - faujasite) and amorphous aluminosilicates. In this study, possibility to use spent FCC as a component of binders with low content of Portland cement (PC) have been investigated.

Binders made with high volume of mineral additions are being developed in order to reduce the amount CO₂ emitted by PC industry. Fly ash (FA), a by-product of coal combustion in thermal power plants, is one of the most commonly used raw material for these binders. The main shortages of binders with high volume of FA are long setting time and low early strength.

In this work, mixtures of PC, FA and spent FCC were prepared. It was found that addition of spent FCC to low PC mixtures significantly shortened setting time and improved early strength of the binders. However, 28-day compressive strength of the ternary binder was lower than the strength of low PC binder synthesized with FA only, which indicated the need for further optimization of the binder composition.

OT-78

EFFECT OF TITANIUM OXIDE ADDITION INTO MAGNESIA REFRACTORIES

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This work contains the results of investigations into the influence of titanium oxide (TiO₂) addition on the properties of refractory magnesia ceramics. The presented research involved adding titanium oxide in a classic way, i.e. directly to the ceramic mix. The conducted laboratory tests revealed a significant impact of this oxide on the functional properties of refractory materials. Addition of a small amount of TiO₂ favoured the ceramic mix sintering whereas adding a bigger amount - more than 10 wt.% resulted in the formation of materials characterised by considerable porosity and low

mechanical strength. Addition of this oxide also slightly improves the corrosive resistance of materials.

OC-79

**THE EFFECT OF FIELD ASSISTED SINTERING PARAMETERS ON
PROCESSING OF IN-SITU FORMED B₄C-TiB₂ CERAMICS**

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In the present study, B₄C/TiB₂ composites containing different contents of TiB₂ were fabricated by in situ reaction of B₄C, TiO₂ (in the form of particles and fibres) and carbon black using a Field Assisted Sintering Technology (FAST). The TiO₂ fibers used in the study were prepared using needle-less electrospinning technique, followed by the heat treatment. The effect of degassing and pressure on the sintering behaviour of B₄C was studied in the temperature range of 1700 to 2000 °C and the pressure of 50–70 MPa.

Phase and morphologies of B₄C/TiB₂ composites were characterized by X-ray diffraction, scanning electron microscope and Raman spectroscopy. The results showed that when the degassing step was applied, homogeneous microstructure and high density were obtained. Only B₄C and TiB₂ phases were identified, although the presence of a small amount of oxygen or carbon could not be ruled out. This confirmed that the reaction between TiO₂ and B₄C was completed and no residual TiO₂ was found in the materials. Fully dense B₄C-10 vol.%TiB₂ materials were obtained using the sintering regime with a degassing step at the temperature of 1800 °C, followed up by the applying of maximum pressure for 30s. Interestingly, when the critical sintering conditions were exceeded, the in-situ formation of graphene in the middle part of the materials was observed. The mechanical properties of B₄C, such as hardness, fracture toughness, were improved by the addition of in-situ formed TiB₂ fibres and particles.

Acknowledgement: This work was supported by the Slovak Research and Development Agency under the contract no. APVV-SK-SRB-0022 and APVV-17-0328. The support of the MVTs project of SAS “UltraCom” within the scheme of “Seal of Excellence projects” is also acknowledged.

OC-80

**FABRICATION OF POWDER BASED CERAMIC INSULATION
MATERIAL FOR HIGH TEMPERATURE APPLICATIONS**

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Increasing energy demand in the world and environmental concerns due to air pollution and CO₂ increase force the energy-intensive industries to use alternative renewable energy sources and take actions for energy efficiency. Energy efficiency in process industries is basically classified into three main groups: Heat integration, reuse of waste heat and application of thermal insulation materials. Inorganic thermal insulation materials, such as rock wool, glass wool, mineral wool, are generally used in energy-intensive industries. These types of material have some disadvantages like high moisture holding capacity, difficulty in the application for equipment with complex geometry (flanges, traps, etc.). Furthermore, the manufacturing process of conventional insulation materials consumes high energy and natural resources. For this purpose, geopolymer technology, which is produced by chemical activation of industrial solid wastes (fly ash, metakaolin, slag, etc.), is seen as an optimal alternative and green solution for thermal insulation due to their excellent thermal stability, fire resistance, longer service life, and low cost. In this study, it is aimed to develop powder-based alternative and competitive insulation coating material that can be directly applied to hot metal surfaces (>250 °C, etc.) by spraying. Fly ash based geopolymer composite filled with inorganic hollow particles were fabricated. The effect of Si/Al, Na/Al, filler/aluminosilicate matrix ratio on the physical, thermal and mechanical properties of insulation coating were investigated. Furthermore, the effect of curing temperature (temperature of metal surface, etc.) and time on microstructural properties of the coating were studied in order to understand how hollow particles distributed in geopolymer matrix. Results showed that hollow particles filled geopolymer coating material has excellent thermal conductivity ($k < 0.06$ W/mK) and reasonable mechanical properties (Adhesion strength > 1.2 MPa). With its high performance, newly developed material will be a promising candidate for thermal insulation coating on hot metal surfaces in energy-intensive industries.

OC-81

**EFFECT OF ANNEALING ON THE STRENGTH RETENTION AT
ULTRA-HIGH TEMPERATURE**

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A HfB₂ ceramic containing SiC and WC was hot pressed and subsequently annealed at 2100 °C for 2 hours. The annealing procedure resulted beneficial for high temperature strength which increased of about 300 MPa in the 1500–1800 °C temperature range as compared to the as-sintered material. Peak strength of 850 MPa at 1500 °C and 650 MPa at 1800 °C were achieved.

To correlate the mechanical properties with the microstructure, fracture and polished surfaces of both the as-sintered and annealed ceramic were analyzed by field-emission scanning electron microscopy (FESEM) coupled to an energy dispersed X-ray micro-analyzer (EDS). Local phase analysis was performed using transmission electron microscopy (TEM) equipped with an EDS X-ray spectrometer.

Two main microstructural changes were observed. Firstly, rounded SiC particles immersed in silica-based glass evolved into platelets with clean grain boundaries after the heat treatment. Secondly the boride matrix underwent a fundamental variation in its typical HfB₂ core/(Hf,W)B₂ rim substructure. During the annealing process the rim zone tended to re-arrange and decompose leaving a W-depleted (Hf,W)B₂ solid solution with homogeneously dispersed metallic tungsten nanoparticles. Those are capable of absorbing fracture energy owing to the ductility and extreme toughness of W above 400 °C. These two features greatly contributed to refractoriness and energy absorption at high temperature.

OC-82

DEVELOPMENT OF ZrC-BASED CERAMIC NANOCOMPOSITES

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Due to characteristics such as high melting temperature (3500 °C), hardness (28.7 GPa), low density and thermal conductivity, ZrC is being studied from the different perspective (nanoparticles, composites, fibers). Moreover, the access to different synthesis route (Carbo-thermal reduction, plasma reaction and sol-gel) adds versatility to this prominent material and their composites.

In one hand, reinforcements such graphene, SiC and Mo compounds improve the sinterability of the particles. On the other hand, it can improve the properties of ZrC ceramics like oxidation and thermal shock resistance, by avoiding the diffusion of oxygen and forming oxide film in the surface correspondly.

In the current work, ZrC nanopowders are synthesized by different methods like as sol-gel and plasma reaction. Both synthesis routes use Zirconium propoxide as Zr source, the main differences in the products lies in the control of the particle sizes, cost and morphology. The as synthesized nano-ceramic powders are consolidated by spark plasma sintering (SPS) method, with the same retention times, in a temperature range between 1500 °C and 1900 °C. The morphologies of ZrC nanopowders and sintered bodies are characterized by scanning electron microscopy (SEM), while the phase compositions by X-ray diffraction (XRD). Surface chemical compositions are determined by X-ray photoelectron spectroscopy (XPS).

OC-83

CHARACTERIZATION OF SiC_f/SiC COMPOSITES DENSIFIED BY MICROWAVE ASSISTED CHEMICAL VAPOUR INFILTRATION

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Silicon carbide (SiC) fibre-reinforced SiC matrix composites (SiC_f/SiC) have potential for a range of high temperature applications, including hot components in gas turbine engines, fuel cladding and radiation blankets for nuclear fusion reactors.

Chemical vapour infiltration (CVI) has been widely used for the densification of SiC_f/SiC; the benefits of CVI compared to other approaches include good near-net shape capability and excellent microstructural control. However, the densification of SiC_f/SiC from conventional CVI is very time-consuming, especially when a large component is required; process times of 1000 hours are not uncommon. When the infiltration is assisted by microwave heating (MCVI), however, process times can be reduced to less than 100 hours. The enhanced densification rate is caused by the inverse temperature profile that develops in the SiC preform during MCVI, i.e. the temperature in the centre of the preform is higher than at the surface. However, different processing temperatures during MCVI in the different areas of the preform could result in crystallographic, microstructural and hence property differences across the as-infiltrated SiC matrix.

In this presentation, SiC_f/SiC composites produced by MCVI using a mixture of methyltrichlorosilane (CH₃SiCl₃, MTS) and H₂(g) will be reported. The phase assemblage, microstructural features and mechanical properties at different locations of SiC_f/SiC composites produced at 950 °C / 20 kPa have been characterised using a combination of techniques including Raman spectroscopy, X-ray diffraction, electron diffraction and transmission electron microscopy.

OA-84

**INORGANIC LI-ION CONDUCTORS FOR ALL SOLID STATE
BATERIES: CRYSTAL CHEMISTRY AND TRANSPORT
PROPERTIES IN THE Li₂S–P₂S₅ SYSTEM**

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Li-ion batteries (LIBs) have been used extensively in portable electronics owing to their high energy densities and energy conversion efficiencies. However, requirements for higher energy density and better safety in demanding applications, such as grid storage and electric vehicles, call for next-generation battery technologies. All solid state batteries (ASSBs) might enhance the safety and the energy density of conventional LIBs

by removing flammable organic electrolytes and allowing the use of metallic lithium anodes [1]. In this context, solid sulfide-based electrolytes have drawn a lot of attention owing to their high ionic conductivities (10^{-2} – 10^{-4} S cm⁻¹ at RT) [2–5]. Few compositions in the Li₂S–P₂S₅ binary system, particularly Li₃PS₄ and Li₇P₃S₁₁, are heavily investigated as glass-ceramic materials synthesized by mechano-chemistry and subsequent annealing [4]. However, varying ball-milling parameters and annealing procedures was rarely systematically investigated [5].

In this work, we will report on the synthesis of various materials in the Li₂S–P₂S₅ system via a systematic synthesis approach: the individual effects of pre-selected ball-milling and subsequent annealing parameters will be presented, as characterized in details through X-ray and neutron diffraction, and various techniques of spectroscopy such as Raman, NMR and complex impedance. The latter technique is quite powerful to discriminate between crystalline and amorphous contributions to the total ionic conductivity, that may be strongly affected as well by the relative amounts of local units such as P₂S₆⁴⁻, P₂S₇⁴⁻, PS₄³⁻ in the material. Additionally, the description of the crystal structure of β-Li₃PS₄, which is still under debate [6–8], will be presented in details.

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OC-85

**ULTRA-SENSITIVE ELECTROCHEMICAL BIOSENSOR REALIZED
USING LTCC TECHNOLOGY**

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Recently, biosensors found a number of applications in point-of-need diagnostics due to their high specificity and sensitivity, low-cost and fast response times. In this paper we propose a novel topology of electrochemical sensor realized using Low Temperature Cofired Ceramic (LTCC) technology.

We proposed electrochemical sensors based on three-electrodes (working, reference, and auxiliary). In the proposed design working electrode was realized in the shape of Sierpinski fractal gasket, while two other electrodes were realized to follow the curvature of the central electrode. In that manner the effective surface area between electrodes is increased resulting in increased sensitivity of the sensor.

The proposed sensor has been realized using a combination of a laser micromachining, screen printing, lamination, firing and spin coating processes. Design process started with the preparation of the substrate for the electrodes and with laser cutting of Ceram tape GC, glass ceramic based material. In the following step, gold conductive paste Heraeus TC7102 was deposited on the previously cut Ceram tape using screen printing process. After stacking of several supporting Ceram tape layers and layer with printed electrodes the whole structure was laminated in uniaxial press Carver 3895CEB under the pressure of 1 t for 3 min at temperature of 75 °C. The sintering of the material has been performed using firing process in the oven at the maximal temperature of 865 °C, in total duration of ten hours. After firing the SU-8 resin was used to delimit the detection area enabling the use of very small sample volume (50 µL) for detection. The 3 µm resin was deposited using spin coating process through protective mask and dried in oven at 95 °C for 2 min.

SEM TM3030 Hitachi was used for micrographs of fabricated electrodes, while the optical profilometer Huwiz HRM-300 was used for profiler analysis. The fabricated electrode was characterized using cyclic voltammetry (CV), and compared with commercial one. The applicability of the proposed sensor was demonstrated for detection of *Campylobacter* DNA sequence. The proposed electrochemical biosensor displayed good reproducibility and high stability, allows good immobilization of DNA and aptamers on the electrode surface and shows more 30 times better sensitivity in comparison with commercially available electrodes.

OA-86

SUITABILITY OF Sr AND Co-FREE LANTHANUM PEROVSKITE MATERIALS AS CATHODES FOR IT-SOFC WITH A LANTHANUM SILICATE APATITE-TYPE ELECTRODE

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Last trends in the field of solid oxide fuel cells have been focused on the development of new electrolyte materials which possess higher ionic conductivity in the intermediate temperature range (600–800 °C) than the classic yttria-stabilized zirconia. It has been long proved that apatite-type lanthanum silicates are good oxygen ion conductors [1] and doping the Si-site with lower valence cations may also enhance its ionic conductivity [2]. Therefore, La_{9.67}Si₅AlO₂₆ has been selected as a proper electrolyte for IT-SOFC.

Few studies about the use of this apatite-type material have been published so far, and those available are focused on classic cathode materials like (La,Sr)MnO_{3±δ} or (La,Sr)(Co,Fe)O_{3±δ}. Nevertheless, there is also a tendency towards disposing of elements such as strontium or cobalt [3,4] in the development of new cathode compositions, due mainly to strontium tendency to segregate towards electrode/electrolyte interfaces [5] and the large thermal expansion coefficient of cobalt-substituted perovskites, which may be twice as high as the apatite TEC [3,6], which can affect the structural integrity of the cell and lead to a degradation in performance.

In this work, some well-known perovskites such as lanthanum manganite, ferrite and chromite have been substituted with nickel or copper in the B-site in order to tailor both the structural and the functional properties of those materials, and a study of suitability with the apatite-type electrolyte has been done.

Solid state synthesis of perovskites, apatite-perovskite chemical compatibility at different temperatures (900 and 1250 °C), dilatometry tests to check thermal expansion coefficients, electric conductivity as a function of temperature of sintered perovskites, symmetrical cell preparation and electrochemical impedance spectroscopy of the cell has been carried out in order to select the most suitable cathode material for this apatite electrolyte IT-SOFC.

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OA-87

**THE DEVELOPMENT OF THE NOVEL TERNARY $\text{Y}_2\text{O}_3\text{-CeO}_2\text{-ZrO}_2$
AND $\text{TiO}_2\text{-CeO}_2\text{-ZrO}_2$ SOLID ELECTROLYTES
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The development of the new ceramic solid electrolytes (SE) for intermediate temperature solid oxide fuel cells (IT-SOFC), effectively operating at 650–800 °C – is a serious scientific problem associated with the possibility of the shift towards the hydrogen energy. So far the operating temperature of SOFC is about 1000°C, when the lifetime of the materials is significantly reduced because of the interaction of the electrolyte and cathode materials causing the formation of low-conductive layers; the rapid growth of ceramic grains and recrystallization of the membrane. However at IT the electrical conductivity of solid electrolytes is insufficient, along with the electrode polarization and increasing of sensitivity to catalytic poisoning. Thus the aim of this research is to develop new ternary solid electrolytes $\text{Y}_2\text{O}_3\text{-ZrO}_2\text{-CeO}_2$ and $\text{TiO}_2\text{-CeO}_2\text{-ZrO}_2$ by the cryochemical technique.

For that, cryochemical technique comprising reversed co-precipitation from 0.1 M aqueous solutions of $\text{TiOSO}_4\cdot 2\text{H}_2\text{O}$, $\text{Ce}(\text{NO}_3)_3\cdot 6\text{H}_2\text{O}$, $\text{ZrO}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ and $\text{Y}(\text{NO}_3)_3\cdot 6\text{H}_2\text{O}$ with the subsequent cryochemical dehydration of gel was applied. As a precipitator 1 M aqueous solution of ammonia was used, solution of salts was added dropwise of 1–2 ml/min, pH = 9–10, $T=1-2$ °C. The precipitate of hydroxides was then freeze-dried (Labconco, 1Lchamber, Kansas City, MO, USA; 293K, 24 h, $P=0.018$ mm Hg). Obtained powders were annealed at 600–1100 °C during 3–24 hours to ensure Ce^{3+} to Ce^{4+} transformation. Precursors after synthesis and calcination were investigated by simultaneous thermal analysis (STA, STA 409 of C/4/G Jupiter, NETZSCH), X-ray diffraction analysis (XRD, Shimadzu XRD 6000), particle size distribution analysis (PSD, Horiba partica LA-950). Thermodynamic behavior of obtained $\text{Y}_2\text{O}_3\text{-ZrO}_2\text{-CeO}_2$ solid solutions was investigated via high-temperature mass spectroscopy. To determine the completeness of Ce^{+3} to Ce^{+4} transformation Electron Spectroscopy for Chemical Analysis (ESCA, Combined Auger, X-ray and Ultraviolet Photoelectron spectrometer Thermo Fisher Scientific ESCA-lab 250Xi) was used.

Via STA, XRD and ESCA it was shown that the use of the reversed co-precipitation technique with following prolonged calcinations of precursors at 1000 °C for 4–10 hours leads to the equilibrium $x\text{Y}_2\text{O}_3\text{-(50-x)ZrO}_2\text{-50CeO}_2$ solid solutions formation ($x = 12.5, 25, 37.5$ mol% Y_2O_3) without an admixture of Ce^{3+} . Using high-temperature mass-spectroscopy technique combined with Knudsen effusion cell it was found that ceria activity is characterized with the stronger deviation from the ideal case comparing to zirconia. For the first time the formation of zirconia based solid solutions was investigated in $\text{TiO}_2\text{-CeO}_2\text{-ZrO}_2$ system at 600–1100 °C. Via XRD and STA it was shown that tetragonal $5\text{TiO}_2\text{-9CeO}_2\text{-86ZrO}_2$ solid solution was formed at 800–1000 °C.

Acknowledgement: The work was supported by the President's grant for young scientists #MK-2703.2019.3.

OC-88

PARTICLE SIZE INFLUENCE ON THE FORMING OF THIN SPINEL LAYERS ON A STEEL SUBSTRATE FOR SOFC APPLICATION

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Solid Oxide Fuel Cells (SOFC) offer efficient generation of energy without emission of harmful compounds for the environment. To reach desired power, the certain number of the fuel cells are joined together to form a stack. The element which is placed between the cells is a bipolar plate - interconnect which also provides the channels for the flow of processing gases - hydrogen and air. Most often, it is made of chromium-alloyed steel (like Crofer 22 APU from VDM Metals). One of the key issues ensuring proper and long-term operation of the SOFC stack is the application of ceramic protective layers on the interconnects, to avoid evaporation of the volatile chromia compounds resulting in the degradation of the SOFC cathode and decrease the efficiency of the unit. The protective layers should have minimal porosity, optimal uniform thickness and low area specific resistance in high temperatures. Such parameters allow to block solid state diffusion of Cr from steel and to demonstrate low area-specific resistivity what is essential for proper SOFC stack operation.

The research was focused on investigation of the process of forming the spinel layer via electrophoretic deposition (EPD), taking into special consideration the influence of the particle size distribution of the ceramic powder on the final layer properties. Commercially available manganese cobalt oxide ($\text{Mn}_{1.5}\text{Co}_{1.5}\text{O}_4$) powder was milled in different conditions to obtain the grain size in range of 0,2-0,6 μm . The sample coatings were deposited on flat Crofer 22 APU substrate. The process parameters were optimised to reach the desired uniform thickness 15 μm of the sintered layers. The layers were subjected to microstructural investigation in green state and after reductive sintering followed by oxidation. Area specific resistance (ASR) for chosen samples was measured in short-term experiment.

OC-89

**PROPERTIES OF ADVANCED YTTRIA- AND SCANDIA-STABILIZED
ZIRCONIA COMPOSITE FOR SOLID OXIDE FUEL CELL
APPLICATION**

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Zirconia is continuing to attract the attention of many scientists due to its excellent mechanical and electrical properties. Among its many applications, its use in Solid Oxide Fuel Cells (SOFCs) is one of the most prominent. SOFCs are electrochemical devices that convert the energy of a chemical reaction directly into electrical energy.

Zirconia stabilized with 8-mol% Y₂O₃ (8YSZ) is the state-of-the-art electrolyte material for SOFC applications. However, despite the extensive use of 8YSZ for SOFCs, within the past decade zirconia stabilized with 10-mol% Sc₂O₃ and 1-mol% Ce₂O₃ (10Sc1CeSZ) has garnered much attention as one of the best ionic conductors for SOFCs operated at lower temperature (600–700 °C). In the search of new electrolyte systems with high ionic conductivity, other parameters such as strength, structural and chemical stability, and cost of materials are very important if such materials are going to be used in commercial devices. In consequence of high price for 10Sc1CeSZ there are many studies are focused on combination of YSZ and ScSZ systems to improve electrolyte properties. Zirconia stabilized with 3.5-mol% Y₂O₃ (3.5YSZ) provides higher strength than 8YSZ and 10Sc1CeSZ, but it doesn't provide the enough ionic conductivity.

The provision of improved electrolyte mechanical strength complete with sufficient ionic conductivity is possible through the use of 3.5YSZ and 10Sc1CeSZ. Thus, the present work is devoted to studying the influence of varying amounts of 3.5YSZ (33-, 40-, 50-wt.%) on the electrical and mechanical properties of a 10Sc1CeSZ-3.5YSZ composite. As a base for comparison with the 10Sc1CeSZ-3.5YSZ properties, 3.5YSZ, 10Sc1CeSZ and 8YSZ have been used.

The most important observation of this study is that the solid state reaction/or redistribution of stabilizers (Sc₂O₃, CeO₂, Y₂O₃) take place in 10Sc1CeSZ-3.5YSZ ceramic samples during sintering at 1400 °C for 2 h. The 10Sc1CeSZ-3.5YSZ (33-, 40-, 50-wt.%) ceramics has had lower normalized electrical conductivity at 600 °C ($3.2 \cdot 10^{-3}$, $2.2 \cdot 10^{-3}$ and $1.2 \cdot 10^{-3}$ S·cm⁻¹, respectively) and fracture strength (111, 117 and 133 MPa, respectively) comparing with 8YSZ ceramics ($4.6 \cdot 10^{-3}$ S·cm⁻¹ and 247 MPa) made at the same rate and tested at the same conditions.

OC-90

**THE STUDY OF CERAMIC COMPOSITES BASED ON ZIRCONIA
AND MANGANITE WITH EXCESS MANGANESE FOR
CATHODE OF SOFCs**

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Nd_{0.67}Sr_{0.33}MnO₃ manganite is an alternative ceramic material for use as a cathode in solid oxide fuel cell (SOFC) operating from 500 to 800 °C that considered the most suitable cathode materials. cathode material of this composition has a coefficient of thermal expansion close to the specific value of the Sc₂O₃ stabilized ZrO₂ electrolytes (ScSZ), high electrode activity and high electrical conductivity at given temperatures. At the same time, there is practically no information about the microstructure, phase stability and durability of this material in the operating mode. Moreover, there are no data on the effectiveness of the cathode in cells with electrolyte based on Sc₂O₃ and CeO₂, which has more specific properties for medium-temperature SOFCs.

This paper presents the results of studying the effect of excess manganese on the structure and conductivity of composite ceramic materials consisting of manganite (Nd_{0.67}Sr_{0.33})_{1-x}Mn_{1+x}O₃ ($x = 0$ and 0.2) and electrolyte 10 mol% Sc + 1 Ce mol% (NSMO-SCSZ) used as the cathode for the medium-temperature SOFCs. To obtain composites, nanosize powders of manganites and electrolytes were mixed in equal mass fractions and sintered at 1000 °C.

According to the research results of NSMO-SCSZ ($x = 0$; 0.2) composites the excess manganese ($x = 0.2$) promotes the formation of the microstructure which appropriated requirements for cathode materials, namely a porous microstructure (open porosity of 20–30%) with necks formed between manganite grains (grain size of 200–300 nm) and an electrolyte, but also the preservation of phase composition of both materials, which is a completely new result in the studies of these composite cathodes. This behavior has affected the electroconductive properties of the samples. It's found that the sample with excess manganese has a electroconductivity twice more than the sample without excess manganese in all temperature range. Moreover the activation energy E_a of conductivity for sample with $x= 0.2$ is 0.38 eV which is almost two times less than for $x = 0$ (0.52 eV). This is due to the fact that the excess manganese located on the surface promotes a sintering and forming of grain boundary that leads to reduction in the energy barrier for charge carriers.

Thus, it is found that excess manganese in manganites significantly influences the formation of the structure and conductivity of composite cathode materials, namely it ensures the formation of the necessary grain size and porosity, the preservation of phase composition and high conductivity of the nanocomposite.

OA-91

**ELECTRICAL PROPERTIES OF $\text{SiO}_2\cdot\text{Na}_2\text{O}\cdot\text{LiO}_2\cdot\text{Bi}_2\text{O}_3$
CONDUCTIVE GLASSES**

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It is common sense that ceramics are non-conductive at room temperature. This is due their occupied valence band is separated from their non-occupied conduction band by a very large spacing between the bands, making it difficult to excite the electrons according to the available thermal energy. In this way, it is possible to increase the electronic driving capacity by increasing the thermal energy of these materials. Some oxide glasses may be conducting electrolytes by the action of alkaline monovalent cations or silver. As ionic conductivity is the product of the concentration of the effective charge carriers by their mobility, the observed increase in conductivity can be attributed to both an increase in mobility and an increase in the concentration of these effective charge carriers. Vitreous materials have been researched as solid electrolyte in solid-state batteries since the 1980s. However, according to the literature on this subject it is evident that this interest still exists today. Therefore, the aim of this work was to evaluate the influence of lithium, sodium and bismuth oxides on the electrical properties of silica glasses. The initial compositions of the glasses were based on patents. Constrained mixture design (DoE) was used to define ten compositions studied in the present work, using SiO_2 , Na_2O , LiO_2 and Bi_2O_3 as main factors, from their starting materials. The compositions were initially studied by DSC/TG to determine their melting temperatures as well as the characteristic temperatures (T_g and T_s) of the glasses and the influence of each oxide on them. The compositions were melted in alumina crucibles at 1200 °C for 2 h. The glasses were poured into graphite mold and after annealing, glass samples were characterized by XRD, FTIR and electrochemical impedance spectroscopy (EIS). From the EIS results, the charge resistances were determined for all glasses, and compared to ITO. Some glasses showed a charge resistance $5\times$ higher than ITO due to ionic conduction.

OA-92

BIOMIMETIC ZINC AND MAGNESIUM SUBSTITUTED CALCIUM PHOSPHATES DERIVED FROM BIOGENIC SOURCE

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Hydroxyapatite (HAp) is the most widely used calcium phosphate (CaP) for hard tissue applications due to its compositional similarity to biological apatite, biocompatibility and bioactivity. Along with HAp, octacalcium phosphate (OCP) and tricalcium phosphates (TCP), are gaining increasing attention. As bone apatite is a multi-substituted carbonated HAp containing trace elements, one of the used approaches to improve the biological and physicochemical properties of CaPs is ionic substitution.

In this study Mg- and Zn-substituted CaPs, with varying magnesium and zinc content (0, 1, 5 and 10 mol%), have been prepared by using cuttlefish bone as precursor of calcium and various trace ions. The effect of the doping on phase composition, crystal structure, morphology, thermal stability, ion release and toxicity of synthesized materials was studied. Mineralogical composition of the as-prepared and the heat-treated powders were determined using X-ray diffraction analysis. The lattice parameters, the cell volume and phase composition of prepared powders were determined by Rietveld refinement using software DIFFRAC.SUITE TOPAS V.5.0. The chemical composition of as-prepared unsubstituted, Mg- and Zn-substituted CaP powders and concentration of released ions were determined by inductively coupled plasma mass spectrometry.

The as-prepared powders were composed of calcium deficient carbonated HAp, OCP and amorphous calcium phosphate (ACP). Heat treated powders were composed of HAp, α -TCP and β -TCP. Along with magnesium and zinc ions, unsubstituted CaP is doped with carbonate, strontium and sodium ions, as they are present in cuttlefish bone. CaP systems are CO₃-substituted by B-type substitution as in natural bone mineral. Decrease of lattice parameters is evident in OCP and β -TCP phase for Zn-substitution, while for Mg-substitution in HAp, OCP and β -TCP phase. Zn²⁺ occupy the Ca(1,3,4,6,7,8) sites in OCP and Ca(1,2,3,4) sites in β -TCP, while Mg²⁺ ion Ca(2) site in HAp and Ca(4,5) sites in β -TCP. The culture of human embryonic kidney cells indicated non-cytotoxicity of the prepared CaP powders.

OA-93

**THE EFFECT OF THE PARTICLE SIZE ON SYNTHESIS AND
CRYSTALLIZATION OF POTASSIUM FLUORMICA
GLASS-CERAMICS**

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Production of sinterable fluormica glass-ceramic has always been a challenging process controlled by many factors. This study investigated the role of the glass powder particle size in synthesis and crystallization of potassium fluormica glass-ceramics. Potassium fluormica glasses were produced with compositions 51.2SiO₂, 10.6Al₂O₃, 6.5K₂O, 12.8MgO, 7.9B₂O₃, 10.7MgF₂ (mol%) using melt-quench method. Glass frits were milled and sieved producing three glass powder batches with different particle size fine (MF), medium (MM) and course (MC). Glass powders were characterized using differential scanning calorimetry (DSC), simultaneous thermal analysis (STA), X-ray diffraction (XRD) and ¹⁹F MAS-NMR then heat treated at 1000 °C for 5 hours. The produced glass-ceramics were characterized using XRD, SEM and ¹⁹F magic angle spinning-nuclear magnetic resonance (MAS-NMR). DSC results showed similar glass transition temperature (*T_g*) for different particle size. STA results revealed that the percentage of weight loss decrease with increasing particle size. XRD results showed that only MF glass-ceramic did not have all peaks of potassium fluorophlogopite. SEM images of MF glass-ceramics showed fewer and less developed mica crystals than others. ¹⁹F MAS-NMR spectrum of glass and all heat treated glass powder showed that the fluorine was presented as F-Mg(3) in the parent glass and glass-ceramics. Particle size of parent glass powder plays an important role in synthesis and crystallization of fluormica glass-ceramics.

OC-94

**THE COMPARISON OF THE BONDING ABILITY OF DENTAL
INSERTS BASED ON STRONTIUM AND MAGNESIUM DOPED
HYDROXYAPATITE WITH RESTORATIVE MATERIALS**

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One of the most common reasons for high failure rate of dental restorations remains the polymerization shrinkage and low mechanical properties of the resin based dental composites. Recently, this problem has been addressed by investigating utilization of inorganic dental inserts based on hydroxyapatite. Additionally, application of dental inserts for restorations of large cavities could simplify the procedure in the clinical protocol. The aim of this study was to compare bonding ability of dental inserts based on hydroxyapatite doped with strontium and magnesium ions (Sr-HAP and Mg-HAP), with commercially available restorative materials for possible application in the restorative dentistry. Nanostructured Mg-HAP and Sr-HAP powders were synthesized hydrothermally and pressed into cylindrical compacts. Inserts were obtained upon sintering of green compacts at 1200 °C. The bonding ability of inserts with different restorative materials was determined by the Shear Bond Strength test (SBS). The results indicated that application protocol of the commercially available restorative materials, i.e. total-etch or self-etch protocol, as well as the inserts nature had significantly influenced the SBS values.

OA-95

EVALUATION OF THE MORPHO-TEXTURAL AND MAGNETIC PARAMETERS OF THE $\text{SiO}_2\text{-Fe}_2\text{O}_3$ SYSTEM

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The sol-gel method is one of the simplest method to obtain silica matrices. Different approaches have been used to obtain the silica particles [1] and iron oxide. [2] In order to limit the increase of iron oxide particle the best way is to introduce this crystalline phase in the silica. The selection of silica dimension is a critical parameter to obtain better result in order to reduce the dimension to 20 nm of the iron oxide. So, in this case we modified the catalyst from the sol-gel reaction in order to change the pH of the sample. For the silica matrix TEOS precursor and water was used. Further, four catalyst were changed: NH_3 , CH_3COOH , HCl 0.1 N. and H_2SO_4 . All used solutions were concentrated. For the iron oxide $\text{Fe}(\text{acac})_3$ precursor was added. After a series of stages 8 samples were obtained. The materials were dried at 60 °C and 250 °C. All samples were evaluated by different physico-chemical methods as N_2 adsorption-desorption, magnetization, etc. The results obtained were primarily observed in color after calcination of samples at 250 °C but also in the specific surface area and magnetization. The BET, Langmuir, V-t plot, Alpha -S, BJH and DFT method was evaluated from the N_2 adsorption desorption isotherms [3]. The obtained results are indicating that when the pH of the samples is increasing the surface area is also increasing to $\sim 450 \text{ m}^2/\text{g}$.

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OC-96

**pH-RESPONSIVE DELIVERY OF ANTICANCER METAL
COMPLEXES BY FUNCTIONALIZED MESOPOROUS
SILICA-BASED NANOCARRIERS**

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Mesoporous silica nanoparticles (MSN) exhibit high potential for constructing stimuli-responsive drug carriers for targeted cancer therapy. The material is characterized by high surface area (ca. 1000 m²/g), various possibilities for surface functionalization, presence of mesopores (2-3 nm in diameter) and high biocompatibility. Drugs can be filled inside the mesopores or attached to the surface, while the surface of MSN can be specifically modified to allow the controlled release of the loaded drugs, triggered by a specific stimulus (change in pH, exposure to light, magnetic field, enzymes, etc.). Herein, novel cancer-targeting nanotherapeutics are developed based on MSN, containing therapeutic Ru(II)- and Ir(III)-coordination compounds covalently linked to the surface of MSN through an acidification-cleavable linker. This effect would enable targeted cancer treatment due to a weakly acidic environment of the extracellular matrix of tumor tissues, in comparison to the neutral pH of healthy tissues. Novel Ru(II) and Ir(III) coordination compounds are also synthesized, containing the same ligands that are attached on the surface of MSN, and their anticancer activity is also evaluated. The constructed series of novel nanomaterials and coordination compounds were characterized by scanning electron microscopy, thermogravimetric analysis, zeta potential and nitrogen sorption analyses, infrared spectroscopy, X-ray crystallography, and elemental analysis. The characteristics of prepared metal complexes and novel nanomaterials demonstrate high potential for their effective application in targeted cancer treatment, as determined by *in vitro* experiments on cancer cells.

OC-97

**SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS AND
MECHANOCHEMICAL SYNTHESIS OF ULTRA-HIGH
TEMPERATURE Hf-C-N CERAMICS FOR EXPLOITATION IN
EXTREME CONDITIONS**

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It has long been established that carbides of transition metals of groups IV and V possess the highest melting points and high hardness, and they can resist erosive flow. Therefore, such materials are widely used in the aerospace industry. However, a new round in the development of ultra-high temperature ceramics (UHTCs) of the new generation was marked by the creation of triple and multicomponent systems based on the original "champions" - carbides and nitrides of Ta, Hf, Zr. It is assumed that ternary compounds as compared with nitrides and carbides of transition metals will have better physical and chemical properties, as well as higher melting points.

At present, works that are devoted to modeling the properties of solid solutions, stoichiometric and non-stoichiometric introduction phases are being published quite actively. For example, according to theoretical calculations of scientists from Brown University (USA), hafnium carbonitride of non-stoichiometric composition $\text{HfC}_{0.51}\text{N}_{0.38}$ will have not only good chemical and mechanical properties, but also the highest melting point among all known compounds, above 4200 °C.

However, the preparation of such compounds is not an easy task due to the high melting points of the main components and the complexity of controlling the carbon-nitrogen ratio in the system. In contrast to the "traditional" methods of synthesis, which require large time and energy costs, self-propagating high-temperature synthesis (SHS) and mechanochemical synthesis (MS) make it possible to obtain materials of the required composition in a short period of time. The purpose of this work was to obtain a new promising UHTC based on hafnium carbonitride powders produced by the MS or SHS, followed by spark plasma sintering (SPS).

As a result, hafnium carbonitride powders of different composition were obtained and sintered into dense ceramics. The maximum density (98.7%) was observed in the sample obtained using the SHS method, along with the highest hardness (21.3 GPa) and crack resistance ($4.7 \text{ MPa m}^{1/2}$) among all samples.

OA-98

SINTERING OF BORON-RICH CARBIDE POWDERS

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In the boron-carbon system, there is only one boron carbide compound with large deviation from the formal stoichiometry, B_4C , in both sides of excess carbon or boron. Boron Carbide (B_4C) has many features such as the highest hardness, superior thermal stability, low density, lightweight, chemically resistant, large absorption of neutrons and high melting point. Several theoretical and experimental studies have suggested that the atomic bonding, electron density, mechanical properties and lattice constants of boron carbide change with boron/carbon ratios [1]. However, a systematic experimental investigation of the influence of boron/carbon ratios, particularly in the boron-rich side, on microstructure and sintering.

Boron carbide powders were usually synthesized by carbothermal reduction of boron oxide in a similar process performed in the presence of magnesium and carbon. Fine-grained boron carbide powders using the direct reaction between boron oxide and some organic carbon precursor-like saccharides can also be obtained. The goal of the present work studies on the direct synthesis of boron carbide powders using boron and soot powders as precursors and on pressureless sintering of prepared powders.

The synthesis was performed in a cylindrically closed graphite crucible. A compact of amorphous boron powder was placed on the bottom of the crucible and similar compact of soot powder was placed on it. The crucible with the powders was placed into a graphite furnace and then heat-treated at 1650 °C for 2 h under argon atmosphere. The powder layer above the boron compact was compound of the rhombohedral boron carbide close to $B_{13}C_2$ stoichiometry, the tetragonal $B_{48}B_2C_2$ phase and a small amount of carbon. The obtained powder and commercial boron carbide to compare with a few percent additions of boron, carbon, YAG, Y_2O_3 and a stoichiometric mixture of boron and carbon were ground, compacted and pressureless sintering at 2250 °C for 2 h. It was stated that the density of the sintered materials and their microstructure strongly depend on using addition and powder used to sintering

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OC-99

**TAILORING A NEAR ZERO TEMPERATURE COEFFICIENT
OF RESISTANCE IN PRESSURELESS SINTERED
MWCNT/Al₂O₃ AND GPO/Al₂O₃ COMPOSITES**

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Near zero temperature coefficient of resistance (TCR) in heating elements is essential for the precise control of temperature and constant power draw hence easy to control electronically. The aim of this work was to form a heater with a TCR of 500 ppm/K in the temperature range room temperature to 500 °C. This was achieved by percolating a network of electrically conductive near zero TCR multiwall carbon nanotubes (MWCNTs) or reduced graphene oxide (GPO) in insulating Al₂O₃. Using MWCNT compositions 1, 2 and 3 wt.%, MWCNT/Al₂O₃ samples were pressureless sintered in Ar at 1400, 1550 and 1700 °C for 4 hours. As a result, 88–96 % theoretical density was achieved for the composites sintered at 1550 and 1700 °C. Once percolation was achieved, the samples showed near zero TCR behaviour. However, thermal cycling revealed some hysteresis behaviour. These near zero TCR composites have also been thermally cycled multiple times to understand its effect on degradation of electrical properties and address the issue of health and safety on use of carbon nanomaterials in consumer products.

OC-100

**SYNTHESIS AND CHARACTERIZATION OF METAL-GLASS
COMPOSITE MATERIAL**

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Parts of industrial machines and structures are often exposed to aggressive environments, which in a short period of time can cause the loss of their integrity. Depending on working conditions, a surface of the material can be exposed to various mechanisms of damage, of which corrosion damage stands out. Commercial stainless

steels have a significant application in making machine parts in the industry because of their outstanding properties. However, the high prices of this material can sometimes be a limiting factor. For this reason, the idea is to replace expensive stainless steel with a composite material with lower price and improved mechanical properties.

Austenitic stainless steel is a material which is widely used in an industry primarily due to good corrosion resistance. Powder of commercial austenitic stainless steel (SURFIT TM 316L) of the diameter from 45 to 63 μm was used, in our experiment. The steel powder has a spherical shape which is obtained by gas atomization. The source of glass was andesite basalt rock from the locality "Donje Jarinje" Leposavic, the Republic of Serbia. Basalt is a hard aluminosilicate rock which has a relatively low melting point and low viscosity. The composite material was manufactured by mechanical mixing of stainless steel powders with freshly crushed basalt rock in diameter about 10 μm . The composite material consists of stainless steel and different content of basalt.

The green compact was obtained by a hydraulic pressing of the mixture, with a pressure of 150 MPa using a steel mold. Sintering is done at 1250 $^{\circ}\text{C}$ in a time of 30 minutes in a high-temperature vacuum furnace.

Semi-quantitative analysis of andesite basalt powder is obtained by energy dispersive X-ray spectrometry (EDS). Starting powder as well as sintered composites were characterized by X-ray diffraction method (XRD). Morphology of powders and microstructure of sintered sample were followed by a scanning electron microscope (SEM) and a light optical microscope (LOM). The hardness of the composite material is determined by the Vickers method.

OA-101

PRODUCTION OF Si_3N_4 CERAMIC TAPES BY TAPE CASTING METHOD FOR ARTIFICIAL BONE APPLICATION

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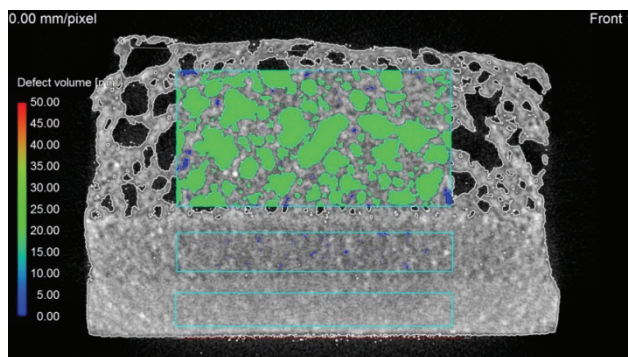
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Porous silicon nitride ceramics having properties similar to human bone have been sintered and characterized with a primary purpose to develop a material to be used as bone substitute. Functional grade materials are preferred to represent the human bone. Tape casting provides a good grading of this type of material. In the concept of this paper, tape casting method was studied for the production of porous Si_3N_4 ceramics containing "bone-like" micro and macro pores. In order to produce Si_3N_4 tapes by tape casting method, recipes were prepared by using different alcohol based solvent system

and adding various dispersing agents, binders, plasticizers and pore-forming agents. Tapes obtained by tape casting method were laminated and pressed according to their properties and sintered under air atmosphere at different temperatures after the removal of binder/



3D X-ray microtomography image of sintered sample

Final samples were characterized by 3D tomography and biological tests after microstructural examination and the results were evaluated. As a result, a bioceramic material, with bone-like microstructure, based on silicon nitride, containing micro and macro pores with gradual transition has been obtained.

OC-102

THE Al_2O_3 -Cu-Cr COMPOSITES – MICROSTRUCTURE AND SELECTED PROPERTIES

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The work investigated the influence of metal components content on the microstructure and selected properties of composites from Al_2O_3 -Cu-Cr system. The following powders were used: Al_2O_3 (Almatis) with density 3.96 g/cm^3 , Cu (Sigma Aldrich) with density 8.94 g/cm^3 and Cr (Createc) with density 7.14 g/cm^3 . The average particle size of initial powders was measured using Dynamic Light Scattering technique. Based on conducted measurements average particle size for Al_2O_3 , Cu and Cr powders were determined as $65.57 \text{ }\mu\text{m}$, $6.86 \text{ }\mu\text{m}$ and $12.59 \text{ }\mu\text{m}$ respectively. While Cu particles can be characterized by spherical morphology, Cr particles have had an irregular and

complex shape of particles. It was assumed that the application of two kinds of metal particles with different morphology as a metal phase can avoid the outflow of metallic phase during the sintering process. The composites were obtained by the slip casting technique. Commercial DURAMAX D3005 was used as a dispersant. Five series of composites with 50 vol% of solid content and 15 vol% of metal content with respect to the total solid phases were prepared. Particular series were characterized by different volumetric composition of metals in respect to total metal content in composite. Selected physical properties were determined using Archimedes method. The phase composition of sintered composite samples was examined by X-ray diffraction analysis. Microstructural observations were carried out with the use of Scanning Electron Microscopy. Actual metallic phase distribution was defined using stereological analysis performed on metallographic samples. Physical properties analysis revealed high porosity of sintered samples, what caused low examined relative density after the sintering process. X-ray analysis showed that all of the obtained samples consisted of three phases: Al_2O_3 , Cu and Cr. SEM observations revealed the homogenous microstructure of obtained samples and conducted stereological analysis showed uniform distribution of metal particles in ceramic matrix. Based on performed research it was concluded that the slip casting technique allows obtaining composites from Al_2O_3 -Cu-Cr system.

Acknowledgments: The study was accomplished thanks to the funds allotted by The National Science Centre within the framework of the research project ‘OPUS 13’ no. 2017/25/B/ST8/02036.

OA-103

SYNTHESIS OF B_4C POWDER VIA DYNAMIC THERMOCHEMICAL METHOD

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Boron carbide B_4C proved itself as a ceramic of a big importance due to its great hardness. Because of the effect of free carbon on the mechanical properties, a high-quality powder, with absence of free carbon, has always been desired. Furthermore, the extremely high synthesis temperature which exceeds 1800°C , and the further milling necessary to reduce the powder size, result an excess of energy, which proportionally increase the powder price. In this study, a reaction temperature of 1500°C for 1h was sufficient to synthesize submicron boron carbide powders from a mixture of boron oxide B_2O_3 and Carbon, via dynamic carbothermal reduction (DCR) method. XRD results showed a lower free carbon content, while SEM analysis demonstrated a reduced particle size of the as-synthesized powders.

Keywords: boron carbide, dynamic synthesis, advanced ceramics, carbothermal reduction

OA-104

HYDROLYSIS PROCESSES AND PHASE EQUILIBRIA IN ZrO_2 AND $4\text{Y}_2\text{O}_3$ -96 ZrO_2 PRECURSORS (mol%), OBTAINED BY REVERSE CO-PRECIPIATIONT. Lomakina, O. Kurapova, V. Konakov*St. Petersburg University, St. Petersburg, Russia*

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Oxide ceramics based on high-symmetric ZrO_2 modifications is widely used in industry as solid electrolytes in different electrochemical devices in automotive industry and for aerospace, as a functional material for prostheses and bone implants production. The use of sol-gel synthesis techniques is beneficial for high-performance ceramics fabrication. The use of co-precipitation from the aqueous solutions leads to the essential shift of phase equilibrium in oxide systems, crystallization of zirconia polymorphs at lower temperatures, than it is indicated on phase diagrams. The hydrolysis processes are taking place along precipitation and are competitive to sol formation. However no detailed investigation was performed so far. Thus the goal of the present work was the investigation of hydrolysis during precipitation and phase equilibriums sequence in the partially stabilized and undoped zirconia precursors in the temperature range of 20–1100°C.

For that, the potentiometric titration was used for hydrolysis processes investigation in $\text{ZrOCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{ZrO}(\text{NO}_3)_2$ salts with various. The cryochemical technique comprising reversed co-precipitation from 0.1M aqueous solutions of $\text{ZrOCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{ZrO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ with the subsequent liquid nitrogen treatment was used. As a precipitator 0.5, 1 and 3 M aqueous solution of ammonia was used, the solution of salts was added dropwise of 1–2 ml/min, pH = 9–10, $T = 1$ –2 °C. The fresh precipitate of hydroxides was placed in Dewar vessel with liquid nitrogen by the small portions, then the frozen granulate was dried in air. The received amorphous powders were annealed for 2 hours at 550, 800, 1000 and 1100 °C. Precursors after synthesis and calcination were investigated by simultaneous thermal analysis (STA, STA 409 of C/4/G Jupiter, NETZSCH), the X-ray diffraction analysis (XRD, Bruker “D2 Phaser”, Cu-K α the radiation, $\lambda = 1.54$ Å, with XRD patterns refinement by the Rietveld method), particle size distribution analysis (PSD, Horiba partica LA-950).

For the first time it was shown that in case of 0.5 and 1 M ammonia precipitator, for both salts the hydrolysis takes place in two steps. The reaction scheme was suggested. When 3M ammonia was used the simultaneous processes of precipitation and hydrolysis takes place. It was found that phase formation in $4\text{Y}_2\text{O}_3$ -96 ZrO_2 (4YSZ) powders takes place according to Y_2O_3 - ZrO_2 diagram. Other situation was observed for undoped zirconia precursors, obtained from various salts, i.e. in all temperature range formation of mixed tetragonal and monoclinic phases takes place with the different ratio. Phase transition from the amorphous to crystalline phase resulted in tetragonal phase content ~87 wt.% and 13 wt.% monoclinic phase. The increase of the temperature up to

1100 °C leads to tetragonal phase content decrease up to ~5 wt.%. The existence of the metastable tetragonal phase ZrO₂, most likely, is due to high powders dispersity.

Acknowledgement: STA and XRD research was conducted in the Research Centre for X-ray Diffraction Studies, Research park of Saint Petersburg State University. The research is conducted within research special grant of the president for young scientists – MK-2703.2019.3

OA-105

STRUCTURE, MAGNETIC PROPERTIES AND INDUCTION HEATING ABILITY STUDIES FOR HYPERTHERMIA TREATMENT OF TUMORS BY Mn SUBSTITUTED CuFe₂O₄ NANOPARTICLES

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In this research synthesizes nanocrystalline CuFe₂O₄ spinel structure using co-precipitation method. The pure copper ferrite is substituted by manganese Mn_xCu_{1-x}Fe₂O₄ nanopowders (x from 0.0 to 1.0). X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), UV-visible-Spectrophotometer and vibrating sample magnetometer (VSM) are utilized in order to study the effect of variation of Mn²⁺ ions substitution and its impact on crystalline size, lattice parameters, microstructure and optical and magnetic properties of the formed nanopowders. Moreover, the induction heating behavior of the formed nanopowders with alternative magnetic field was also determined based on the change of specific absorption rate (SAR) or specific loss power (SLP) with the synthesis conditions. Indeed, heating properties of the Mn_xCu_{1-x}Fe₂O₄ nanoparticles in an alternating magnetic field at 150–300 kHz were estimated. The specific heat rate SAR values were in the range from 125 to 350 W/g at different Mn²⁺ ions substitution. Finally, large SAR values are obtained using low magnetic field, making Cu-Mn ferrite appropriate for hyperthermia treatment of cancer.

Keywords: CuFe₂O₄, spinel ferrites, doped Mn ions, optical properties, magnetic properties, heat induction

OA-106

LASER SURFACE MODIFICATION OF CP-Ti IN DIFFERENT GAS ATMOSPHERES

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Aiming to enhance the biointegration properties of the metallic implant materials, different surface modification techniques were developed over the years. Laser surface irradiation is considered to be the most promising surface modification method since it enables modifications of the implant materials surface chemistry and morphology avoiding, at the same time, any possible surface contamination. In order to examine the possibility of the successful surface modification of the widely used biometallic material in regard to its enhanced osseointegration properties, the effect of the picosecond Nd:YAG laser irradiation on the commercially pure titanium (CP-Ti) surface characteristics was studied. In that purpose the Nd:YAG laser was operated at 1064 nm wavelength and with 150 ps pulse duration, while the repetition rate was 10 Hz. The CP-Ti was subjected to laser irradiation in air and argon atmosphere for different fluence values (7.1 and 42.4 J/cm²) and irradiation durations. Scanning electron microscopy (SEM) was used to analyze the resulting surface morphology and obtained results indicated that the interaction of the laser irradiation with the CP-Ti surface influenced the melting and vaporization of the material leading to the surface craters appearance. Moreover, the presence of the microcracks and hydrodynamic effects in the form of droplets and wave-like structures was identified. Formation of the passive Ti-oxide film, which can protect CP-Ti from further degradative oxidation, was identified at the surface by energy dispersive spectrometry (EDS). Presence of this film was more pronounced after irradiation in air. The changes emerged on the CP-Ti surface during the low-fluence and short-term irradiation were characterized as only superficial. Optical profilometry was utilized in order to additionally analyze the resulting surface modifications. Increase in irradiation duration and fluence value led to the deeper surface craters appearance. Comparison of the surface modifications achieved during the laser irradiation under the same irradiation conditions revealed that the higher damage degree was achieved during the irradiation in argon atmosphere. Obtained results indicated that the laser surface modification method could be used for the formation of a specific surface morphology suitable for the improvement of the biocompatible and osseointegration properties of the hard-tissue implant materials.

OA-107

**STRUCTURAL AND THERMIC INVESTIGATION ON A NOVEL
METHOD OF SYNTHESIS FOR HYDROXYAPATITE**

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Synthetic hydroxyapatite (HA: $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) has been extensively used as a bone substitute material due to its chemical and structural similarity with natural bone mineral [1,2].

In the present investigation, a novel method is developed to synthesize pure, stable, stoichiometric hydroxyapatite starting from $\text{Ca}(\text{NO}_3)_2$ and the source of phosphate being a mixture of two phosphate salts, $\text{NH}_4\text{H}_2\text{PO}_4$ and $(\text{NH}_4)_2\text{HPO}_4$. This type of method is not mentioned in literature and it is a work in progress.

Preliminary results have been obtained by using FT-IR and thermal analysis. The synthesis method was tested at different pH values, from 8 to 12. Finally, the first method mentioned of synthesis was most preferred due to the confirmation of obtaining pure hydroxyapatite from the FT-IR spectra and the thermal analysis of the dried samples and the samples sintered at 950 °C [3].

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OC-108

**SYNTHESIS, STRUCTURE AND THE POSSIBILITY OF USING
DIFFERENT MATERIALS FROM THE CALCIUM
PHOSPHATE GROUP**

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Materials which belongs to group of Calcium-phosphates (CaP) have wide range of applications, due to their good structural characteristics, chemical stability and simple synthesis. The materials from CaP group are used in the field of dentistry and medicine as implant materials, but they are increasingly used as adsorbents and solid electrolytes. In our paper, the results of synthesis, characterization, determination of structural properties and possibilities of hydroxyapatite, doped hydroxyapatite, brushite and monetite are presented. Modified solution precipitation method was used to prepare pure and doped Mg, Sr and Na hydroxyapatite type materials. Modification consisted of partial substitution of nitrates by acetate solution in order to achieve a more soluble and cost effective synthesis. All powders were characterized by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). Calcined hydroxyapatite samples were densified at 1000 °C in an air for 3 h, and characterized also by complex impedance methods. The highest conductivity was found for the multi-doped phosphate sample (CaSrNaP₁₀₀₀) at 700 °C ($1.90 \times 10^{-3} \Omega^{-1} \text{cm}^{-1}$). The corresponding activation energies of conductivity amounted to 0.31 eV in the temperature range 500–700 °C. Mesoporous Monetite (CaHPO₄), obtained by mechanochemical treatment of previously synthesized Brushite (CaHPO₄·2H₂O), was used as efficient adsorbent for the organic pesticide malathion. It was found that five minutes of milling induces Brushite-Monetite phase transformation. Adsorption of malathion from aqueous solutions showed that this pesticide can be successfully adsorbed on surface of these material. The results showed that simple and economically cost-effective methods of synthesis can provide materials of the desired structural properties that can be used in environmental protection and as solid fuel cell electrolytes at medium temperatures.

OC-109

**IN SITU SYNTHESIS AND CHARACTERIZATION OF
HYDROXYAPATITE/TITANIUM OXIDE COATINGS DERIVED BY
ANODIZATION AND ANAPHORETIC DEPOSITION**

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Titanium substrates were electrochemically treated by novel *in-situ* synthesis method to produce anaphoretic hydroxyapatite/titanium oxide (HAp/TiO₂) coating. This synthesis method was performed *via* anaphoretic deposition of hydroxyapatite and simultaneous anodization of Ti in order to produce highly adherent and strengthened composite coating, where the influence of electric potential, time, electrolyte concentration and pH value of the anodization process on titanium surface roughness and adhesion strength of composite HAp/TiO₂ coatings was examined. *In situ* synthesis was performed in different solutions containing different concentrations of NaOH, HAp and ethanol at different values of time and constant voltage. The effects of anodizing voltage on the morphology and bioactivity of the HAp coating and on the bonding strength between the HAp coating and the anodized substrates were investigated. A modified chemical precipitation method was used to prepare hydroxyapatite powder by the reaction of stoichiometric amount of calcium oxide and phosphoric acid. Prior to novel *in situ* synthesis of HAp/TiO₂ composite coatings, optimization of anodization process of titanium was performed. Anodization was executed under different electric potentials and different distances of counter electrodes from working electrodes, but all experiments had constant quantity of electric charge. Results indicated that highly ordered amorphous TiO₂ surface is formed on the Ti substrate after anodic oxidation. Characterization of titanium samples having rectangular contact surfaces of 10×10×0.89 mm included SEM/EDS analyses, X-ray diffraction analyses, AFM surface topography, morphology and roughness analyses and linear measurements of roughness. The prepared coatings were characterized by FE-SEM, XRD and EDS. Adhesion was investigated by ASTM D 3359–97 Test method B. Uniform and adherent HAp/TiO₂ composite coating on Ti was obtained. The results of simulated body fluid immersing experiments suggest that obtained HAp coating exhibits promising bioactivity. The obtained coating can be good candidate for bone implants due to improved adhesion.

OA-110

**ToF-ERDA/RBS ANALYSIS OF ANNEALED TiO₂ NANOTUBES
GROWN ONTO FTO GLASS**

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Different semiconductors have been studied as photoanode material for solar cells. Among them, TiO₂ has been shown to be the best material due to its chemical stability and good charge transport capability. It is usually deposited onto FTO glass (F-SnO₂), and then heated at high temperature in order to obtain inter-crystalline electric contact for improving electron conductivity. The focus of this research was chemical analysis of the TiO₂ nanotubes (NTs) obtained by anodization of titanium film on FTO glass using NH₄F in ethylene glycol electrolyte. There are only few studies suggesting that the electrolyte is a source of N dopant in TiO₂, but also Sn diffusion from FTO support was reported. This study aims to show the chemical distribution of elements of interest along the nanotubes depth. For that purpose the time-of-flight elastic recoil detection analysis (ToF-ERDA) and Rutherford backscattering (RBS) methods were used as complementary techniques for chemical analysis of both light, such is N, and heavy elements, such is Sn. The TiO₂ NTs films were annealed at different temperatures (450–630 °C) and the impact of the heating temperature on the chemical distribution was followed.

OA-111

STRUCTURE AND PHOTOCATALYTIC PROPERTIES OF SOL-GEL SYNTHESIZED PSEUDOBROOKITE

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Fe₂TiO₅ nanoparticles were synthesized by modified sol-gel method with aid of Fe(NO₃)₃·9H₂O and Ti(OC₃H₇)₄ as starting reagents, oxalic acid as chilate agent and cetyltrimethylammonium bromide as surfactant. The aim of this study was to asses the photocatalytic degradaton of the antibiotic Oxytetracycline (OTC) using visible light irradiation. As prepared nanoparticles were characterized by XRD, BET, FESEM and UV-vis DRS. The optimal operating conditions of oxytetracycline photocatalytic degradation were achived by changing the pH of the solution and changing the concentration of photocatalyst.

OA-112

PRECURSORS EFFECT ON Ni_{0.3}Co_{2.7}O₄ OXIDE ELECTROCATALYTIC ACTIVITY

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To develop an eco-friendly, stable, low cost, efficient, and selective material for Oxygen Reduction Reaction electrocatalysis, mesoporous Ni_{0.3}Co_{2.7}O₄ spinel oxide has been effectively synthesized using sol-gel process with different precursors. The Precursors nature and molecular components effect on the resulted oxides electrocatalytic properties was evidenced for the first time. The resulted mesoporous oxides exhibit an average particle size of about 9 nm with cauliflowers-like structure. The specific surface areas was found to be sensitive to the precursor nature, 56.49, 25.70, and 3.66 m²/g for the oxide obtained using, Chloride, Nitrate and Sulfate precursors, respectively. Interestingly, linear sweep voltammetry (LSV) and Rotating-ring-disk

electrode measurement reveals that all elaborated oxides exhibit excellent stability and electrocatalytic activity towards O_2 reduction. For a potential range up to 0.8 V (from 0.6 V to - 0.2 V vs RHE), $Ni_{0.3}Co_{2.7}O_4$ from nitrate salts exhibit preferential 4 e^- O_2 reduction pathway, with H_2O production higher than 90%. This result was found to be close to that obtained with commercial Pt graphitized carbon. However, $Ni_{0.3}Co_{2.7}O_4$ made from chloride and sulfate precursors display a selective 2 electron reduction of O_2 , with H_2O_2 production higher than 95% and 70%, respectively, for a potential range up to 0.8 V (from 0.6 V to - 0.2 V vs RHE). Electrode formed with $Ni_{0.3}Co_{2.7}O_4$ -Cl can effectively degrade 92 % of methylene blue within 2 h electrocatalysis.

The synthesized oxides are selective and very promising for several electrochemical applications including energy production and storage, also for wastewater treatment.

Keywords: Precursor, $Ni_{0.3}Co_{2.7}O_4$ oxide, O_2 reduction, electrocatalytic properties.

OA-113

TiO₂-BASED PHOTOCATALYTIC COATING FOR CERAMIC MATERIALS

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Air pollution is becoming an alarming issue, especially in industrialized and highly urbanized areas in both developing and developed countries. The most common atmospheric pollutants are carbon dioxide, sulfur dioxide, nitrogen oxides and dusts. A large share of nitrogen oxides pollution is related to road transport. Most urban agglomerations are struggling with over-normative concentrations of NO_x, especially in the vicinity of busy streets and main roads. The need to improve air quality leads to solutions focused on eliminating the pollutants and limiting their transfer from gas to liquid phase. Degradation of pollutants with the use of photocatalysts has been proved to be very effective in purifying the air. Out of the group, TiO₂ remains the most popular due to its non-toxicity, good chemical stability and high photocatalytic activity. The size of band gap energy is approximately 3.2 eV, which allows the absorption of radiation from UV range.

To help purify the air in urban agglomerations, an attempt was made to obtain a TiO₂-based coating with photocatalytic properties. This coating could ultimately be applied to ceramic surfaces, for example building facades, significantly contributing to lowering NO_x concentration in the air. Preparation of thin TiO₂ film is possible with the use of sol-gel method, which is based on alkoxide hydrolysis reaction. The main reaction substrate is titanium alkoxide (Ti(OR)₄), which is dissolved in an organic solvent. The formation of an active coating composed of surface-bonded nanocrystallites depends on

the method of obtaining the sol and the calcination temperature of the ceramic material with the TiO₂ coating.

The crystallite size and the photocatalytic capacity of the TiO₂ coatings change as the calcination temperature increases. TiO₂ crystallizes in the form of anatase, which transitions to rutile as the temperature rises. Photocatalytic ability depends on the polymorphic form of the crystallites formed. Particle size and phase composition of the formed nanocrystalline TiO₂ can be controlled by the temperature of the firing process.

OC-114

**STUDY ON THE PHOTOCATALYTIC BEHAVIOR OF Zn BASED
SEMICONDUCTOR IN THE PRESENCE OF ORGANIC
STABILIZATOR AND CO-CATALYST**

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Hydrogen has a great potential taking into account the limited resources of fossil fuels and the increased need for new renewable and eco-friendly energy [1]. Due to the increased photocorrosion of the semiconductors with the narrow band gap and thus photocatalytic activity in the visible range, introduction of organic stabilizator and co-catalyst represents a good solution for the occurring problem. The final products were characterized by powder X-ray diffraction (XRD), Raman spectroscopy, transmission electron microscopy (TEM), Fluorescence spectroscopy (PL), Brunauer-Emmett-Teller (BET) and UV-visible spectroscopy. Raman measurements [2] together with BET [3] and fluorescence analysis provided a better understanding of the process, indicating the adsorptive, charge separation and transportation abilities of the used materials. Hydrogen evolution of the compounds was performed under visible light and determined by gas chromatography.

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OA-115

**VISIBLE LIGHT PHOTOCATALYTIC ACTIVITY OF
NANOCRYSTALLINE $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0-1$)**

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The sol-gel combustion method was applied for synthesis of spinel magnesium cobalt ferrites $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$, with varying cobalt and magnesium content, $x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9$. Magnesium nitrate, cobalt nitrate and iron nitrate were used as oxidizers and citric acid was used as a reducing agent. Structural and morphological properties of the obtained ferrite powders were investigated and characterized by X-ray diffraction (XRD), Raman spectroscopy, Field emission scanning electron microscope (FESEM) and Fourier transform infrared (FT-IR) spectroscopy. Optical properties were investigated by UV-VIS spectrophotometry. A cubic spinel structure with multigrain agglomerates formed. Visible light photocatalytic activity of the spinel ferrite powder samples was evaluated by measuring the rate of photodegradation reaction of methylene blue (MB). After 240 min $\text{Co}_{0.1}\text{Mg}_{0.9}\text{Fe}_2\text{O}_4$ showed the best rate of photodecomposition of MB resulting in 90% of its initial concentration in an alkaline environment.

OC-116

**HUMIC ACID REMOVAL FROM WATER USING $\alpha\text{-Fe}_2\text{O}_3$
NANOMATERIAL BY SORPTION AND PHOTOCATALYTIC
UNDER VIS IRRADIATION**

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In recent years, nanomaterial-based technologies have emerged as promising alternatives to current water treatment techniques at lower costs and high efficiencies

which should meet the increasingly stringent water quality standards. In this paper, the synthesis, characterization and testing performance of α -Fe₂O₃ nanomaterial for humic acid (HA) sorption and photocatalysis experiments has been presented. The Fe(acac)₃/silica/PVA nanocomposite was synthesized via sol-gel method at room temperature. The reactant molar ratio between reactants used for the synthesis was: TEOS : H₂O : PVA : Fe(acac)₃ : MeOH : HNO₃ = 1 : 10 : 1.2·10⁻⁵ : 0.20 : 18 : 0.01 was used. The synthesized material was characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) spectra and transmission electron microscopy (TEM). Textural properties of nanocomposite were analysed using nitrogen sorption/desorption measurements at liquid nitrogen temperature (77K). The morphology of iron oxide nanoparticles was examined by scanning electron microscopy (SEM). The photocatalytic experiments were conducted in a photocatalytic reactor under visible irradiation (VIS light set between 460 and 510 nm). The sorption experiments were carried out under similar conditions of photocatalysis without irradiation. Pseudo-first- and pseudo-second-order kinetic models were tested for fitting the sorption and photocatalysis experimental data. The pseudo-second-order kinetic model fit very well the experimental data for the humic acid (HA) sorption and photocatalysis. The calculated q_e values agree very well with the experimental data, and the correlation coefficients for the pseudo-second-order kinetic model are higher than 0.90. It can be concluded that the photocatalytic effect of α -Fe₂O₃ is manifested in special from kinetics point of view and higher dose led to better sorption performance. The results of this study recommend α -Fe₂O₃ as potential material for the design of advanced process to remove HA from water.

OC-117

INFLUENCE OF FERRITES PHASE ON PROPERTIES OF THE BARIUM ZIRCONIUM TITANATE BASED MULTIFERROIC COMPOSITES

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Multiferroic composites with general formula Ba(Ti_{0.95}Zr_{0.05})O₃-Ni_{0.7}Zn_{0.3}Fe₂O₄/CoFe₂O₄/Ni_{0.7}Cu_{0.01}Sm_{0.05}Zn_{0.29}Fe_{1.95}O₄, (BTZr(95-5)-NZF/CF/NCuSmZF) were prepared by mixing chemically obtained different types of ferrites and BTZ(95-5) powders in the planetary mill for 24 h. The optimization of sintering process was performed and powders were pressed and sintered at 1300 °C for obtained composites samples. From X-ray analysis for single phase and composites ceramics can be noticed the formation of crystallized structure of ferrites and barium zirconium titanate. SEM

analyses indicated the formation of two types nanosized grains, polygonal ferromagnetic and rounded ferroelectric grains.

The electrical properties of these materials were investigated using impedance spectroscopy and analysis of ferroelectric measurements. Impedance analysis of all investigated samples has shown different relaxation processes that originated from the grain and grain boundary contributions. The results of polarization vs. electric field measurements have shown the influence of magnetic phase type and its concentration on the ferroelectric properties of the composites. Due to high conductivity of ferrite phases and presence of interfacial polarization, the shapes of these curves differed from the conventional ferroelectric materials.

OC-118

FINITE DIFFERENCE METHOD FOR MODELLING THE DIELECTRIC PROPERTIES OF CERAMIC COMPOSITES

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Composite electroceramics (e.g. magnetoelectrics, porous materials, core-shell structures) contain at least two distinct phases with different chemical and physical properties, separated by well-defined interfaces. These interfaces usually collect uncompensated free or bounded charges, due to the fact that the constituent phases have contrasting permittivity and conductivity. Therefore, their effective dielectric properties are not simple *sum properties* derived from ones of the parent phases, but they contain important contributions from the interfaces. To describe the overall electrical response of such inhomogeneous systems, one should consider that interfaces between di-similar materials introduce local field inhomogeneities, which are specific to a microstructure. While the simple effective field theories provide information about the static permittivity, the numerical approaches allow to compute effective dynamic complex permittivity for realistic microstructures.

In the present work, we propose the Finite Difference Method to solve the Poisson equation to compute local electric field, charge distribution at different variable voltages and to extract the time-dependent $I(V)$ and impedance response of ferroelectric-magnetic composite ceramics with different types of compositions and phase interconnectivity. By considering thermally activated mechanisms for conduction (Arrhenius) in the calculation of local charge distributions (in the continuity equation), the frequency and temperature response were simulated and compared with experimental ones for composites based on ferroelectrics (BaTiO_3 , $\text{Pb}(\text{Zr,Ti})\text{O}_3$) and ferrites (CoFe_2O_4 , NiFe_2O_4) [1,2]. The model describes the role of different parameters as filling factor, permittivity and conductivity values of the parent phases, frequency and temperature, as

well as the role of microstructure, on the effective complex dielectric response of various types of ceramic composites.

Acknowledgements: The work was supported by Romanian grant PN-III-P4-ID-PCCF-2016-0175 grant (HighKDevice).

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OC-119

MAGNETIC AND FERROELECTRIC PROPERTIES OF SCTO-(C,M)FO MULTIFERROICS

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Two-component electroceramic materials of $\text{SrCu}_{1/3}\text{Ta}_{2/3}\text{O}_3\text{-(Co,Mn)Fe}_2\text{O}_4$ [SCTO-(C,M)FO] type were investigated due to the magnetic and ferroelectric properties. In contrast to natural multiferroics (multiferroicity state existing in a single-phase material), the various kinds of composites (particulate ceramics, multilayer laminates, 3D-heterostructures, *etc*) could significantly enhanced the magnetoelectric properties observed in this class of materials. Above aspect is very important as someone considers a large application potential for multiferroic materials in different scientific fields (e.g. optoelectronics, spintronics or solar energy devices and sensor technologies).

In this study, both the SCTO relaxor and (C,M)FO cobalt ferrite will be synthesized by a solid-state reaction method and then combined together to form the particulate composites of different proportions between the magnetic and ferroelectric

constituents, as well as the various contributions of Co and Mn elements in (C,M)FO ferrite.

The elementary physicochemical characterization of SCTO-(C,M)FO system will be presented. For this reason, SEM inspection of a local microstructure, as well as the structural investigation (XRD) of composites will be discussed. Also, a research will be involving the static and dynamic magnetic properties (VSM, *ac* susceptibility), as well as the magnetostriction parameters.

On the other hand, the presented work will be explore the *P-E* hysteresis loops, as well as transportation parameters involving the specific electrical conductivity (σ) and impedance spectroscopy (EIS) measurements. All above methods are convenient for a characterization of the ferroelectric properties of SCTO-(C,M)FO materials.

OC-120

CHARACTERIZATION OF SUPERCONDUCTOR (YBa₂Cu₃O_{7- δ}) - MULTIFERROIC (YMnO₃) HETEROSTRUCTURES

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In this work, we present results on three ceramic heterostructures based on YBa₂Cu₃O_{7- δ} (YBCO) and YMnO₃ (YMO). The former is the first high-temperature superconductor found to exhibit critical temperature above liquid nitrogen [1], whereas the latter is a class (I) multiferroic with a strong coupling between magnetism and ferroelectricity below 72 K [2,3].

Here, we explore the interplay between the magnetism in YMO and the superconducting state of YBCO by combining these compounds into macroscopic layered structures: YBCO/YMO, YBCO/YMO/YBCO and YMO/YBCO/YMO, which

were then annealed in a pure oxygen atmosphere [5–7]. The superconducting phase in YBCO was probed by the Raman spectroscopy [6,7].

The specific behavior of the different heterostructures was probed with different experimental techniques, such as magnetic susceptibility, electrical resistivity, specific heat and impedance spectroscopy [8]. We analyze our results in the framework of the DFT (Density Functional Theory) model [9,10].

A modulation of the ferroelectric ordering temperature in YMO is observed in the different structures probed. We discuss the interaction between the ferromagnetic and superconducting layers (for example in the structure YBCO/YMO/YBCO), within the context of the superconducting proximity effect [11].

This work opens routes towards the study of the interplay between superconductivity and magnetism in thin (mesoscopic) YBCO/YMO heterostructures.

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OC-121

ELECTRICAL CHARACTERIZATION OF METAL OXIDE COMPOSITES PREPARED THROUGH PHYSICAL AND CHEMICAL ROUTES

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Metal oxides are primarily wide band gap semiconductors with potential applications in several fields, such as optoelectronic devices, piezoelectric transducers, gas sensors, and transparent conducting films. Zinc oxide (ZnO) is an intrinsically *n*-type oxide, while nickel oxide (NiO) is an intrinsically *p*-type oxide. Thus, an oxide-based *p-n* junction can be fabricated using them.

In this work, we compare two different routes to synthesize these junctions, a solid-state reaction route and a wet chemical synthesis route. In solid-state synthesis, different percentages of NiO were mixed with ZnO and cold compacted, after which they

were annealed. The annealing temperature and time was optimized based on the I-V data. X-ray diffraction results showed only two phases, ZnO and NiO, and the morphology changes were studied using scanning electron microscopy. The I-V behavior of the composite exhibited non-linear behavior dependent on the NiO concentration. The optimum composition was identified for preparation by chemical synthesis.

A chemical precipitation route was optimized for the synthesis. This provided a more uniform distribution of the two phases and lead to an improvement in the non-linear I-V behavior. This route can also be integrated with printing techniques to prepare patterned composite deposition. A printed varistor (variable resistor) was fabricated as part of this work, and its electrical response was characterized.

OA-122

CERAMICS MICROSTRUCTURE SIMULATION ON THE ISOTHERMAL SECTIONS OF T-x-y DIAGRAM BY MEANS OF EXCEL SHEETS AND AutoCAD SOFTWARE

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To imitate a process of the heterogeneous material microstructures forming by the method of Frederick N. Rhines [1, Fig. 199, 202-203; 2], it is convenient to use the isothermal sections (Fig. 1), which were produced by means of the Excel sheets and AutoCAD software [3]. Adequacy of sections were checked by the graphics of T-x-y diagrams [4] of different topological types with the linear contour of the surfaces.

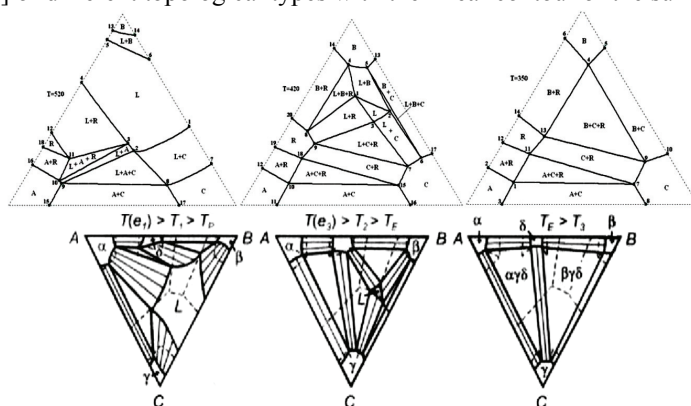


Figure 1. Isothermal sections at $T_Q < T = 520 < T_{eAC}$, $T_E < T = 420 < T_{eBC}$, $T = 350 < T_E$. Below – the same sections in [4, Fig. 4.39]

Acknowledgments: This work has been performed under the program of fundamental research SB RAS (project 0336-2019-0008), and was supported by the RFBR project 17-08-00875.

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OA-123

PROCESS PARAMETERS OPTIMIZATION OF ALUMINIUM-TRIHIDRATE SYNTHESIS

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Alumina is product which can be produced in several ways, whereby different quality can be derived. Although it is very complicated, Bayer process is most abundant because it gives alumina with very good quality. Main raw material for this process is bauxite, whose extraction, using sodium hydroxide as solvent, sodium aluminate is produced. Using the processes of decantation and filtration sodium aluminate is separated from red mud and it undergoes further processing. Depending of alumina type which has to be produced, solution is treated ie not treated with appropriate additive, and later crystallization condition determines other properties and usage of alumina. In this paper, as well as the title hints, effect of additive for whiteness is examined on quality of derived aluminium hydroxide. Under that, effect of temperatue, concentracion of additive, and mixing time, is included. Also, by reviewing literature conditions of decomposition of sodium aluminate is processed as well as it's effects on product quality. Obtained white aluminium tri hydrate is stored and sell or it can be used for production of white aluminum oxide by calcination. Main application of this product is for manufacturing alumina or ordinary ceramics, and also it can be used as filler for paper, dyes, glues, for manufacture of catalyst and so on. Due to it's good physical-chemical and mechanical properties alumina represents one of the most used engineering material. Thus ceramics on the basis of alumina is widely used for production of fine ceramics which have excellent mechanical and electrical properties and chemical resistance. In combination with other refractory compounds alumina builds wide spectrum of different refractory ie ceramic products.

Keywords: alumina, additive for whiteness, Bayer process, crystallization, sodium aluminate

OC-124

EFFECT OF ZrSiO₄ ADDITION ON SINTERING OF GLASS-CERAMIC MATERIALS FROM THE SiO₂-Al₂O₃-Na₂O-K₂O-CaO-MgO-BaO SYSTEM

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Analysis of the sintering process allows for the appropriate design of the sintering curves in order to obtain a material with a high degree of crystallization. Both, chemical composition and heat treatment determine the final phase composition, which results in the functional properties of these materials. Zirconium silicate (ZrSiO₄) is commonly used in the ceramic industry as an opacifier. Furthermore, it provides excellent mechanical and chemical resistance. The presented research results concern the influence of various quantities of zirconium silicate on the sintering of glass-crystalline materials from the SiO₂-Al₂O₃-Na₂O-K₂O-CaO-MgO-BaO system.

OC-125

MICROFLUIDIC SENSORS REALIZED USING LOW-TEMPERATURE CO-FIRED CERAMIC TECHNOLOGY

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Microfluidics deals with the control and manipulation of fluids on sub-millimeter scale in a single or network of micro channels. Manipulation of fluid at the micro scale found the applications in many different practical applications such as: particle separators, droplet generators, point-of-need diagnostics, etc.

Microfluidic devices can be fabricated in ceramic-based Low-Temperature Co-fired Ceramic (LTCC) technology, thanks to the simplicity and rapid forming of complex channels within a single ceramic layer. For the realization of a microfluidic chip in LTCC process, LTCC tapes with different thicknesses and physical characteristics are usually used from various producers (CeramTec, DuPont, Heraeus), laser for formation of micro channels in desired geometry, and lamination and sintering process (in a furnace, using carefully selected thermal profile). Before firing, non-baked LTCC tapes are mechanically flexible and numerous geometrical shapes can be cut. LTCC-based microfluidic chips have chemical and temperature stability, very good mechanical properties. On the other hand, LTCC allows printing of conductive and restive material

directly on the tape, and therefore the detection circuit and electronics can be easily integrate with a microfluidic devices. For some applications, drawback of LTCC technology is non-transparency, thus it is necessary to perform bonding of LTCC structure with other transparent materials, such as PDMS or glass. In this paper we propose a several different microfluidic devices realized using LTCC technology, such as microwave sensors, microfluidic sensors for biochemical diagnosis, potentiometric microfluidic device for biparametric analysis, etc.

In this paper, we present also a novel recently proposed technique for the rapid prototyping of low-cost microfluidic chips that rely on a combination of laser micromachining and xurographic technique. The proposed chip combines two materials, PVC foil and LTCC tape, a glass ceramic base material composed of an anorthite glass (calcium aluminosilicate) with ceramic filler. The complete fabrication process will be demonstrated and several microfluidic chips, including 3D multilayered microfluidic chip for particle filtration, microfluidic mixers and microfluidic microwave sensors will be presented. The advantages of the proposed technology will be demonstrated thought comparison with other microfluidic technologies in term of biocompatibility, optical and mechanical properties, cost, etc.

OC-126

Ni_{0.3}Co_{2.7}O₄ SPINEL OXIDE IMMOBILIZED IN TEFLON CAVITY ELECTRODE FOR ENVIRONMENTAL APPLICATIONS

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Mesoporous Ni_{0.3}Co_{2.7}O₄ oxide nanoparticles were effectively synthesized by sol-gel process, using sulfate precursors and citric acid as chelating agent. The obtained nanopowders were characterized, immobilized in Teflon Cavity Electrode (TCE) to investigate their catalytic properties, using Ethylene Glycol and Methylene Blue as contaminants models in various conditions. Spinel mesoporous Ni_{0.3}Co_{2.7}O₄ nanocrystallites of about 11 nm with regular cauliflower-like shape agglomeration and high surface area equals 62 m²/g were obtained. Interestingly, the TCE exhibits excellent electrochemical stability and ability to remove both pollutants with a rate of more than 92%, by pseudo-first order mechanism, without significant adsorption and can be used again for several cycles.

Low cost, simple preparation, long-term storage, operational stability and ability to degrade recalcitrant organics can make this electrode a candidate for industrial use.

Keywords: spinel nanopowders, Sol-gel process, immobilization, catalytic properties

OT-127

PRIMARY RESEARCH ON PORCELAIN WASTE UTILISATION

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Due to low sinterability, hard porcelain waste is mainly used as an additive (up to 10% by weight) to the bodies for the production of sanitary wares. The increase of recyclability for this waste could result in economical and environmental benefits. In this primary study, sinterability was examined depending on the grain size distribution. Several sintering activators, such as ZnO, CaO and MgO was introduced into porcelain waste up to 10 wt%. In this work, influence on various technological parameters i.a. shrinkage, bulk density, flexural strength and water absorption were studied. Phase composition of obtained materials was studied by X-ray diffractometry (XRD) and scanning electron microscopy (SEM) with EDS microanalysis. All of obtained samples showed significantly increased flexural strength comparing to samples made from unprocessed porcelain waste.

OT-128

MICROSTRUCTURAL CHARACTERIZATION OF SOLIDIFIED/STABILIZED MATRICES OF POLLUTED SEDIMENT AND CLAY IN LONG-TERM TREATMENT

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The application of stabilization/solidification technology (S/S) to low-cost and easily available materials and binders today has wide application in the treatment of contaminated sediments with heavy metals. This research conduct extremely contaminated sediment from European environmental hotspot “Great Bačka Canal” to several tests in order to increase concentration and toxic capacity of priority substances of 6 heavy metals (Cr, Ni, Cu, Zn, Cd, Pb) and As. The investigated matrix represents the S/S sediment mixture with kaolinite as immobilization agent followed by changes in the measured concentrations of heavy metals and arsenic after 7, 28 days and after 7 years. The changes in the performance of the tested matrices and rapidly decrease trend of concentration from investigated substances after treatment were observed. For the purpose of monitoring the testing of long-term performance of the treated mixtures,

various techniques such as SEM/EDS, FTIR, XRD analyses and leaching tests were applied in order to test the leaching concentrations of heavy metals and arsenic. Using standard DIN and TCLP toxicity tests, a reduction in heavy metal concentrations after 7 years of aging was determined as a consequence of pozzolanic reaction and the formation of CSH gels. In terms of microstructural characteristics, the presence of CSH gels was confirmed using SEM analysis identified as an amorphous structure. The composition of the C-S-H gel is variable and the incorporation of the anion or cation is related to the molar ratio Ca/Si determined by the EDS analysis. Based on the XRD analysis, different mineral fractions were identified, and the most common scores were CSH, muscular, dolomite, illite, quartz and calcite. It was found that after seven years the crystalline structure prevails and that the sediment is more stable. FTIR analysis identified Si-O-Al and O-Si-O bands, which confirms the presence of silicates that are very important in the immobilization of metals.

Acknowledgement: This work has been produced with the financial assistance of the Ministry of Education, Science and Technological Development of the Republic of Serbia (III43005)

OT-129

CEMENTLESS CONCRETE COMPOSITE FROM KAOLINITE AND ALUMINO-SILICATE WASTE MATERIALS

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In presented paper the results of research carried out in the Łukasiewicz Institute of Ceramics and Building Materials are shown. The main aim of this work is obtaining cementless geopolymer high durability concrete from inorganic waste materials. Geopolymer materials are modern, inorganic, amorphous, synthetic alumino-silicate polymers, with specific composition and properties. The basic ingredients for obtaining the binding geopolymer material are low calcium content alumino-silicates, which can be derived from natural raw materials or industrial wastes (such as silica fly ashes).

The basic compounds, used in presented research, to develop cementless geopolymer concrete, apart from the aggregate, were fly ashes, derived from coal combustion. Conditions of alkali activation were determined based on literature review and preliminary research. However cementless geopolymer concrete based only on fly ashes, despite of achieving high compressive strength, after alkaline activation, was characterized by pure freeze-thaw resistance. Optimization of the concrete composition through introduction thermal activated kaolinite waste clay allowed to get cementless concrete composite with enhanced properties. Created composite is resistant to carbonization, low temperatures and shows the stability of certain properties, while storage in air-dry conditions as well as in the water.

The properties of obtained composites, were tested with mutually complemented methods such as: spectroscopy electron microscopy, RTG powder diffraction and differential thermal analyzes. Results obtained from preliminary research lead to the statement, that alkali activation of fly ashes with burned clay, by NaOH solution and sodium water glass, with proper curing conditions (low-pressure infusing process) allows to achieve high strength geopolymer concrete.

Based on results of phase research, can be supposed that a binding and hardening process of tested composite is a result of dissolution of active components of fly ashes and burned kaolinite clay in alkaline solutions. Alumino-silicates gels crystallized and create hydrated alumino-silicates of sodium like a sodalite. Also it is possible that the cementless geopolymer concrete made by modified fly ashes and thermal activated clay waste can be used in wide applications in civil engineering what allows to reduced usage of natural mineral resources and energy.

OT-130

STUDY OF THE EFFECT OF CALCIUM SUBSTITUTION BY MAGNESIUM IN A GLASS-CERAMIC FERTILIZER

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Since conventional phosphate glasses have poor chemical durability, their application was limited. Thus, many efforts have been focused on increasing their chemical durability. However, new applications are lately emerging for environmental materials such as controlled release fertilizers. The aim of this work is to elaborate a new glass ceramic fertilizer and to study the effect of Calcium substitution by Magnesium on thermal properties and the chemical durability of this. The samples were characterized by DSC, Density, FTIR Spectroscopy and SEM. The dissolution behaviour was investigated using ICP.

Keywords: ceramic, glass, chemical durability.

OA-131

APPLICATION OF ICP AES METHOD AND MICROWAVE DIGESTION TO ROUTINE ANALYSIS OF CORUNDUM MATERIALS

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Corundum is a natural polymorphic form of Al_2O_3 , and it is known for its superior hardness (9 in Mohs' scale). Thanks to this property, $\alpha\text{-Al}_2\text{O}_3$ is often utilised as a grinding material. Its high thermal and chemical resistance explains its wide application in the refractory industry. Moreover, the biocompatibility of corundum allows it to be implemented in the field of tissue engineering. The technologies used to produce refractory as well as abrasive materials and to perform tissue engineering set different requirements concerning the chemical composition of corundum.

The determination of the chemical composition of corundum raw materials is a difficult and tedious analytical task. This is mainly due to problems connected with the total decomposition of the material. In the presented study, two different microwave systems were used to accelerate the digestion procedure. One of them, namely the microwave digestion of corundum samples in a closed, single-mode focused microwave energy system proved to be effective at achieving the total decomposition of the samples. The optimal analytical conditions for the ICP AES determination of alumina and other components (silica, iron, titanium, calcium, magnesium, sodium and potassium oxides) with satisfactory precision were also chosen. The total time of sample digestion and ICP AES determination of the main component - Al_2O_3 - and the accompanying 7 components (silica, iron, titanium, calcium, magnesium, sodium and potassium oxides) does not exceed 3 hrs, whereas the determination of these components by means of the conventional method requires about 5 days. The proposed ICP AES analytical procedure was validated by analyzing the corundum certified reference material and by comparing the results with those obtained via standard methods and XRF, and a good agreement between the ICP AES results and reference values was obtained.

OC-132

PHASE RELATIONS AND ADVANCED MATERIALS IN THE ZrO_2 - La_2O_3 - Gd_2O_3 SYSTEM

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The ZrO_2 -based materials are practically important as the thermal barrier coatings for high temperature gas turbines, due to their low thermal conductivity, high temperature thermal stability and excellent interfacial compatibility. Studies of the phase equilibria of the ZrO_2 -based systems can provide the necessary basic knowledge to develop the next generation TBC materials.

The objective of this work is to investigate the phase relations in the ternary ZrO_2 - La_2O_3 - Gd_2O_3 system at 1500, 1600 °C in air in the whole concentration range. Zirconium oxide nitrate, $\text{ZrO}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, powders of La_2O_3 , Gd_2O_3 (99.99 %) and analytical-grade nitric acid were used as the starting materials. The samples were prepared from nitrate solutions with their subsequent evaporation and decomposition at 1000 °C for 2 h. Thermal treatments at 1500 °C for 50 h and at 1600 °C for 10 h in air were carried out. The phase compositions were investigated by X-ray diffraction, petrography, microstructural phase and electron microprobe X-ray analyses.

New phase in the ternary system was not revealed at 1500, 1600 °C. The structure of the boundary binary systems defines the phase equilibria in the ZrO_2 - La_2O_3 - Gd_2O_3 system. Solid solutions based on cubic with fluorite-type structure (F) and tetragonal (T) modifications ZrO_2 , cubic (C) and monoclinic (B) modification Gd_2O_3 , hexagonal (A) modification of La_2O_3 as well as intermediate phases with pyrochlore type structure $\text{Ln}_2\text{Zr}_2\text{O}_7$ (Py) were determined. The refined lattice parameters of the unit cells and the boundaries of the homogeneity fields for solid solutions were determined.

In the zirconia-rich corner, the solid solutions based on tetragonal modification of ZrO_2 are formed. The solubility of La_2O_3 in the T- ZrO_2 is low and amounts to ~0.5 mol %, as evidenced by XRD analysis results (1500, 1600 °C). The solid solutions based on tetragonal modification of zirconia cannot be quenched from high temperatures due to low stability of T- ZrO_2 under cooling with furnace conditions. The diffraction patterns recorded at room temperatures included the peaks of monoclinic phase M- ZrO_2 .

The isothermal section of the ZrO_2 - La_2O_3 - Gd_2O_3 system at 1600 °C contains four three-phase regions (T+F+Py, A+Py+B, Py+F+B, F+C+B) and nine two-phase regions (T+Py, F+Py, F+T, F+C, F+B, B+Py, A+Py, B+A, B+C). The isothermal section of the ZrO_2 - La_2O_3 - Gd_2O_3 system at 1500 °C contains four three-phase regions (T+F+Py, A+Py+F, A+B+F, B+C+F) and ten two-phase regions (Py+A, A+F, A+B, F+B, B+C, C+F, two regions F+Py, F+T, Py+T).

OC-133

**CONTRIBUTION TO THE IMPROVEMENT OF THE PROPERTIES OF
SiO₂-BASED POLYMER COMPOSITE MATERIALS**

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In recent years, silica has found interesting applications in a variety of disciplines including concrete, catalysis, clean technology, separations science and microelectronics devices. Silica is also a good candidate as filler in composite polymers. In fact, polymer composites are the advanced materials alternative to traditional materials such as metals or ceramics and consist of at least two constituents of different phase, one of them being continuous polymeric matrix phase and other is reinforcements (fibers, filler). Interaction between the filler and the polymer matrix is a key of the properties of the polymer composites. The control of the interface of the filler is the very important without the addition of chemical agent. Although it is challenging to determine the true surface of SiO₂ compounds in order to avoid hazardous additions.

The specific aim of this work is to study the dependence between structure, surface state and reactivity of silica for different heterogeneous SiO₂ compounds and evaluate the behaviour of their surface subject to chemical stress, to increase reactive ability of theirs surfaces to be able to interact with the molecules of modifiers. The surface morphology of silica is examined by Variable Pressure Scanning Electron Microscope (VP-SEM) and showed the original fibrous surface of silica quartz. The FTIR frequency shift of the bridging oxygen stretching vibration Si–O–Si is observed and the intensity ratio between Si–OH band and Si–O–Si increases is determined. Furthermore, X-ray diffraction showed that the quartz lattice was conserved during the treatment with a shift of the main peak 101 in agreement with the infrared results on the Si–O–Si peak shift and the increase in the intensity ratio of Si–OH / Si–O–Si. The phase obtained will be used to prepare polymer composites with high thermal and mechanical performances

OC-134

**THE EFFECT OF TITANIUM BORIDE ADDITIVES ON THE
MECHANICAL AND TRIBOLOGICAL PROPERTIES OF MATERIALS
IN Fe-Cr-C SYSTEM**

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The effect of the addition of titanium borides (0.38–2.2 wt.%) on phase - structure formation, physicommechanical and tribological properties has been investigated. Titanium boride additives cause the formation of multiphase structure of a matrix-filled type which consisting of an iron-based matrix and solid inclusions of complex chromium-iron carbides such as Me_7C_3 and Me_{23}C_6 and carboborides, as well as electron and X-ray microscopic analysis showed the presence of small spherical phases based on titanium in matrices. The titanium boride additives intensify the interaction of components in the system, leading to additional doping of the material, as well as to the formation of solid inclusions like Fe_2B and FeB and TiC , which significantly increase the microhardness of the solid phases.

It is determined that the optimal content of alloying additives is 0.74 wt.%. With an increase in the amount of TiB_2 to 2.2 wt.% volumetric shrinkage of materials from 35% to 21.5% deteriorates, and also this leads to a slight decrease in hardness from 74 to 71.8 HRA and a flexural strength from 1940 to 690 MPa.

Resistance to abrasive wear paired with a diamond ring and wear resistance under dry friction together with steel 100Cr6 was studied. Material 64.63%Fe - 34.8%FH800 - 0.74% TiB_2 is distinguished by high wear resistance under abrasive wear (11.3 km/mm), and the material 64.26%Fe - 34.26%FH800 - 1.48% TiB_2 has high wear resistance with dry friction paired with 100Cr6 (44.7 km/mm).

Such materials are recommended for use in conditions of intensive abrasive wear for static loads and for the manufacture of parts of friction pairs.

AUTHOR INDEX

Abdelghany A.M.	58	Bobić J.	78, 90, 137
Abedi M.	73	Bobrowski A.	149
Abramović B.F.	94	Bochenek D.	76, 82, 139
Aciksari C.	87, 103	Boffa V.	44
Adamczyk-Habajska M.	75, 76, 80, 84	Boros A.	96
Afqir M.	151	Borrell A.	70
Agostino R.G.	45, 47, 48	Bošković M.	37
Ajdys L.	111	Bossini D.	23
Akarcay O.	103	Boteva T.	68
Akimov G.	113	Boussebha H.	125
Albu P.	129	Boyer L.	42
Aleksić J.	37	Božanić D.K.	57
Almásy L.	95	Braida M.-D.	106
Alzahrani A.	59	Brodnikovska I.	112
Andrievskaya E.R.	150	Brodnikovskiy D.	112
Anglada M.	64	Brodnikovskiy Y.	112
Anselmi-Tamburini U.	69	Brukhsanska D.	147
Antonova M.	83	Brychevskiy M.	112
Antunović M.	115	Brzezińska D.	82
Arivazhagan P.	26	Bučko M.M.	85, 121
Baciu A.	136	Buinevich V.S.	120
Bajac B.	43	Burdin B.	76
Bakan S.	125	Buscaglia M.T.	25
Bakić G.	122	Buscaglia V.	25, 81
Balaban D.	143	Bykov O.I.	150
Balauca S.R.	138	Camargo B.C.	140
Bara M.	75, 84	Camuka H.	24
Baran S.	139	Can Tatlisu G.	103
Bartkowska J.A.	75	Candelario V.	44
Barudžija T.	37, 131	Canikoğlu N.	51
Baščarević Z.	101	Canu G.	25, 81
Bečelić-Tomin M.	146	Cappuyns V.	97
Behara S.	88	Čebela M.	74
Belkessam C.	133, 145	Celebi S.	103
Bellettini G.C.	99	Chambart H.	97
Belous A.	112	Changdong Z.	86
Bernardin A.M.	99, 114	Chen L.	24
Beškoski V.	39	Chiriac V.	129
Bhattacharya S.S.	26	Chiu Y.-L.	105
Binner J.	22, 35, 105	Cieply Ł.	80
Birks E.	83	Ciganović J.	128
Bjelajac A.	132	Ciopec M.	95
Blaskova-Kochnitcharo.	76	Conte G.	45, 47, 48
Blesznowski M.	62	Curecheriu L.	77

Ćurković L.	42, 92	Fleutot B.	106
Cvetnić M.	115	Geiss J.	38, 54
Cvijović-Alagić I.	63	Ghilane J.	133, 145
Cygan R.	128	Gil L.	70
Czaja P.	76, 139	Gilli N.	104
Dal Bó A.G.	114	Glukharev A.G.	52, 110
Dalmacija B.	146	Goncharov E.	61
Damazio M.	99	Gonciarczyk J.	149
Danilović D.	57	Goryczka T.	84
Delcioiu C.	136	Gorynski C.	69
Deluca M.	81, 91	Goulart S.	99
Diblíková P.	68	Gozde Tuncolu I.	87
Dinic I.	54	Gross S.	24
Djelali N.	133, 145	Großmann P.F.	46
Djerdj I.	24	Groszewicz P.B.	81
Djoković V.	57	Grudzień-Rakoczy M.	63
Dojčilović R.	57	Guillén R.	70
Dojcinovic M.	133, 136	Gyergyek S.	37
Dolcet P.	24	Hahn H.	26
Dorey R.A.	122	Hanzel O.	102
Dudek K.	41	Harech M.A.	148
Dulski M.	41	Hasik M.	49
Durand J.-O.	18	Hill R.G.	116
Durand-Panteix O.	42	Hilmas G.E.	104
Duša A.	144	Hinrichsen O.	89
Dusza J.	67, 102	Hříbalová S.	55
Dzik J.	80	Ianăși C.	36,45,47,48,95, 118, 135, 136
Džunuzović A.	90, 137	Idiri N.	133, 145
Efremova A.	93	Ilić N.	90, 137
Eglite L.	83	Ilies S.	36, 136
Einarsrud M.-A.	30	İşlek E.	50
Elaatmani M.	151	Ismail M.M.	127
Elbashar Y.H.	56	Ivanković M.	115
Ellinghaus R.	24	Jagličić Z.	37
Elshourbgy A.	58	Jahanara Mohammad M.	141
Elyseu F.	99, 114	Jakšić M.	132
Engelke L.	69	Janačković Đ.	117, 132
Eraković S.	131	Jankovic-Castvan I.	133
Eray E.	44	Janus G.	147
Ercuța A.	36	Jaramillo Nieves L.J.	99, 114
Erić S.	130	Jastrzębski C.	140
Fachikov L.	76	Jiménez-Pique E.	64
Fahrenholtz W.G.	104	Jones J.L.	79
Fakralmobasheri N.	71	Jugović D.	37
Famprikis T.	106	Kai Z.	86
Feliksik K.	75		

Kaczmarczyk K.	144, 146	Kuskov K.	65
Kaliciak A.	147	Labbilta T.	148
Kaluđerović G.	119	Lach R.	85
Kanas N.	30	Laketić S.	128
Karaimchuk E.S.	152	Łańcucki Ł.	85
Karakaya C.	103	Lazău R.	36
Karolus M.	76	Lefterova E.	76
Karpukhina N.	116	Lelièvre J.	84
Kaszuwara W.	39, 40, 124	Len A.	95
Keles E.	103	Leonelli C.	29
Kerkez Đ.	146	Levish A.	34
Keszler A.M.	105	Ležaja Zebić M.	117
Khalid O.	24	Li C.	24
Kılıç A.	123	Livinsh M.	83
Kitić G.	108	Ljubas D.	92
Klar P.J.	24	Lomakina T.	126
Klaser T.	74	Lu X.-Y.	105
Kljajević L.	130	Lukacs V.A.	79
Kluczewska-Chmielarz	139	Luković J.	122
Knežević N.	28, 119	Lutsyk V.	142
Kocijan M.	92	Lysunenکو N.	112
Kojić S.	144	M. Mladenović	119
Konakov V.	52, 126	Macias G.G.	57
Konakov V.G.	110	Mackert V.	51
Konieczny P.	139	Madej D.	100
Konopka K.	40	Magnacca G.	44
Kónya Z.	46, 64	Mahmoud G.	53
Korim T.	90, 96	Makó É.	97
Korniienko O.A.	150	Maksimović V.	122
Kosiorek M.	62	Mancic L.	54
Kostić D.	143	Manea F.	136
Kostić Lj.	37	Manisa V.K.	122
Kosyanov D.	61	Marchet P.	42, 84
Kotwica Ł.	98	Marijanović I.	115
Kovač S.	101	Markovic S.	136
Kovács A.	97	Martins S.	99
Kozielski L.	20	Maslarević A.	122
Kozień D.	121	Martiz A.	105
Kratzer M.	81	Maslyuk V.A.	152
Krčmar D.	146	Masquelier C.	106
Królicka A.	149	Matić T.	117
Küçüktürk G.	86	Matović B.	39, 122, 130
Kukovecz Á.	20, 46, 64	Matovic J.	43
Kurapova O.	52, 110, 126	McDonald N.	112
Kurt A.O.	125	Mechouet M.	133, 145
Kusiorowski R.	101	Mercier T.L.	106

Merino R.I.	109	Nowak A.	41
Miazga A.	124	Orera A.	109
Michałek J.	144, 146	Orosz-Ábel M.	93
Migliori A.	104	Oufakir A.	151
Miklós M.	90	Over H.	24
Milanovic M.	78	Özdemir B.	51, 125
Milenković I.	39	Özer İ.Ö.	50
Miletić V.	117	Pabst W.	55, 68
Milosavljević A.	57	Padurariu L.	77, 138
Milosevic O.	54	Pahomi A.	129
Mirković M.	122, 130	Pajić D.	74
Mitoseriu L.	77, 79, 138	Panić V.	131
Mitrić M.	37	Pankratova D.	65
Mladenova E.	76	Pantović Pavlović M.	131
Möller M.	24	Papynov E.	61
Momčilović M.	128	Parfenova M.	142
Moscovskikh D.	65	Partyka J.	49, 144, 146
Moskovskikh D.	73, 120	Pasiut K.	144, 146
Moustafa S.H.	58	Pavkov V.	122
Mrówka J.	49	Pavlović M.	131
Múdra E.	102	Pavlovic V.P.	136
Mukasyan A.	65, 73, 120	Pęczkowski P.	139, 140
Murthy T.S.R.C.	35	Pejović B.	143
Mužina K.	92	Pelin Erden S.	87
Muzyk M.	140	Penaranda-Foix F.	70
My Bui H.	89	Perušić M.	143
Nahon L.	57	Petkov P.	68
Naicker L.	122	Petkova T.	68, 76
Nandhini J.U.	26	Petrović R.	101, 117
Narkiewicz U.	60	Pichaniarczyk P.	98
Nascimento de Souza B.	114	Pichór W.	134
Naumovich Y.	111	Picioruș E.-M.	36, 95
Nečina V.	68	Pikula T.	80
Negrea A.	95	Piotrkiewicz P.	39, 124
Nenadović S.	130	Pitir F.	86
Nepapushev A.A.	120	Podunavac I.	108, 144
Nesterovic A.	78	Polat M.	50
Nicola R.	36, 95	Polat Ş.	123
Nicolas C.	57	Policicchio A.	45, 47, 48
Niemiec P.	82	Polishko I.	112
Nikiforov P.	61	Popov M.	81
Nikolic M.	54	Porter M.	105
Nikolic M.V.	133, 136	Poyraz H.B.	50
Nomel I.	42	Prekajski M.	39
Nourmohammadi A.	71	Prorok R.	100
Novokhatska A.	113	Provatas G.	132

Putz A.-M.	36,45,47,48,95	Staciwa P.	60
Qadir A.	67	Stan M.	85
Rađenović D.	146	Stanojev J.	43
Radonić V.	108, 144	Steinberger-Wilckens R.	112
Radotić K.	39	Stelitano S.	45, 47, 48
Radović M.	108	Stec K.	149
Rajczyk K.	147	Stevanović J.	131
Rakić J.	101	Stijepovic I.	78
Rakin M.	128	Stojanović B.	90, 137
Rakoczy Ł.	63	Stojmenović M.	130
Ramachandran H.	141	Suard E.	106
Ramult J.	100	Suchanicz J.	76
Rashwan M.	116	Suvaci E.	87
Rayan D.A.	53, 56, 58, 127	Svera P.	135
Ressler A.	115	Swaminathan P.	141
Rieger B.	46	Szalbot D.	75, 80
Robles A.	109	Szamosvölgyi Á.	46
Rogachev A.	65, 120	Szczytko J.	140
Rosić A.	130	Szenti I.	64
Salamci M.U.	86	Szoldra P.	134
Salvador M.D.	70	Tabak Y.	123
Sameljuk A.V.	150	Tadic N.	133, 136
Sápi A.	46, 91, 93	Tahiri N.	151
Sarkar A.	26	Talskikh K.	61
Savaci U.	87	Taranova A.	65
Schulte J.	38	Tatarko P.	102
Sebők D.	64	Teichert C.	81
Sedegov A.	65	Teke Y.	103
Šenjug P.	74	Temnikova M.	52
Shepa I.	102	Thomas T.	88
Shichalin O.	61	Tingting L.	86
Sibera D.	60	Tomašević Pilipović D.	146
Siketić Z.	132	Torić F.	74
Silvestroni L.	27, 104	Tovar-Vargas D.	64
Simão F.	97	Troll C.	46
Skawińska A.	98	Trusov G.V.	120
Skoko Ž.	74	Tsybulin V.	65
Skulski R.	82	Turan S.	87, 103
Slater P.R.	109	Turcan I.	77
Slijepčević N.	146	Turón-Viñas M.	64
Smarsly B.	24	Tyrała K.	100
Śnieżek L.	40	Uhlířová T.	68
Šoškić A.G.	94	Ulaş Kudu Ö.	106
Souza M.	99	Ünsal H.	102, 123
Spitaler J.	81	Ur Rehman A.	86
Srdic V.V.	43, 78	Urban S.	24

Uzelac M.M.	94	Żurawska A.	62, 111
Vandemeulebroecke L.	97	Zygmuntowicz J.	39, 40, 124
Varchenko V.T.	152		
Vásárhelyi L.	64		
Vasiljevic Z. Z.	133, 136		
Vasylyev O.	112		
Vatansever B.K.	123		
Veerapandiyam V.	81, 90		
Velasco L.	26		
Veljović Đ.	117, 128		
Venkatachalam V.	35		
Vidić J.	108		
Vijatović Petrović M.	90, 137		
Vilarinho P.M.	22, 28		
Vladikova D.	76		
Vlase G.	129		
Vlase T.	129		
Vodnik V.	57		
Vorotylo S.	65		
Vujancevic J.	133, 136		
Vujančević J.	132		
Vukmirovic J.	78		
Vukovic M.	54		
Vukšić M.	42		
Vulic P.	54		
Wachowski M.	40, 124		
Watts J.	104		
Wenhe L.	86		
Whiting M.J.	122		
Wiik K.	30		
Williams T.	122		
Winterer M.	19,34,38,51,54, 69		
Wodecka-Duś B.	84		
Wojciechowski K.	85		
Wolf D.E.	69		
Yurchenko Y.V.	150		
Zabot E.	99		
Zachariasz P.	139, 140		
Zagorac D.	74		
Zavjalov A.	61		
Zayed M.A.	53		
Zilli L.	99		
Živković L.	39		
Žmak I.	42		
Zou J.	35, 105		

NOTES

NOTES

NOTES

NOTES

NOTES

NOTES