Can X-ray Spectrum Imaging (XSI) Replace Backscattered Electron (BSE) Imaging for Compositional Contrast in the Scanning Electron Microscope?

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The silicon drift detector (SDD) has advanced energy dispersive x-ray spectrometry (EDS) in the scanning electron microscope (SEM) in remarkable ways, especially in the output count rate (OCR) capability, which has been improved by a factor of 10 to 100 over the OCR performance of the Si(Li)-EDS for the same spectral resolution [1]. SDD-EDS enables recording an x-ray spectrum image (XSI), wherein a complete spectrum of 4096 5-eV channels with full intensity range is stored at each pixel, in the same time that it takes to record a backscattered electron (BSE) image with high signal-to-noise, e.g., 30 seconds or less. The BSE image has traditionally been the SEM microscopist's favorite tool for visualizing compositional contrast, which arises because of the nearly monotonic increase of the BSE signal with increasing atomic number. The emergence of high speed SDD-XSI, coupled with elegant and efficient software tools to "mine" the resulting large databases for compositional information, raises the question whether SDD-XSI can replace BSE for detecting compositional contrast in the SEM [2]. SDD-XSI has the obvious advantage of the elemental specificity of x-ray spectrometry compared to the non-specific BSE response. To assess the relative merits of BSE and SDD-XSI imaging, the available contrast, limits of resolution, and speed of acquisition must be considered. (1) Can XSI compensate when BSE compositional contrast is weak? BSE compositional contrast is strong (C > 0.1) when the difference in atomic number, ΔZ , between imaged areas is large. When $\Delta Z = 1$, the BSE contrast is weak and decreases as Z increases: e.g., for Al-Si, C=0.060; Ni-Cu, C=0.026; Pt-Au, C=0.004. In the threshold contrast-current visibility equation, C enters with an exponent of 2, so that imaging weak contrast (C < 0.05) requires high beam current. More seriously, weak compositional contrast can be overwhelmed by BSE topographic contrast and BSE range effects [3]. For imaging weak compositional contrast, SDD-XSI imaging has a special advantage because the x-ray compositional contrast arises from differences in characteristic peak intensities, providing those peaks are resolvable and background corrected. An example is shown in Figure 1, where the BSE image from a leaded-brass particle is compared with Cu, Ni, and Pb maps from an SDD-XSI, both recorded in 20 seconds. The BSE image reveals the strong contrast of the lead-rich area vs. the rest of the brass particle but fails to show the unexpected Ni component for which $\Delta Z \sim 1$ relative to the Cu-Zn brass and C ~ 0.03 . The Ni is easily recognized in the XSI sum spectrum from which the Ni elemental map is derived. In this example, the Ni, Cu, and Zn K-family peaks are adequately separated. However, for $\Delta Z = 1$ in the range Z = 38 to 52, such as Pd-Ag or Sn-Sb, severe x-ray Lfamily peak overlaps occur so that peak deconvolution must be applied to the pixel-level spectra. In such cases, longer XSI acquisition times are inevitably needed to accumulate sufficient counts in the pixel spectra to enable robust peak deconvolution. (2) Spatial resolution: When boundaries due only to compositional change are compared, BSE images and XSI images convey similar spatial resolution. However, many practical

boundaries actually involve both compositional and fine scale topographical changes, and for these more complex boundaries, the BSE image usually preserves finer details. (3) Speed of acquisition: Compared to backscattering of 0.25 BSE/e⁻ for a Mn target at E₀=20 keV, the total measured X-ray emission is 1.8 x 10⁻⁴ photons/e⁻ (MnK- and Lshell characteristic and bremsstrahlung, 0.2–20 keV) with relatively uniform angular emission. Ignoring the cosine angular distribution of the BSE, any unit of solid angle thus contains about 1400 times more BSEs than x-rays. Inevitably, then, the BSE signal is much superior when the time speed of acquisition is considered. Useful BSE images can be recorded even at TV scan rate, which is not yet practical with SDD-XSI, so that XSI replacing the BSE image is not likely. Nevertheless, the advanced OCR performance of the SDD has reduced the time penalty to perform XSI mapping to less than 30 s, so the enormous advantage of the specific compositional information available from the XSI makes the combination of BSE and SDD-XSI ideally complementary tools for evaluating compositional microstructure. The microscopist will always record a BSE image, but now the corresponding SDD-XSI should be also recorded every time to avoid missing critical information like that discovered with the XSI in the example of Figure 1.

References

- 1. Newbury, D., SCANNING, 27 (2005) 227.
- 2. Newbury, D., et al., Microsc Microanal 13, Suppl 2 (2007) 1340CD.
- 3. Goldstein, J.I. et al., Scanning electron Microscopy and X-ray Microanalysis, 3rd ed., (Springer, New York, 2003) 175.

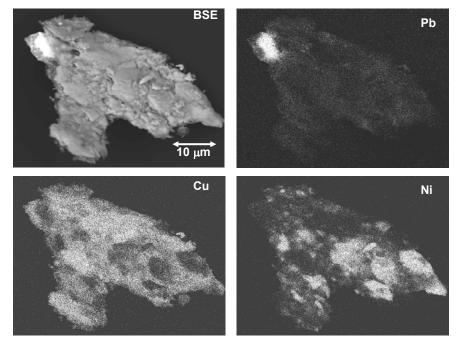


Figure 1 Leaded-brass particle: comparison of BSE (solid state detector) and SDD-EDS XSI; 640x480 pixels, $64 \mu s$ dwell = $\sim 20 s$ total scan; $E_0 = 20 keV$, $i_B = 25 nA$.