Computed Tomographic Spectral Imaging: 3D STEM-EDS Spectral Imaging

P.G. Kotula[1], L.N. Brewer[1], J.R. Michael[1], and L.A. Giannuzzi[2]

[1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185-0886
[2]FEI Company, 5350 NE Dawson Creek Drive, Hillsboro, OR 97124

Spectral imaging, where complete x-ray spectra are acquired from 2D or 3D arrays of points, is a powerful microanalytical technique, especially when combined with multivariate statistical analysis [1]. Chemical imaging, typically spectroscopic imaging or mapping with energy-loss electrons or x rays has been performed on flat [2-3] or pillar-shaped specimens [4] in the TEM to achieve 3D chemical images. In this paper we describe results from acquisition and analysis, via multivariate statistical analysis (MSA), of x-ray spectral images acquired from a needle-shaped FIB specimen, tilted from -90 to +90 in a newly available TEM specimen holder from Fischione Instruments [5], with full EDS coverage and constant thickness throughout the tomographic series.

A needle-shaped specimen of aluminum oxide (~10-20nm particles) dispersion-strengthened Ni (matrix) was prepared by FIB to a thickness of ~100nm near the tip and ~200nm near the base, and extracted *in situ* and placed upon a special cartridge designed originally for atom-probe tips. The cartridge was then placed in the Fischione Model 2050 On-Axis Tomography holder [5]. Figure 1a shows the tip of the holder while Figure 1b is an SEM image of the needle-shaped specimen. The specimen was imaged in a FEI Tecnai F-30ST operated at 300kV in STEM mode (3nm-diameter FWTM electron probe) with an EDAX SUTW Si (Li) EDS. Drift-corrected spectral images were then acquired at 10° intervals over the range from -90° to +90° from a 400 nm by 2000 nm area and 100 by 500 pixels (4nm/pixel). The dwell time per pixel was 50 msec, resulting in a spectral image acquisition time of about one hour or a total acquisition time of about 19 hours. After acquisition, the series of spectral images, consisting of a total of almost one million spectra, were analyzed with Sandia's Automated eXpert Spectral Image Analysis (AXSIA) software [1] in one minute on a dual Itanium 64-bit computer equipped with 6Gb RAM. Three components resulted from the analysis of the 0-2kV region of the series of spectral images: Ni, Al_2O_3, and an absorption component reflecting the use of soft x-rays for this analysis. Figure 2a are the Ni (red) and alumina (green) component images overlaid for +90°, 0°, and 90°. Figure 2b shows the corresponding spectral shapes. Ni and alumina components are clearly deconvolved in the MSA analysis shown, even though the x rays used are soft (<1.5kV). Reconstruction of the tilt-series data into 3D renderings is underway.

References

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[6] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States National Nuclear Security Administration, part of the Department of Energy (DOE), under contract DE-AC04 94AL85000.

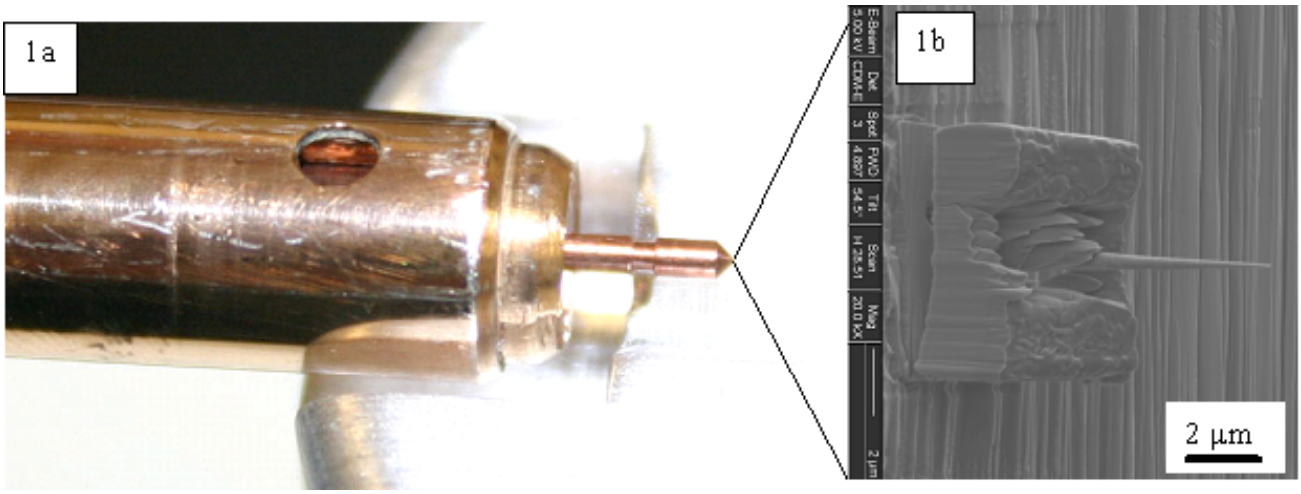

FIG. 1a and b. TEM specimen holder tip and SEM image of the FIB-produced needle-shaped specimen.

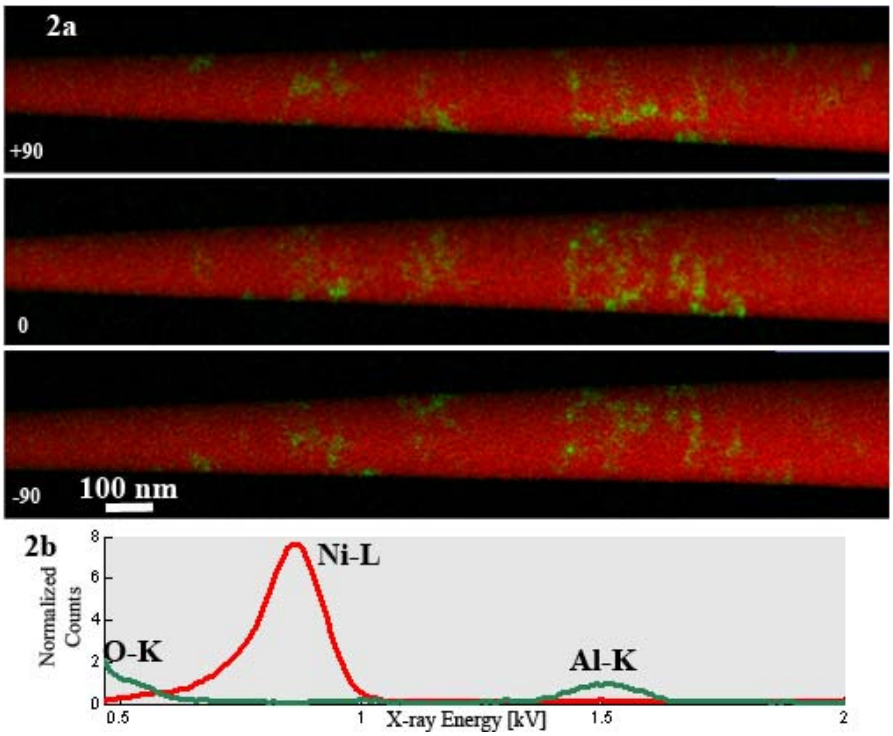

FIG. 2a and b. Component images corresponding to Ni (red) and Al_2O_3 (green) at +90, 0, and -90 degrees of eucentric tilt, and corresponding spectral shapes respectively.