DOI: 10.1017/S1431927603445029
Microsc Microanal 9(Suppl 2), 2003

Tomographic Spectral Imaging: Comprehensive 3D X-ray Microanalysis

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Microanalysis in the SEM is a very powerful tool for materials characterization but is typically only a measurement of the surface. Spectral imaging, where a series of complete x-ray spectra are typically collected from a 2D area, combined with efficient statistical analysis algorithms has more recently made the technique even more powerful and comprehensive [1]. 3D microanalysis, via some sort of sectioning followed by Auger microanalysis, has been performed [2] but suffers from the same shortcomings/potential artifacts of mapping techniques in general [3]. In this paper, we present the results of comprehensive 3D microanalysis performed via serial sectioning in the focused ion-beam (FIB) tool combined with spectral imaging for each slice–tomographic spectral imaging (TSI). The entire 4D (x, y, z, E) spectral image data set was automatically analyzed utilizing a modified version of our software developed previously [1]. The result was a compact, easily interpreted solution that, as with previous applications of the technique [1], is far more straightforward to interpret than the raw data itself or using other conventional approaches.

The tomographic spectral images were acquired in a FEI DB-235 focused ion-beam tool (FIB)/scanning electron microscope (SEM) equipped with a Thermo NORAN EDS detector and Vantage (Digital Imaging with Spectral Imaging) system. Automatic x-ray spectral image analysis calculations were performed on a PC workstation equipped with dual 2.4Ghz Pentium IV Xeon processors and 2Gb of system RAM although this amount of computation power is by no means required. The analytical geometry is shown in Figures 1a and b. Firstly, a trench is milled to expose the initial analysis surface, then an additional trench is milled perpendicular to the first allowing the x-ray detector to ‘see’ the analysis surface. In addition, fiducial markers were milled into the original surface for slice-to-slice alignment. Two specimens were analyzed in this way, a Cu-Ag eutectic and a sulfide bloom on a corrosion coupon. The analysis trenches for both were 50μm wide of which spectral images were acquired from a region 40μm wide (less in the vertical direction) at 128x128 pixels by 512 (sulfide) or 1024 (eutectic) energy channels by 10 or 12 slices with an electron potential of 5 or 10kV respectively. For the eutectic specimen the automated spectral image analysis software identified 4 components that described all of the chemical information in the specimen: Cu, Ag, Pt (from the FIB to protect the top surface), and Ga (implanted into the Pt by the FIB). Figure 2a shows a 3D rendering of the Cu component image as well as its corresponding component spectrum showing Cu-L and K peaks. The second example of TSI was from an Au- and Ni-plated Cu coupon that was subjected to a corrosive environment resulting in localized sulfide blooms on the specimen surface. TSI analysis was made from a region including one such bloom. The data set analyzed was 128x128x10voxels by 512 energy channels per pixel. Our software automatically identified seven components as seen in Figures 2b and c: Au (red), Ni (yellow), Cu (green), Cu-S (cyan), Si-O-Cl (blue), Pt (not shown), and Ga (not shown). The result is a comprehensive analysis of over 160,000 spectra or 80 million data points from volume elements. The analysis of this ~340Mbyte raw data set took 29 seconds and was distilled down to ~9Mbyte (128x128x10voxels x 7 component images (3D) plus 7 component spectra).

[1] P.G. Kotula et al., *Microsc. Microanal.* 9[1] (2003)1-17.
[2] Cheng, Z. et al. *J. Vac. Sci. Tech.* 16 (1998) 2473.

[3] Newbury, D.E., Bright, D.S. *Microsc. Microanal.* **5**, (1999) 333–343.

[4] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United Stated Department of Energy (DOE) under contract DE-AC04-94AL85000.

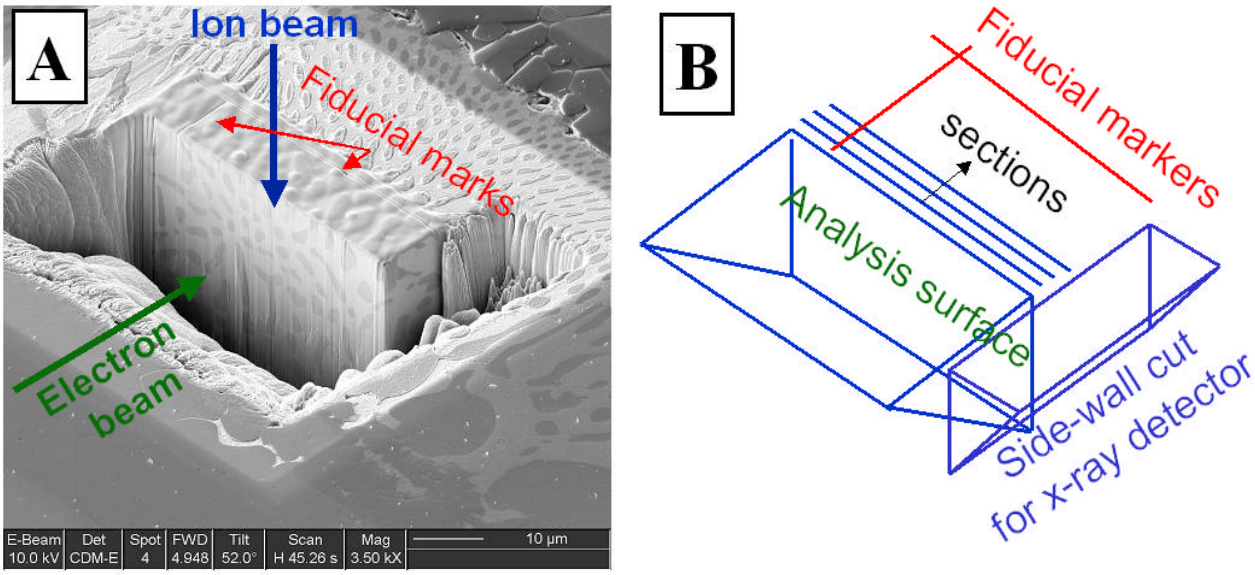

FIG. 1. Analytical geometry of FIB serial sectioning tomography. A. SEM image showing the view of the analysis surface from perspective of the x-ray detector. B. Schematic of the FIB trenches/markers.

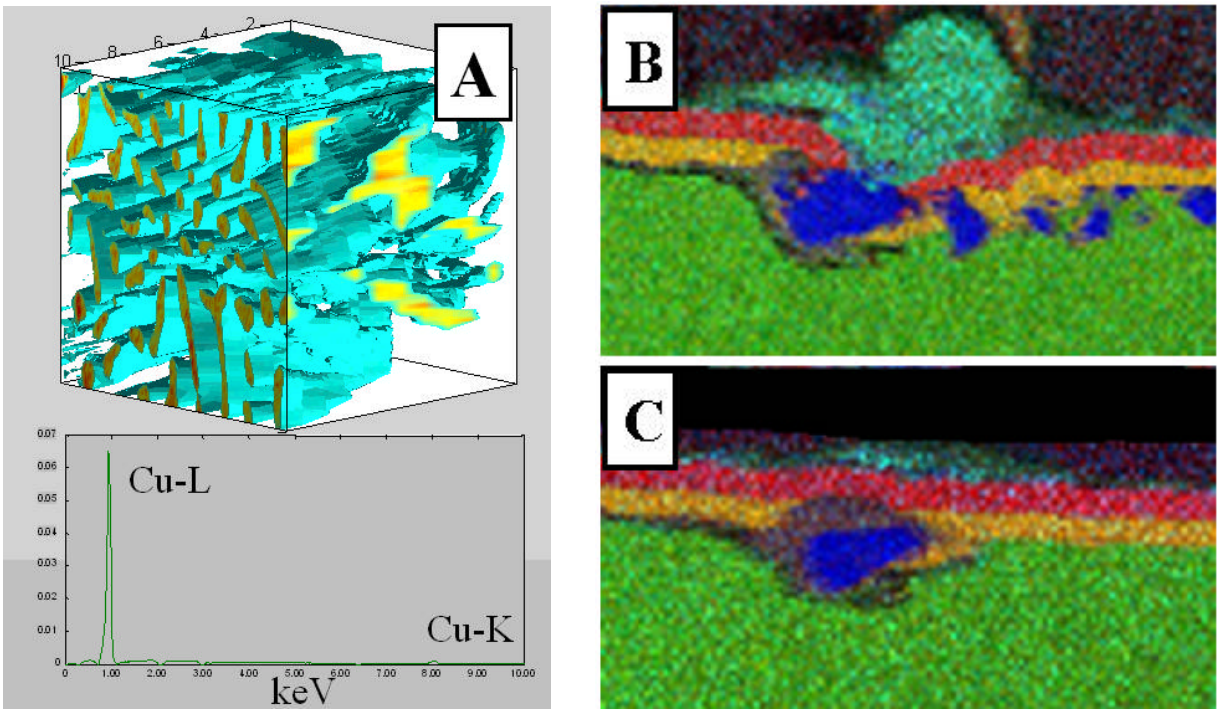

FIG. 2. Results of automated spectral image analysis of two tomographic spectral images. A. Cu-component 3-D image and spectrum from the Cu-Ag eutectic sample. B,C. RGB composites of two slices showing five components from the corrosion sample.

https://doi.org/10.1017/S1431927603445029 Published online by Cambridge University Press