Correlative STEM and SEM Imaging of Nanostructured Materials in a Scanning Electron Microscope

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Transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) with low-energy electrons has been recognized as an important addition to the family of electron microscopies to prevent knock-on damage and increase the contrast of weakly scattering objects. However, the focus of low-energy (S)TEM has been predominantly on operating transmission electron microscopes at low acceleration voltages. Scanning electron microscopes, although limited to 30 keV electron energies, have been up to now rarely considered in a low-energy context because they are mainly used as a standard characterization tool for topography imaging of bulk samples. However, the implementation of a STEM detector, a double-tilt holder for TEM specimens, recent improvements in resolution, and the installation of a CCD-camera for acquiring on-axis transmission electron diffraction patterns make scanning electron microscopes interesting for structure analysis of electron-transparent specimens (not only beam-sensitive ones), which are traditionally investigated in transmission electron microscopes. An interesting aspect is correlative SEM and STEM imaging of the same specimen region. Only few studies, all of them performed at 200 keV, have emphasized the benefits of combined SEM surface topography and bulk TEM imaging, e.g., [1].

Correlative SEM and STEM imaging in a scanning electron microscope (Thermo Fisher Helios G4 FX) is exemplified by Figure 1 which shows Pt nanoparticles on a porous Al_2O_3 carrier for catalysis. The secondary-electron (SE) SEM image Figure 1a shows topography while material contrast by backscattered-electron (BSE) SEM imaging (Figure 1b) reveals larger Pt particles, not only at the top surface but also from the bulk or lower surface of the agglomerate. The same region is imaged by HAADF-STEM (Figure 1c) where porosity of Al_2O_3 is visualized. The larger Pt particles show, against intuition, dark contrast which is due to pronounced large-angle scattering at low electron energies. With increasing thickness, electrons are scattered beyond the HAADF-STEM detection angle range which leads to the decrease of HAADF-STEM intensity. The proper interpretation of low"energy dark-field STEM intensities requires simulations of the STEM intensity due to the strong thickness dependence of the (HA)ADF intensity which can be favorably exploited for, e.g., sample thickness determination. High-magnification HAADF-STEM imaging (Figure 1d) from a thin region (frame in Figures 1a,c), shows the presence of Pt nanoparticles with a size clearly below 3 nm while topography contrast of this region is seen in the insert of Figure 1a.

We will present examples that show that correlative SEM and STEM including on-axis transmission electron diffraction in a scanning electron microscope is particularly interesting for nanostructured materials where sample thickness is typically not a limiting factor.

[1] H. Inada et al., Ultramicroscopy 111, 865 (2011).
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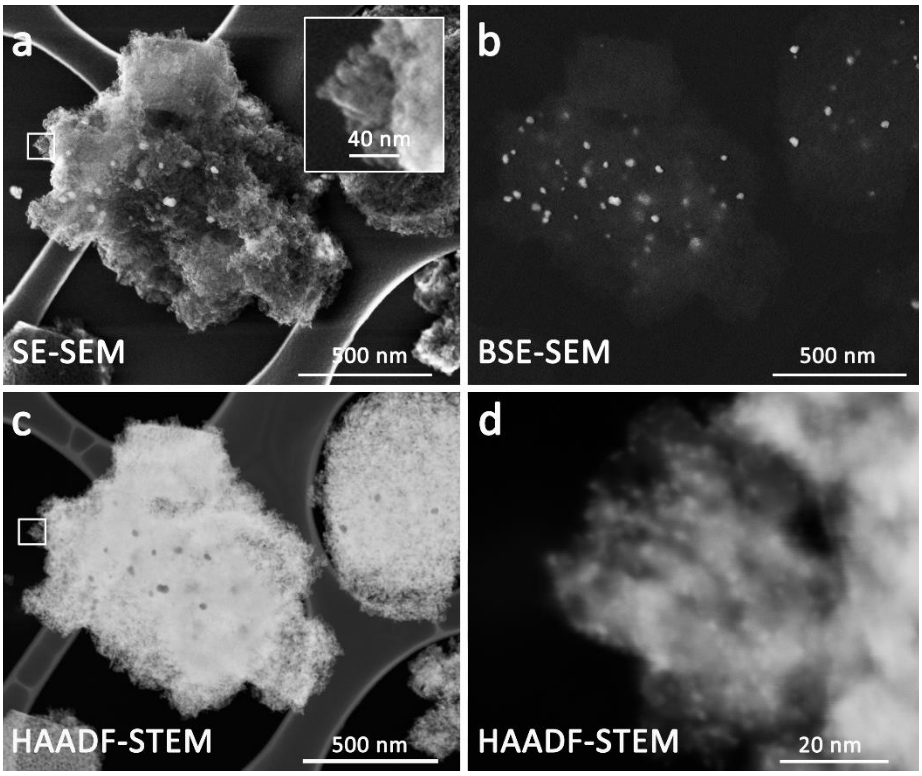

Figure 1. Pt nanoparticles on a porous Al_2O_3 carrier imaged at 30 keV by a) SE-SEM, b) in-lens BSE-SEM, c,d) overview and high-magnification HAADF-STEM.