CENTRE FOR ELECTRON MICROSCOPY AND MICROANALYSIS (CEMM)

The Centre for Electron Microscopy and Microanalysis (CEMM) is an instrumental centre at the JSI that combines analytical equipment in the field of electron microscopy and microanalysis. Access to the research equipment of CEMM is provided to other JSI departments as well as other research institutions, universities and industrial partners. The equipment at the CEMM is used by researchers, interested in the morphology and structural and chemical characterization of materials between the micrometre and the atomic levels. At the CEMM there are three scanning electron microscopes (SEM) JSM-5800, JXA-840A, JSM-7600F, two transmission electron microscopes (TEM) JEM-2100 (CO NIN) and JEM-2010F, and the equipment for the TEM and SEM sample preparation. Centre of Excellence NAMASTE contributed to the equipment for electron microscopy with a CCD camera and an ADF (annular dark field) detector for JEM-2010F microscope and an EBSD system for the JSM-7600F. Additionally, the IJS is a co-owner of a JEM-ARM200F (transmission electron microscope with atomic resolution) at the Chemical Institute. Furthermore, CEMM helps with the maintenance of the dual beam SEM/FIB (focused ion beam) microscope (Helios Nanolab 650) at Nanocenter department.

The research involving the staff and equipment at the CEMM is diverse regarding the investigated materials and the methods used. Scanning electron microscopy is employed to observe the morphology and structure of the surfaces and for the microstructural investigation and determination of the chemical composition. Sample that are mostly investigated are ceramics (polycrystalline oxide and non-oxide compositions), nanostructured materials, metallic magnetic materials, metals, alloys glass, etc. All of the scanning electron microscopes in the CEMM are equipped with an energy-dispersion (EDXS) and / or wavelength dispersion (WDXS) spectrometer for X-rays, allowing non-destructive determination of the chemical composition of the investigated materials. The scanning electron microscope JSM-7600F is additionally equipped with an electron back-scattered diffraction (EBSD) detector and an electron lithography system.

Transmission electron microscopy (TEM) provides an insight into the structure of the material on the nano-scale (atomic level). Transmission electron microscopy enables structural and chemical analyses of the grain boundaries and study of precipitates, planar defect and dislocation determination. Instead of ceramic samples also different material and structures are investigated like thin film on different substrates, alloys, delicate metallic magnetic materials, polymers, etc. Transmission electron microscope JEM-2100 is equipped with an EDXS spectrometer and a CCD camera, and the JEM-2010F is additionally equipped with a scanning transmission electron (STEM) unit, EDXS and EELS (electron energy loss) spectrometers, and a CCD camera. The CEMM also manages the necessary equipment for the SEM and TEM sample preparation.

The operation of the Centre is managed by properly trained employees. Besides maintenance of the equipment, among other CEMM activities are trainings for new operators, organization of workshops and conferences on the topic of electron microscopy, providing services for industrial partners and the introduction of new analytical techniques. CEMM personnel are also responsible for the demonstration of electron microscopy to the general public in the scope of organized visits to the IJS, as well through publications in traditional and digital media. For users of microscopes CEMM organized the 6th and 7th workshop (SEM sample preparation and scanning electron microscopy with microanalysis - EDS and WDS). The aim of the workshops is to present the operation and handling of the equipment, moreover presenting properly prepared SEM samples (Figure 1).

Figure 1: 7th CEMM Workshop (Koblar M, CEMM)

CEMM workshops for new operators.
The aim of the workshops is to present the operation and handling with the equipment and to present properly prepared samples.

ISO-FOOD Spring School

The ISO-FOOD Spring School and Workshop was devoted to various perspectives of nanoparticles in food that may appear during production, packaging or cooking. They can be added inadvertently or deliberately to improve the taste, color, etc. Due to their size they are difficult to detect with simple experiments in the laboratory, so the electron microscopes were used. The size evaluation and chemical identification was defined using SEM,TEM and EDXS. Figure 2. shows today's popular fountains that are used at birthday parties. Particles that are produced can either pass into the respiratory system and a part of them can be transferred on the food – e.g. cake. We have shown that particles are in different ranges of magnitude, from a few nanometers (TEM image) to micrometers (SEM image).

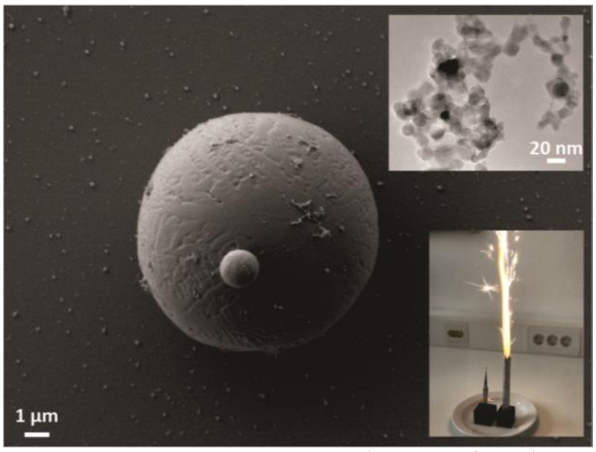


Figure 2: Fontana's experiment. (Koblar M, CEMM).

In a collaboration with projects, ISO-FOOD and SmartNanoTox, we have studied and analysed nanoparticles, that are harmful for health.

SmartNanoTox

SmartNanoTox project exploring interactions between nanomaterials and cell (the identification of molecular initiating events). The production of new materials, in a world of science, is growing and their impact on health is often unexplored. Project includes research of nanoparticles inhalation and cardiovascular disease. Major part of research is molecular mechanism explanation. CEMM has collaborated with TiO_2 nanoparticles analysis using transmission electron microscope JEM-2100. We have been focused on initiating characteristics and typical morphology of host material. With F-5 department (Department of Solid State Physics) few TEM samples of LA-4 epithelial cell layers were prepared and analysed. TEM study indicated typical aggregate of LA-4 cell membrane and nanoparticles after 2 days incubation of LA-4 cell layer with nanoparticles in a complete cell-culturing medium. Nanoparticles, lipids and proteins can be identified with crystalline tube structures, surrounding amorphous layer and the almost-round dark objects. (Figure 3)

Results and discussions, of electron microscopy and other new technology combinations, are published in a renowned journal (Urbancic. I.; Garvas. M.; Kokot. B.; Majaron. H; Umek. P; Cassidy. H.; Škarabot. M.; Schneider, F.; Galiani, S.; Arsov. Z.; Koklic. T.; Matallanas. D.; Čeh. M.; Muševič. I.; Eggeling. C.; Štrancar. J. Nanoparticles Can Wrap Epithelial Cell Membranes and Relocate Them Across the Epithelial Cell Layer. Nano Lett. 2018, 18, 5294-5305).

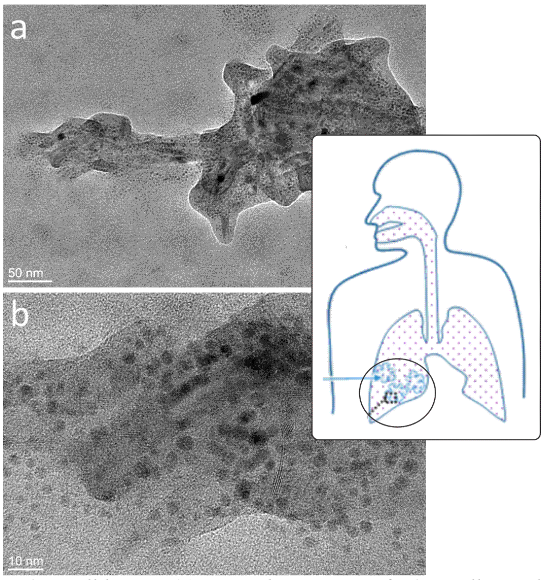


Figure 3: TEM analysis of proteins form LA-4 cell layers. (a) Typical aggregate of LA-4 cell membrane and (b) nanoparticles after 2 days of incubation *(Drev S, CEMM)*

Individual research works and analysis using CEMM equipment

Multilayer of Ag and Ni sputtered on Si substrate. TEM sample with cross-section technique was prepared to analyse preferential sputtering effect in depth profiling of multilayers. Transmission electron microscope JEM-2010F was used to detected eight layers of Ag and Ni. Multilayer profile showed constant thickness of particular layers. According to AES (Auger electron spectroscopy) of (Ag/Ni)x4/Si multilayer structure, the thickness of layers are similar. Results of AES and TEM techniques confirmed a thickness of layers (20-25nm). (Hofmann. S.; Zhou. G.; Kovač. J.; Drev. S.; Lian. S. Y.; Lin. B.; Liu. Y.; Wang. J. Y. Preferential sputtering effects in depth profiling of multilayers with SIMS, XPS and AES. Applied Surface Science (in press). (Figure 4)

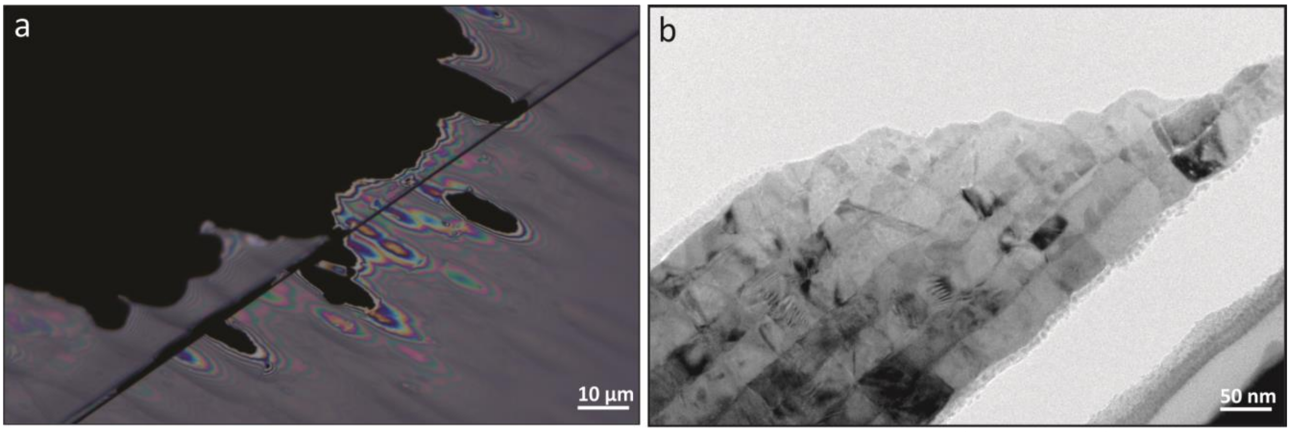


Figure 4: Study of preferential sputtering effects in depth profiling of multilayers. (Drev S, CEMM)

Solid solution of $Pb(Mg_{1/3}Nb_{2/3})O_3$–$xPbTiO_3$ is a perovskite relaxor ferroelectric system with large polarization and strain responses to external fields. These are assigned to its special polar nanoscale distortions, which influence the movement of microscale ferroelectric domains. Transmission electron microscope JEM-2100 was used to detect characteristic 90° tetragonal domain structure. Within the 90° domains, 180° domains (magnification: characteristic zig-zag pattern) and polar nanoregions are embedded. (Otonicar. M.; Ursic. H.; Dragomir. M.; Bradesko. A.; Esteves. G., Jones. J. L.; Bencan. A.; Malic. B.; Rojac. T. Multiscale field-induced structure of (1-x)Pb(Mg1/3Nd2/3)O3-xPbTiO3 ceramics from combined techniques. Acta materialia. 2018, 154, 14-24). (Figure 5)

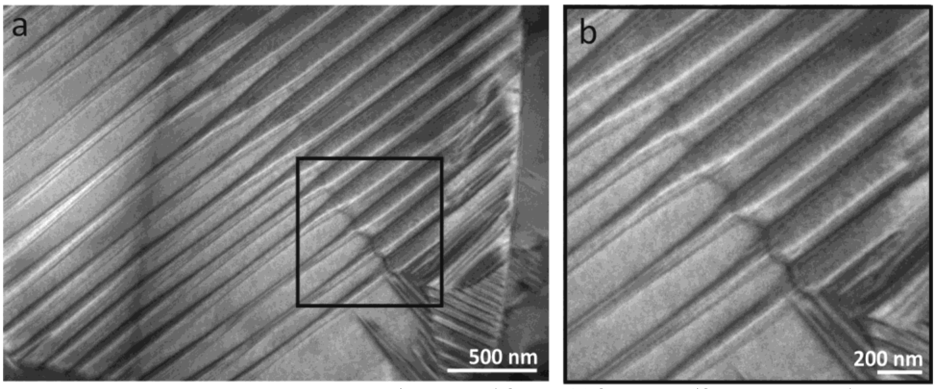
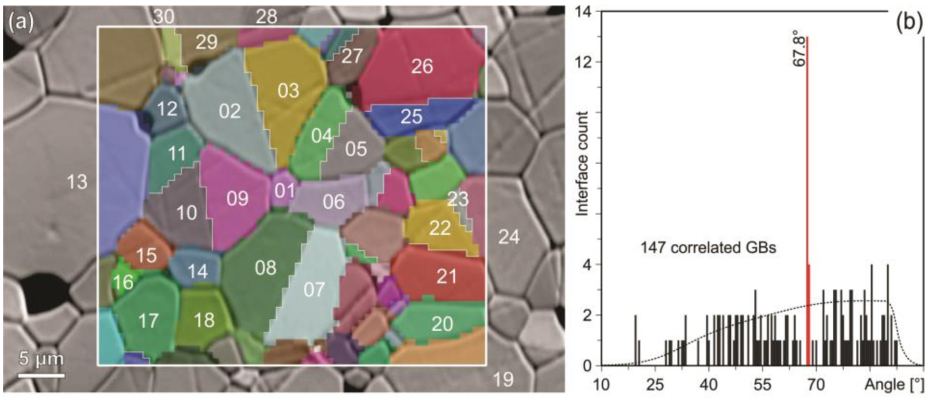


Figure 5: Domain structure of $Pb(Mg_{1/3}Nb_{2/3})O_3$–$xPbTiO_3$ sample (Otoničar M, K5)

Scanning electron microscopy JSM-7600F was used to analyze twin relation in SnO_2 ceramics. EBSD map produced by crystallographic orientation data from 3231 Kikuchi patterns recorded within the outlined area. Selected grains are indicated by numbers and their angular relations are listed below. White lines indicate boundaries where the grains are met in {101} twin orientation. Also MacKenzie plot of relative frequency of incidences among the SnO_2 grains has been done. Red lines indicate the incidence corresponding to the complementary angle of {101) twins in SnO_2. (Tominc. S.; Rečnik. A.; Samardžija. Z.; Dražić. G.; Podlogar. M.; Bernik. S.; Daneu. N. Twinning and charge compensation in Nb_2O_5–doped SnO_2–CoO ceramics exhibiting promising varistor characteristics. Ceramics International. 2018, 44, 1603-1613). (Figure 6)



Pair	01-02	02-03	03-04	04-05	07-08	08-09	09-10	10-11	09-11	11-12	20-21	21-22	22-23	23-24	22-24
Angle	80.2	67.8	85.9	67.7	67.7	72.6	67.6	68.3	44.2	33.1	33.9	68.0	67.9	68.3	90.0

Figure 6: Twin relation analysis in SnO_2 ceramics (Samardžija Z, K7)

Superparamagnetic nanoparticles are now a day in intensive investigation, being used for a variety of biological applications (bio separations), *in vitro* cell manipulation, and magnetically targeted drug delivery. (Kralj. S. and Makovec. D. The chemically directed assembly of nanoparticle clusters from superparamagnetic iron-oxide nanoparticles. Royal Society of Chemistry. 2014, 4, 13167-13171). Chains of superparamagnetic nanoparticles are coated with a silica layer containing large, radially-oriented pores. Study of superparamagnetic samples have been made by transmission electron microscope JEM-2100. (Figure 7)

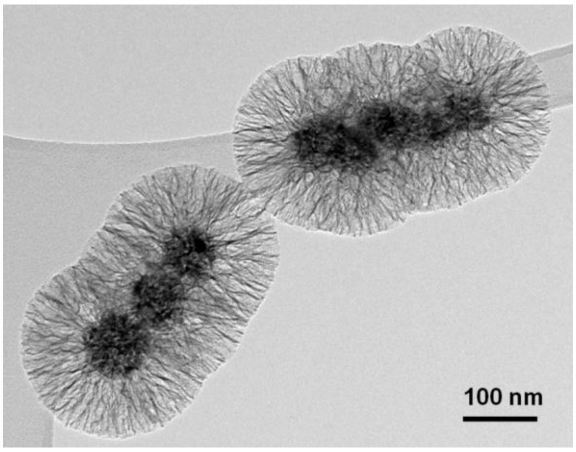


Figure 7: Superparamagnetic nanoparticles (Kralj S, K8)

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BIBLIOGRAPHY