CENTRE FOR ELECTRON MICROSCOPY AND MICROANALYSIS (CEMM)

The Centre for Electron Microscopy and Microanalysis (CEMM) is an instrumental centre at the JSI that comprises analytical equipment for electron microscopy and microanalysis. Access to the research equipment within the CEMM is available for the JSI departments as well as for other research institutions, universities and industrial partners. The equipment in the CEMM is used by researchers, interested in the morphology and structural and chemical characterization of materials on micrometre and atom level. CEMM comprises four scanning electron microscopes (JSM-7600F, Verios G4 HP, Quanta 650, JSM-5800), two transmission electron microscopes (JEM-2100 (CO NiN) and JEM-2010F), and the equipment for the TEM and SEM sample preparation. Additionally, the IJS is co-owner (20%) of a JEM-ARM200CF at the Chemical Institute.

Figure 1. Centre for Electron Microscopy and Microanalysis (CEMM).

Figure 2. Scanning electron microscope JSM-7600F.

High-resolution scanning electron microscope Verios G4 HP, Thermo Fisher Scientific (Figure 3) is unique in this part of Europe and provides extremely high imaging resolution at low accelerating voltages. It also features automatic sample insertion and the ability to observe non-conductive specimens with exceptional Z-contrast even at low voltages. In addition to the highly sensitive EDXS detector, the microscope is equipped with a transmission detector (STEM) as well.

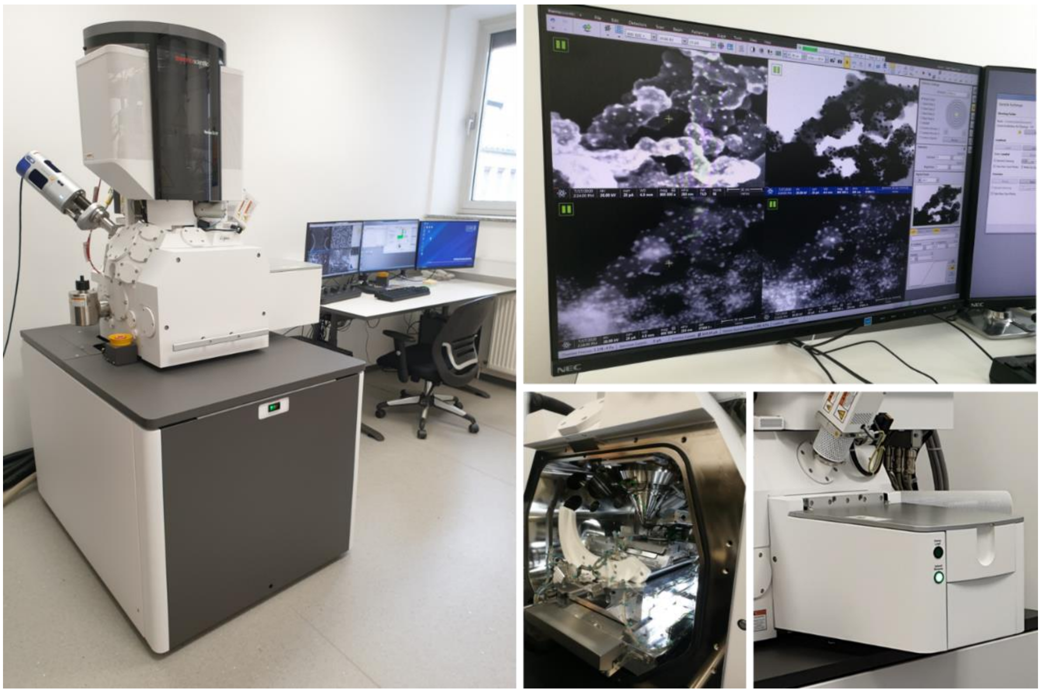

Figure 3. Scanning electron microscope Verios 4G HP.

Scanning electron microscope Quanta 650, Thermo Fisher Scientific (Figure 4) is operational in three vacuum ranges that are achieved through differential pumping. This allows us to investigate a wide range of materials, both conductive and non-conductive.

Figure 4. Scanning electron microscope ESEM Quanta 650.

The research carried out using the equipment in the CEMM is diverse due to many different research topics of the JSI departments:

- Scanning electron microscopy is employed to observe the morphology and the structure of surfaces and for the microstructural investigation and determination of the chemical composition of investigated materials. Samples that are most frequently 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. The equipment of the Verios 4G HP microscope enables the observation of the morphology of nanoparticles and samples which are sensitive to electron doses. The Quanta 650 microscope allows the observation of larger, conductive or non-conductive samples.
- Transmission electron microscopy (TEM) provides an insight into the structure of the material on the nano-scale (atom level). Transmission electron microscopy enables structural and chemical analyses of nanostructured phenomena, such as grain boundaries, precipitates, planar defect, dislocations, etc.. Materials which are investigated include thin films on different substrates, alloys, metallic magnetic

materials, dielectric materials, ferroelectrics, 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 ARM200CF is a dedicated scanning transmission electron microscope with ADF, HAADF, ABF STEM detectors and GIF system.

- The CEMM also manages the necessary equipment for the SEM and TEM sample preparation.

The operation of the Centre is managed by the CEMM personnel. Besides maintenance of the equipment, other CEMM activities include, among other, training of new operators, organization of workshops and conferences on the topic of electron microscopy, providing services for industrial partners and implementation of new analytical techniques. CEMM personnel are also responsible for the dissemination of electron microscopy techniques to the general public in the scope of organized visits to the IJS, as well through publications in traditional and digital media.

Examples of microstructural and nanostructural investigations of materials using the CEMM equipment

The examples of analyses of structural and chemical characterisations of different materials using electron microscopy techniques were performed by the operators from different JSI departments and by the CEMM personnel.

1. Dissolved active pharmaceutical substance

Analysis of dissolved pharmaceutical active substance was performed in the scanning electron microscope Verios 4G HP in the SEM mode, STEM bright field mode and STEM HAADF mode (Figure 5).

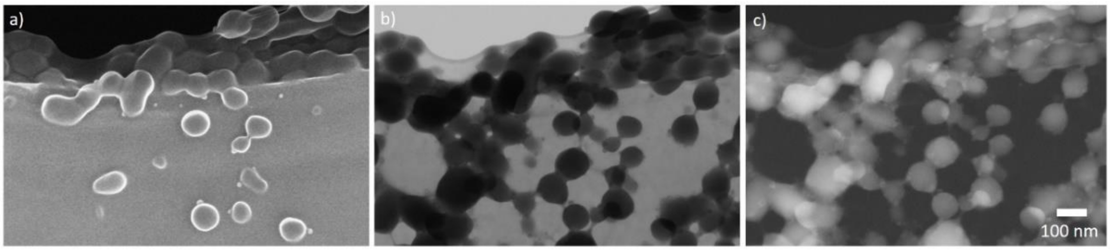

Figure 5. Dissolved active substance image a) SEM mode b) STEM bright field mode c) STEM HAADF mode (Jitka Hreščak, CEMM, SEM Verios G4 HP).

2. Sliding surface of a cross-country ski

Polymer coatings for skis were analysed in a scanning electron microscope before and after wax coating. The work was done in collaboration with the Faculty of Polymer Technology in Slovenj Gradec and the Ski Association of Slovenia (Figure 6).

Ref.: M. Plesnik, diploma work, 2021, 56 pages

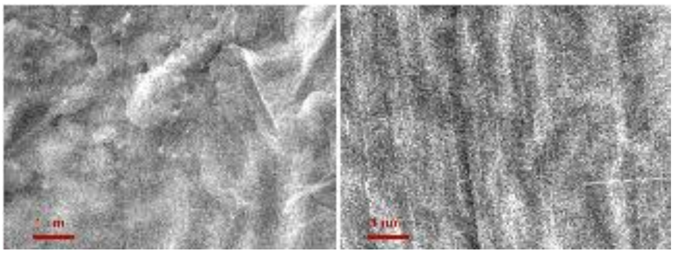

Figure 6. SEM images of sliding surface of cross-country ski. Before (left) and after (right) the ski surface was waxed with a wax for dry snow (Polona Umek, IJS-F5, Miha Plesnik).

3. Silane snowflake

SEM micrograph of silane polycondensation on a glass substrate is shown in Figure 7.

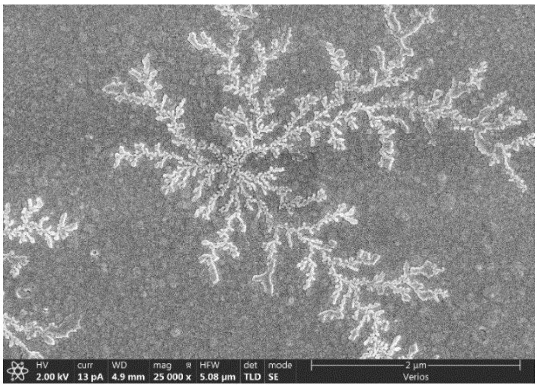

Figure 7. SE SEM image of 'silane snowflake' (Darja Lisjak, K8).

4. Rutile nanorods

High-resolution SE micrograph of the termination of rutile nanorods is shown in Figure 8. Image was recorded at 200.000x magnification in the scanning electron microscope Verios 4G HP.

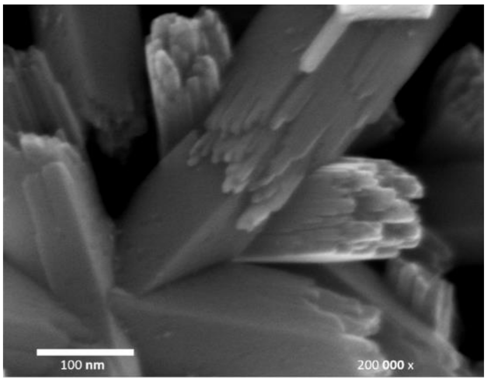

Figure 8. SEM image of rutile nanorods (Zoran Samardžija, K7, Verios G4 HP).

5. Characterization of nanocomposite based on graphene and polypyrrole nanoballs

The morphological features of PPy@Graphene were observed using a high-resolution transmission electron microscope (HRTEM) (JEM 2100, JEOL Ltd.) and an AG-Ultra 55 (Zeiss) field-emission scanning electron microscope (FESEM) (Figure 9).

Ref.: Vir: J. Casanova-Chafer, P. Umek, S. Acosta, C. Bittencourt, E. Llobet, ACS applied materials & interfaces, ISSN 1944-8244, 2021, vol. 13, 34, str. 40909%40921.

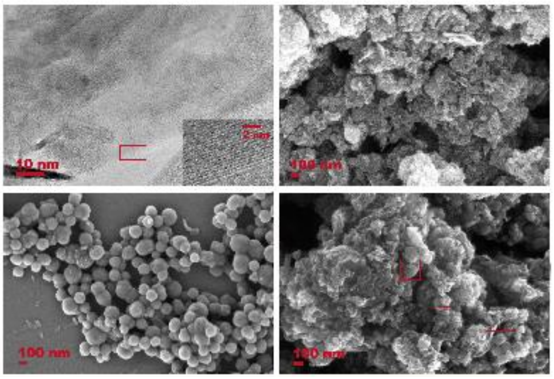

Figure 9. Up: TEM and SEM images of bare graphene. The inset in the TEM image shows a layered graphene structure (the red frame in the image indicates the area of the inset). Bottom left: SEM images of synthesized polypyrrole nanoparticles (PPy NPs), Bottom right: graphene nanoflakes decorated with PPy NPs.Red squares help spotting PPy nanoparticles on loaded graphene (Polona Umek, JSI-F5, AG-Ultra 55, JEM-2100).

6. Morphological characterization of WS_2 nanostructure

The microstructure of the WS_2 was characterized using scanning electron microscopy SEM-FEI Quanta 600. High-resolution transmission electron microscopy (HRTEM) was used to analyse crystal structure at the atomic level (Figure 10).

Ref.: A. Alagh, F.E. Annanouch, P. Umek, C. Bittencourt, A, Sierra-Castillo, E. Haye, J.F. Colomer, E. Llober, Sensors and actuators. B, Chemical, 2021, vol. 326, str. 128813-1-128813-11.

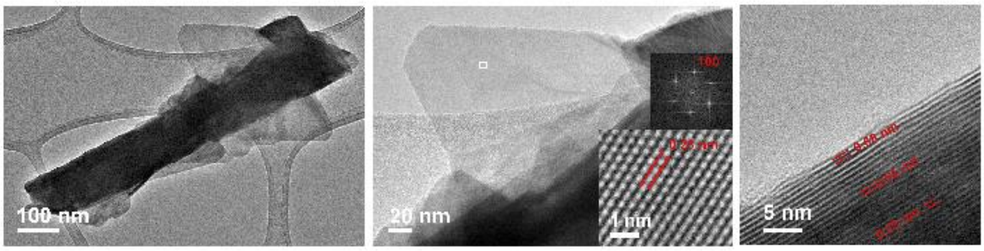

Figure 10. Left: TEM image of WS_2 nanoneedle with diameter of 125 nm and length of 800 nm.; Middle: triangular WS_2 nanosheet growing from the nanoneedle's longest side. White frame in the image indicates the area where HRTEM image was taken (bottom inset), upper insert is the corresponding FFT pattern. Right: HRTEM images of a side wall of the WS_2 nanoneedles (Polona Umek, JSI-F5, Quanta 600, JEM-2100)

7. Ferroelectric domains in ceramics

SEM analysis of ferroelectric domains in $Pb(Mg_{1/3}Nb_{2/3})O_3$-$0.4PbTiO_3$ ceramics in virgin state and after ex-situ applied electric field of ~10 kV/cm is shown in Figure 11.

Ref.: M. Otoničar et al., Open Ceramics, 7 (2021), 100140

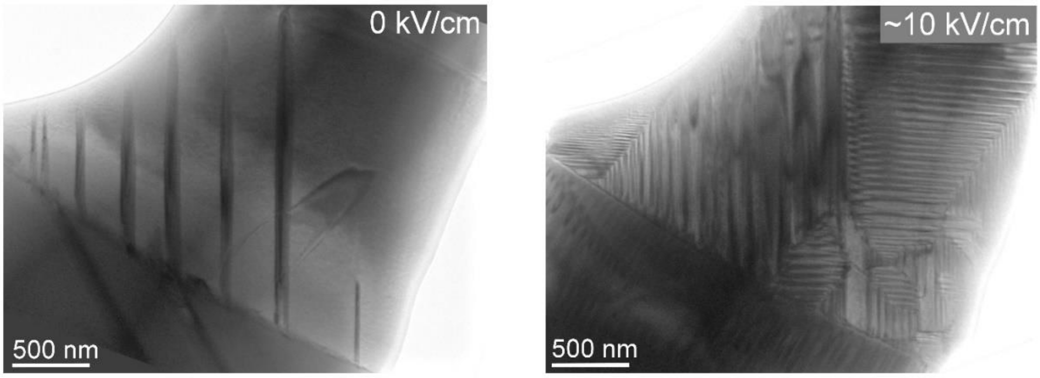

Figure 11. SEM image of ferroelectric domains in $Pb(Mg_{1/3}Nb_{2/3})O_3$-$0.4PbTiO_3$ ceramics in virgin state (left) and after ex-situ applied electric field of ~10 kV/cm (right) (Mojca Otoničar, K5).

8. EDS analysis of Ti/V layer in a multilayer ceramic component

EDS mapping was used to detect Ti/V layers within the multilayer ceramic component (Figure 12).

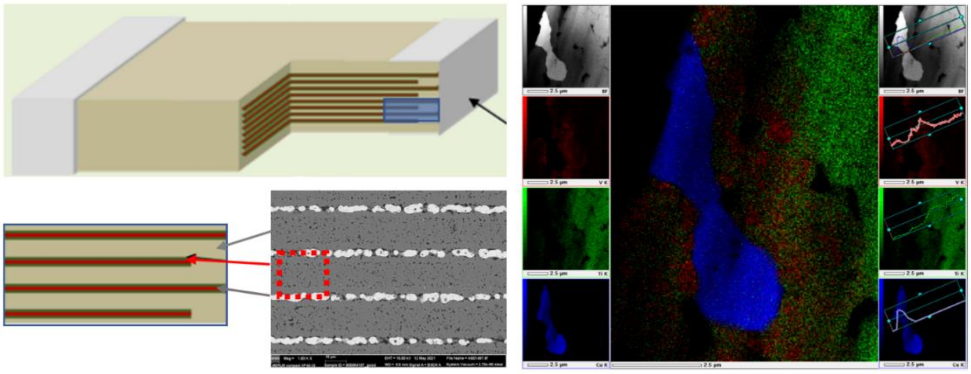

Figure 12. EDS mapping of the multilayer (Sandra Drev, CEMM, JEM-ARM200F).

9. STEM analysis of LSCO/(100)LSGM thin film

High-angle annular dark-field (HAADF) STEM and Annular bright-field (ABF) of LSCO/(100)LSGM thin film deposited by PLD technique showed ordering free zone between LSCO and LSGM layers (Figure 13).

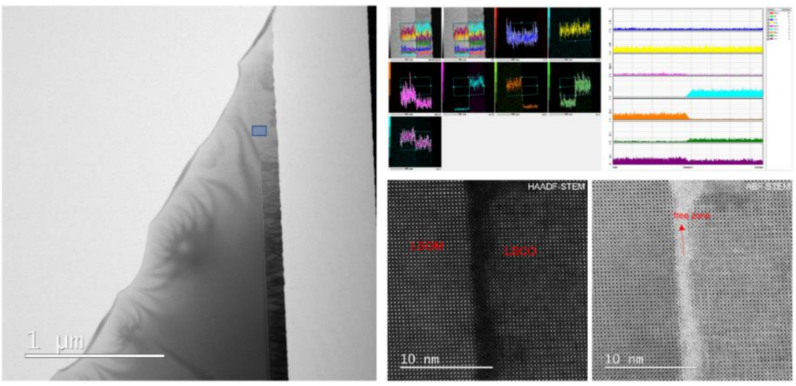

Figure 13. HAADF STEM and ABF STEM images of free zone in LSCO/(100)LSGM thin film (Sandra Drev, CEMM, JEM-ARM200F).

10. CeO_2 nanoparticles dissolution

TEM analysis of CeO_2 nanoparticles taken from freshwater shrimp was performed to study the differences between the radiolabelled nanoparticles from the original batch with the original nanoparticles (Figure 14).

Ref.: S. Schymura, I. Rydkin, S. Uygan, S. Drev, R. Podlipec, T. Rijavec, A. Mansel, A. Lapanje, K. Franke, M. Strok, Environmental Science Nano, 2021, 8, 1934

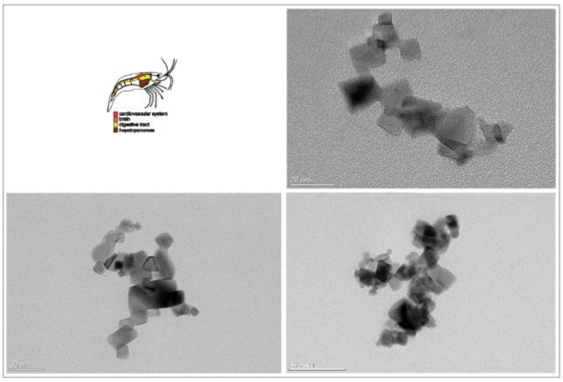

Figure 14. TEM images of CeO_2 NPs, (top left) pristine CeO_2 NPs, (top right) [Ce-141]CeO_2 and (bottom) [Ce-139/Ce-141]CeO_2 NPs (Sandra Drev, CEMM, JEM-2100).

11. A study of Sn-rich inversion boundary in ZnO

Quantitative HRTEM study of Sn-rich inversion boundary in ZnO showed two different short-range arrangements of cations in the IB-plane. Cation arrangements (stripes and zigzag) are controlled by the IB chemistry and 6-fold in-plane symmetry limitations (Figure 15).

Ref: Ribić et al., Acta materialia, 53 *(2021), 237-252.*

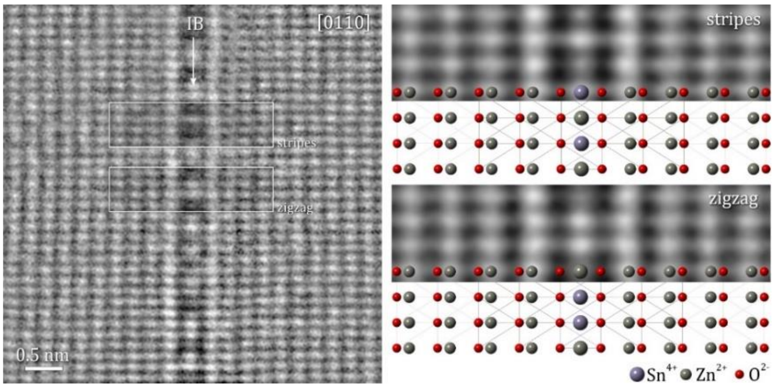

Figure 15. The quantitative HRTEM study of the Sn-rich IB in [0110] projection. (a) Highresolution HRTEM image with the evident cation distribution along the IB-plane. Two typical sequences are observed (outlined). The simulated HRTEM images based on (b) zigzag, and (c) stripes cation ordering with overlaid structure models. Simulated images were calculated for 2.8 nm thick crystal and a focus value of -72 nm. (Tina Radošević, F7/F9, Vesna Ribić).

12. TEM study of Pt/STO

TEM study of the position of Pt/STO interfacial region with the superimposed structural models viewed in the $[110]_{Pt}$ and $[110]_{STO}$ zone axes (Figure 16).

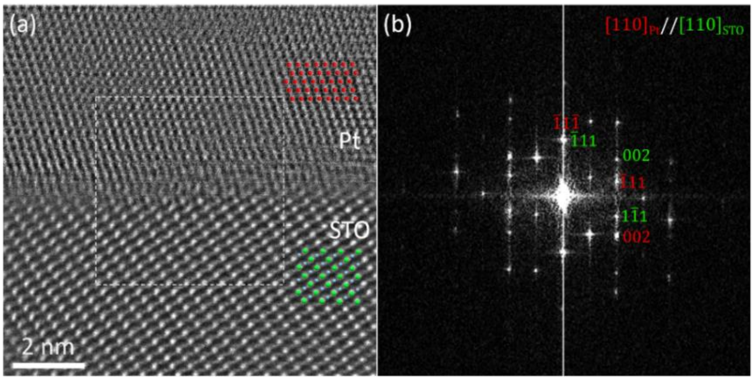

Figure 16. HAADF-STEM image of the Pt/STO interfacial region with the superimposed structural models viewed in the [110]Pt and [110]STO zone axes. Pt, Sr and Ti atoms are shown in red, green and blue. (b) The corresponding FFT pattern obtained from the marked area at (a) (Sorour Semsari Parapari, K7).

13. TEM study of $Al_{13}Co_4$ intermetallic alloy

Atomically-resolved HAADF-STEM image of $Al_{13}Co_4$ intermetallic showing the atom positions within the structure (Figure 17).

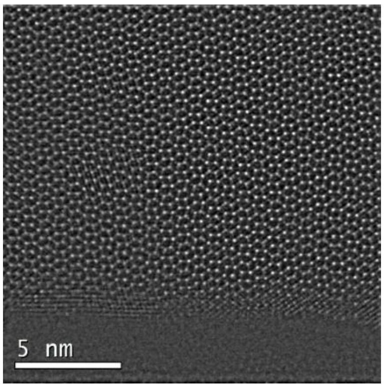

Figure 17. Atomically-resolved HAADF-STEM image of $Al_{13}Co_4$ intermetallic alloy (Sorour Semsari Parapari, K7).

14. TEM study of Wulfenite ($PbMoO_4$) crystal

Atomically-resolved HAADF-STEM image of Wulfenite ($PbMoO_4$) crystal showing the arrangement of atoms in the unit cell. Heavier Pb atoms are resolved as brighter dots (Figure 18).

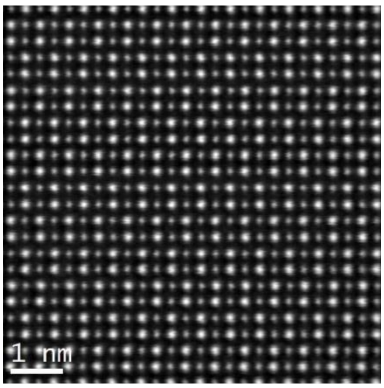

Figure 18. Atomically-resolved HAADF-STEM image of Wulfenite ($PbMoO_4$) crystal. Pb and Mo atoms are visible as bigger and smaller dots, respectively (Sorour Semsari Parapari, K7).

EMPLOYEES

1. **Prof. Miran Čeh, head**
2. Dr. Sandra Drev
3. Dr. Jitka Hreščak
4. Andreja Šestan Zavašnik, univ. dipl. inž. kem. inž..
5. Petra Drnovšek, dipl. graf. in med. teh.
6. Aleksander Učakar, mag. inž. metal. in mater.