# DEPARTMENT FOR NANOSTRUCTURED MATERIALS K-7

***The basic and applied research in the Department for Nanostructured Materials includes ceramic materials, metals, intermetallic alloys and minerals. Our research encompasses conventional processing as well as the development of new technologies and methods for preparing new materials with novel properties. It includes the experimental and theoretical investigations of structures, analyses of chemical compositions at the atomic level, and measurements and calculations of physical properties, all of which help us to improve the properties of micro- and nanostructured materials.***

Head:
***Prof. Spomenka Kobe***

2014 was the second year of the EU FP7 project "Nanocrystalline permanent magnets based on hybrid metal-ferrites" (**NANOPYME**). Because Sr-ferrite particles prepared via high-energy ball milling did not exhibit sufficiently high magnetic properties, a hydrothermal synthesis was implemented. Furthermore, hydrothermally synthesized Sr-ferrite particles were used for the preparation of hard-soft magnetic composites. Such composites are particularly interesting due to the possibility of an exchange-coupling interaction between the phases, which leads to an increase in the maximum energy product of the magnet. The increase of latter in ferrite-based magnets is also one of the goals of the project.

In this year we finished our study on the effect of coatings on **Sm–Co magnets** to improve the corrosion resistance. We coated Sm–Co magnets using two technologies: standard galvanic nickel, and PVD sputtering (TiN, CrN or TiAlN). A combination of both was tried as well (galvanic and PVD), with bare magnets being used as a reference. The oxidation resistance was tested in air at three different temperatures (350, 450 and 550 °C). By periodical weighing of the magnets during annealing we were able to follow the progress of the oxidation, and finally extract the parabollic oxidation coefficients. All the coatings improved the oxidation resistance, with the best performance observed for the Ni/CrN combination. These results were confirmed by optical and scanning electron microscopy observations. Before and after the annealing experiments, the magnetic properties were measured too, showing that the magnetic properties deteriorate more slowly when coated.

In the scope of the European project Replacement and Original Magnet Engineering Options ROMEO one of the tasks is to produce a prototype electromotor based on Nd–Fe–B permanent magnets. A crucial demand of such magnets is to meet the stringent requirements of the industrial partners, which will build the prototype (VALEO, SIEMENS and DAIMLER).

**Post-sintering technology, developed at the Department for Nanostructured Materials was the only proposed technology that was able to satisfy all of the required conditions at once. It is called the grain-boundary diffusion process (GBDP), based on the electrophoretic deposition (EPD) of $TbF_3$ powder. The process optimization was focused on temperature/time modification, which revealed the optimum temperature at 875°C for 10 h. Calculated values for the coercivity and the remanence of the GBDP magnets at room temperature were 2027 kA/m and 1.31 T. With these achievements, the main goal was achieved and the industrial partners will build a demonstrator on the basis of our technology.**

The architecture of the magnets needs to be designed in such a way that the outer part of the magnets is more resistant to the demagnetizing field to which the magnets are exposed during the motor-operation at high-loads.

Goal 1 of the project is to achieve ambitious magnetic properties: a coercivity of 2000 kA/m or more and a remanence of at least 1.3 T at room temperature.

The Nd–Fe–B magnets need to be a representative of the family with a reduced amount of heavy rare earths (HREs). i.e., Dy and Tb. This means that the sintered magnets produced only via a conventional powder metallurgy process are not capable of achieving low concentrations of HRE and retain the magnetic properties at the extreme level of Goal 1.

Within the scope of the EU project ROMEO we also developed **highly coercive ($H_{ci}$) isotropic permanent Nd–Fe–B-based magnets.** The high $H_{ci}$ was achieved by addition of $DyF_3$ suspension to commercially available MQU-F rapidly quenched ribbons. The ribbons were coated with $DyF_3$ in isopropanol, spark plasma sintered and heat treated. The highest $H_{Ci}$ enhancement was obtained at 2.2 wt.% Dy-fraction, i.e., from 1580 to 2025 kA/m, which is a 25 % improvement. To understand the diffusion process of Dy into the Nd–Fe–B ribbons we performed microstructural and chemical analysis of the high $H_{ci}$ ribbons, using scanning (SEM) and transmission (TEM) electron microscopy. The EDXS-TEM mapping of nano-sized grains showed that the samples prior to the heat treatment do not contain Dy. After the heat-treatment process the Dy diffuses into the grains and forms core-shell-like structures,

which are crucial for the major increase in $H_{ci}$. Further research will be based on a quantitative EELS-TEM analysis of samples before and after the heat treatment.

Within the 7th EU FP we coordinate another international research project called "**Mag-Drive**", the aim of which is the development of new technologies for the production of rare-earth-based permanent magnets for high-efficiency electro-motor applications. Besides the coordination, our tasks in the project are spark-plasma sintering and microstructural and magnetic characterization of the samples.

**Nanostructured materials** based on **Fe–Pd, Co–Pt and Fe–Pt** exhibit unique properties, depending on their crystal structure and size, applicable in bio-sensing, actuation, cell separation or targeted drug delivery and advanced cancer treatments. Electrochemical synthesis was applied for the processing of Fe–Pd and Co–Pt nanowires, where a kinetic study revealed the systems properties can be tailored with high reproducibility. Single Fe–Pd nanowires were investigated using magnetic force microscopy and the magnetization reversal mechanism study revealed the dominant contribution of the shape anisotropy and a square like hysteresis behaviour typical for magnetic mono-domain nanostructures. We have to emphasize that the magnetic properties of a single magnetic nanostructure is, due to its low volume and low magnetic signal, challenging to measure, and that these measurements represent an excellent basis for the magnetic nanostructures applications. Fe–Pd nanowires were successfully transformed into highly coercive tetragonal or $L1_0$ phase with the highest coercivities achieved for this system. In Co-Pt nanowires, it was discovered that the crystallographic orientation of both phases texture, such that the [00–1] of the hcp phase and the [111] of the fcc phase are pointing almost perpendicular to the nanowire axis. The fitting of the experiment to these calculations results in a transverse effective anisotropy constant (Keff= 2.6 x 104 J/m$^3$) in the nanowires, which can be ascribed to the strong magnetocrystalline anisotropy of the hcp phase. A MFM study revealed that a spatial magnetization modulation was found to be length dependent. In order to correlate the magnetization distribution in nanostructures, we have started with modelling in order to investigate the magnetization distribution aspects and to translate theory into practice.

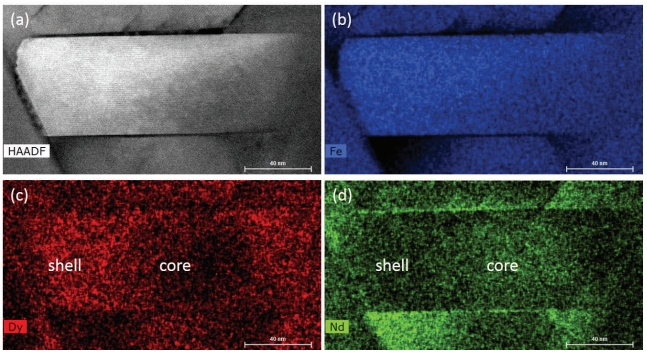


*Figure 1 : EDXS-TEM mapping of Dy-treated Nd-Fe-B grain (a). Figure shows Fe (b), Nd (c) and Dy (d) maps, where Dy forms (Dy,Nd)-Fe-B shell around Nd-Fe-B core matrix.*

We have continued the work on multifunctional **Fe–Pt–$SiO_2$–Au core/shell nanoparticles (NPs)** suitable for novel magneto-photo medical curing. The Fe–Pt nanoparticles were produced in a single and multicore version with improved magnetic properties, without affecting their superparamagnetic nature necessary for biomedical usage. Fe–Pt–$SiO_2$ NPs were further functionalized with biocompatible zwitterionic catechol ligands which introduce excellent hydrophilicity. Fe-Pt NPs were coated with $SiO_2$/Au and show large absorption the range between 550 and 850 nm, which make them superior to commercially available Au NPs with a discrete absorption peak. The suspension of these Fe–Pt–$SiO_2$–Au core/shell nanoparticles was irradiated with a laser at the wavelength λ=810nm at P=1W. The temperature increased with a rate of 2 K/min, which makes this material suitable for photo thermal cancer treatments.

We investigated the isotropic-strain influence on magnetic ordering in systems with strongly-correlated 4f electrons of rare-earth elements. We demonstrated the relation between the topology of the Fermi surface and the type of magnetic ground state. Within the framework of the density-functional theory we explored the existence of magnetic phases in the system $Ca_xGd_{1-x}Cu_5$. We determined the magnitude of the magneto-elastic coupling of the geometrically-frustrated ferromagnet $NaMnO_2$.

In 2014 the European programme **EUROFusion** (Horizon 2020) has been established. The department is involved with two projects aimed at the development of structural materials for the demonstration fusion reactor DEMO. In the project within the "Enabling research" we continued the development of the ceramic-matrix composite ($SiC_f$/SiC). The second project (WPMAT) was focused on tungsten-based composites, where the goal is to improve the mechanical properties of tungsten under operational conditions by the addition of nanoparticles and/or fibres. The research has been partly performed in collaboration with the UK company TISICS Ltd.

In the final year of the project **BioTiNet** (FP7-ITN) we performed the last part of the study of nanocrystalline titania coatings applied to Ti-based alloys using a hydrothermal treatment of the alloy. Previous studies have confirmed the improved physico-chemical properties of the coatings as well as their beneficial interaction with proteins and human adipose cells. In the further study, performed in collaboration with Biotechnical Faculty, University of Ljubljana, and the Faculty for Mechanical Engineering, University of Ljubljana, ***E-Coli*** was used as a model system. The main goal was to understand the effects of surface charge, wetting and micro-roughness on the adhesion of bacteria. Using the specific hydrothermal treatment we succeeded in reducing the adhesion by 40 %, which attracted the attention of the producers of Ti-based biomaterials.

In the frame of the COST Action **Namabio** (From nano to macro biomaterials and applications to stem cells regenerative orthopaedic and dental medicine), the research of materials for tissue engineering has been continued. The research work has been aimed at the development of 3-dimensional biodegradable and bioactive scaffolds with bioactive glass for the osteochondral implants. We studied the effect of the bioactive glass nanoparticles embedded into gellan gum or a silk fibroin matrix on hydroxiapatite formation, biodegradability and the mechanical properties of the composite material. In collaboration with the Educell and Animacell (SMEs) we also started with the cell tests. In collaboration with the Spanish group CIDETEC we have also confirmed ability to prepare injectable bioactive scaffolds.

In the field of **photocatalysis** we designed and assembled various (micro)reactors based on photocatalytic titanium dioxide for chemical oxygen demand (COD) measurements in water in order to determine the overall content of organic compounds, as well as for the decomposition of the specific organic pollutants in water. The active part of a (micro)reactor is made of $TiO_2$ anatase nanotubes that are prepared by the anodic oxidation of titanium foil. The specific design of the photocatalytic reactor was protected by a patent.

Within the investigations of n-type **oxide thermoelectric materials** we synthesized Nb-doped $SrTiO_3$. The basic microstructure of the solid solution $Sr(Ti_{0.8}Nb_{0.2})O_{3-x}$ was modified with the addition of $Sr_3Ti_2O_7$ nucleation seeds and/or with the addition of SrO-excess. The $Sr_3Ti_2O_7$ seeds were synthesized using a molten-salt approach. In the case when the $Sr_3Ti_2O_7$ seeds were added to a $Sr(Ti_{0.8}Nb_{0.2})O_{3-x}$ solid solution we were able to improve the thermoelectric properties of the material due to the lower thermal conductivity and the increased electrical conductivity. The figure of merit ZT thus increased from 0.09 to 0.14. By using high-resolution STEM HAADF imaging we were able to determine the Nb content on the B sites in the $Sr(Ti_{0.8}Nb_{0.2})O_{3-x}$ solid solution with the perovskite structure.

**Perovskite materials** such as $BaTiO_3$ and Fe-$SrTiO_3$ nanostructures are suitable for many applications, such as humidity or oxygen sensing and tunable HTS (high temperature superconducting) microwave filters. The potential advantages of the nanostructured forms have been, however, scarcely explored compared to other oxides. The synthesis of perovskite nanostructures via sol-gel electrophoretic deposition (EPD) into anodic aluminium oxide (AAO) membranes has proven to be very successful and useful. In the last year we submitted a postdoc project that was granted in the middle of the year. The project entitled "From the synthesis of metal oxides to the humidity and oxygen prototype nanosensors" focuses on the synthesis and gas-sensing device formation of the one-dimensional (1D) metal oxide materials such as $BaTiO_3$, Fe-doped $SrTiO_3$ and Y-doped $ZrO_2$ nanostructures.

We developed homogeneous coarse-grained low-voltage **ZnO-based varistor ceramics** doped with $Bi_4Ti_3O_{12}$ or $Bi_{12}TiO_{20}$ and other varistor dopants having breakdown voltages in the range from 20 to 40V/mm and a nonlinearity coefficient $\alpha$ above 20. The development was based on the discovery that the rapid decomposition of pre-reacted phases from the $Bi_2O_3$-$TiO_2$ system into the $TiO_2$-rich $Bi_2O_3$ liquid phase enhances formation of inversion boundaries in the ZnO grains, which results in the homogeneous grain growth and the microstructure development.

The influence of the organic vehicle and the amount of added varistor powder filler on the rheological characteristics of the pastes and their screen-printing performance was studied. A paste with a high solids load of 70% and good printing characteristics was developed and enables the preparation of dense layers of varistor ceramics with good electrical characteristics at a low sintering temperature of 900 °C, typical for the screen-printing hybrid circuit technology.

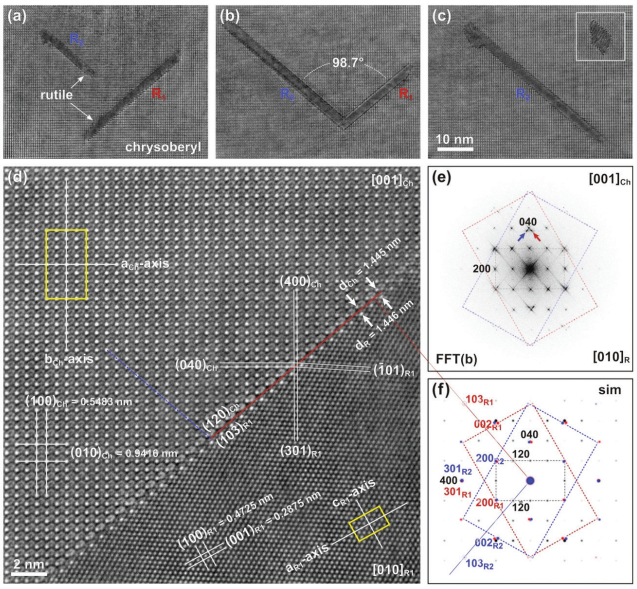


*Figure 2: TEM study of rutile precipitates in chrysoberyl crystals from Pratinhas. (a) Two rutile orientations are observed (R1 and R2). (b) L-shaped cluster of two impinging rutile precipitates enclosing an angle of 98.7°. (c) Isolated rutile precipitate. (d) Structural analysis of chrysoberyl-rutile (Ch|R1) interface. Crystallographic axes of chrysoberyl and rutile are used to determine the orientation relationship [001]Ch(120)Ch ‖ [010]R(103)R. Real space 4x4 unit-cells are outlined in yellow. (e) Fast Fourier transform of HRTEM image from Fig. 8b displaying additional reflections from rutile R1 and R2 precipitates (arrows). (f) Reconstructed EDP of chrysoberyl and rutile precipitates calculated with lattice parameters of deformed rutile from the HRTEM analysis (Fig. 8d), with (120)Ch ‖ (103)R1 and (12-0)Ch ‖ (103)R2. Reciprocal 2x2 unit-cells are outlined (chrysoberyl - grey, R1 - red, R2 - blue).*

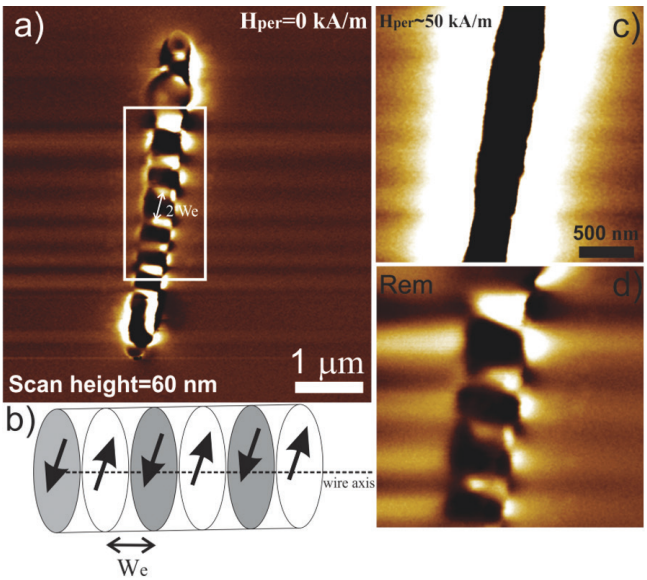


*Figure 3: (a) The MFM image of an individual Co-Pt nanowire. (b) Schematic picture of possible magnetization modulation in the nanowire. (c) Saturation magnetization state of an individual Co-Pt nanowire at applied field of Hper≈ 50 kA/m. (d) Remanent magnetization of an individual Co-Pt nanowire.*

Applications involving **transparent conducting films** (TCFs), such as flat-panel displays and touch screens technologies, are dominated by indium-tin-oxide (ITO). The increasing prices of indium make a strong argument for alternative TCFs with competitive characteristics and a lower price. We demonstrate the potential of highly oriented zinc oxide (ZnO) films grown on glass substrates under low-temperature hydrothermal (HT) conditions at 90 °C from aqueous solutions of Zn-nitrate and Na-citrate. The formation of a continuous ZnO seed-layer with the proper thickness, grain size, connectivity and orientation of the seed-grains on the glass is shown to be essential to achieve the conditions for the growth of highly oriented (0001), smooth, transparent and conductive ZnO films according to the spatially confined oriented growth (SCOG) mechanism. The film grown on a homogeneous seed-layer with a grain size of about 20 nm showed an optical transmittance of up to 82% and a relatively low resistivity for the undoped ZnO ceramic of the order of a few 100 Ω $sq^{-1}$. Such characteristics are explained by a highly oriented crystalline texture and a high coalescence of the ZnO crystals in these films.

In collaboration with the company VARSI we continued the development of special varistors for the overvoltage protection of renewable energy systems (solar panels and wind-turbine generators). The result is new types of varistors with a high stability under a dc field for operation under harsh climate conditions (temperature, humidity).

In the development of cost-effective and **environment friendly photovoltaic systems** the $Cu_2ZnSnS_4$-type films (CZTS) showed very promising. CZTS is p-type semiconductor with preferred optical band gap at 1.5eV and a high absorption coefficient made of well abundant and hence cheap elements, which are also nontoxic. We studied the preparation of CZTS films by sol-gel spin coating and annealing in a sulphur-rich atmosphere at 500°C on flexible metal substrates of Al, Ti, Cu and Mo.

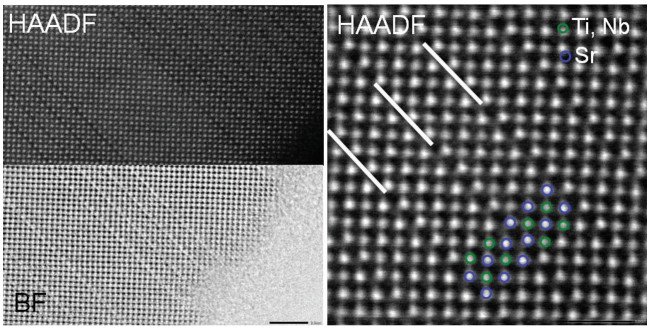

*Figure 4: HRSTEM micrograph of RP faults within $Sr_3Ti_2O_7$ as seen in the [001] zone axis.*

We continued our studies of tropochemical defects, epitaxies and phase transformations in **natural and synthetic minerals**. We found that small Cu additions stabilize the formation of mackinawite and the sphalerite (Cu, Fe)S modification. In ***American Mineralogist*** we published a paper on (101) twins in rutile, where we proposed the formation mechanism of twins in rutile by progressive recrystallization after oxyhydroxide precursor; the (101) twin boundaries are stabilized by the presence of Al-hydroxide and the (301) twin boundaries by the presence of Fe-hydroxide. We also continued our investigations of twinning in spinel and taaffeite-type modulated structures in the spinel-chrysoberyl ($MgAl_2O_4$-$BaAl_2O_4$) system. The PhD student Sandra Drev conducted a three-month specialization at the Max Planck Institute in Stuttgart, where she focused on the ordering of taaffeite-type modulated structures in Be-doped spinel. In cooperation with dr. Goran Dražič from the National Institute of Chemistry we successfully determined the presence of a single atomic layer of beryllium at the (111) twin boundaries in spinel. In addition to being a major analytical challenge, this was crucial for the confirmation of our theory on tropochemically induced twinning. We also determined the local atomic structure of the (130) twin and rutile precipitates in chrysoberyl. The initial atomic model of the twin boundary was proposed based on the HRTEM analyses and a detailed Rietveld analysis of the chrysoberyl structure, performed in cooperation with dr. Matjaž Mazaj from the National Institute of Chemistry. The local atomic model was further optimized by density functional theory (DFT) calculations in cooperation with dr. Matej Komelj. The results of this work were described in a paper that received excellent reviews and is accepted for publication in ***American Mineralogist***. In the system ilmenite-hematite we continued our studies of topotaxial transformations during the oxidation of ilmenite to rutile and hematite. First we investigated natural oriented rutile/hematite intergrowths from Mwinilunga in Zambia, where we learned of a complex nature of topotaxial reactions. After a detailed analysis of the rutile/hematite interfaces, nano-inclusion of ilmenite in rutile and precipitates of hematite in rutile, we were able to reconstruct the progressive topotaxial reactions that led to the formation of complex rutile/hematite intergrowths. This work is accepted for publication in ***Contributions to Mineralogy and Petrology*** (IF=3.02), which is to be the first article published in this prestigious journal by Slovenian authors. The mechanism of ilmenite oxidation was further studied by heating single crystals of ilmenite in an atmosphere of air. We found that during the heating of ilmenite, rutile exsolutions follow a different crystallographic law compared to that of the natural specimens. Based on our studies of naturally and synthetically oxidized ilmenite samples we are able to explain topotaxial crystallographic relationships as a function of temperature and oxygen fugacity. In the past year we also started synthesis procedures for the enhanced growth of multiply twinned rutiles in several subsequent generations, which has potential application value in the development of fractal supports for separation processes and catalysis. In 2014 we started a new research area of twinning in cassiterite, which is isostructural with rutile, and has many potential applications in catalysis and sensor technology due to its excellent optical and electrical properties. We started our investigations by studying natural cassiterite twins, where we have indications that their formation is related to the topotaxial recrystallization over some Fe-oxides, probably magnetite. The identified mechanism of twinning will be further verified by the synthesis under controlled laboratory conditions.

In collaboration with research group from Vinča Institute of Nuclear Sciences, Serbia, we studied the relationship between the kinetics of the sorption process and the structure-chemistry properties of **$MgH_2$ thin films**, which are considered to be one of the most promising candidates **for the reversible hydrogen storage**. To elucidate the rate-limiting step for desorption thin films of $MgH_2$ were prepared and irradiated with argon ions. Detailed high-resolution transmission electron microscopy (HRTEM) and selected-area electron diffraction (SAED) studies showed different microstructures between non-irradiated and irradiated films. The non-irradiated thin films are homogeneous, consisting of randomly orientated crystallites. In contrast to the previous case, the irradiated film was signified by large crystal grains imbedded in a crystallites matrix. The SAED analysis showed the coexistence of $MgH_2$, MgO and Mg crystal phases.

In collaboration with the research group from Sabanci University, Turkey, we have studied **long persistence phosphors**, which enable the storage and slow discharge of light. It is anticipated that the incorporation of such a delayed light release source will improve the light-harvesting efficiency for photovoltaic applications. We focused on strontium aluminate (SA) phosphors doped with $Eu^{2+}$ and $Dy^{3+}$ ions, which are the promising long persistence phosphors candidates for photovoltaics. We showed that the incorporation of boron into the compounds extends the afterglow from minutes to longer than 8 hours. To determine how the electronic structure is modified by the incorporation of boron into the matrix structure we applied electron energy-loss spectroscopy (EELS). Spectral features were interpreted qualitatively as the coordination fingerprints, using reference crystal phases, revealing that the boron is planar 3-fold coordinated, forming $BO_3$ units, therefore occupying the interstitial sites in this SA powders.

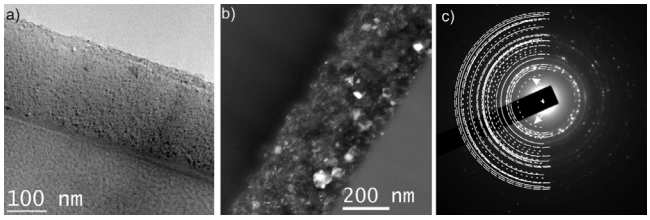


*Figure 5: TEM image of a) non-irradiated film and of the b) irradiated film. c) SAED pattern acquired from the irradiated film shows the presence of MgH2 (short dashed line), MgO (full line) and Mg (long dashed line) crystal phases.*

For the investigations of innovative materials on the micrometre and sub-micrometre scales we have implemented advanced analytical methods: high-resolution scanning electron microscopy (FEGSEM), qualitative and quantitative elemental electron-probe microanalysis (EPMA) with energy-dispersive and wavelength-dispersive X-ray spectroscopies (EDS, WDS) and electron backscatter diffraction (EBSD). These methods were modified and improved, taking into account the specific characteristics of individual materials in order to achieve precise, accurate and reliable analytical results.

Optimized analytical methods were applied for the research of a variety of materials: metallic and ceramic thin films, nanorods, nanoparticles, complex metallic alloys, magnetic materials, perovskite ferroelectrics. Using EBSD we have investigated the microcrystallographic properties of materials, i.e., the crystallinity, crystallographic orientation of phases and texture. In this way we have directly verified the presence of quasicrystals with the so-called "forbidden" 5-fold and 10-fold symmetries, which form in complex alloys, e.g., Al–Mn–Cu–Mg–Si.

We have collaborated with partners from industry and other research institutions performing the analyses and expertise related to the microstructural, quantitative elemental and crystallographic characterization of various materials. The main collaborations were realized with SwatyComet Maribor, Cinkarna Celje, UL-NTF Department of Materials and Metallurgy, University of Ljubljana, RC SIMIT Kidričevo, and the Faculty of Medicine, University of Ljubljana.

One of the important research areas of the department is the implementation of various electron-microscopy analytical techniques within **the existing EU project ESTEEM2**, such as electron-energy-loss spectroscopy (EELS), high-resolution scanning transmission electron microscopy (STEM, HAADF-STEM), electron holography and the mechanical preparation of the TEM samples. The implementation of various electron microscopy analytical techniques and the possibility for the researchers to access research infrastructure for electron microscopy within the Centre for Electron Microscopy and Microanalysis (CEMM) is of utmost importance for the research.

The researchers at our department are also strongly involved in the promotion of science. One of the activities is a series of scientific lectures for the general public within the action "**Science on the street, knowledge and ideas on the go**".

## Organization of conferences, congresses and meetings

1. AdSTEM 2014, European Workshop on Quantitative STEM Imaging, EELS and EDXS, Piran, Slovenia, 29 September–3 October 2014
2. 22nd International Conference on Materials and Technologies, Portorož, Slovenia (co-organizers), 20–22 October 2014
3. REPM2014, Rare Earth and Future Permanent Magnets and Their Applications Workshop 2014, Annapolis, USA, 15–23 August 2014 (members of the International Advisory Board)

4. EMAS 2014, 14th European Workshop on Modern Developments and Applications in Microbeam Analysis, Leoben, Austria, 21–24 September 2014, (members of Management Board of the European Microbeam Analysis Society)

## Awards and appointments

1. **Martina Lorenzetti**, Thomas Luxbacher, Spomenka Kobe, Saša Novak: »Zeta Potential: A Useful Tool to Interpret the Hydrothermally Treated Titanium Behaviour as Biomaterial«, Best oral presenation at the ESB2014 (European Society of Biomaterials) Conference, Liverpool, United Kingdom, 3 September 2014
2. **Ana Gantar**, Rok Kocen, Saša Novak: »Nanoparticulate bioactive glass-reinforced gellan-gum hydrogels for bone tissue engineering«", Best oral presentation at the 22nd Conference on Materials and Technology, Portorož, Slovenia, 20–22 October 2014

## Patent granted

1. Kristina Žužek Rožman, Paul McGuiness, Marko Soderžnik, Dejan Mir, Passive magnetic cradle with THWE mechanism of stopping and positioning, SI24202 (A), Urad RS za intelektualno lastnino, 30.4.2014.

# INTERNATIONAL PROJECTS

1. 7FP - ROMEO, Replacement and Original Magnet Engineering Options
   Prof. Spomenka Kobe
   European Commission
2. Selective Laser Melting (SLM) and Spark Plasma Sintering (SPS) of Cost Effective Rare-earth based Permanent Magnets for Electrical Machines
   Prof. Spomenka Kobe
   ABB Switzerland Ltd
3. 7FP - BioTiNet; Academic-Industrial Initial Training network on Innovative Biocompatible Titanium-based Structures for Orthopaedics
   Prof. Spomenka Kobe
   European Commission
4. 7FP - Fusion Expo; Fusion Expo Support Action under EFDA Work Programme, Task Agreement WP10-PIN-FUSEX
   Prof. Saša Novak Krmpotič
   Ministry of Education, Science and Sport
5. 7FP - NANOPYME; Nanocrystalline Permanent Magnets Based on Hybrid Metal-Ferrites
   Asst. Prof. Kristina Žužek Rožman
   European Commission
6. 7FP - ESTEEM 2; Enabling Science and Technology through European Electron Microscopy
   Prof. Miran Čeh
   European Commission
7. 7FP - MAG-DRIVE; New Permanent Magnets for Electric-Vehicle Drive Application
   Asst. Prof. Matej Andrej Komelj
   European Commission
8. 7 FP; ERA CHAIR ISO-FOOD - Era Chairs for Isotope Techniques in Food Quality, Safety and Traceability
   Prof. Saša Novak Krmpotič
   European Commission
9. Materials-PPPT-FU, EUROFUSION: WC and SiC Reinforced Tungsten
   Prof. Saša Novak Krmpotič
   European Commission
10. Enabling Research-1-FU, EUROFUSION: Advanced SiC/SiC Toward Implementation in Fusion Power Plants
    Dr. Aljaž Iveković
    European Commission
11. MODEF - Creazione e Sperimentazione Congiunta di Modelli per l'Ottimizzazione dell'Utilizzo di Energia Fotovoltaica
    Dr. Zoran Samardžija
    Unindustria Rovigo
12. COST MP1005, NAMABIO; From Nano to Macro Biomaterials (Design, Processing, Characterization, Modelling) and Applications to Stem Cells Regenerative Orthopaedic and Dental Medicine
    Prof. Saša Novak Krmpotič
    COST Office
13. COST ES1205; The Transfer of Engineered Nanomaterials from Wastewater Treatment & Stormwater to Rivers
    Prof. Saša Novak Krmpotič
    COST Office
14. CALGAD-X: New Calcium-Gadolinium-X Complex Metallic Alloys
    Prof. Spomenka Kobe
    Slovenian Research Agency
15. Study of Chemical Strain in Perovskites Doped With Aliovalent Cations by Applying In-Situ X-Ray Diffraction, Dilatometry and Advanced Transmission Electron Microscopy Techniques
    Asst. Prof. Sašo Šturm
    Slovenian Research Agency
16. Biomimetic Characterisation of Bioactive Composit Scaffolds for Bone and Osteochondral Tissue Repair
    Prof. Saša Novak Krmpotič
    Slovenian Research Agency
17. Development of Oxide Thermoelectric Materials for Waste-heat Recovery into Electricity
    Asst. Prof. Slavko Bernik
    Slovenian Research Agency
18. Characterisation of Growth Features and Planar Defects in Crystals Grown Under Hydrothermal Conditions
    Asst. Prof. Nina Daneu
    Slovenian Research Agency
19. Advanced Methods and Technologies for Processing of a New Generation of ZnO-based Varistor Ceramics
    Asst. Prof. Slavko Bernik
    SICCAS, Shangai, China
20. NSFM: Novel Smart Filtration Media
    Asst. Prof. Kristina Žužek Rožman
21. Services for the Exports
    Dr. Zoran Samardžija

# RESEARCH PROGRAM

1. Nanostructured Materials
   Prof. Spomenka Kobe

# R & D GRANTS AND CONTRACTS

1. Twinning, Epitaxy and Phase Transformations in Minerals
   Asst. Prof. Nina Daneu
2. Atomic-scale Studies of Initial Stages of Phase Transformations in Minerals
   Asst. Prof. Nina Daneu
3. Near-net Shape Nanoparticle-reinforced Polymer-composites for Highly-loaded Advanced Mechanical Components with Superior Tribological Performance
   Prof. Saša Novak Krmpotič
4. Electron Microscopy and Microanalysis of Materials on Submicrometer Scale
   Dr. Zoran Samardžija
5. Hydrothermal Synthesis of Strongly Adhered TiO2 Photocatalytic Coatings on Metallic Substrates
   Asst. Prof. Goran Dražić
6. Novel Functionalized Nanomaterials for Applications as Nano- or Biosensors/Actuators/Bioresponsive (Carrier) Systems
   Asst. Prof. Kristina Žužek Rožman

7. Structure and Chemical Composition Study of Surfaces and Interfaces with High Resolution Scanning Transmission Electron Microscopy at Atomic Level
   Asst. Prof. Aleksander Rečnik
8. Bio-responsive Magneto-optically Coupled Nanomaterial-based Systems for Innovative Skin Cancer Treatments
   Asst. Prof. Sašo Šturm
9. Microbial Adhesion Management on Material Surfaces
   Asst. Prof. Goran Dražić
10. High-coercivity Nd-Fe-B Bonded Magnets for Automotive Applications
    Prof. Spomenka Kobe
11. Protected Permanent Magnets for Advanced High-Temperature Applications
    Asst. Prof. Paul John McGuiness
12. Materials and Technologies for Applications of ZnO-based Thick-film Varistors and Oxide Thermoelectrics
    Asst. Prof. Slavko Bernik
13. Colour, Absorption and Protective Nanolayer Coatings for Aluminium Alloy
    Prof. Miran Čeh
14. Innovative Production Systems for Vaccines and Regenerative Medicine
    Asst. Prof. Aleksander Rečnik
15. Development of the Model of the System for Intelligent Support of the Selection of Suitable Powder Material when Developing Sintered Products
    Prof. Saša Novak Krmpotič
16. Modification of TiO2 Nanoparticle Surface: Prevention of Agglomeration and Preservation of Intrinsic Properties
    Asst. Prof. Aleksander Rečnik
17. Tridimensional Bioactive Glass and Biopolymer Composite Scaffolds for Treatment of Osteochondral Defects Developed due to the Articular Cartilage Lesions
    Dr. Nataša Drnovšek
18. Irradiation and Analysis of Si Samples
    Prof. Saša Novak Krmpotič
19. From the Synthesis of Metal Oxides to the Humidity and Oxygen Prototype Nanosensors
    Dr. Kristina Žagar

# NEW CONTRACTS

1. The Study of Self-cleaning and Abrasion Resisting Properties of Carbon-, Para-aramid- or Glass-fibre-based Composite Materials by Applying Photocatalytic and/or Mechanical Resistant Nanoparticles
   Asst. Prof. Sašo Šturm
   CDT Group, d. o. o.
2. Multipole Magnetisation of NdFeB Bonded Magnets for Rotor Application
   Prof. Spomenka Kobe
   Kolektor KFH, d. o. o.
3. Cooperation within the R&D Program of the Company Akripol
   Prof. Saša Novak Krmpotič
   Akripol d. o. o.

# VISITORS FROM ABROAD

1. Yibo Zhou, Shanghai Institute of Ceramics, Chinese Academy of Science – SICCAS, Shanghai, China, 16 January–11 July 2014
2. Francesco Gucci, Università di Torino, Turin, Italy, 4 March–31 May 2014
3. Prof. Jean-Marie Dubois, Institut Jean Lamour, Nancy, France, 17–22 January 2014, 30 March–4 April 2014, 3–4 June 2014, 17–19 July 2014, 27 August–3 September 2014
4. Prof. Michael Zehetbauer, Fakultät für Physik, Universität Wien, Vienna, Austria, 24 April 2014
5. Federica Pirani, Università di Torino, Turin, Italy, 2 April–30 June 2014
6. Luisa Belardi, Università di Torino, Turin, Italy, 2 April–30 June 2014
7. Prof. Michael Gasik, Aalto University Foundation, Aalto, Finland, 3–4 April 2014
8. Prof. Ikka Kangasniemi, ID Creations oy, Turku, Finland, 3–4 April 2014
9. Idris Sorar, Mustafa Kemal University, Department of Physics, Antalya/Hatay, Turkey, 19 May 2014
10. Dr. Thomas Luxbacher, Anton Paar, Graz, Austria, 3 June 2014
11. Prof. Kazuki Nakanishi, Department of Chemistry, Kyoto University, Kyoto, Japan, 4 June 2014
12. Stephen Kyle-Henney, TISICS Limited, Hampshire, United Kingdom, 16–17 June 2014
13. Prof. Frans Kools, Technische Universiteit Eindhoven, Eindhoven, The Netherlands, 26 June 2014
14. Dr. Richard Wheeler, Edinburgh Scientific, Edinburgh, United Kingdom, 11 August 2014
15. Michele Carenini, Edinburgh Scientific, Edinburgh, United Kingdom, 11 August 2014
16. Prof. Mehmet Ali Gulgun, Prof. Cleva Ow Yang, Meltem Asilturk in Yener Kuru, Sabanci University, Istanbul, Turkey, 29 September–8 October 2014
17. Samed Çetinkaya, Technology and R&D Application and Research Center, Mustafa Kemal University, Hatay, Turkey, 7–15 October 2014
18. Dr. Heike Schlörb, Institut für Metallische Werkstoffe – IMW, Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden - IFW Dresden, Germany, 22–27 October 2014
19. Dr. Milivoj Plodinec, Institut Rudjer Bošković, Zagreb, Croatia, 17–22 November 2014
20. Dr. Andreja Gajović, Institut Rudjer Bošković, Zagreb, Croatia, 22–28 December 2014
21. Koichi Sakuta, Technology Research Association of Magnetic Materials for High-Efficiency Motors - MagHEM, Tokyo, Japan, 18 November 2014
22. Hirokazu Kubo, Intermetallics Co. Ltd., Kyoto, Japan, 18 November 2014
23. Hiroyuki Kobayashi, T&T Innovations Inc., Otake, Japan, 18 November 2014
24. Noritsugu Sakuma, Toyota Motor Corporation, Shizuoka, Japan, 18 November 2014
25. Sho Goto, Denso Corporation, Kariya, Aichi, Japan, 18 November 2014
26. Shintaro Arakai, Daikin Industries Ltd., Osaka, Japan, 18 November 2014
27. Masaki Yasuoka, AIST, Nagoya, Japan, 18 November 2014
28. Kaoru Iizuka, Nedo, Japan, 18 November 2014
29. Dr. Laszlo Peter, Institute for Solid State Physics and Optics, Hungarian Academy of Sciences, Budapest, Hungary, 27–28 November 2014
30. Prof. Mariana Calin, Institut für Komplexe Materialien, Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden - IFW, Dresden, Germany, 18–20 December 2014

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6. Asst. Prof. Paul John McGuiness
7. Prof. Saša Novak Krmpotič
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27. Rok Kocen, B. Sc.
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34. Rok Rudež, B. Sc.
35. Nadežda Stanković, B. Sc.
36. Luka Suhadolnik, B. Sc.
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**Technical staff**
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## ORIGINAL ARTICLE


1. Muhammad Shahid Arshad, Darja Pečko, Sašo Šturm, Juan Escrig, Matej Komelj, Paul J. McGuiness, Spomenka Kobe, Kristina Žužek Rožman, "Angular dependence of the coercivity in electrodeposited Co-Pt nanostructures with a tube-wire morphology", In: Proceedings of the INTERMAG 2014, IEEE International Magnetics Conference, May 4-8, 2014, Dresden, Germany, *IEEE trans. magn.*, vol. 50, no. 11, pp. 2302904-1-2302904-4, 2014.
2. Muhammad Shahid Arshad, Sašo Šturm, Janez Zavašnik, Alvaro P. Espejo, Juan Escrig, Matej Komelj, Paul J. McGuiness, Spomenka Kobe, Kristina Žužek Rožman, "Effect of magnetocrystalline anisotropy on the magnetic properties of electrodeposited Co-Pt nanowires", *J. nanopart. res.*, vol. 16, pp. 2688-1-2688-16, 2014.
3. Muhammad Shahid Arshad, Janez Zavašnik, Sašo Šturm, Paul J. McGuiness, Spomenka Kobe, Kristina Žužek Rožman, "Microstructure and magnetic properties of electrodeposited Co-Pt nanowires (NWs) with diameters below 100 nm", *IEEE magn. lett. (Print),* vol. 5, art. no. 6700204, 2014.
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5. Nina Daneu, Aleksander Rečnik, Werner Mader, "Atomic structure and formation mechanism of (101) rutile twins from Diamantina (Brazil)", *Am. mineral.*, vol. 99, no. 4, pp. 612-624, 2014.
6. Ana Gantar, Lucilia P. da Silva, Joaquim M. Oliveira, Alexandra P. Marques, Vitor M. Correlo, Saša Novak, Rui L. Reis, "Nanoparticulate bioactive-glass-reinforced gellan-gum hydrogels for bone-tissue engineering", *Mater. sci. eng., C, Biomim. mater., sens. syst.*, vol. 43, pp. 27-36, 2014.
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9. Petra Jenuš, Darja Lisjak, Danjela Kuščer, Darko Makovec, Mihael Drofenik, "The low-temperature cosintering of cobalt ferrite and lead zirconate titanate ceramic composites", *J. Am. Ceram. Soc.*, vol. 97, no. 1, pp. 74-80, 2014.
10. Sanja Jevtić, Iztok Arčon, Aleksander Rečnik, Biljana Babić, Matjaž Mazaj, Jelena Pavlović, Danka Matijašević, Miomir Nikšić, Nevenka Rajić, "The iron(III)-modified natural zeolitic tuff as an adsorbent and carrier for selenium oxyanions", *Microporous and mesoporous materials*, vol. 197, pp. 92-100, Oct. 2014.
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14. Nina Kostevšek, Kristina Žužek Rožman, Darja Pečko, Boris Pihlar, Spomenka Kobe, "A comparative study of the electrochemical deposition kinetics of iron-palladium alloys on a flat electrode and in a porous alumina template", *Electrochim. acta*, vol. 125, pp. 320-329, 2014.
15. Matic Krivec, Ralph Dillert, Detlef W. Bahnemann, Alma Mehle, Janez Štrancar, Goran Dražić, "The nature of chlorine-inhibition of photocatalytic degradation of dichloroacetic acid in a $TiO_2$-based microreactor", *PCCP. Phys. chem. chem. phys.*, vol. 16, issue 28, pp. 14867-14873, 2014.
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17. Darja Lisjak, Petra Jenuš, Alenka Mertelj, "The influence of the morphology of ferrite nanoparticles on the directed assembly into magnetically anisotropic hierarchical structures", *Langmuir*, vol. 30, issue 22, pp. 6588-6595, 2014.
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19. Martina Lorenzetti, Eva Pellicer, Jordi Sort, Maria D. Baró, Janez Kovač, Saša Novak, Spomenka Kobe, "Improvement to the corrosion resistance of Ti-based implants using hydrothermally synthesized nanostructured anatase coatings", *Materials (Basel)*, vol. 7, no. 1, pp. 180-194, 2014.
20. Danijela Luković Golić, Jovana Ćirković, Maja Šćepanović, Tatjana Srećković, E. Longo, José A. Varela, Nina Daneu, Vojislav Stamenkovic, Goran Branković, Zorica Branković, "The modification of structural and optical properties of nano- and submicron ZnO powders by variation of solvothermal syntheses conditions", *J. nanopart. res.*, vol. 16, no. 10, pp. 2670-1-2670-11, 2014.
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25. Milivoj Plodinec, Andreja Gajović, Gregor Jakša, Kristina Žagar, Miran Čeh, "High-temperature hydrogenation of pure and silver-decorated titanate nanotubes to increase their solar absorbance for photocatalytic applications", *J. alloys compd.*, vol. 591, pp. 147-155, 2014.
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34. Magdalena Wencka *et al.* (19 authors), "Physical properties of the InPd intermetallic catalyst", *Intermetallics (Barking)*, vol. 55, pp. 56-65, 2014.
35. Janez Zavašnik, Nadežda Stanković, Muhammad Shahid Arshad, Aleksander Rečnik, "Sonochemical synthesis of mackinawite and the role of Cu addition on phase transformations in the FeS system", *J. nanopart. res.*, vol.16, no. 2, pp. 2223-1-2223-13, 2014.
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37. Daša Zupančič, Rok Romih, Horst Robenek, Kristina Žužek Rožman, Zoran Samardžija, Rok Kostanjšek, Mateja Erdani-Kreft, "Molecular ultrastructure of the urothelial surface: insights from a combination of various microscopic techniques", *Microsc. res. tech.*, vol. 77, issue 11, pp. 896-901, 2014.
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## Published Conference Contribution (invited lecture)

1. Zoran Samardžija, "Quantitative EPMA of $(1-x)Pb(Mg_{1/3}Nb_{2/3})O_3 xxPbTiO_3$ (PMNT) ferroelectric ceramics", In: *Book of tutorials and abstracts: practical aspects: including a session on Rare and noble elements: from ore deposits to high-tech materials* , Antwerp, EMAS, 2014, pp. 241-252.
2. Marko Soderžnik, Kristina Žužek Rožman, Spomenka Kobe, Paul J. McGuiness, "The grain-boundary diffusion process in Nd-Fe-B sinteerd magnets based on the electrophoretic deposition", In: *Proceedings*, 2014 International Conference on NdFeB Magnets: Supply chain, critical properties, & applications, March 2-5th, 2014, Ningbo, China, [S. l., s. n.], 2014, pp. 166-170.

## Published Conference Contribution

1. Slavko Bernik, Nives Novak Gramc, Matejka Podlogar, Aleksander Rečnik, Nina Daneu, "Low-voltage varistor ceramics with the addition of pre-reacted $Bi_2O_3 - TiO_2$ phases", In: *Conference 2014, proceedings*, 50th International Conference on Microelectronics, Devices and Materials, October 8 - October 10, 2014, Ljubljana, Slovenia, Marko Topič, ed., Polona Šorli, ed., Iztok Šorli, ed., Ljubljana, MIDEM - Society for Microelectronics, Electronic Components and Materials, 2014, pp. 221-226.
2. Mateja Košir, Nina Daneu, Aleksander Rečnik, Miran Čeh, Emmanuel Guilmeau, Slavko Bernik, "Thermoelectric ceramics based on the ZnO – $In_2O_3$ system", In: *Conference 2014, proceedings*, 50th International Conference on Microelectronics, Devices and Materials, October 8 - October 10, 2014, Ljubljana, Slovenia, Marko Topič, ed., Polona Šorli, ed., Iztok Šorli, ed., Ljubljana, MIDEM - Society for Microelectronics, Electronic Components and Materials, 2014, pp. 203-208.
3. Mojca Presečnik, Slavko Bernik, "Effect of processing methods on the microstructural development and thermoelectric properties of $Ca_3Co_4O_9$", In: *Conference 2014, proceedings*, 50th International Conference on Microelectronics, Devices and Materials, October 8 - October 10, 2014, Ljubljana, Slovenia, Marko Topič, ed., Polona Šorli, ed., Iztok Šorli, ed., Ljubljana, MIDEM - Society for Microelectronics, Electronic Components and Materials, 2014, pp. 209-214.
4. Rok Rudež, Piotr Markowski, Andrzej Dziedzic, Slavko Bernik, "Development of oxide-based thermoelectric microgenerators based on *p*-type $Ca_3Co_4O_9$ and *n*-type $(ZnO)_5 \cdot In_2O_3$ thermopiles", In: *Conference 2014, proceedings*, 50th International Conference on Microelectronics, Devices and Materials, October 8 - October 10, 2014, Ljubljana, Slovenia, Marko Topič, ed., Polona Šorli, ed., Iztok Šorli, ed., Ljubljana, MIDEM - Society for Microelectronics, Electronic Components and Materials, 2014, pp. 215-220.
5. Maša Zalaznik, Mitjan Kalin, Saša Novak, "Tribological behavior of PEEK/$MoS_2$ composites: influence of $MoS_2$ particles concentration and processing temperature", In: *Nordtrib 2014*, 16th Nordic Symposium on Tribology, 10-13 June 2014, Aarhus, Denmark, [Aarhus, Danish Technological Institute], 2014 (6 f.).
6. Maša Zalaznik, Mitjan Kalin, Saša Novak, Aljaž Iveković, "Improvement of tribological properties of PEEK with addition of $MoS_2$ particles", In: *Polytrib 2014: proceeding*, 1st International Conference on Polymer Tribology, 11th - 12th September 2014, Bled, Slovenia, Ljubljana, Slovenian Society for Tribology, 2014 (5 f.).
7. Maša Zalaznik, Mitjan Kalin, Saša Novak, Aljaž Iveković, "Tribological properties of poly-ether-ether-ketone (PEEK) produced at different processing temperatures", In: *Polytrib 2014: proceeding*, 1st International Conference on Polymer Tribology, 11th - 12th September 2014, Bled, Slovenia, Ljubljana, Slovenian Society for Tribology, 2014 (5 f.).

## Patent Application

1. Luka Suhadolnik, Matic Krivec, Miran Čeh, Kristina Žagar, Goran Dražić, *Reactor,* PCT/EP2014/069011, European Patent Office, 5.9.2014.

## Patent

1. Kristina Žužek Rožman, Paul McGuiness, Marko Soderžnik, Dejan Mir, *Passive magnetic cradle with THWE mechanism of stopping and positioning,* SI24202 (A), Urad RS za intelektualno lastnino, 30.4.2014.

## Mentoring

1. Matic Krivec, *Synthesis and characterization of nanoparticles, nanostructures and micro-devices based on photocatalytic $TiO_2$:* doctoral dissertation, Ljubljana, 2014 (mentor Goran Dražić).
2. Alenka Lenart, *Structural and chemical investigations of twinning in natural and synthetic crystals of a low-temperature form of quartz:* doctoral dissertation, Ljubljana, 2014 (mentor Breda Mirtič; co-mentor Sašo Šturm).
3. Martina Lorenzetti, *Synthesis and characterisation of nanostructured bioactive anatase coating on ti-alloys for biomedical applications:* doctoral dissertation, Ljubljana, 2014 (mentor Saša Novak Krmpotič; co-mentor Spomenka Kobe).
4. Darja Pečko, *Electrochemical study and synthesis of Fe-Pd ferromagnetic nanowires for future magnetic applications:* doctoral dissertation, Ljubljana, 2014 (mentor Spomenka Kobe; co-mentor Kristina Žužek Rožman).
5. Matejka Podlogar, *Synthesis of micro- and nano-sized ZnO particles from solutions:* doctoral dissertation, Ljubljana, 2014 (mentor Slavko Bernik).
6. David Sojer, *Protection of Nd-Fe-B-based melt-spun ribbons using nanoscale sol-gel derived films of $SiO_2$ and $Al_2O_3$:* doctoral dissertation, Ljubljana, 2014 (mentor Paul J. McGuiness; co-mentor Irena Škulj).
7. Blaž Goričar, *Applying the grain-boundary diffusion process using electrophoretic deposition to selected regions of a Nd-Fe-B magnet:* master's thesis, Maribor, 2014 (mentor Matjaž Kristl; co-mentor Paul J. McGuiness).