DOI: 10.1017/S1431927605500734
Microsc Microanal 11(Suppl 2), 2005

Multivariate Statistical Analysis of Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) Spectral Images - Unbiased Analysis of the Entire 3-Dimensional Data Cube

Vincent S. Smentkowski

General Electric Global Research, 1 Research Circle, Niskayuna, NY, 12309

Time of flight secondary ion mass spectrometry (ToF-SIMS) has a number of desirable characteristics for surface analysis/microscopy including: high surface specificity (analysis of the top 0.3 to 1.0 nm of samples), high sensitivity (ppm to ppb for all elements), high mass resolution ($M/\Delta M > 10{,}000$), high lateral resolution (< 100 nm), and the ability to detect all elements plus molecular fragments during a single measurement [1]. At each pixel in a ToF-SIMS image an entire mass spectrum is saved in the raw data file generating a three-dimensional data cube that is often referred to as a spectral image. The spectral image data files contain approximately 8×10^{10} spectral channels, which translate to a wealth of information regarding the measurement. Few tools are available to assist the analyst in visualizing the entire raw data set and as a result, most of the data are not utilized. The ToF-SIMS analyst typically relies on information known about the material, results obtained from similar past analysis, high peak intensities, and/or unusual species detected in the ToF-SIMS spectrum to manually select a few species for further analysis. The ToF-SIMS analyst is expected to perform rapid, cost effective analysis, and provide a complete evaluation of the dataset. Figure 1 shows a typical ToF-SIMS spectrum collected on a polymer sample. Note that, unlike an EDS spectrum from an SEM for example, there are significantly more peaks corresponding to the elements, molecules, and fragments detected.

Automated, non-biased, multivariate statistical analysis (MVSA) techniques are useful for converting the massive amount of data into a smaller number of chemical components (spectra and images) that are needed to fully describe the ToF-SIMS measurement [2-3]. At GEGR, we are using the AXSIA (Automated eXpert Spectrum Image Analysis) toolkit developed at Sandia National Laboratories [4] to perform MVSA on ToF-SIMS spectral images. The advantages of AXSIA include: the ability to select the mass range, the ability to bin the mass spectra from 0.001 amu to 1.0 amu, optimally scaling the data to account for Poisson counting statistics [5], and intuitive results (e.g., negative peaks are not allowed in the spectral response). The AXSIA analyses were performed on a Dell Latitude D400 laptop computer with a 1700 MHz processor and 1.046 GB RAM using Microsoft Windows 2000. All of the AXSIA analyses that will be presented required less than 30 seconds.

AXSIA analysis of the ToF-SIMS spectrum shown in Figure 1 provided 5 components. The component spectra and images generated by AXSIA are shown in Figure 2. The AXSIA analysis was performed using the entire mass spectrum (0 to 1,200 amu); for clarity, only the low mass (0 to 150 amu) regions of the spectra are shown in Figure 2. In order to decrease the file size (increase computation speed) this data set was binned to 1 amu. The five components fully describe the data. Component 1 is characteristic of the polymer, component 2 is rich in alkali species, component 3 represents polymer additive (Bn POSS) rich region of the sample, component 4 is characteristic of silicone oils (sample contaminant) and component 5 is characteristic of hydrocarbon species (contaminants) on the sample.

In the presentation, numerous additional examples will be discussed, stressing the analytical insight provided by AXSIA analysis.

References:

[1] J. C. Vickerman, and D. Briggs, D., *ToF-SIMS Surface Analysis by Mass Spectrometry*, Surface Spectra/IMPublications: UK, 2001.
[2] J. A. Ohlhausen, M.R. Keenan, P.G. Kotula, P.G., and D.E. Peebles, *Appl. Surf. Sci.*, 231-232 (2004) 230.
[3] V.S. Smentkowski, J.A. Ohlhausen, P.G. Kotula, and M.R. Keenan, *Appl. Surf. Sci*., 231-232 (2004) 245.
[4] M.R. Keenan, and P.G. Kotula, U.S. Patent No. 6,584,413, June 24, 2003; M.R. Keenan, P.G. Kotula, U.S. Patent No. 6,675,106, Jan 6, 2004.
[5] M.R. Keenan, and P.G. Kotula, *Surf. Interface Anal*., 36 (2004) 203.

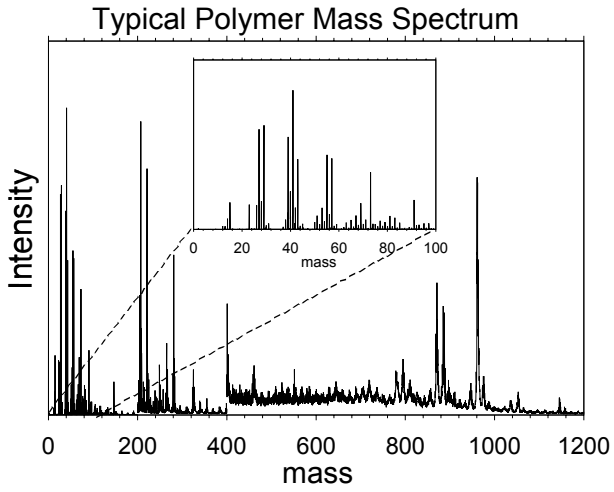

Fig. 1. ToF-SIMS spectrum of a typical polymer.

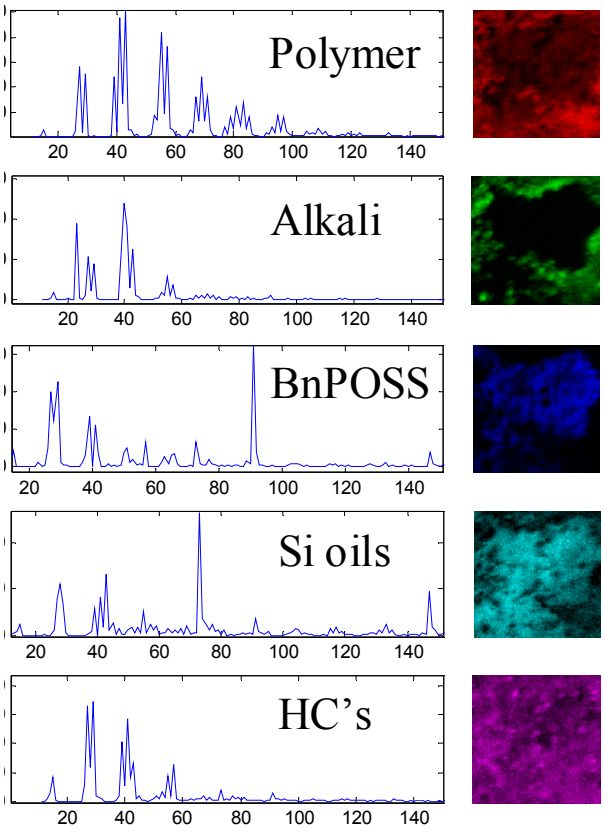

Fig. 2. Five components (spectra and images) identified by AXSIA. These five components fully describe the data shown in Figure 1. Imaged area = 500 micron x 500 micron.

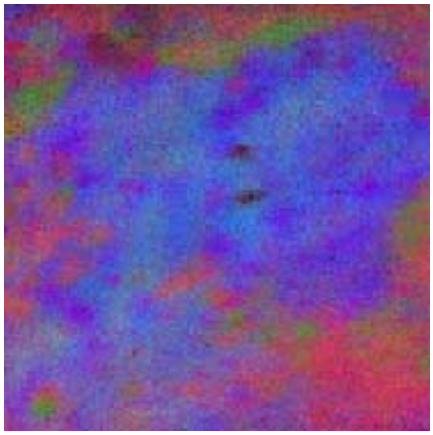

Fig. 3. Color overlay of the AXSIA analysis (imaged area is 500 micron x 500 micron).

https://doi.org/10.1017/S1431927605500734 Published online by Cambridge University Press