DEPARTMENT FOR ADVANCED MATERIALS K-9

The main activities of the department encompass basic and applied research within the fields of energy materials, biomaterials and electronic materials. Among the important objectives are the development of new, efficient oxides for high-temperature thermoelectric energy conversion, materials with improved antibacterial and photocatalytic effects and the development of thin films and nanostructured powders of functional oxides for various electronic applications.

Head:
Prof. Danilo Suvorov

Thermoelectric oxides

Continued research on thermoelectric layered cobaltates $Ca_{3-x}Na_xCo_4O_9$ showed that intergrown structures exhibit a Seebeck coefficient higher than both the end members, Na_xCoO_2 and $Ca_3Co_4O_9$. With an increase in x the electrical conductivity can be tailored from semiconducting to metallic behaviour, which enables control of the power factor of the material. The highest measured power factor was at compositions with x ~ 1, which also exhibit environmental stability as opposed to pure Na_xCoO_2. In the scope of the industrial project with Epcos, a part of TDK-EPC company, we performed a study of the development of intergrown structures and their influence on the texturing and, consequently, the electrical conductivity. We found that the formation of an intergrown structure prior to the sintering stage crucially influences the microstructure and, consequently, the electrical conductivity. We have also found that intergrown structures are stable in an air atmosphere up to 930°C, above which a reversible loss of oxygen takes place, resulting in the formation of a secondary phase CoO. In an inert atmosphere the reversible decomposition takes place already at temperatures below 500°C. The findings indicate that based on coherently intergrown structures $Ca_{3-x}Na_xCo_4O_9$ high-temperature p-type thermoelectric materials with an operating range up to ~900°C can be synthesized. At 700°C ceramics with the nominal composition $Ca_{2.2}Na_{0.8}Co_4O_9$ exhibit a figure of merit zT~0.3. The results obtained so far also indicate that a further improvement of the conversion efficiency is possible by reducing thermal conductivity and so controlling the atmosphere during processing and thus influencing the formation of nano-inclusions within the coherently intergrown matrix.

Antibacterial and photocatalytic materials

Nanocomposite materials based on TiO_2 and Pt particles were prepared via two syntheses, i.e., hydrothermal synthesis and sonochemical synthesis, followed by thermal treatment in a reducing atmosphere at 400°C for 3 h. The hydrothermally synthesized TiO_2/Pt consisted of TiO_2 (average particles size 16 nm) in the anatase and the rutile crystal modification. TiO_2/Pt prepared by the sonochemical synthesis crystallized as the TiO_2 (average particle size 9 nm) anatase and brookite. The hydrothermal synthesis enabled the formation of two types of Pt particles: the Pt particles (12-17 nm) that were attached on the TiO_2 surface and the Pt particles that existed as an isolated phase (up to 45 nm). In the case of the sonochemical synthesis the Pt particles (up to 5 nm) formed a composite with the TiO_2 particles. The XPS surface analysis of the prepared TiO_2/Pt revealed that the formed Pt existed as Pt^0 and Pt^{2+}. The presence of the free hydroxyl groups was identified utilizing the FTIR spectroscopy. The free surface hydroxyl groups were detected only in the case of the hydrothermally synthesized TiO_2/Pt. The photocatalytic activity was

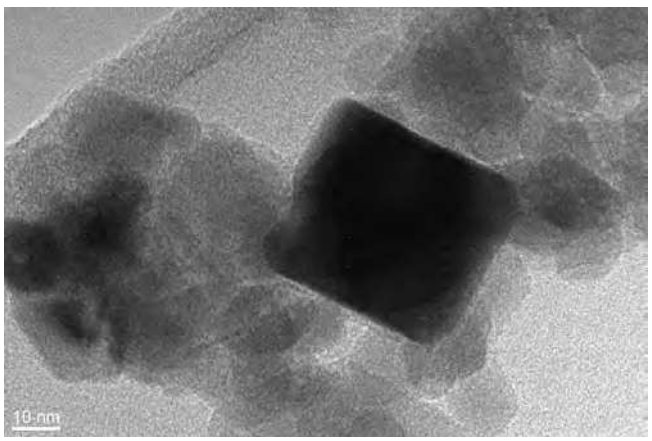

Figure 1: Hydrothermally synthesized TiO_2/Pt.

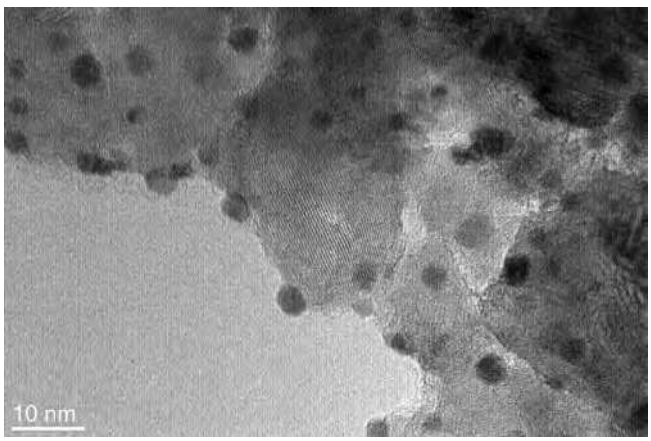

Figure 2: Sonochemical synthesized TiO_2/Pt.

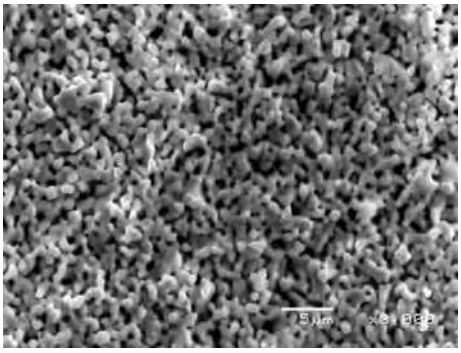

Figure 3: SEM image of BSO thin films deposited on $Si/SiO_2/TiO_2/Pt$ substrate annealed at 700°C/1h.

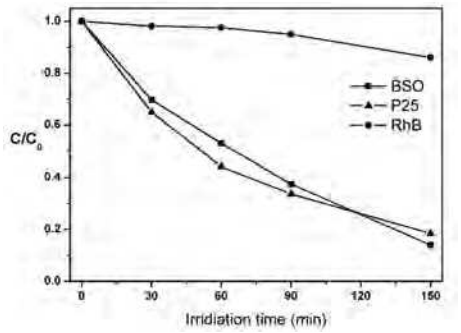

Figure 4: The degradation rate of the RhB solution under UV irradiation with the photocatalyst BSO film and P25 film

determined by monitoring the degradation of the aqueous solution of the azo dye methylene blue under UV and Vis irradiation. The measurements revealed that the UV photocatalytic activity of the hydrothermally synthesized TiO_2/Pt was 16-times higher than the activity of the sonochemically synthesized TiO_2/Pt. Under Vis irradiation the photocatalytic performance of the hydrothermally synthesized TiO_2/Pt exceeded the activity of the sonochemically prepared TiO_2/Pt by 5-times. The higher photocatalytic efficiency of the hydrothermally synthesized TiO_2/Pt was ascribed to the presence of the free surface hydroxyl groups. Such hydroxyl groups form with the photogenerated holes from the TiO_2 valence band strong hydroxyl radicals, which degraded the organic compounds adsorbed on the surface of the TiO_2.

Photocatalytic films based on the sillenite compound $Bi_{12}SiO_{20}$ (BSO) were prepared using the polymerizable complex method. The obtained BSO films had a porous microstructure with an average size of the grains equal to 1μm. Its photocatalytic activity was evaluated with the degradation of the aqueous organic pollutant Rhodamine B under UV-light irradiation. The $Bi_{12}SiO_{20}$ films showed comparable photocatalytic performance in the degradation of the RhB solution under UV-light irradiation as the reference Degussa P25 film. Namely, both photocatalytic films BSO and P25 degraded over 82% of the RhB solution within the irritation time of 150 min.

In 2013 we also focused on the syntheses of new antibacterial materials that include a gallium component. This should increase the efficiency of the functionalized gold nanoparticles on hydroxyapatite, which have recently been developed at this department, but avoid the increase of their harmfulness towards eukaryotic (human) cells. Using a sonochemical method we successfully prepared nanocomposites of gold nanoparticles, functionalized with amino acids, and hydroxyapatite with Ga(III) ions, most probably in its interstitials. All the nanoparticles in the composite are spherical, of very similar size (with a diameter of approx. 5 nm with histidine, approx. 10 nm with arginine and approx. 20 nm with glycine) and well separated on the hydroxyapatite nanorods. We also managed to obtain a composite of hydroxyapatite with spherical gallium nanoparticles, covered with GaOOH. A gold nanoshell with different amino acids attached is being added to these nanoparticles, in order to obtain functionalized core-shell nanodevices for targeted drug delivery of an antibacterial (gallium) core.

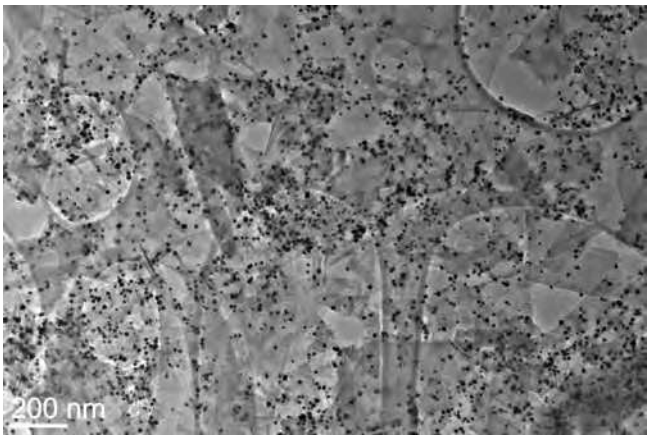

Figure 5: Nanocomposite of arginine-functionalized Au nanoparticles and Ga(III)-containing hydroxyapatite under a transmission electron microscope.

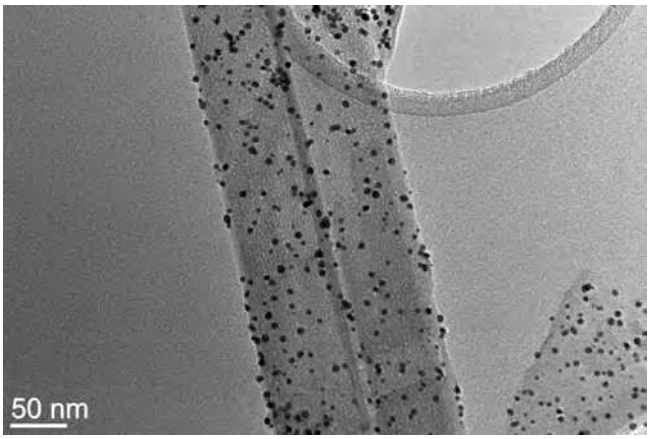

Figure 6: TEM picture of histidine-functionalized Au nanoparticles on hydroxyapatite nanorods with incorporated Ga(III) ions

Functional oxides for electronic applications

In the field of the investigation of phase relations in ternary oxide systems where new compounds and/or solid solutions are stable and exhibit pronounced electric properties, we determined phase relations in the ternary systems La_2O_3-TiO_2-CaO at 1400°C in previous research. This year we continued with investigations and we determined the crystallographic structure of the solid solution $CaTiO_3$-$Ca_3La_4Ti_3O_{15}$, which is stable along the whole tie line. We described a transition from one crystal structure into another and determined the microstructural and dielectric properties of these ceramics. For the synthesis of the single-phase ceramics a modified Pechini method was used.

We have also investigated ceramics based on the compound $CaCu_3Ti_4O_{12}$, which exhibit interesting dielectric properties and ceramics based on the solid solution $CaCu_3Ti_4O_{12}$- $CaCu_3Ru_4O_{12}$.

The research of $BaTiO_3$ particle formation was focused on (i) the topotactic transformation of various titanate precursors into $BaTiO_3$, (ii) the characterization of tetragonal $BaTiO_3$ particles, prepared under moderate hydrothermal conditions, (iii) the sintering of variously shaped $BaTiO_3$ particles and a determination of the dielectric properties. The aim of the first part was to explore possibilities for the preparation of defined shaped $BaTiO_3$ particles by preserving the shape of the titanate precursor. This kind of transformation is possible in liquid phase (hydrothermal and molten salt synthesis) under conditions that favour the epitaxial growth of $BaTiO_3$ on a titanate precursor. These conditions include a low lattice mismatch and a low density of surface defects. The comparison of the $BaTiO_3$ lattice parameters with those of the titanate precursors ($Na_2Ti_3O_7$ (NT) belts, $K_2Ti_6O_{13}$ (KT) wires, $K_{1.33}Li_{1.33}Ti_{3.33}O_8$ (KLT) plates) showed a mismatch greater than 4%. Epitaxial growth could occur, when the high strains are relieved by the formation of dislocations and grain boundaries. Our investigations revealed that the topochemical transformation of the titanate precursor into $BaTiO_3$ was better approached by the NT belts than by the KLT plates and KT wires. The morphology of the $BaTiO_3$ particles formed from the last precursor at low temperature (80-100°C) was found to be very similar to that obtained with the NT belts. The differences appeared at T≥150°C, where $BaTiO_3$ with a considerably higher degree of tetragonality was formed from KT compared to that from the other precursors. Raman spectroscopy, which gave information about the local structure, showed that the asymmetry within [TiO_6] the octahedra was present already in BaTiO3, prepared at 100°C. According to the result of this technique, $BaTiO_3$ with a high degree of tetragonality formed from KT at 150≤T≤240°C. The differences in tetragonality for $BaTiO_3$ formed at different high-temperature (150≤T≤240°C) conditions could be better detected by XRD and DSC, because they gave information about long-range order. In XRD pattern, the tetragonality is evident from the splitting of the (200) diffraction line, while DSC gave the enthalpy of tetragonal to cubic phase transition. We found that tetragonality increased with an increase of the temperature and synthesis time. When the NaOH content exceeded the concentration needed for $BaTiO_3$ formation, the tetragonality decreased with an increase of the NaOH content.

The sintering studies of variously shaped (star- and square-like) cubic $BaTiO_3$ particles revealed that particle shape had an important influence on the grain growth and phase transformation. The sintering of square-like particles led to tetragonal ferroelectric and coarse grained (2-10 μm) $BaTiO_3$ ceramics, while star-like particles preserved the cubic crystal structure without a significant increase in the grain size (1 μm). High relative densities of 98% were achieved by two-step sintering, which is regarded as a promising method for controlling the grain growth. Based on this fact we assume that dielectric, ferroelectric and piezoelectric properties of $BaTiO_3$ ceramics could be tailored by the selection of the particle shape and the proper sintering conditions.

We have investigated the synthesis of $Ag(Nb_xTa_{1-x})O_3$ (x = 0.2-1) ceramics by a solid-state reaction method. Pure-phase ceramics with a relative density higher than 96% can be obtained, except for x = 0.2. As x decreases from 1, the dielectric constant at 2.9-4.4 GHz first increases from 222, reaches the maximum value of 491 for x = 0.65, and then decreases to 206 for x = 0.2. While the Q×f value increases monotonically from 72 GHz for x = 1 to 1.550 GHz for x = 0.2, although the ceramic with x = 0.2 is relatively porous and not single phase. The $Ag(Nb_xTa_{1-x})O_3$ (x = 0.5-1) ceramics show interesting tunable dielectric properties. When x = 1, unique "W"-shaped dependences of dielectric constant and dielectric loss on DC bias are observed, indicating the coexistence of ferroelectricity and anti-ferroelectricity. Similar results are observed for x = 0.8 and 0.65, while only anti-ferroelectricity is indicated for x = 0.5. The anti-ferroelectricity can be observed based on a tunability measurement with the maximum DC of 125 kV/cm or even lower. However, much higher electric field of 175 kV/cm is needed for observing an anti-ferroelectric-like hysteresis loop. It is indicated that the tunability measurement is a more sensitive tool than the hysteresis loop for determining the anti-ferroelectricity.

$Ag(Nb_{0.5}Ta_{0.5})O_3$ thin films have also been deposited on (0001) Al_2O_3 single-crystal substrates by pulsed laser deposition with a fluence of 1.5 J/cm² and an oxygen pressure of 0.1 mbar, and they are characterized by XRD and RHEED. When the target-to-substrate distance is 55 mm, the repetition rate is 5Hz and the deposition time is 1 hour, polycrystalline $Ag(Nb_{0.5}Ta_{0.5})O_3$ primary phase is indicated from XRD for the deposition temperatures of 550-625°C. However, a small concentration of secondary phase can also be

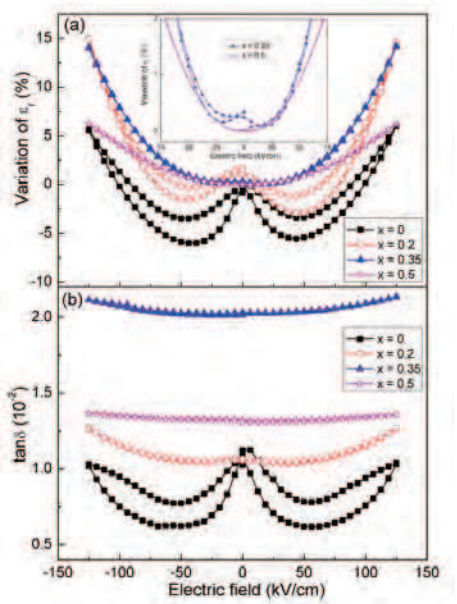

Figure 7: Tunable dielectric properties for $Ag(Nb_xTa_{1-x})O_3$ ($x = 0.5$-1) ceramics with "W"-shaped dependences of dielectric constant and dielectric loss on DC bias, indicating the coexistence of ferroelectricity and anti-ferroelectricity.

Figure 8: Plume created during laser ablation of $Ag(Nb_{0.5}Ta_{0.5})O_3$ ceramic target at 1.5 J/cm² laser energy and 0.1 mbar oxygen partial pressure in pulsed laser deposition system.

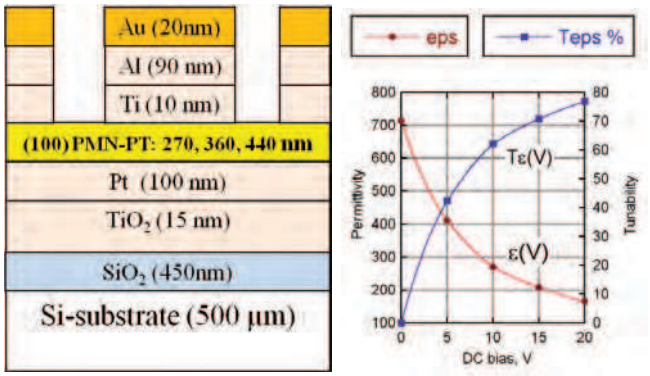

Figure 9: Cross-sectional view of the test resonators (a) and DC bias dependences of permittivity (ε(V)) and relative tunability of permittivity (Tε(V)), measured at 3 GHz, of a test structure with the 440-nm-thick PMN-PT film (b).

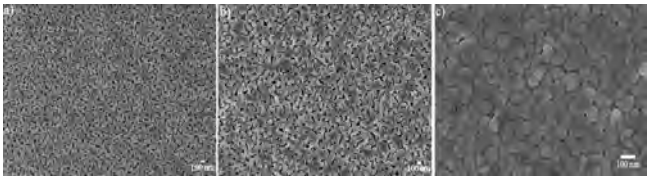

Figure 10: Microstructural development of PMN-PT thin films pyrolysed at temperature a) 200°C, b) 300°C in c) 430°C and annealed at 650°C/20 min.

In the scope of the Center of Excellence in Nanoscience and Nanotechnology we installed the Empyrean X-ray diffractometer at the Advanced Materials Department, which is utilized mainly for the detailed structural characterization of single-crystal thin films. The delivered system enables the study of the epitaxial layers in terms of reciprocal space-map measurements and a determination of epitaxial relationship with respect to the substrate, while the reflectivity measurements provide us information about the thickness, surface/interface roughness and density of single or multilayered structures on a substrate. The above-mentioned applications of the system are enabled by a sample stage with five computer-controlled and programmable axes and very powerful 2D area detector, capable of measuring in the 0D mode (standard point detector), 1D mode (fast linear detector) and 2D mode (area detector). In addition to a structural characterization of thin films, standard powder-diffraction measurements can also be performed, optionally with a diffracted beam monochromator for sample fluorescence removal. Furthermore, the system is equipped with high/low-temperature sample stages, which make it possible to measure samples over a broad temperature range from -261°C to 1200°C and in different gas atmospheres.

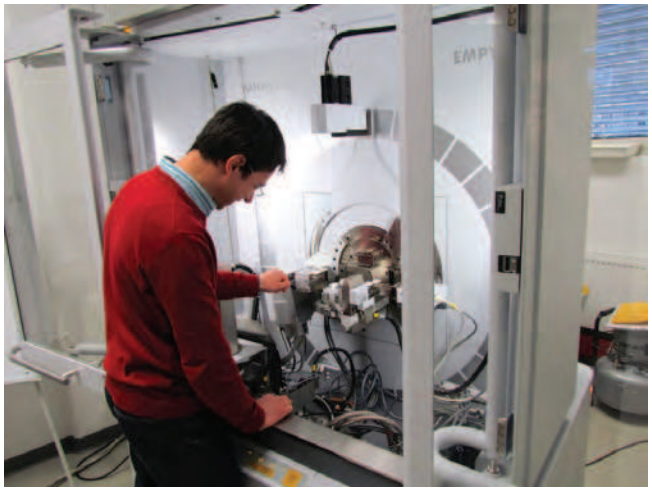

Figure 11: Empyrean X-ray diffraction system

observed in the patterns. When the target-substrate distance increases to 60 mm, a pure-phase $Ag(Nb_{0.5}Ta_{0.5})O_3$ thin film is formed with the thickness of 150 nm, which nevertheless should be doubled for measuring the microwave dielectric properties. When the deposition time increases to 2 hours, a small concentration of $Ag_2(Nb_{0.5}Ta_{0.5})_4O_{21}$ secondary phase appears due to the decomposition of $Ag(Nb_{0.5}Ta_{0.5})O_3$ after long-time deposition. In the final part of the work, the repetition rate increases to 10 Hz and the deposition time is set at 1 hour to reduce the decomposition of $Ag(Nb_{0.5}Ta_{0.5})O_3$. In this way, a pure-phase polycrystalline $Ag(Nb_{0.5}Ta_{0.5})O_3$ thin film with a thickness of 300 nm is obtained.

In the scope of NAFERBIO project we investigated the synthesis of PMN-PT thin films. In short, we systematically varied the conditions of the reagents in order to determine the influence of the coordination chemistry on the formation of the perovskite phase. Results revealed that the major contribution to the formation of single-phase perovskite PMN-PT thin films comes from the coordination of the Pb reagent. A pyrochlore-free PMN-PT film with (100) orientation was formed when the steric hindrance of the Pb precursor was increased by using polyvinylpyrrolidone (PVP). The (111) orientated PMN-PT thin films were prepared using sol-gel-derived TiO_2 as a nucleation layer. Our research was further focused on the influence of the pyrolysis treatment on the phase formation and microstructural development of PMN-PT thin films. The XRD results showed that a different pyrolysis treatment has no influence on the phase formation of thin films. In contrast, the microstructural development of the PMN-PT thin films strongly depends on the used pyrolysis temperature.

Based on PMN-PT films with (100) orientation FBAR devices were subsequently fabricated and tested. Films were deposited on platinized silicon substrates. More than 4% tuning of resonance frequency under DC field less than 15 V/μm was demonstrated. In comparison with the $Ba_xSr_{1-x}TiO_3$ based FBARs, this tunability was achieved at 2-3 times lower applied DC field. The other advantage of PMN-PT is the high electromechanical coupling coefficient that allows for development of wide-band tunable filters. Even though FBARs with this performance may be used in microwave circuits, the achieved tunability is not as high as we would anticipate from the large electrostriction coefficient of PMN-PT reported in the literature. In these experiments the lower electrostriction coefficient and lower tunability are due to the lower density of the PMN-PT films, which is the subject of forthcoming studies. An additional reduction of the tunability is due to large negative nonlinear electrostriction coefficient.

In addition, a part of our activities, in collaboration with an industrial partner, were focused on the development of aluminum foams. For such foams TiH_2 is used as a foaming agent. As an alternative forming agent we proposed in the past the application of dolomite. A drawback of the dolomite is its high decomposition temperature, which is above 820°C. In order to decrease its decomposition temperature we mechanically and chemically treated the dolomite and such products start to decompose at 400°C.

Within the cooperation with industrial partner Knauf Insulation d.o.o, the research work on the joint project was focused on the morphology and chemical composition of mineral fibres and their composites, crystallization and melting behaviour, aging process, determination of specific heat and thermal stability of mineral wools.

Organization of Conferences, Congresses and Meetings

1. Workshop on MATERA ERA-NET project "Novel inorganic inks for hybrid printed electronic demonstrators", Ljubljana, 16. 10. – 17. 10.2013.

2. Materials Science & Technology 2013 Conference and Exhibition, Montreal, Canada, 27. 10. – 31. 10. 2013 (co-organizers).
3. 21st Conference on Materials and Technologies, Portorož, 13. 11. – 15. 11. 2013 (co-organizers).
4. Institue of Science and Technology for Ceramics, Faenza, Italy and Institut "Jožef Stefan" Workshop on Materials, Ljubljana, 11. 12. – 12. 12. 2013.

Awards and Appointments

1. Aničić Nemanja: Award of the Henkel Slovenia Foundation for B. Sc. Thesis, Faculty of Chemistry and Chemical Engineering, University of Maribor, Maribor, "Application of the population balance model for the prediction of concentrated emulsion droplet size distribution".

Patent granted

1. Marija Vukomanović, Srečo D. Škapin, Danilo Suvorov, Composites materials based on ceramic phase and metal with functionalized surface as environmentally-friendly materials with antibacterial activity, a process for preparing and use thereof, SI24094 (A), Urad RS za intelektualno lastnino, 31.12.2013.

Figure 12: New laboratory for antibacterial materials analysis

We established a new laboratory for the analysis of antibacterial materials, where we will be able to grow bacterial and mammalian cells and examine their survival after being exposed to the prepared materials as well as their interactions with these materials.

INTERNATIONAL PROJECTS

1. Thermoelectric Oxide Materials
   EPCOS OHG Ceramic Components Division
   Prof. Danilo Suvorov
2. Microwave Tunable Materials, Composites and Devices
   NATO - North Atlantic Treaty Organisation
   Asst. Prof. Boštjan Jančar
3. The Synthesis of Dielectric Materials by Chemical Solution Deposition and Characterization of their Dielectric Properties
   Slovenian Research Agency
   Prof. Danilo Suvorov
4. Nanostructural Designing of Multifunctional and Sintered Electrical and Biological Functionally Graded Materials
   Slovenian Research Agency
   Asst. Prof. Srečo Davor Škapin
5. Multifunctional Ferroelectric Materials based on $Ag(Nb,Ta)O_3$
   Slovenian Research Agency
   Prof. Danilo Suvorov

RESEARCH PROGRAM

1. Contemporary Inorganic Materials and Nanotechnologies
   Prof. Danilo Suvorov

R&D GRANTS AND CONTRACTS

1. Nanoengineering of Self-Assembled Materials
   Prof. Danilo Suvorov
2. New Materials for Power Conversion: Oxide Semiconductor Thermoelectrics
3. Prof. Danilo Suvorov
4. INNOINKS: Novel Inorganic Inks for Hybrid Printed Electronic Demonstrators
   Prof. Danilo Suvorov
5. NAFERBIO: Nanostructured Ferroelectric Films for Biosensor
6. Prof. Danilo Suvorov

NEW CONTRACTS

1. Development and Characterisation of Mineral Wool Fibres
   Knauf Insulation, d. o. o., Škofja Loka
   Prof. Danilo Suvorov
2. New Materials for Energy Conversion: Oxide Semiconducting Thermoelectrics
   Gorenje Household Appliances, d. d.
   Prof. Danilo Suvorov

VISITORS FROM ABROAD

1. Hermann Gruenbichler, B. Sc., Dr. Manfred Schweinzger, Dr. Yongli Wang, TDK EPCOS, Deutschlandsberg, Austria, 6. 3. 2013.
2. Dr. Markus Mente, Gorazd Šebenik, B. Sc., Borut Vezočnik, B. Sc., Knauf Insulation, Škofja Loka, 9. 5. 2013.
3. Prof. Dr. Dragoljub Uskoković, Institut of Tecnical sciences of the Serbian Academy of Sciences and Arts, Belgrade, Serbia, 16. 7. – 17. 7. 2013.
4. Prof. dr. Ivan Sondi, Faculty of mining, geology and petroleum engineering, University of Zagreb, Zagreb, Croatia, 9. 4. 2013.
5. Dr. Smilja Marković, Institut of Tecnical sciences of the Serbian Academy of Sciences and Arts, Belgrade, Serbia, 7. 6. – 19. 6. 2013.
6. Dr. Jyoti Prosad Guha, Missoury University of Science and Technology, Rolla, USA, 10. 6. – 14. 8. 2013.
7. Dr. Smilja Marković, Institut of Tecnical sciences of the Serbian Academy of Sciences and Arts, Belgrade, Serbia, 5. 8. – 14. 8. 2013.
8. Prof. Dr. Suk-Joong L. Kang, Korea Advanced Institute of Science and Technology, Daejeon, South Korea, 28. 8. - 30. 8. 2013.
9. Dr. Hiroyuki Enomoto, Dr. Kesaku Sonoda, Research Laboratories of NOF Corporation, Tsukuba, Japan, 16. 10. – 17. 10. 2013.
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15. Dr. Damir Dominko, Dr. Damir Staresinić, Institute of Physics, Zagreb, Croatia, 19. 12. 2013.
16. Dr. Maja Đekić, Amra Salčinović, Faculty of natural sciences and mathematics, University of Sarajevo, Sarajevo, Bosnia and Herzegovina, 19. 12. 2013.

Visiting researchers

1. Dr. Ismael Fabregas, Centro de Investigaciones en Sólidos, CITEFA, Buenos Aires, Argentina, 1. 1. 2013 – 31. 8. 2013.
2. Dr. Zoran Jovanović, Faculty of Physical Chemistry, University of Belgrade, Belgrade, Serbia, 1. 1. 2013 – 31. 12. 2013.
3. dr. Lei Li, Zhejiang University, Hangzhou, China, 1. 1. 2013 – 31. 12. 2013

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4. Dr. Matjaž Spreitzer
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17. Andreja Šestan, B. Sc.
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21. Maja Šimaga, M. Sc.
22. Silvo Zupančič

BIBLIOGRAPHY

Original Article

1. Gabriela Ambrožič, Jernej Šribar, Srečo D. Škapin, Majda Žigon, Zorica Crnjak Orel, "An antibacterial macroporous polyurethane hybrid material with a high content of zinc ions: a template to uniform ZnO nanoparticles", *Mater. res. bull.*, vol. 48, no. 4, pp. 1428-1434, 2013.
2. In-Sun Cho, Chi Hwan Lee, Yunzhe Feng, Manca Logar, Pratap M. Rao, Lili Cai, Dong Rip Kim, Robert Sinclair, X. Zheng, "Codoping titanium dioxide nanowires with tungsten and carbon for enhanced photoelectrochemical performance", *Nature communications*, vol. 4, no. 3, pp. 1723-1-1723-8, 2013.
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