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IT-5-O-1718 Towards Quantitative EDX Results in 3 Dimensions.

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Over the last 10 years, electron tomography has evolved into a versatile tool to investigate (hetero)nanostructures [1]. Nevertheless, resolving their chemical composition in 3D remains challenging. In principle, energy dispersive X-ray (EDX) mapping can be combined with electron tomography since the number of generated X-rays increases with sample thickness. However, early attempts to perform 3D EDX experiments were complicated by the specimen-detector geometry [2]. Recent efforts therefore led to a novel EDX detection system, enabling the extension of EDX mapping to 3D [3]. An example of a 3D EDX reconstruction is shown in Figure 1, showing a Au@Ag nanocube of which the Au core yields an octahedral shape. This example clearly illustrates the potential of 3D EDX mapping, but one needs to be careful when extracting quantitative information from such reconstructions. In order to obtain quantitative 3D reconstructions using EDX, different steps in the experiment need to be optimized.

The Super-X detection system consists of 4 EDX detectors that are symmetrically arranged around the sample. As a result, it is expected that shadowing effects are minimized and that the total number of detected characteristic X-rays for a spherical nanoparticle is independent of tilt angle. Figure 2 presents the EDX counts that were acquired from a Au particle using a Model 2030 Fischione tomography holder. Using this dedicated holder, shadowing is kept at a strict minimum, but even in this case, an asymmetric collection efficiency of the detector is still observed. This problem, caused by remaining shadowing of the sample grid, can be overcome by combining EDX signals, unaffected by shadowing, that are collected by different detectors during the tilt series.

Quantification of the EDX maps is typically performed using the “Cliff-Lorimer” method, originally developed for the investigation of thin films. Here, we evaluate the use of the “ζ (zeta)-factor” method to obtain quantitative 3D chemical data using the following equation [4]:

$\rho t = \zeta \, I / (C D_e)$

In this formula, ρ is the density of the material and t equals sample thickness, which can be obtained from 3D high angle annular dark field STEM (HAADF-STEM) reconstructions. First, the ζ-factor can be determined by measuring the intensity I and the electron dose for monometallic nanostructures. After estimation of the ζ-factors for different elements, quantitative 3D elemental analysis becomes possible for heteronanomaterials having unknown composition.

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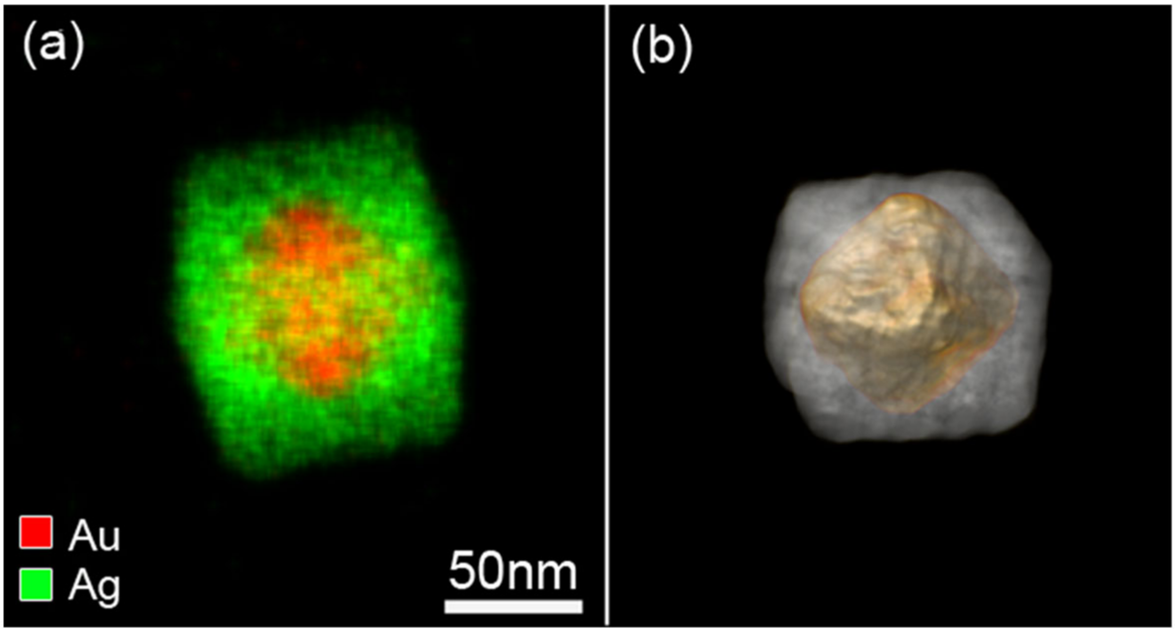

Fig. 1: (a) 2D EDX map of a Au@Ag nanocube. Based on a tilt series of such 2D EDX maps, 3D reconstructions (b) could be obtained showing the 3D distribution of the different chemical elements.

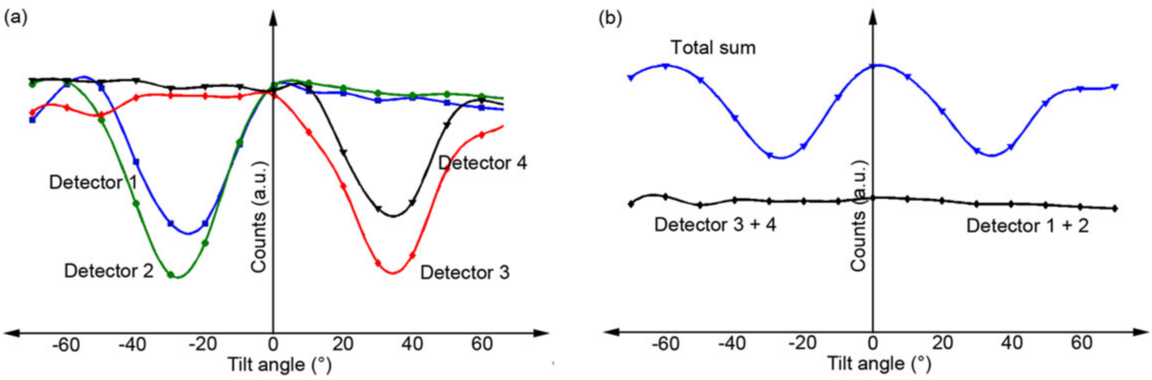

Fig. 2: (a) Detected X-ray counts as function of tilt angle for each individual detector of the super-X system. At certain tilt angles, shadowing effects may block the X-rays preventing them to reach the detectors. (b) Total X-ray count when adding the signal from different detectors.