# Improved Data Analysis and Reconstruction Methods for STEM-EDX Tomography

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Energy dispersive X-ray (EDX) tomography is a valuable technique for the 3D compositional mapping of nanomaterials, but has been limited in the past by poor collection efficiency, long acquisition times and unreliable reconstructions [1,2]. With the recent development of high-brightness X-FEG guns and super-X EDX systems comprising four detectors, there has been a renewed interest in the technique and promising applications reported (e.g. [3-4]). While the acquisition of the EDX-STEM tilt series is still performed manually, progress has been made in data processing and algorithms for quantitative reconstruction. As an example, here, we have applied these advances to a Tazewell IIICD meteorite sample for the 3D chemical analysis of tetrataenite (FeNi) islands embedded in an Fe-rich matrix. A focused ion beam was used for the preparation of a needle-shaped sample, and a Fischione 2050 on-axis holder employed for the EDX-STEM tilt series acquisition using an FEI Osiris (S)TEM operating at 200kV. EDX-STEM maps (and the corresponding HAADF-STEM images) were recorded every 5°, with a dwell time of 30ms/pixel corresponding to 8min/map. Hyperspy, a python-based software for hyperspectral data processing [5], has been used for the batch-processing of the 4D dataset: the spectra were denoised using principal component analysis (PCA), and X-ray intensities extracted after background subtraction. The obtained tilt series of Ni and Fe maps were aligned using ImageJ, and exported to Matlab for tomographic reconstruction using compressed sensing (CS) [6]. This algorithm takes advantage of any prior knowledge about the sparsity of the object in a chosen transform domain and seeks a sparse solution that is consistent with the acquired data. CS has been shown to yield reliable reconstructions from a limited number of projections (e.g. [7-9]). Applied to the current dataset, sparsity in the image and gradient domains was promoted to generate Fe and Ni volumes, and the 3D (voxel-wise) quantification was achieved in Hyperspy using the Cliff-Lorimer quantification method. Figure 1 (a-b) show the obtained volumes, displayed in weight percent. Figure (c-d) are slices through the volumes, highlighting the presence of Fe-rich secondary precipitates in the tetrataenite islands. These quantitative results were confirmed by atom probe tomography, performed on the same needle.

Multivariate statistical analysis methods, such as independent component analysis (ICA) [10] and non-negative matrix factorization (NMF) [11], can also be applied to decompose 4D EDX datasets into a reduced number of component loadings, or phases, present in the material, thus speeding up both the data analysis and the reconstructions [12].

References:

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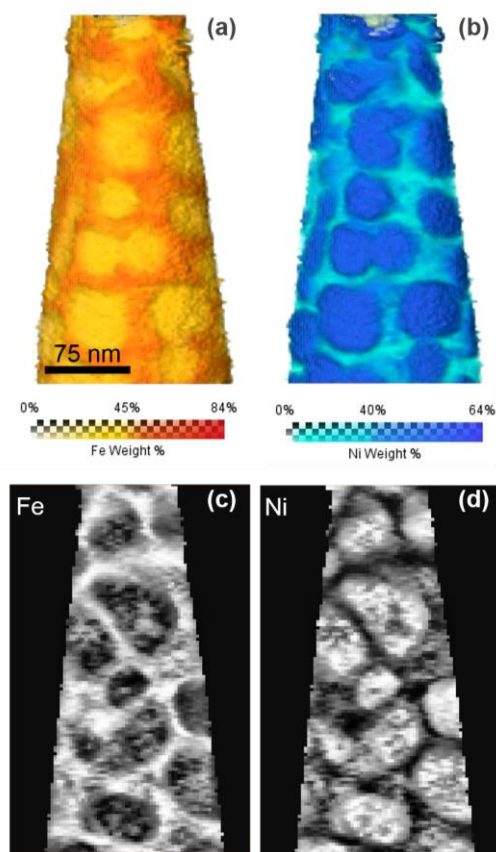[11] S. Ikeshita et al., Micron **82** (2016), p.1.

**Figure 1.** 3D chemical mapping of a Tazewell IIICD meteorite. (a-b) Voxel projection rendering of the Fe and Ni volumes, displayed in weight %. (c-d) Orthoslices through the volumes showing the presence of Fe-rich secondary precipitates in the tetrataenite.