

Quantitative Aspects of 3D Chemical Tomography in the Scanning Transmission Electron Microscope

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Modern fabrication techniques have enabled the production of ever smaller, ever more complex nanoscale architectures for the development of new technologies. This has created increased demand for robust characterization methods in order to advance our understanding of the relationship between structure, properties, and processing in such devices and systems. Electron tomography (ET) in the scanning transmission electron microscope (STEM) offers a method to reveal the 3-D structure within small volumes of material and is capable of nanoscale or better spatial resolution in all three dimensions. In ET, the 3-D structure of a specimen is mathematically calculated from a series of 2-D projections acquired over a range of specimen orientations. Recent advances in specimen preparation, automation of data collection, and improved reconstruction methods have also made it feasible to couple tomographic data acquisition with X-ray energy dispersive and electron energy-loss spectroscopies (XEDS and EELS, respectively) in order to carry out 3-D elemental and chemical analysis at similar length scales [1-7].

In this talk, quantitative aspects of 3-D chemical tomography in the STEM using XEDS and EELS will be discussed. As an example, Figure 1 shows a cross sectional specimen fabricated via focused-ion beam milling of NIST standard reference material (SRM) 2135c which consists of alternating layers of Ni and Cr supported on a Si substrate. The individual layers and the substrate are readily visible in the HAADF image, as are the protective layers deposited on the surface to protect the underlying material from damage during milling. In order to explore the effects of varying X-ray absorption paths in this system, the cross section was then further milled [8] so as to produce pillar shaped specimens in two distinct geometries: one where the layer modulation direction was orthogonal to the pillar axis (Figure 1, top-right) and another where this direction was parallel to the pillar axis (not shown). Samples with this geometry can be analysed over a full 180° tilt range in order to eliminate reconstruction artifacts due to missing information which often plague STEM-based tomography data. Reconstruction of both STEM-HAADF based tomography data (Figure 1) as well as XEDS hyperspectral tomography data (Figure 2) was carried out using the simultaneous iterative reconstruction technique (SIRT) as implemented in the ASTRA toolbox [9]. The image data is less noisy than the XEDS data and therefore the quantified features in the former can be used as ground truth in assessing the fidelity and limitations of the reconstructions from resulting from the latter. The effects of sample geometry and choice of data processing methods, reconstructions algorithms, and quantification approaches will be discussed for these model specimens as well as for more complex, industrially relevant structures.

References:

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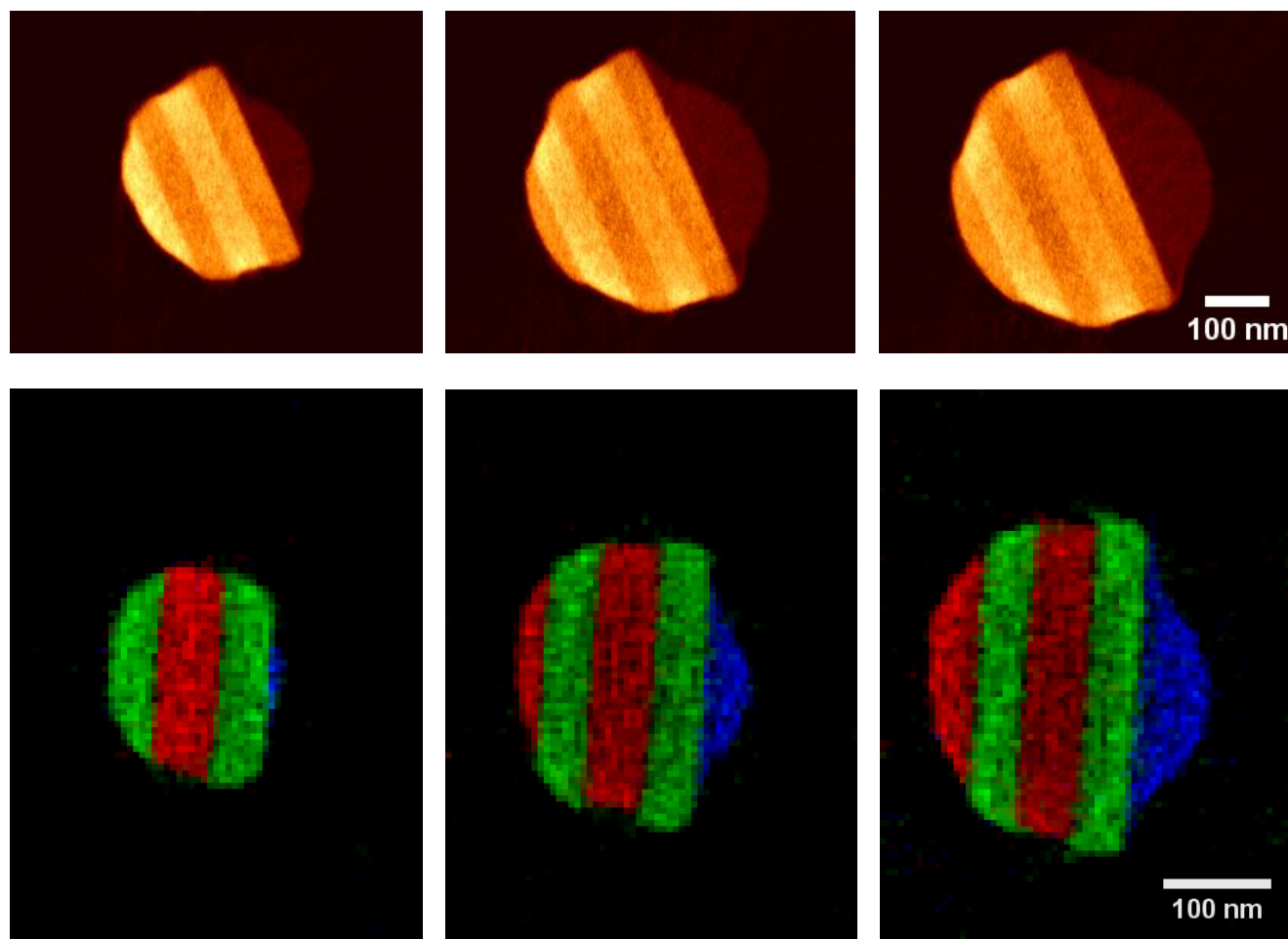
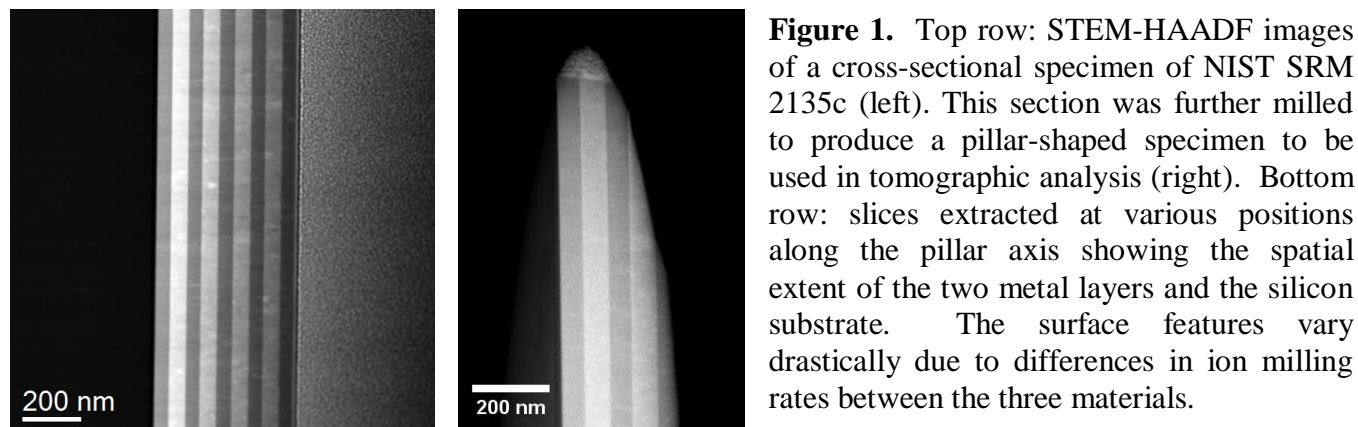


Figure 2. Color overlays of slices extracted after SIRT reconstruction of XEDS hyperspectral tomography data of NIST SRM 2135c pillar-shaped specimen. Elemental maps were extracted and reconstructed separately for the Ni-K α (red), Cr-K α (green), and Si-K α (blue).