

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 electronic oxides for various applications.

Thermoelectric oxides

The research of oxides as possible thermoelectric materials was triggered by the discovery that metallic layered cobaltate Na_xCoO_2 exhibits a large Seebeck coefficient combined with a high electrical conductivity and a low thermal conductivity, which was attributed to its layered crystal structure consisting of two-dimensional sheets of edge-sharing CoO_6 octahedra intercalated by Na ions. The highest reported zT values of Na_xCoO_2 are ~ 1.0 for single-crystalline and ~ 0.8 for polycrystalline material at temperatures in the vicinity of 800°C . With such properties it was considered to be a good candidate for the high-temperature p-type thermoelectric material. However, the chemistry of layered sodium cobaltates is governed by the high mobility of interlayer sodium, which reacts with atmospheric moisture and carbon dioxide. Furthermore, the layered crystal structure of Na_xCoO_2 enables the intercalation of molecules such as water, which can lead to exfoliation and thus degradation of the material. Because of this the focus of the research turned to a semiconducting misfit-layered cobaltate $\text{Ca}_3\text{Co}_4\text{O}_9$, the structure of which consists of triple Ca_2CoO_3 layers and single layers of CoO_2 analogous to CoO_6 sheets of Na_xCoO_2 compounds. The highest zT reported for this structural type was ~ 0.6 . We found that the sheets of octahedrally coordinated Co ions, which are the common structural element of the Na_xCoO_2 and $\text{Ca}_3\text{Co}_4\text{O}_9$ phases, allow the spontaneous intergrowth of the two structures (Fig. 1) leading to a significant improvement of environmental stability. Furthermore, the coherent intergrowth of the two structural types results in effective texturing in polycrystalline material with the preferred grain growth aligned in-plane with common CoO_6 layers, thus allowing high electrical conductivity. The nanostructured intergrowths also result in a significant reduction of the thermal conductivity, which was at 700°C measured to be ~ 0.3 W/mK for the “out-of plane” and ~ 0.6 W/mK for the “in-plane” direction. With the measured power factor of $\sim 6.5 \cdot 10^{-4}$ W/mK² the calculated “in-plane” zT of the intergrowth structure material with the nominal composition $\text{Ca}_{2.2}\text{Na}_{0.8}\text{Co}_4\text{O}_9$ is ~ 1.0 at 700°C , which is higher than any so-far reported value for oxide thermoelectrics.

Antibacterial and photocatalytic materials

An innovative concept has been applied to develop human and environmentally friendly material with antibacterial properties. The material is a composite formed of bioceramic, metallic and organic phases that contain amino and thiol groups. Metallic nanoparticles have a functionalized surface and they are carriers of antibacterial activity. The efficacy of the antibacterial action of the composites depends on the type of surface functionalization that provides activity against Gram-positive and Gram-negative bacteria. The materials were prepared by a sonochemical method that was developed as a novel route for the synthesis of this type of material and belongs to green chemistry. The developed materials have a stronger antibacterial activity in comparison to silver-based materials that are frequently used in practice, which indicates the possibility of their replacement by novel, more effective and safer materials developed in our laboratory. The morphological properties of the newly developed material and its antibacterial activity are shown in Fig. 2.



Head:
Prof. Danilo Suvorov

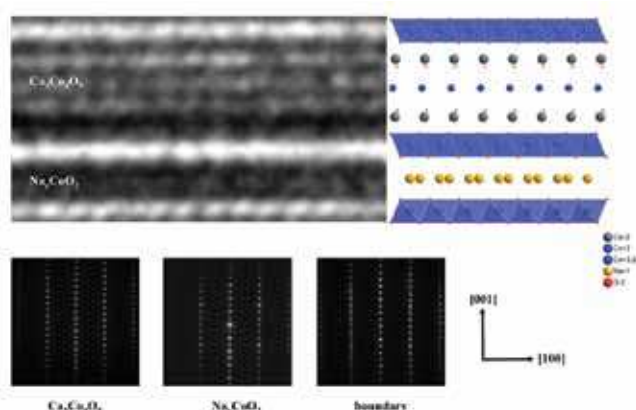


Figure 1: HRTEM image and SAED pattern of coherently intergrown Na_xCoO_2 and $\text{Ca}_3\text{Co}_4\text{O}_9$ structure types.

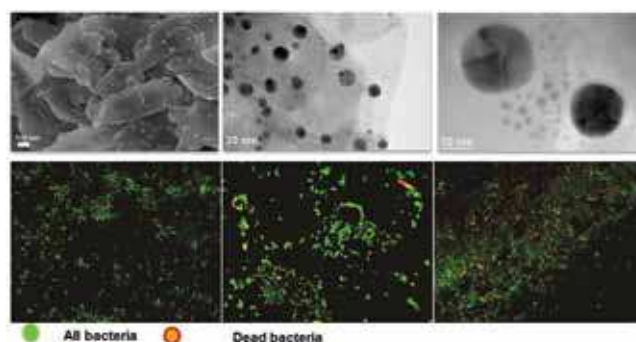


Figure 2: Morphological properties of a novel antibacterial material and its antibacterial effect on *E. coli*, developed at the Advanced Materials Department.

In the field of photocatalytic materials, hierarchically assembled nanostructured spherical anatase particles in the size range from 3 to 7 nm, using a solvothermal synthesis method, were prepared. The spherical particles are composed of nanosized primary crystallites with a size below 30 nm. The prepared anatase exhibits good photocatalytic properties and a high temperature stability, over 1000°C. Due to the large particle size, these particles are less harmful, compared to nanosized commercial anatase.

Functional electronic oxides

In the scope of the European project "Nanostructured Ferroelectric Films for Biosensors" we studied the synthesis and characterization of $\text{Pb}(\text{Mg}_{1/3}/\text{Nb}_{2/3})\text{O}_3$ - PbTiO_3 thin films prepared on $\text{Pt}(111)/\text{TiO}_2/\text{SiO}_2/\text{Si}$ substrates using the sol-gel method for Film Bulk Acoustic Resonator (FBAR) sensor applications. In order to determine the influence

In collaboration with Epcos OHG, a member of the TDK-EPC Corporation, we developed a p-type thermoelectric oxide with $zT \sim 1.0$ at 700°C, which makes it superior to so-far reported polycrystalline thermoelectric oxides. The invention is EU patent pending.

of the coordination chemistry on the formation of the perovskite the conditions of the reagents were systematically varied. As a source of Mg-precursor $\text{Mg}(\text{CH}_3\text{COO})_2 \times 4\text{H}_2\text{O}$, $\text{Mg}(\text{AcAc})_2 \times 2\text{H}_2\text{O}$ and $\text{Mg}(\text{NO}_3)_2 \times 6\text{H}_2\text{O}$ were applied to reduce the concentration of the undesired pyrochlore phase that forms in addition to the perovskite phase. $\text{Pb}(\text{NO}_3)_2$, $\text{Pb}(\text{CH}_3\text{COO})_2$, $\text{Pb}(\text{PVP})_2$ and $\text{Pb}(\text{AcAc})_2$ were used as a source of Pb. Changing the coordination sphere of Mg does not increase the reactivity of Mg ions towards Nb to the point that they will preferentially react, forming the Mg-O-Nb heterometallic structure.

A pyrochlore-free $\text{Pb}(\text{Mg}_{1/3}/\text{Nb}_{2/3})\text{O}_3$ - PbTiO_3 film was formed when the steric hindrance of the Pb precursor was increased. In this way the reactivity of Pb ions towards Nb ions is decreased, resulting in the formation of Mg-O-Nb heterometallic clusters, leading to the formation of a perovskite phase. Thus, $\text{Pb}(\text{PVP})_2$ and $\text{Pb}(\text{AcAc})_2$ were shown to be effective in the formation of pyrochlore-free thin films.

It was observed that during the direct casting of the film on the $\text{Pt}(111)/\text{TiO}_2/\text{SiO}_2/\text{Si}$ substrate the films grow into a dense and crack-free microstructure. The relative permittivity and dielectric losses for the pyrochlore-free PMN-PT thin film were found to be 1650 and 0.12, respectively.

We further investigated the tunable properties of $(1-x)\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ - $x\text{NaTaO}_3$ thin films ($0.05 < x < 0.3$) prepared by a modified sol-gel method. A dense and homogenous microstructure, with the average grain size ranging between 70 and 110 nm, was obtained for the NBT-NTa thin films by using a Bi-propionate precursor in the sol-gel synthesis. It was observed that the dielectric permittivity increases with the annealing temperature for all the prepared NBT-NTa thin-film compositions. The decrease in the average grain size below 150 nm caused the appearance of

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single-domain grains, which then strongly affected the polarization behavior of the 5NTa thin films, giving them a relaxor-type response. The relaxor-type and paraelectric-type responses were observed for the 10NTa and 30NTa thin films, respectively. Comparable dielectric permittivity and relative tunability values were obtained for the 5NTa ($\epsilon = 441$, $n_r = 42\%$) and the 10NTa ($\epsilon = 440$, $n_r = 40\%$) thin films, whereas the 30NTa thin films showed lower values ($\epsilon = 370$, $n_r = 23\%$).

In the field of ferroelectric thin films, research is driven by demands to improve and tailor the inherent material's electrical response. The motivation behind our investigation was directed towards tuning the temperature-dependent dielectric behavior and improving the dielectric loss characteristics. In this attempt, we designed $\text{SrTiO}_3/\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3/\text{SrTiO}_3$ (ST/NBT/ST) structured thin films, where the ferroelectric NBT phase is embedded within a low-dielectric-loss ST component. Using repeated thermal treatment we managed to prepare thin films with a temperature flat dielectric permittivity (t_e of 780 ppm/K in the temperature range between -50°C and 200°C), decreased dielectric losses, and a frequency undispersed response at room temperature. The obtained results were attributed to the compositional gradient between particulate ST and NBT layers, as determined by X-ray diffraction and X-ray photoelectron spectroscopy. As-structured thin films exhibit promising properties for functional devices that are required to produce stable performance in a broad temperature range.

Beyond sol-gel techniques we utilized diblock-copolymers (BCs) to synthesize multifunctional thin films. Due to the different chemical nature of the separate blocks they self-organize into different phases, where the type of the phase depends on the block fraction ratio, the monomer chemical nature, the temperature, etc. In our work we take advantage of a polystyrene-polyethylene oxide (PS-b-PEO) block-copolymer to obtain $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ (NBT) / SrTiO_3 (ST) or $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ - PbTiO_3 (PMN-PT) / CoFe_2O_4 (CF) multifunctional thin films with in-plane interfaces. Using a different polarity of the precursors or specific casting procedures we can selectively direct inorganic components into the PS or PEO block. In the first part of the study we investigated the self-assembling properties of the BC solely. We observed that the morphology of the films is highly dependent on the casting conditions. Films cast under ambient conditions presented no preferential orientation. A short anneal in toluene vapor orients the PS-b-PEO into a hexagonal order. Subsequently, multifunctional thin films were prepared from the NBT and ST sols

or the PMN-PT sol and CF nanoparticles. Using the proper casting conditions and thermal treatment we managed to prepare films, for which grazing incidence X-ray diffraction confirmed the presence of NBT/ST or PMN-PT/CF. This is especially important for NBT/ST, since they easily form a solid solution across the entire compositional range.

Within the European project “Novel Inorganic Inks for Hybrid Printed Electronic Demonstrators” we investigated the synthesis of differently shaped ferroelectric particles. The experimental conditions for the growth of shape-controlled BaTiO_3 particles in NaOH and barium salt ($\text{Ba}(\text{NO}_3)_2$, BaCl_2 or $\text{Ba}(\text{CH}_3\text{COO})_2$) aqueous and water/ethanol solutions using various TiO_2 -containing precursors (Ti-precursor) were studied. We found that different chemistries and physical characteristics of the Ti-precursors resulted in different BaTiO_3 formation rates, morphologies and phase compositions. Nanocrystalline anatase, TiO_2 aerogel and sodium titanates (NT) belts led to cubic BaTiO_3 at temperatures of 80-230°C, while tetragonal BaTiO_3 formed from potassium titanate (KT) at 150-230°C. The morphology of the BaTiO_3 , prepared from KT at low temperatures (80-100°C), did not differ significantly from that obtained from NT belts and TiO_2 aerogel. These precursors, which reacted slowly in alkaline aqueous media, produced single-crystalline star-like particles. The fastest BaTiO_3 formation rate was observed for nanocrystalline anatase, which led to irregularly shaped BaTiO_3 particles. According to TEM investigations, the growth of the single-crystalline star-like BaTiO_3 particles occurred via the oriented attachment of nanocrystals, which formed from the dissolved barium and titanium species. The modification of the water solution by the addition of ethanol or excess of NaOH caused the morphological change from star- to square-like particles, which similar to stars became irregularly shaped above 100°C. The modifications of the solution are believed to influence both the nucleation and aggregation process and consequently changed the particle shape from star- to square-like.

In the scope of the Center of Excellence in Nanoscience and Nanotechnology we set up the first Pulsed Laser Deposition (PLD) system in Slovenia, which is a powerful technique for the thin-film growth of inorganic materials (Fig. 3). The delivered system is dedicated for layer-by-layer growth and thus enables the preparation of high-quality thin films and structuring on a nanoscopic level. The system is equipped with the following major components: heater stage for laser and resistive heating of substrates, target scanning stage, loadlock for sample and target transfer, high-pressure reflection high-energy electron diffraction system, upgrade with UHV pumps (titanium sublimation pump, ion pump), oxygen plasma source, sputter source (1 x 1.3" target), connection with a glovebox. For the ablation of the target material a KrF excimer laser is used with an energy up to 700 mJ per pulse and a maximum repetition rate of 50 Hz. The system is mainly utilized for the deposition of functional oxides on silicon substrates. Interfacing an oxide with silicon is a great challenge that has attracted a lot of interest in the industrial and scientific community so far. Solving the interface problem would enable the further scaling of microelectronic devices to smaller dimensions and the growth of high-quality oxides with different functionalities on a silicon platform, which can be exploited in micro-electro-mechanical systems, random-access memories, and other oxide-based nano-electronic devices. In our study, pulsed laser deposition (PLD) was used to study the interfaces between SrTiO_3 (STO) thin films and silicon. Before the deposition the native oxide on the silicon was removed by HF dip, while in order to understand the interfacial structure in more detail, we performed kinematical simulations of the reflection high-energy electron diffraction (RHEED) patterns. The RHEED patterns were calculated for unreconstructed and reconstructed $\text{Si}(001)$ surface with up to four atomic layers and for each of these models we used two different azimuth directions, [100] and [110].

Using a HF treatment of silicon substrate we obtained H-terminated surface, which prevents silicon from oxidising. On such substrates STO was deposited directly or using SrO as a buffer layer. The results show that the optimum conditions involve a two-step procedure, in which the initial vacuum and the lower deposition temperature have an important role. In the case of the direct deposition of STO the obtained films are preferentially textured with a (100) orientation. The application of SrO enabled partially epitaxial growth of STO with $\text{STO}(110) \parallel \text{Si}(100)$ and $\text{STO}[100] \parallel \text{Si}[110]$ (Fig. 4). The change of the growth orientation induced by SrO was governed by the formation of the $\text{SrO}(111)$ intermediate layer and subsequently by the minimization of the lattice misfit between the STO and SrO.



Figure 3: Pulsed laser deposition (PLD) system with laser and optical cabinet.

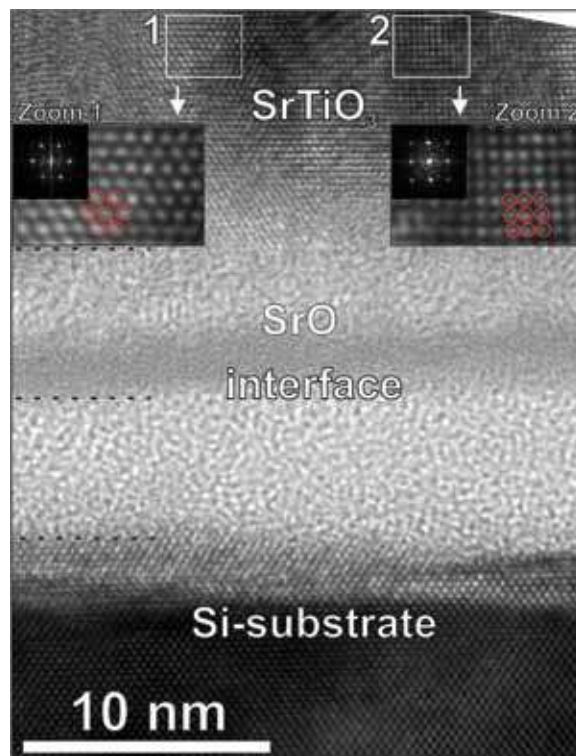


Figure 4: HRTEM image of STO deposited on SrO-buffered Si substrate.

Organization of conferences, congresses and meetings

1. Journal of European Ceramic Society Trust Meeting, Ljubljana, Slovenia, 2. 2. 2012.
2. Permanent Executive Committee of the European Ceramic Society Meeting, Ljubljana, Slovenia, 3. 2. 2012.
3. Materials, Science and Technology 2012 Conference and Exhibition, Pittsburgh, USA, 7.-11. 10. 2011, co-organizers
4. 20th Conference on Materials and Technologies, Portorož, Slovenia, 17.-19. 10. 2012, co-organizers

Patents granted

1. Aleš Dakskobler, Andraž Kocjan, Manca Logar, Method for the preparation of carrier colloidal powder with high specific surface area, SI23502 (A), Urad RS za intelektualno lastnino, 30.4.2012.
2. Aleš Dakskobler, Andraž Kocjan, Manca Logar, Method for the preparation of carrier colloidal powder with high specific surface area, SI23580 (A), Urad RS za intelektualno lastnino, 26.6.2012.

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

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. Functionalization of the surface of organic pigments for durable, efficient and colour-stable paints
Asst. Prof. Srečo Davor Škapin
3. Self-cleaning antibacterial photocatalytic coatings in whitewear production
Prof. Danilo Suvorov
4. Physics and chemistry of porous aluminium for Al panels, capable of highly efficient energy absorption
Prof. Danilo Suvorov
5. New materials for power conversion: oxide semiconductor thermoelectrics
Prof. Danilo Suvorov
6. INNOINKS: Novel inorganic inks for hybrid printed electronic demonstrators
Prof. Danilo Suvorov
7. NAERBIO: Nanostructured ferroelectric films for biosensor
Prof. Danilo Suvorov

NEW CONTRACTS

1. New materials for energy conversion: oxide semiconducting thermoelectrics
Gorenje Household Appliances, d. d.
prof. dr. Danilo Suvorov
2. Development and characterisation of mineral wool fibres
Knauf Insulation, d. o. o.
Prof. Danilo Suvorov

VISITORS FROM ABROAD

1. Dr. Tim Jackson, School of Electronic, Electrical and Computing Engineering, University of Birmingham, Birmingham, Great Britain, 18.-20. 1. 2012
2. Prof. Anatolii Bilous, Dr. Oleg Ovchar, Vernadskii Institute of General and Inorganic Chemistry, Ukrainian National Academy of Sciences, Kiev, Ukraine, 18.-20. 1. 2012
3. Prof. Ivan Sondi, University of Zagreb, Zagreb, Croatia, 24. 2. 2012
4. Dr. Marija Vukomanović, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 2.-6. 4. 2012
5. Dr. Christoph Auer, Hermann Gruenbichler, Dr. Yongli Wang, TDK EPCOS, Deutschlandsberg, Austria, 14. 6. 2012
6. Dr. Jae Ho Jeon, Korea Institute of Materials Science, Changwon, Korea, 20.-22. 6. 2012
7. Prof. Dragoljub Uskoković, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 21.-22. 6. 2012
8. Prof. Jose Varela, São Paulo State University, Araraquara, Brazil, 22. 6. 2012
9. Dr. Marcelo Orlandi, São Paulo State University, Araraquara, Brazil, 29. 6.-15. 7. 2012
10. Dr. Smilja Marković, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 23. 7.-3. 8. 2012
11. Prof. Dragoljub Uskoković, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 26.-27. 7. 2012
12. Dr. Dragana Jugović, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 25. 10. 2012
13. Dr. Miodrag Mitrić, Vinča Institute of Nuclear Sciences, Belgrade, Serbia, 25. 10. 2012
14. Dr. Jyoti Prosad Guha, Missouri University of Science and Technology, Rolla, ZDA, 9. 10.-15. 11. 2012
15. Miodrag Lukić, Dr. Smilja Marković, Institute of Technical Sciences, Serbian Academy of Sciences and Arts, Belgrade, Serbia, 31. 10.-14. 11. 2012

Visiting researchers

1. Dr. Ismael Fabregas, Centro de Investigaciones en Sólidos, CITEFA, Buenos Aires, Argentina, 1. 10.-31. 12. 2012
2. Dr. Zoran Jovanović, Faculty of Physical Chemistry, University of Belgrade, Belgrade, Serbia, 10. 9.-31. 12. 2012
3. Dr. Lei Li, Zhejiang University, Hangzhou, China, 31. 8.-31. 12. 2012

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11. Dr. Marko Udovič*
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14. Dr. Ines Bračko, left 01.11.12

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Note:

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BIBLIOGRAPHY

ORIGINAL SCIENTIFIC ARTICLE

1. Mostafa Baghbanzadeh, Srečo D. Škapin, Zorica Crnjak Orel, C. Oliver Kappe, "A critical assessment of the specific role of microwave irradiation in the synthesis of ZnO micro- and nanostructured materials", *Chemistry (Weinheim, Print)*, vol. 18, issue 18, pp. 2724-2731, 2012.
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