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Benefits of Using a 4 srad XEDS Detector in Quantitative 3D-Compositional Analysis of Core@shell Nanoparticles

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The chemical composition and distribution of elements in core@shell structures determine largely the properties of these materials. To reveal their structure-property relationship, three-dimensional (3D) information is required. Electron tomography, often based on HAADF STEM, is a very valuable tool to investigate the structure of nanomaterials, even with atomic resolution. Also, the 3D investigation of the composition is nowadays possible through XEDS tomography. The approach works well for systems with smooth and repeatable stage tilt to high angles, high XEDS count rates at all tilts, and effective software for the acquisition, alignment, reconstruction and visualization of the tomography tilt series [1]. Unfortunately, 3D XEDS is significantly more challenging than 3D HAADF STEM imaging, in particular because of the much longer acquisition time and larger electron dose required. In practice, a 5 to 10 minute map at each tilt angle is collected, leading to tilt series for which typical data acquisition takes several hours. This severely restricts the use of XEDS tomography, especially for the investigation of beam sensitive materials, for which sample integrity is crucial.

The acquisition time and signal to noise ratio of the compositional analysis using XEDS is strongly influenced by the collection efficiency of the XEDS detector. Therefore, we here quantitively compare two types of XEDS detectors and we measured the gain in performance due to larger collection angles and a symmetric detector design. To illustrate the progress in performance, tilt series with a SuperX (0.9srad) [2] and UltraX (4.4srad) detector [3] were recorded for similar particles and the same dose conditions. The method applied for generating quantified 3D compositional map data is described in reference [1].

Two examples are presented in this contribution. First, we compare relative beam stable core shell Au@Ag nanoparticles to illustrate the S/N ratio improvement of the UltraX detector versus the SuperX system, using the same electron dose. Second, we present results for Au@Pt particles, which are more challenging because of the small features in the dendritic Pt shell. We hereby observed that the high dose requirements in XEDS leads to damage during the tilt series when using the classical SuperX detector. The superior collection efficiency of UltraX allows to record reliable full tilt series to obtain 3D elemental distributions for these particles. The collection efficiency of the UltraX detector increases the signal by a factor of 6-7 over the entre tilt range and is best suited to provide quantitative 3D chemical information for these complex nanoparticles [4].

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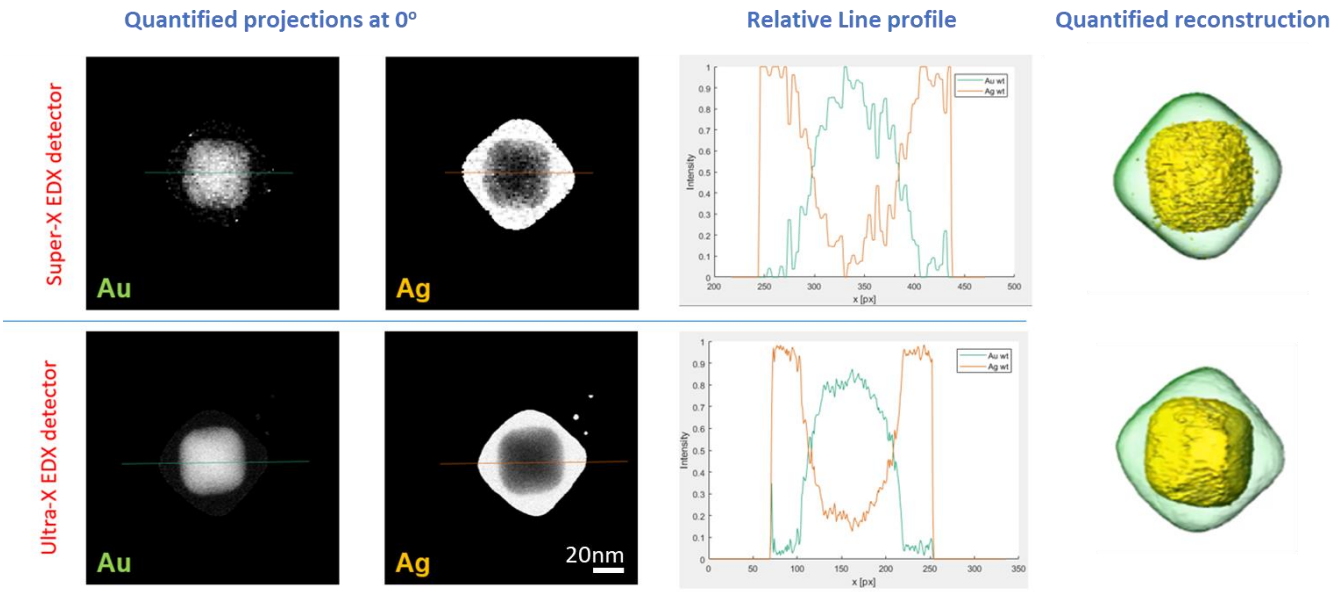

Figure 1. Quantitative comparison of UltraX versus SuperX data with the same experimental conditions, acquired for Au@Ag core-shell nanoparticles. In the quantified projections of Au and Ag the gain in S/N can be clearly illustrated and quantitatively measured. 3D visualizations of the XEDS reconstruction of Au@Ag nanoparticles using SuperX and UltraX, respectively, are shown on the left.

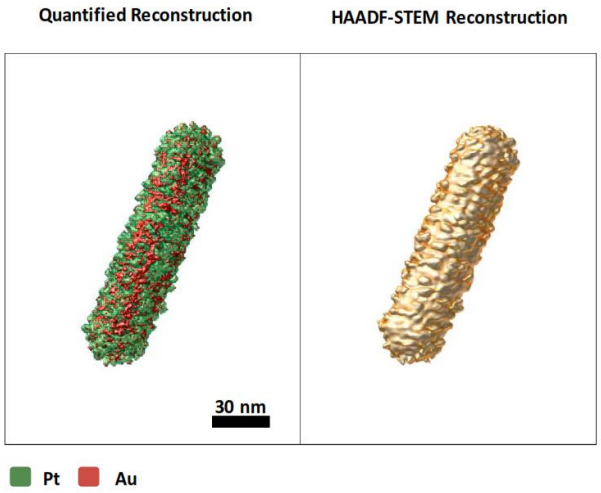

Figure 2. 3D visualization of the XEDS tomography reconstruction of an Au@Pt nanoparticle using the UltraX detector. Interpretation of the position of the Pt spikes becomes possible, which was not the case due to damage caused by the higher dose requirements using detectors with lower collection efficiency.

References:

[1] Particle and Particle Systems Characterization **33(7)** (2016). DOI:10.1002/ppsc.201600021
[2] Microscopy Today **18(04)** (2010). DOI: 10.1017/S1551929510000404
[3] Microscopy and Microanalysis, **27(S1)** (2021), p. 2070. DOI: 10.1017/s1431927621007492
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