PERIODICUM BIOLOGORUM
VOL. 125, No 1–2, 11–14, 2023
DOI: 10.18054/pb.v125i1-2.25775

UDC 57:61
CODEN PDBIAD
ISSN 0031-5362

Review

SEM and TEM investigations of materials at the Centre for Electron Microscopy and Microanalysis (CEMM)

MIRAN ČEH[1,2*]
JITKA HREŠČAK[1]
SANDRA DREV[1]

[1] Centre for Electron Microscopy and Microanalysis (CEMM), Jožef Stefan Institute, Ljubljana, Slovenia

[2] Nanostructured Materials, Jožef Stefan Institute, Ljubljana, Slovenia

***Correspondence:**
Miran Čeh
E-mail address: miran.ceh@ijs.si

Received March 15, 2023
Accepted March 16, 2023

INTRODUCTION

Modern *state-of-the-art* electron microscopy (EM) techniques are among the most versatile and useful analytical methods for non-destructive morphological, structural, and chemical characterization of materials. Both, scanning electron microscopy techniques (SEM) and transmission electron microscopy techniques (TEM), in essence complementary analytical methods, are capable of providing insight into the surface morphology, structure and chemical composition of materials from micrometer range to sub-atomic range. The importance of electron microscopy techniques in materials science have been readily recognized by the researchers of the Jozef Stefan Institute very early after its foundation in 1949 which resulted in the purchase of the first transmission electron microscopy Carl Zeiss EM-8 already in 1954. During the following years many researchers and institute directors have put a lot of effort into building and developing EM research infrastructure at the institute with accompanying expertise.

Today, the Centre for Electron Microscopy and Microanalysis (CEMM) is a modern infrastructure instrumental facility that comprises all relevant analytical equipment for electron microscopy and microanalysis at the institute level. 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 not only on national level but also internationally, based on an open access principle. The international component of the CEMM is additionally stressed by being a member of the ESTEEM consortium through participation in all three Infraia EU projects, i.e., ESTEEM, ESTEEM2 and current ESTEEM3 projects. CEMM comprises four scanning electron microscopes (JSM-7600F, Verios G4 HP, Quanta 650, JSM-5800), four transmission electron microscopes (JEM-2100, JEM-2010F, Spectra 300, Talos), and the equipment for the TEM and SEM sample preparation. Additionally, the IJS is co-owner (20%) of a JEM-ARM-200CF at the Chemical Institute.

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 is also responsible for the dissemination of electron microscopy techniques to the general public in the scope of

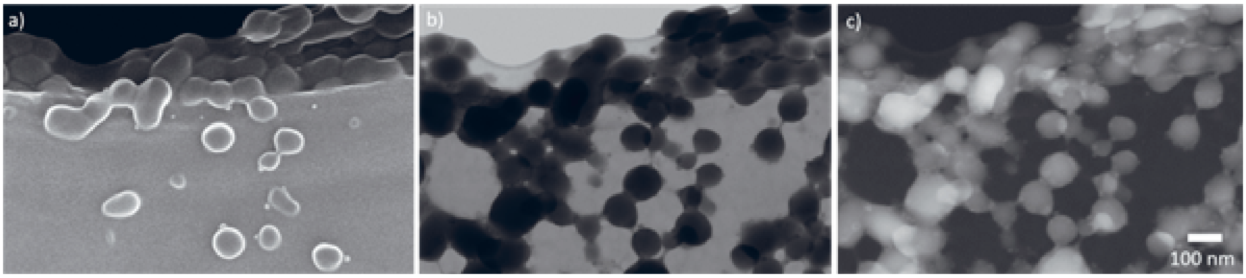

Figure 1. *Study of a drug dissolution in SEM Verios 4G HP, a) SEM image b) STEM-BF image c) STEM-HAADF. Images of the same area of interest were acquired simultaneously.*

organized visits to the IJS, as well through publications in traditional and digital media.

The research carried out using the CEMM equipment is diverse due to many different research topics of the JSI departments. To illustrate the versatility of various available techniques a very few recent microscopy analyses carried out on the CEMM equipment are presented.

SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy is employed to observe the morphology of surfaces and for the microstructural investigation and determination of the chemical composition of investigated materials. The available SEM techniques at the CEMM include SE and BE imaging, EDS/WDS analysis, EBSD, STEM in SEM, e-lithography, low-voltage imaging, low-vacuum imaging, and in-situ heating and fracture testing.

Samples that are most frequently investigated are ceramics (polycrystalline oxides and non-oxide compositions), nanostructured materials, metallic magnetic materials, metals, alloys glass, etc. Additionally, the demand for investigation of biological samples has also increased during the last years. The most advanced SEM microscope within the CEMM is a state-of-the-art SEM Verios 4G HP (Thermo Fisher Scientific). This instrument allows extreme high-resolution imaging as well as good Z-contrast even at voltages below 1 kV. A retractable, highly efficient STEM detector enables imaging of thin TEM samples as well as biological and pharmaceutical samples which are very sensitive to the electron beam damage, but at the same time require very high resolution. As an example of such analysis, figure 1 shows various SEM/STEM images of active drug dissolution process which is crucial for the absorption of the drug by a patient. Due to simultaneous acquisition of SEM and STEM images, not only the information about the particle size, but also about the morphology and topography could be obtained.

For the chemical analysis, new generation of large surface ESD detectors (e.g., 65 mm^2) allow quick and sensitive elemental mapping and high precision EDS quantitative analyses due to high P/B ratio even at low electron beam currents. The combination of STEM and EDS elemental mapping in SEM has proved to be a useful combination even when investigating nanoparticles, as shown in Figure 2.

TRANSMISSION ELECTRON MICROSCOPY

Transmission electron microscopy (TEM) can provide an insight into the structure and chemical composition of materials down to atom scale. The available TEM tech-

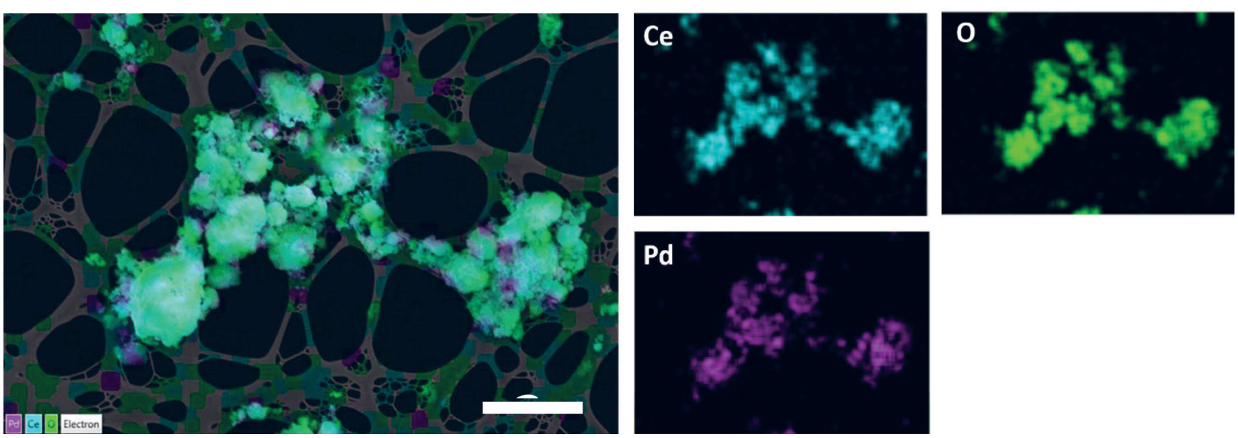

Figure 2. *EDS map of the catalyst CeO_2 with added Pd nanocubes.*

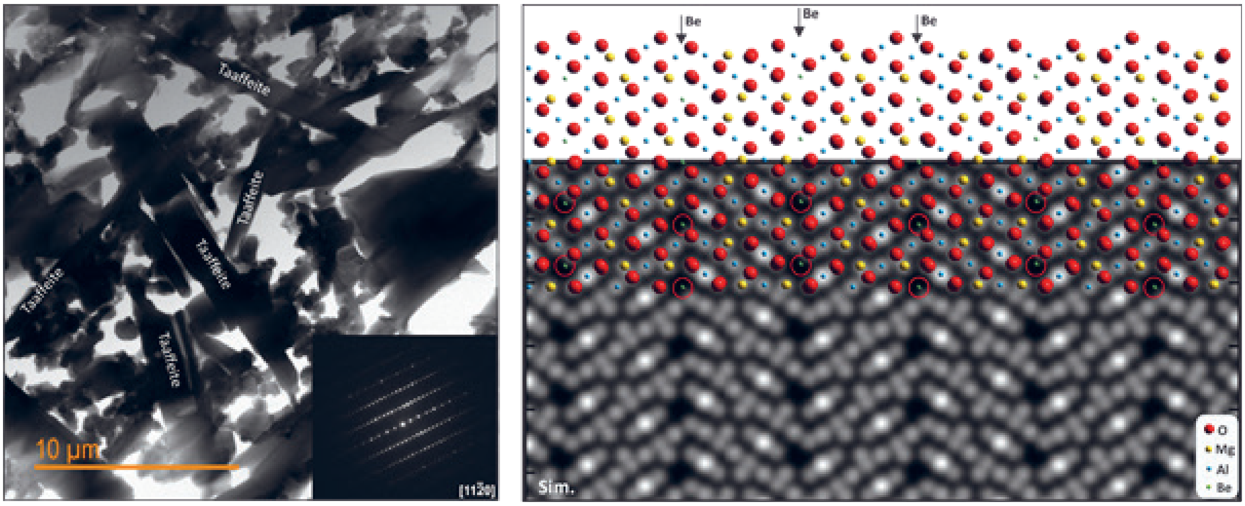

Figure 3. *TEM image with SAED pattern (left) and structural model (right) of taaffeite ($BeMg_3Al_8O_{16}$) grains.*

niques at the CEMM include: HRTEM, HRSTEM (ADF, HAADF, ABF), EDS, 4D STEM, EELS, in-situ heating, in-situ LCTEM, in-situ electrochemical cell, iDCP-STEM. Materials which are investigated include (nano)powders, thin films, alloys, metallic magnetic materials, dielectric materials, ferroelectrics, etc., while the most studied structural phenomena include grain boundaries, precipitates, planar defect, dislocations, etc. TEM analyses are illustrated by investigation of elongated taaffeite mineral ($BeMg_3Al_8O_{16}$) grains and by the EDS analysis of Ti/V layer in a multilayer ceramic component. Bright-field TEM image of $BeMg_3Al_8O_{16}$ grains shows elongated shape of the grains (Figure 3 left). The width of taaffeite lamella ranges from a few layers up to several hundreds of nanometers. HAADF-STEM simulated image (Figure 3 right) is of a periodical sequence of *hcp* (hexagonal) and *ccp* (cubic) spinel stacking typically for taaffeite-type structures. For the simulated images on the atomic level, we used a combination of QSTEM (created by C. Koch) and a DFT Quantum expresso.

An example of EDS analysis in TEM is illustrated by the EDS analysis of a multilayer ceramic component in an all-ceramic multilayer battery. The interface of multilayer ceramic components and electrode ceramic co-sin-

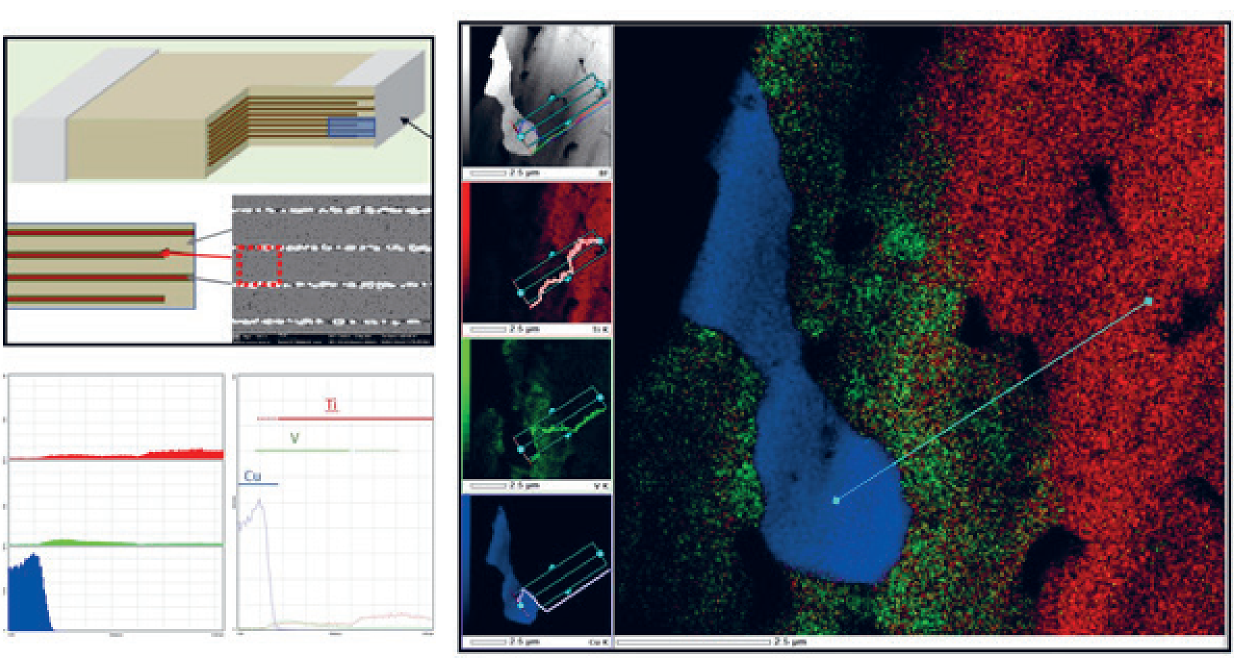

Figure 4. *Detailed scheme (left up) of the Ti/V layer and EDS mapping (left bottom, right) of the Ti/V distributions in a multilayer ceramic component.*

tered Cu layer was studied (Figure 4). EDS mapping of ceramic interface revealed different interdiffusion of Ti and V that has a significant effect on different electric behavior in a battery.

CONCLUSION

In conclusion we can justly stress the vast importance of modern electron microscopy analytical techniques for assessing the structure and the chemical composition of materials down to atomic range. Furthermore, development of new electron microscopy hardware and software is among the fastest among all the analytical research infrastructures. This requires a strong collaboration between centres for electron microscopy on national and also international level in order to fully exploit this expansive research equipment that requires expensive maintenance and personnel with high expertise.