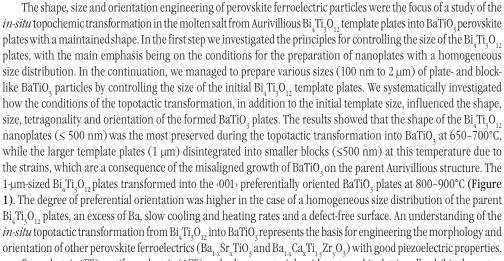
DEPARTMENT FOR ADVANCED **MATERIALS**

In the Department for Advanced Materials we investigate novel materials through an understanding of the mutual dependence of their structural, microstructural and functional characteristics. Modern technologies that enable the synthesis of materials with atomic- and microscale precision are used to prepare pre-designed structural ceramics, thin films, and nanoparticles with the desired crystal structure, chemical composition, microstructure and morphology. Among our important objectives is the development of: i) novel functional oxide materials for various electronic applications, ii) new materials with improved antibacterial and photocatalytic effects, and iii) new oxide materials for efficient high-temperature thermoelectric energy conversion.

Functionalized oxides for electronic applications

Ferroelectric perovskite nanoparticles with well-defined anisotropic shapes are attracting increasing attention because of their unique shape- and size-dependent properties at small dimensions (below 200 nm). In addition, these particles with well-defined shapes and uniform size distributions have the potential to be used as the building Head: blocks for the fabrication of functional nanodevices.



Ferroelectric (FE), antiferroelectric (AFE) and relaxor materials with a perovskite lattice all exhibit characteristic domain structures, which are a result of the lattice strains from phase transformations into lower-symmetry

phases. When external fields (electrical or mechanical) are applied to these materials, changes to the crystal lattice dimensions (intrinsic contribution) and/or changes to the domain structure (extrinsic contribution) can be detected. In order to determine the extrinsic effects on the tunable materials from the Pb_{1-x}La_x(Zr_xTi_{1-x})_{1-x/4}O₃ (PLZT) system, in-situ environmental scanning electron microscopy (ESEM) was employed for the first time. With the in-situ ESEM method the movement of domain walls during pressing or applying an electric field can be directly observed. From these experiments we determined that the mechanical force has the most influence on the domain structure of AFE materials, whereas the domains of FE materials are mainly affected by an electric field. Thus, extrinsic effects play an important role in the electromechanical response of FE and AFE materials. In relaxors, on the other hand, the main contribution to the enhanced electromechanical properties was determined to be intrinsic, from the distortion of the crystal lattice.

In the scope of an investigation of the phase relations in ternary oxide systems where new compounds and solid solutions form and exhibit interesting electric properties, we determined the phase equilibria in La_2O_3 - TiO_2 - GeO_2 at a with the retention of the initial particle size at 900°C (a,b) and at 1000°C. The samples were prepared by the wet-precipitation method from a 660°C (c, d).



Prof. Danilo Suvorov

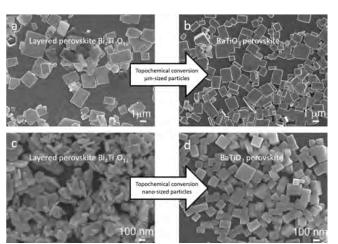


Figure 1: Topochemical conversion from Bi₄Ti₂O₁₂(a,c) into BaTiO₂(b,



soluble precursor in order to achieve good homogenization of the starting compositions. We identified in the system a new compound and several solid solutions and determined their crystal structures and ranges of solid solubility. We found that in the compound $\text{La}_{10x}\text{Ge}_6\text{O}_{26}\pm_{\delta}$ with an apatite crystal structure that forms in the La_2O_3 – GeO_2 system and exhibits high ionic conductivity, it is possible to incorporate Ti, resulting in the solid solution $\text{La}_{9,33}\text{Ge}_{6x}\text{Ti}_x\text{O}_{26}$ that is stable for $0 \le x \le 4$.

We used the pulsed-laser deposition (PLD) technique to grow Pb[Mg_{1/3}Nb_{2/3}]O₃-PbTiO₃ (PMN-PT) thin films on TiO₂-terminated (001) SrTiO₃ (STO) substrates from single-crystal and ceramic targets with compositions in the vicinity of the morphotropic phase boundary (MPB). We prepared the ceramic targets in our laboratories via the columbite method, and we systematically added varying proportions of PbO excess (10–20 %). PbO excess compensates for the loss of highly volatile lead in syntheses at high temperatures, which are necessary for the formation of the perovskite PMN-PT. By using PbO excess and by optimizing the remaining process parameters we managed to avoid the occurrence of the non-ferroelectric pyrochlore phase. Specifically oriented single-phase layers of PMN-PT on STO represent a good starting point for the preparation of multilayered structures for integration into piezoelectric micro-electromechanical systems (piezo MEMS). Namely, the values of the piezoelectric

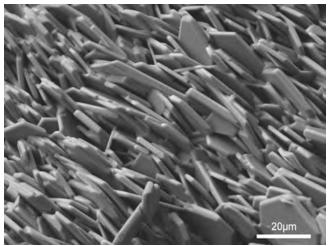


Figure 2: Morphology of trigonal TiS_2 plate-like grains aligned in the (001) direction prepared by a solid-state synthesis of the constituent elements.

 d_{33} constant in rhombohedral PMN-PT single crystals are five times higher than the ones in lead zirconate titanate (PZT). PMN-PT is furthermore distinguished by very low dielectric losses and a high electromechanical factor k_{33} . By optimizing the conditions of the syntheses, and by using appropriate buffer layers and oxide electrodes, these properties can be transferred into the form of thin films.

In the scope of pulsed-laser deposition (PLD) we focused on the epitaxial integration of ${\rm SrTiO}_3$ (STO) with ${\rm Si(001)}$. The high-quality epitaxial growth of STO thin films on a Si platform is essential for many technological applications, since it serves as an excellent template for the growth of different functional oxides. The Si surface is first cleaned by flash annealing up to $1200^{\circ}{\rm C}$, followed by the deposition of metal Sr. The growth of epitaxial STO on the bare ${\rm Si(001)}$ surface is not feasible because of the high oxygen reactivity with the clean Si surface. However, the deposition of a Sr ultra-thin layer passivates the Si surface and enables the further epitaxial growth of STO. The process of PLD deposition of a Sr buffer layer with a coverage up to 1/2 ML has been studied: we have confirmed that Sr grows with the characteristic two-domain $(2\times3)+(3\times2)$ pattern at 1/6 ML Sr coverage and $(1\times2)+(2\times1)$ pattern at 1/2 ML Sr coverage. Furthermore, we have been optimizing the

parameters for the epitaxial growth of STO on such a template, using techniques like Reflection High-Energy Electron Diffraction (RHEED), X-Ray Photoelectron Spectroscopy (XPS), Atomic Force Microscopy (AFM) and X-Ray Diffraction (XRD). Satisfactory values for parameters like the Ar background pressure, laser fluency and frequency, as well as the recrystallization temperature and the sample's suitable film thickness for a recrystallization step have been determined. The PLD system has been modified for the use of an ultra-high vacuum (UHV) suitcase that enables in-situ studies of the different stages of the process by surface techniques, like Scanning Tunneling Microscopy (STM) or XPS. A Temperature Programmed Desorption (TPD) system is also being implemented in order to extract additional chemical information about the investigated sample.

Thermoelectric materials

In the scope of the research on new materials for energy conversion we synthesized new thermoelectric materials based on compounds with a layered crystal structure. Weak interlayer bonding in such compounds enables the intercalation of various atoms, ions and molecules, which contributes to lowering the thermal conductivity and

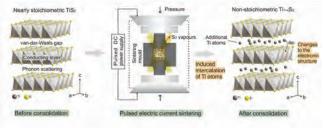


Figure 3: Schematic representation of the pulsed-electric-current-sintering-induced intercalation of Ti atoms in a ${\it TiS}_2$ layered host structure.

consequently to improving the efficiency of the direct conversion of heat to electricity. Layered structures in which individual layers exhibit a high electrical conductivity are thus suitable for the implementation of the phonon glass-electron crystal concept, which is essential for the development of new, efficient thermoelectric materials. We applied ion exchange as a route to new materials based on layered cobaltates and high-pressure pulsed electric current sintering (PECS) to precisely control the nano-stoichiometry and consequently the intercalation of titanium between the layers of titanium disulphide (TiS $_2$). We were the first to show that using a gas-tight model with PECS, a highly stoichiometric TiS $_2$ with a high degree of texturing can be synthesized (Figures 2 and 3). Such a material is interesting for the

development of new thermoelectrics that will replace materials based on rare and toxic elements in near-room- and mid-temperature applications.

Antibacterial and photocatalytic materials

In 2015 the Biomaterials Group was engaged in research work in the

- The development of new antimicrobial biomaterials: We have developed new materials based on Ga-apatite and 1D structures of MgO (Figures 4-6) and showed that they are very effective against *Escherichia coli* (E. coli) and Pseudomonas aeruginosa, (P. aeruginosa). We showed that they are nontoxic to human fibroblast cells and do not produce reactive oxygen species (ROS).
- The development of smart scaffolds as suitable carriers of stem cells and applicable in tissue engineering (in this area we have developed apatite doped with Mg2+, Sr2+, Ga3+ and Zn2+ ions, and functionalized its surface using the BMP-2 protein, which promotes the growth of osteoblastic cells). The material was incorporated within the micro- and nano-porous 3D structure of the PLLA polymer. We showed that the developed material slowly released incorporated bioactive components under physiological conditions and confirmed that the release of doped ions brings a strong antimicrobial activity against *E*. coli and P. aeruginosa.
- The development of innovative nanosensors for the detection of bacteria. In this area we have developed nanomaterials based on Au nanoparticles, functionalized their surface with proteins and confirmed the possibility of their specific interactions with *E. coli* and *P.* aeruginosa bacterial cells.

In 2015 we started to work on the development of new antibacterial materials in which we used the antibacterial peptide nisin (Ni) as an antibacterial component. As a carrier of the antibacterial peptide we prepared spherical gold (Au) nanoparticles with an average size of 20 nm. In order to increase the yield of the antibacterial component (Au-Ni), micron-sized carbon spheres (C) were used as supports. Such prepared hybrid materials exhibit improved activity against the bacteria strains *E. coli* in *P. aeruginosa*.

Materials for heat-insulation applications

The aim of our work on foam glass is to develop a new preparation procedure for a foam glass with improved thermal insulation properties. The new method should have a smaller dependence of the foaming process on the composition of the glass cullet. A new preparation procedure was developed that shows a high potential for further improvements. At a density of 150 kg/m³, we reached a thermal conductivity of 40 mW/(m·K), which is almost 20 % better than in a conventional foam glass. A new project on foam glass aims to reach a thermal conductivity of 37 mW/(m·K). This could be achieved with an understanding of the processes and reactions taking place in the softened glass during the foaming process. We continued with research work focused on the investigation of interactions between foaming additives and glass, and optimizing of the viscosity, surface tension and glass stability at the foaming temperature.

Projects

TDK-Epcos

For the industrial partner EPCOS OHG, Deutschlandsberg, Austria we work on the project: "Thin-Film-Energy-Storage Devices on the basis of PLZT and Cu-electrodes", the focus of which was to investigate the growth decomposition and release of the intracellular content).

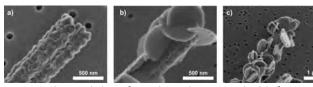


Figure 4: The morphology of a synthesized MgO particle a) before exposure, b) after 2 h of exposure and c) after 24 h of exposure to an aqueous solution. SEM study demonstrates the progress of MgO hydrolysis.

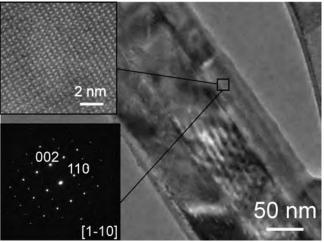


Figure 5: TEM analysis of the hydroxyapatite with incorporated Ga^{3+} ions (4 wt %), which was synthesized by the crystallization of sonochemically prepared hydroxyapatite in the presence of Ga^{3+} ions. This material exhibits an efficient antibacterial activity against P. aeruginosa, a low toxicity for mammalian cells and good control over the release of Ga³⁺ ions. Ga ions are not incorporated into the crystal structure of hydroxyapatite, but rather into the outer thin amorphous layer, which was shown by the NMR analysis.

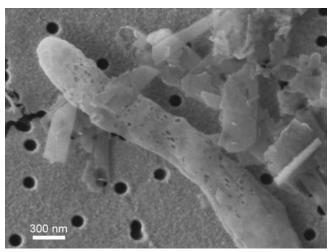


Figure 6: A P. aeruginosa bacterium in contact with the galliumcontaining hydroxyapatite (scanning electron microscope). The bacterium exhibits noticeable holes in the cell wall that most probably lead to bacterial death-cell lysis (destruction of the cell membrane, cell



of $Pb_{(1-x)}La_x(Zr_yTi_{(1-y)})O_3$ (PLZT) thin films using pulsed-laser deposition. The purpose of the project is to develop new materials and technology for advanced energy-storage applications.

ENPIEZO

In the scope of the M-ERA.NET project ENPIEZO we develop piezoelectric-based energy-harvesting (EH) devices to provide a remote source of electricity from waste vibrations with countless applications. For instance, EH devices can be powered by a heartbeat to operate pacemakers or it can provide electricity for sensors at remote locations like wind-turbine air blades. We investigate the fabrication-friendly pulsed-laser deposition of high-quality $Pb(Mg_{1/3}Nb_{2/3})$ O_3 -PbTiO $_3$ thin films on silicon, based on the delicate engineering of silicon-oxide interfaces. The study is performed on laboratory- and industrial-scale systems, and it is the first of its kind in the world. In the project, the preparation of EH devices using aerosol deposition is also investigated. The project, which is coordinated by the JSI, brings together four partners with expertise in a very diverse field of research and development.

SCOPES

During the realization of the SCOPES 2014–2017 project, the laboratory for biomaterials purchased new equipment for the processing of biomaterials (liofilizator), equipment for the cultivation of bacteria and the simulation of physiological conditions (incubator shaker) and equipment for biological work under sterile conditions (biological safety cabinet, class 2).

Some outstanding publications in the past year

- 1. Dejan Klement, Matjaž Spreitzer, Danilo Suvorov, "Formation of a strontium buffer layer on Si(001) by pulsed-laser deposition through the Sr/Si(001)(233) surface reconstruction", *Appl. phys. lett.*, vol. 106, issue 7, str. 071602-1-071602-6, 2015,
- 2. Marjeta Maček, Dejan Klement, Boštjan Jančar, Danilo Suvorov, "Hydrothermal conditions for the formation of tetragonal BaTiO[sub]3 particles from potassium titanate and barium salt", *Ceram. int.*, vol. 41, no. 10, part B, str. 15128-15137, 2015, [COBISS.SI-ID 28836903]
- Jakob Koenig, Danilo Suvorov, "Evolution of the electrical properties of K_{0.5}Bi_{0.5}TiO₃ as a result of prolonged sintering", *J. Eur. Ceram. Soc.*, vol. 35, no. 10, str. 2791-2799, 2015, [COBISS.SI-ID 28539687]
- Vojka Žunič, Srečo D. Škapin, Danilo Suvorov, "The assembly of TiO₂ nanoparticles into micrometer-sized structures, photocatalytically active under UV and Vis light", J. Am. Ceram. Soc., vol. 98, iss. 10, str. 2997-3005, 2015, [COBISS.SI-ID 28722983],
- Vukomanovic Marija, Repnik Urska, Zavasnik-Bergant Tina, Kostanjsek, Rok, Skapin Sreco D., Suvorov Danilo, "Is Nano-Silver Safe within Bioactive Hydroxyapatite Composites?" ACS BIOMATERIALS-SCIENCE & ENGINEERING, Vol 1, Issue 10, pp 935-946, October 2015

Organization of conferences, congresses and meetings

- Workshop on NATO Sfp 984091 project "Microwave Tuneable Materials, Composites and Devices", Ljubljana, 23. 2. – 27. 2. 2015.
- Workshop on SCOPES project "Intelligent Scaffolds as a Tool for Advanced Tissue Regeneration", Ljubljana, 26. 5. - 29. 5. 2015.
- 3. Materials Science & Technology 2015 Conference and Exhibition, Columbus, USA, 4. 10. 8. 10. 2015 (co-organizers).
- 4. 23. International Conference on Materials and Technology, Portorož, 27. 9. 30. 9. 2015 (co-organizers).

INTERNATIONAL PROJECTS

- Thin-Film-Energy-Storage Device on the basis of PLZT and Cu-Electrodes Prof. Danilo Suvorov Epcos Ohg
- Technological Characterisation Test of OGK-5 (RGRES) Ashes for Verification of Usability in the Process of Rock Wool Production Prof. Danilo Suvorov
- Enel Ingegneria E Ricerca S.p.a.

 3. Investigation of NdDyCoCuFe Rare Earth Alloys and Related Compounds Prof. Danilo Suvorov
 Urban Mining Company
- 4. Production and Characterisation of Coal Ash Fibres

Prof. Danilo Suvorov Enel Ingegneria E Ricerca S.p.a.

- Microwave Tunable Materials, Composites and Devices Asst. Prof. Boštjan Jančar
- Nato North Atlantic Treaty Organisation
- 3D Composite Plasmonic Metal/Semiconductor Photo-catalyst for Efficient Solar to Fuel Energy Conversion Asst. Prof. Srečo Davor Škapin Slovenian Research Agency
- Biomineralization at the Nanoscale: from the Natural Systems to the Laboratory Asst. Prof. Srečo Davor Škapin Slovenian Research Agency

RESEARCH PROGRAM

Contemporary Inorganic Materials and Nanotechnologies Prof. Danilo Suvorov

Cleantech Block II - Energy Saving Cladding Dr. Jakob König EDDP (EUDP) Energy Technology Development

Enabling technology for high-quality piezoMEMS Dr. Matjaž Spreitzer

R & D GRANTS AND CONTRACTS

- Engineering of structural and microstructural characteristics in contemporary dielectrics and ferroelectrics with perovskite and perovskite-like crystal structures Prof. Danilo Suvorov
- Growth of high quality piezoelectric thin films on silicon using pulsed laser deposition Dr. Matjaž Spreitzer
- SCOPES; Intelligent Scaffolds as a Tool for Advanced Tissue Regeneration Dr. Marija Vukomanović SNF- Swiss National Science Foundation

NEW CONTRACTS

- Development and characterisation of mineral wool fibres Prof. Danilo Suvorov Knauf Insulation, d. o. o., Škofja Loka
- Development and characterisation of mineral wool fibres Prof. Danilo Suvorov Knauf Insulation, d. o. o., Škofja Loka

VISITORS FROM ABROAD

- 1. Dr. Christoph Auer, TDK EPCOS, Deutschlandsberg, Austria, 10. 2. 2015.
- Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 10. 2. 2015.
- Dr. Manfred Schweinzger, TDK EPCOS, Deutschlandsberg, Austria, 10. 2. 2015.
- Dr. Anatolii Bilous, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine 23 2 - 27 2 2015
- 5. Dr. Tim Jackson, University of Birmingham, Birmingham, Great Britain, $23.\ 2.\ -$ 27. 2. 2015.
- Dr. Oleg Ovchar, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 23. 2. - 27. 2. 2015
- Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 2. 3. 4. 3. 2015.
- Prof. dr. Heli Jantunen, Microelectronics and Materials Physics Laboratories, University of Oulu, Oulu, Finland, 25. 3. - 27. 3. 2015
- Dr. Jari Juuti, Microelectronics and Materials Physics Laboratories, University of Oulu, Oulu, Finland, 25. 3. - 27. 3. 2015.
- Dr. Dmytro Durylin, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 6. 4. - 30. 4. 2015.
- Dr. Oleg Ovchar, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 6. 4. - 30. 4. 2015.
- 12. Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 7. 4. 10. 4. 2015.
- 13. Prof. dr. Yun Liu, Research School of Chemistry, Australian National University, Canberra, Australia, 15. 6. - 19. 6. 2015
- 14. Dr. Hua Chen, Research School of Chemistry, Australian National University, Canberra, Australia, 15. 6. - 19. 6. 2015.
- 15. Dr. Smilja Marković, Institut tehničkih nauka Srbske Akademije Znanosti i Umetnosti, Belgrade, Serbia, 15. 6. - 23. 6. 2015.

- 16. Prof. dr. Dragan Uskoković, Institut tehničkih nauka Srbske Akademije Znanosti i Umetnosti, Belgrade, Serbia, 8. 7. - 13. 7. 2015.
- Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 13.7. 15.7. in 20.7. -24. 7. 2015
- 18. Dr. Oleg Ovchar, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 26. 7. - 13.8. 2015.
- Olexander Suslov, B. Sc., V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 26. 7. - 13.8. 2015.
- 20. Dr. Chun-Liang Kuo, National Taiwan University, Taipei City, Tajvan, 23. 8. 5. 9. 2015.
- 21. Dr. Chao-Ting Chen, National Taiwan University, Taipei City, Taiwan, 23. 8. 5. 9. 2015.
- 22. Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 31. 8. 4. 9. 2015.
- 23. Prof. dr. Anatolii Bilous, V. I. Vernadskii Institute of General and Inorganic Chemistry, Kiev, Ukraine, 10. 9. - 15. 9. 2015.
- 24. Prof. dr. Wen-Jong Wu, National Taiwan University, Taipei City, Taiwan, 10. 9. -14.9. 2015.
- 25. Prof. dr. Gertjan Koster, MESA+ Institute for Nanotechnology, University of Twente, Enschede, Netherlands, 29. 10. - 30. 10. 2015.
- 26. Dr. Jari Juuti, Microelectronics and Materials Physics Laboratories, University of Oulu, Oulu, Finland, 2. 11. - 11. 12. 2015.
- 27. Dr. Kerstin Schmoltner, TDK EPCOS, Deutschlandsberg, Austria, 7. 12. 9. 12. 2015

Visiting Researchers

- Prof. dr. Jianjiang Bian, Department of Inorganic Materials, Shanghai University, Shanghai, China, 2. 6. - 28. 8. 2015.
- Dr. Jyoti Prosad Guha, Missouri University of Science and Technology, Rolla, USA, 14. 7. - 30. 9. 2015.
- Dr. Daniel Diaz Fernandez, Universidad Autónoma de Madrid, Madrid, Spain, 1, 10 2015 - 30. 9. 2016.

STAFF

Researchers

- Asst. Prof. Boštjan Jančar
- Dr. Špela Kunei
- Dr. Marjeta Maček Kržmanc
- 4. Dr. Matjaž Spreitzer
- Prof. Danilo Suvorov. Head
- Asst. Prof. Srečo Davor Škapin
- Dr. Marija Vukomanović

Postdoctoral associates

- Dr. Jakob König
- Dr. Mojca Otoničar

10. Dr. Vojka Žunič Postgraduates

11. Nemanja Aničić, B. Sc.

- 12. Urška Gabor, B. Sc.
- 13. Dr. Dejan Klement, left 01.11.15
- 14. Mario Kurtjak, B. Sc.
- 15. Tjaša Parkelj, B. Sc.
- 16. Tilen Sever, B. Sc.

Technical officers

- 17. David Fabijan, B. Sc.
- 18. Dr. Jana Ferčič
- 19. Damjan Vengust, B. Sc.
- Technical and administrative staff
- Maja Šimaga, M. Sc.
- 21. Silvo Zupančič



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ORIGINAL ARTICLE

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- 5. Jakob Koenig, Danilo Suvorov, "Evolution of the electrical properties of $K_{0.5} Bi_{0.5} TiO_3$ as a result of prolonged sintering", *J. Eur. Ceram. Soc.*, vol. 35, no. 10, pp. 2791-2799, 2015.
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- 13. Anastasia Samodurova, Damjan Vengust, Andraž Kocjan, Tomaž Kosmač, "The sintering-temperature-related microstructure and phase assemblage of alumina-doped and alumina-silica-co-doped 3-mol%-yttria-stabilized tetragonal zirconia", Scr. mater., vol. 105, 50-53, 2015.
- Andrijana Sever Škapin, Luka Škrlep, Danilo Suvorov, Vojka Žunič, Srečo
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- 15. Melita Sluban, Nejc Rozman, Matej Pregelj, Carla Bittencourt, Romana Cerc Korošec, Andrijana Sever Škapin, Aleš Mrzel, Srečo D. Škapin, Polona Umek, "ransformation of hydrogen titanate nanoribbons to ${\rm TiO_2}$ nanoribbons and the influence of the transformation strategies on the

- photocatalytic performance", *Beilstein j. nanotechnol.*, vol. 6, pp. 831-844, 2015.
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- 17. Srečo D. Škapin, Vida Čadež, Danilo Suvorov, Ivan Sondi, "Formation and properties of nanostructured colloidal manganese oxide particles obtained through the thermally controlled transformation of manganese carbonate precursor phase", *J. colloid interface sci.*, vol. 457, pp. 35-42, 2015.
- 18. Srečo D. Škapin, Urša Pirnat, Boštjan Jančar, Danilo Suvorov, "Microwave dielectric properties, crystal structure, and microstructure of the Bi₃Nb_{1-x}Ta_xO₇ solid solution", *J. Am. Ceram. Soc.*, vol. 98, no. 12, pp. 3818-3823, 2015.
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MENTORING

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