doi:10.1017/S1431927610056205
Microsc. Microanal. 16 (Suppl 2), 2010

Applying Multivariate Statistical Analysis to Atom Probe Tomography

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Atom probe tomography (APT) is a powerful method for studying the microstructure and chemistry of materials at the unit-cell level [1]. Because APT produces datasets containing up to billions of atom identities, new methods of analysis are being developed to rapidly datamine information from the APT data. Spectrum imaging (SI) methods utilizing multivariate statistical analysis (MVSA) are becoming standard for acquiring and interpreting analytical electron microscopy (AEM) data [2-4]. Here, the hypothesis that SI techniques can elucidate APT data is explored.

As an example, PM 2000 steel was analyzed in an Imago local electrode atom probe (LEAP). Standard isoconcentration surfaces of two populations of precipitates, α′ Cr and β′ Fe(Ti,Al), in the Fe-rich matrix are shown in Fig. 1. The size of the selected volume is approximately 31×30×65 nm. Using a custom MATLAB code, a 31×30×65 grid of cubic voxels each 1 nm on a side was created. Each detected ion was assigned to a voxel based on the ion's (x,y,z) position. A mass-spectrum was created at each voxel based on mass-charge state ratio (m/n) of the ions. In this case, m/n values from 0 to 50 u were binned into 100 channels. Conceptually, this is no different than recording an electron energy loss spectroscopy (EELS) or energy dispersive spectroscopy (EDS) spectrum at each pixel or voxel in an electron microscopy spectrum image [2-4]. Poisson-scaled [5] principal component analysis (PCA) was performed on the PM 2000 dataset gridded into 1 nm^3 voxels. An eigenvalue plot from PCA indicating four significant PCs is shown in Fig. 2. The resultant PCA scores were thresholded and rendered into isoscore surfaces, shown in Fig. 3 for the first three PCs. The first PC is the mean spectrum and mean image, and when thresholded shows approximately the matrix (Fig. 3, top row). The second PC shows Cr-rich, Al- and Fe-depleted precipitates corresponding to the α′ phase (Fig. 3, middle row). The third PC shows Al- and Ti-enriched regions corresponding to the β′ precipitates (Fig. 3, bottom row). The fourth PC is more difficult to interpret; it varies from a positive to negative score across the Y-direction of the reconstructed volume, and the loading spectrum for PC#4 shows derivative-like peaks at each ion's m/n. This is likely an instrumental artifact arising from the APT data analysis, and is not rendered. Reducing the voxel size from 1×1×1 nm (1 nm^3) down to 0.5×0.5×0.5 nm (0.125 nm^3) provides the same results but at much higher spatial resolution, despite the fact that each voxel contains only a mean count of 4.2 ions at the smaller voxel size. This is possible because of the small-signal aggregation native to PCA-based methods.

These preliminary results indicate that the SI method may be a good candidate to extract information from APT data. The PCA results clearly differentiated the spatial locations of the two ultrafine precipitate populations and gave a qualitative picture of the precipitates' elemental disrtibution. Future work will involve application of methods such as spatial simplicity [6] or segmentation [4] to attempt to extract quantifiable mass spectra for the MVSA-delineated phases. [7]

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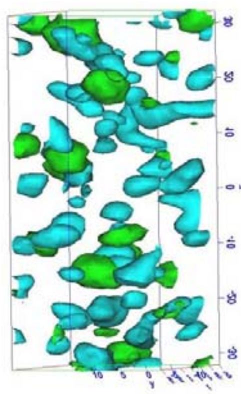

Figure 1: Isoconcentration surfaces in PM 2000. Green: β′, cyan: α′

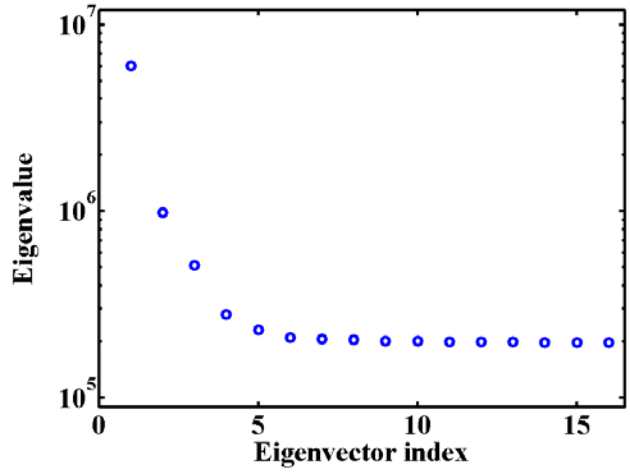

Figure 2: Eigenvalue vs. eigenvector index plot. Four significant PCs are indicated.

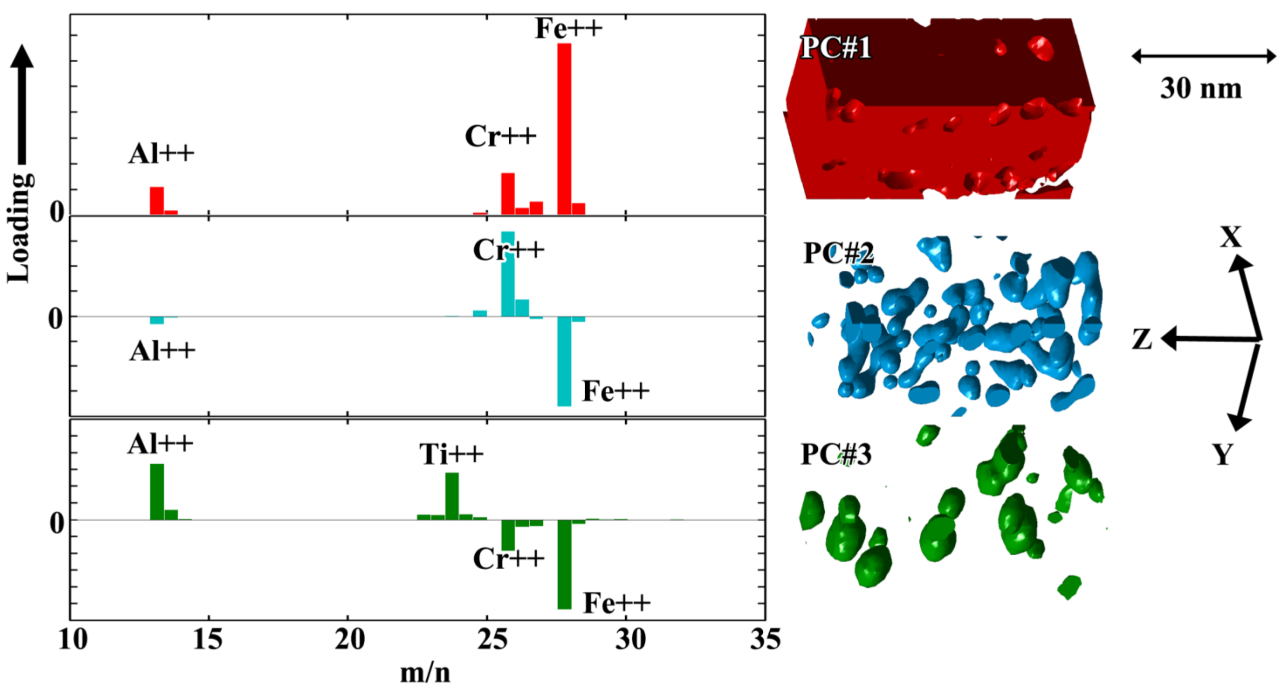

Figure 3: Loading spectra (left) and isoscore surfaces (right) for the first 3 PCs of the PM-2000 APT data. PC#1 is the mean, PCs #2 and #3 describe variance arising from precipitates. An artifact-associated PC (#4) is not rendered.

https://doi.org/10.1017/S1431927610056205 Published online by Cambridge University Press