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Advances in Acquisition and Analysis of Hyperspectral Images

P. G. Kotula

Materials Characterization Department, Sandia National Laboratories, PO Box 5800, MS 0886, Albuquerque, NM 87185

In less than 20 years the microanalysis community has seen incredible changes in technology for acquisition [1] and analysis [2] of x-ray spectral data otherwise with the advent of hyperspectral imaging where complete x-ray spectra area acquired from a 2D array of points. More recently advanced silicon-drift detectors (SDD) have almost completely replaced the Si (Li) detectors with greatly improved spectral resolution at high count rates [3]. Additionally SDDs and their resultant relaxed cooling requirements have resulted in the development of multi-sensor configurations including annular geometries [4-5] exceeding 1 sr solid angle of collection. Multi-SDD systems have more recently been fitted to scanning transmission electron microscopes (STEM) resulting in solid angles of 0.8 sr and higher [6]. This has allowed atomic-resolution x-ray data to be acquired [7] and quantified [8].

Fig. 1 shows results from a Bruker (pN Sensor) 4-channel annular SDD on a Zeiss Supra 55VP SEM operated at 30kV. The sample consisted of *Bacillus anthracis* spores fixed in gluteralehyde and then embedded in epoxy and microtomed to a thickness of about 80nm. The sample was mounted in a specially designed low background transmission holder. At nominal beam currents of 5 nA the output count rate from the SDD was approximately 150 kcps. Figure 1a shows a secondary electron image of the analysis region. An x-ray spectral image 512 by 384 pixels was acquired in 20 minutes. The data were analyzed with Sandia's Automated eX pert Spectral Image Analysis (AXSIA) multivariate statistical analysis software [2] with the component image overlay shown in Fig. 1b with an inset enlargement. This example illustrates the improvements hyperspectral imaging, x-ray spectrometers, data analysis and a STEM-in-SEM analytical geometry with a thin sample to improve spatial resolution as well as analysis throughput.

Figure 2 shows the results of analysis of a fine-scale spinodal decomposition in Paliney 7 (an electrical contact material consisting of Cu, Zn, Pd, Ag, Pt, and Au. Fig. 2a is the AXSIA analysis (component image overlay) of x-ray spectral image data acquired on a FEI Company Tecnai F30-ST operated at 300kV equipped with an EDAX R-TEM x-ray detector with a nominal solid angle of 0.07 sr. in about 2 hours at 2nm/pixel (the approximate probe size). The spinodal microstructure is just visible with the red consisting of Cu, Pd, Pt and Zn. The Green consists of Ag, Pd and Au. Illustrating the power of both new electron microscopes and multi-channel SDDs, Fig. 2b is the AXSIA analysis (component image overlay) of x-ray spectral image data acquired on a FEI Company Titan G2 80-200 with ChemiSTEM Technology and equipped with a high-brightness source, spherical aberration corrector on the probe-forming optics and a 4-SDD array with a combined solid angle of 0.8 s r. the data were acquired in 7 minutes with a probe smaller than 2 Å and a significantly higher pixel density than in Fig. 2a. Taking into account spatial resolution and analysis time this represents a 70-fold improvement over the older electron microscope. The colors in Fig. 2a follow those of Fig. 2a.

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[9] Joseph R. Michael from Sandia was involved with the *Bacillus anthracis* research which was part of the FBI's Amerithrax investigation of the 2001 Anthrax attacks. The Paliney research was a collaboration with Donald Susan and Zahara Ghanbari at Sandia. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockeed Martin Company, for the US Department of Energy (DOE) under contract DE- AC0494AL85000.

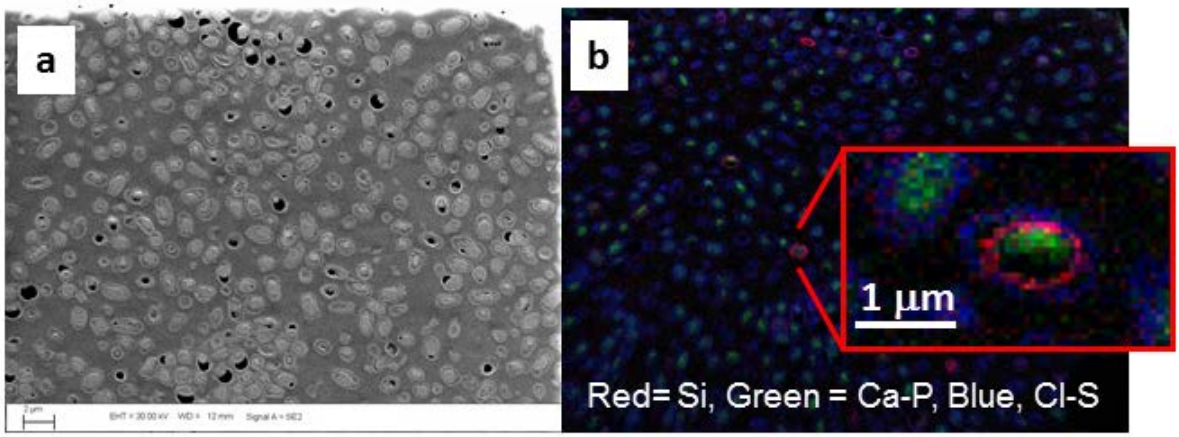

Figure 1. a. SEM secondary electron image of a microtomed section of *Bacillus anthracis* spores. B. AXSIA analysis of an x-ray spectral image acquired (STEM-in-SEM) with an annular-geometry SDD array showing identification of Si incorporation into the sport coat (red) as well as Ca an P associated with the cortex and CI and S associated with the exosporium.

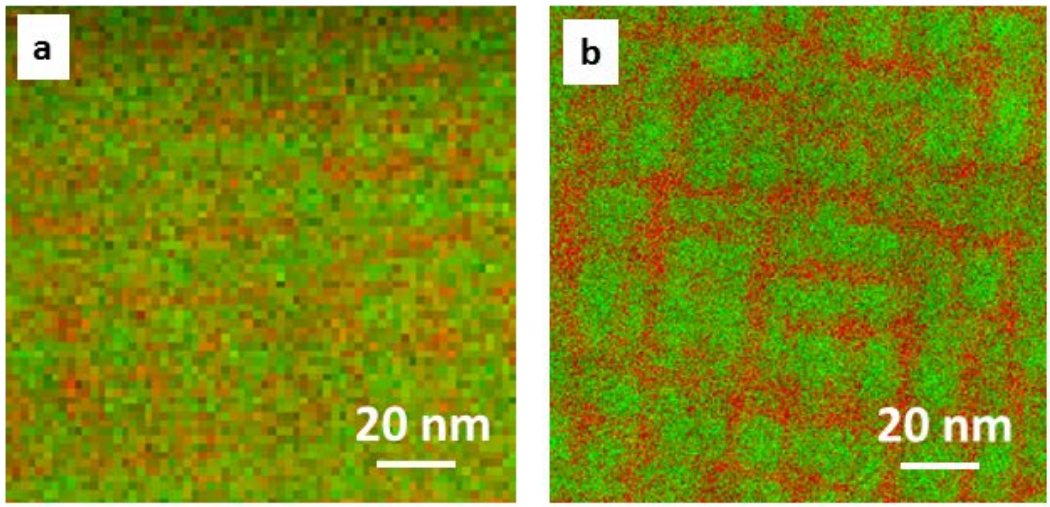

Figure 2. AXSIA analysis of spectral images of a spinodal decomposition in Paliney 7 from data acquired on a. an FEI Company Tecnai F30ST and b. an FEI Company Titan G2 80-200 with ChemiSTEM technology resulting in a 70-fold improvement in combined spatial resolution and analysis time.

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