Considering absorption in EDS TEM tomography of nanocomposite materials

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Electron tomography is a standard and well"established method for 3D imaging of various materials on the nanoscale. Commonly used algorithms like WBP or SIRT demand image intensities to be a monotonic functions of a physical property. Using spectrum images of x-rays as a data set for tomographic reconstruction is not as straightforward, as the spectrum images are often noisy [1] and the often"assumed absence of x-ray absorption in thin TEM samples does not apply to generally thicker tomography specimen.

Furthermore, the absorption varies with x-ray energy, specimen thickness and elements in the absorbing material between x-ray source and x-ray detector (as shown in Figure 1) and leads to "shadowing" artefacts in tomographic reconstructions [2].

In this work, we examine x-ray absorption during acquisition of spectrum images of needle shaped specimens at different tilt angles. By using a sophisticated EDS system with four detectors arranged at different azimuth angles around the microscope axis (FEI Super-X) and comparing the individual detector signals in correlation with their orientation relative to the specimen, the influence of signal attenuation due to absorption should be more discernible.

We apply this approach on a nanocomposite material composed of nano"crystalline iron oxide and oleic phosphate (IOOP). The combination of hard ceramic particles with a soft organic matrix results in a material with superior mechanical properties compared to the single components, showing great potential in many fields like biomedicine or consumer electronics [3]. The combination of low Z (organic linker) and high Z (ceramic particles) however, make EDS tomography troublesome due to the effects mentioned earlier.

The proposed model of the IOOP nanocomposite exhibiting an FCC structure of the spherical ceramic particles covered with the organic linker is shown in Figure 2. The orientation of the supercrystal relative to the EDS detectors corresponds to the acquisition conditions used for the elemental maps in Figure 3b"d. The carbon and phosphorous maps should be congruent as both elements are only present in the organic linker. Due to the lower energy of C-K (0.28 keV) in relation to P-K (2.01 keV), absorption effects are more distinct in the carbon map than in the phosphorous map, containing valuable information about the spatial distribution of elements. Comparison of Figure 3 c and d shows that carbon appears to be concentrated in the interparticle void, whereas the phosphorous map displays the expected ring"shaped pattern expected from a close packed structure. This discrepancy visualizes the need to consider absorption in EDS tomography and to adapt signal acquisition and reconstruction algorithms.

References:

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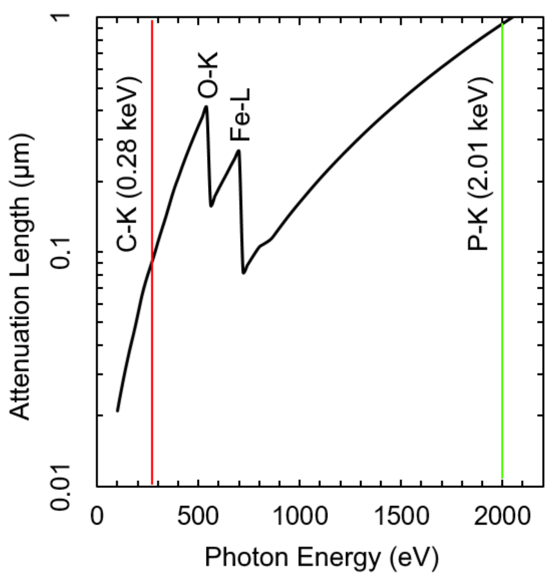

Fig. 1: Calculated attenuation length of x-rays in Fe_3O_4.

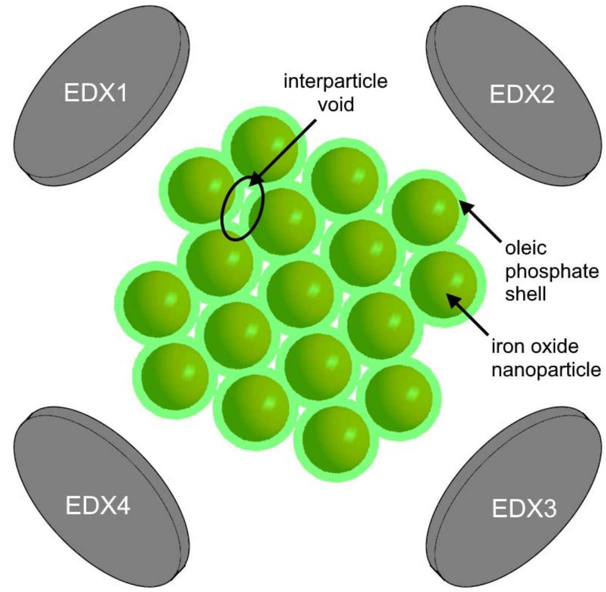

Fig. 2: FCC packed iron oxide nanoparticles with oleic phosphate shell in viewing direction [110]. Sample and detector layout corresponds to acquisition parameters of Fig. 3

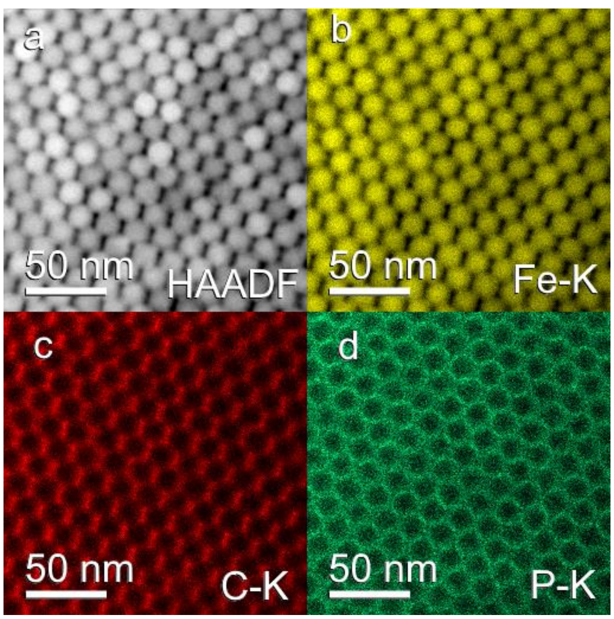

Fig. 3: HAADF and spectrum image of iron oxide - oleic phosphate nanocomposite. All four EDS detectors used.