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Visualizing Low Contrast Compositional Microstructures with SDD-EDS X-ray Spectrum Imaging in the Scanning Electron Microscope

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The extraordinary x-ray throughput that can be achieved with the silicon drift detector energy dispersive spectrometer (SDD-EDS), e.g., an output count rate (OCR) exceeding 1 MHz with a spectral resolution of 145 eV (at MnKα) and with excellent peak stability (width and position) over the full range of input count rate, opens new possibilities for elemental mapping [1]. Elemental mapping has always been a popular mode of operation for scanning electron microscope-EDS evaluation of microstructures, but the limited OCR of the Si(Li)-EDS, even with a short time constant and resolution poorer than 160 eV, results in a severe time penalty when mapping minor and trace constituents or revealing small changes in concentration [2]. X-ray elemental imaging for such applications has previously been restricted to wavelength dispersive x-ray spectrometry (WDS), which still retains special advantages over EDS such as higher peak-to-background and no increase in counting deadtime due to high abundance matrix constituents. However, WDS is inevitably limited to mapping only the elements chosen, whereas EDS mapping in the x-ray spectrum imaging (XSI) mode of operation effectively captures all possible elemental information within the restrictions imposed by the primary dose and the EDS performance. Unexpected constituents can be identified in the XSI database with a variety of software tools and mapped without the need to make additional measurements [3, 4]. With SDD-EDS XSI, mapping of minor and trace constituents and low concentration contrast can be achieved with modest time expenditure. Figure 1 shows an example of imaging a complex Zr-Ni alloy with Ti, V, Mn, and Co present as minor constituents. The XSI was prepared with an SDD time constant that provided resolution of 127.5 eV, at an OCR of 300 kHz, and a pixel density of 512x384 with a dwell of 8192 μs for a mapping time of 27 minutes. The concentration ranges depicted in the gray scale images (background and interference corrected) represent minor V (0.013 to 0.098 mass fraction) and Cr (0.006 to 0.101), and major Ni (0.34 to 0.42). Sufficient x-ray counts were recorded to depict subtle concentration features with high spatial resolution. Figure 2 demonstrates SDD-EDS XSI imaging of low contrast of major constituents. Aluminum wire in a steel electrical junction box can fail through a high temperature process that forms a complex interaction zone containing Al-Fe intermetallic compounds. The XSI maps (512x384, 4096 μs dwell; 1.1 MHz at 145 eV resolution) in Figure 2 show the concentration contrast between Al_3Fe and Al_5Fe_2. The concentration contrast is 7.6% in the Al map and 9.9% in the Fe map.

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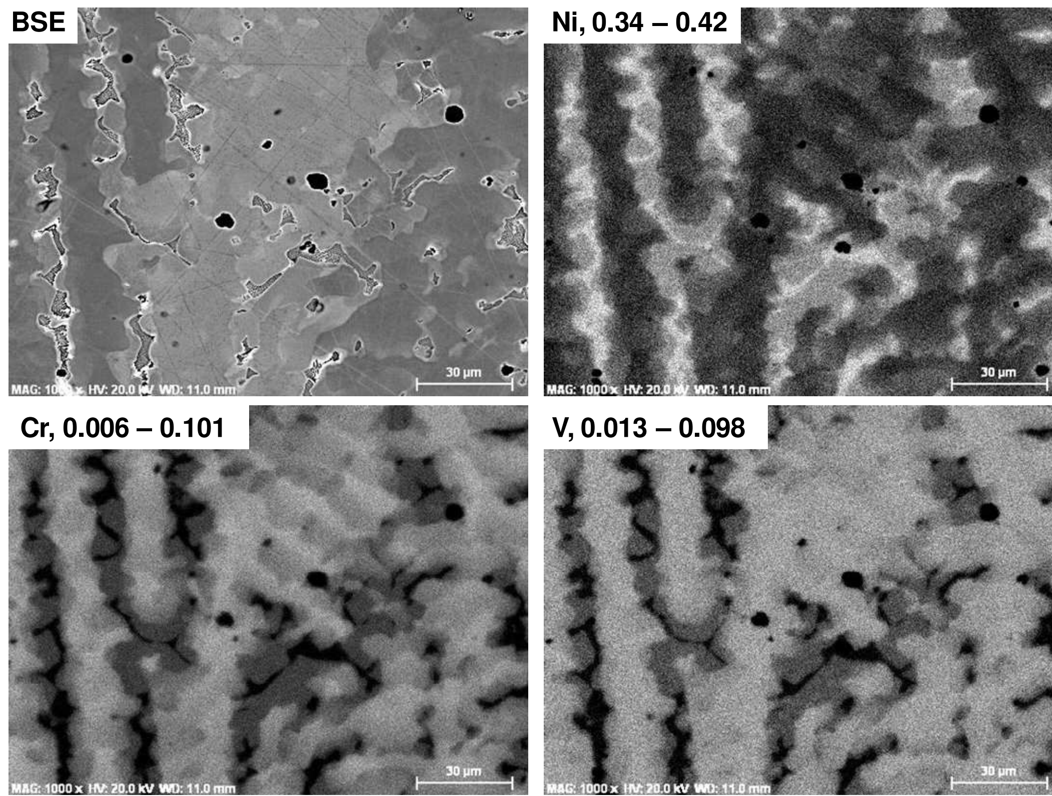

FIG1 Zr-Ni alloy with minor Ti, V, Cr, Mn, and Co. XSI: 512x384 pixels (8192 µs)

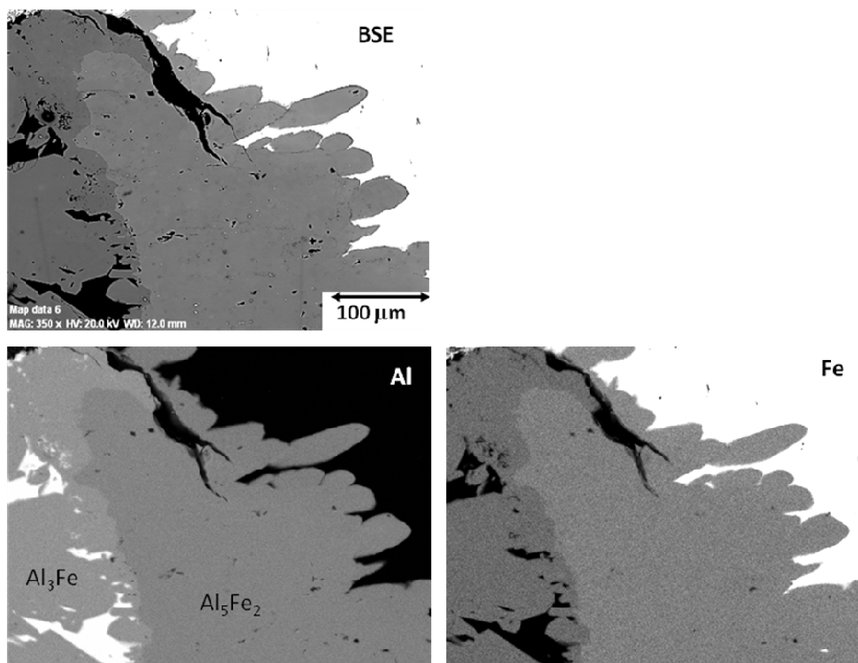

FIG. 2. Al wire – steel screw interface after thermal failure: 512x384 (4096 µs). In the Al map, the phase contrast is 7.6%, while in the Fe map the contrast is 9.9%.

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