

X-ray Mapping in the Spectrum Image Mode at Output Count Rates above 100 kHz with the Silicon Drift Detector (SDD)

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The emergence of the silicon drift detector (SDD) has resulted in a remarkable advance in x-ray throughput in energy dispersive spectrometry (EDS) [1, 2]. Figure 1 shows the output count rate (OCR) vs. input count rate (ICR) performance of a third design generation SDD where an OCR above 100 kHz can readily be achieved with useful resolution (163 eV, FWHM at MnK α), and an OCR above 500 kHz is possible for the shortest peaking time constant tested (250 ns) [3]. Such extraordinary count rate performance reduces the time for conventional x-ray mapping with defined windows, but it is especially useful for the x-ray spectrum image (XSI) data acquisition mode, wherein a complete EDS spectrum is stored at each pixel. XSI acquisition captures, in principle, all possible compositional information about the area being analyzed within the performance limitations of the spectrometer. There has been a simultaneous advancement of software tools that can efficiently extract information from the large databases created by XSI operation [4, 5, 6].

This combination of high EDS throughput, XSI operation, and software tools for data mining allows us to extend x-ray mapping into areas where it has previously been impractical. Several examples have been examined: (1) Detection of rare, unexpected inclusions, down to the single pixel level: Figure 2 shows an example of using the SUM spectrum tool [5] to detect a low concentration feature, iron, in Raney nickel alloy, and the MAXIMUM PIXEL spectrum [5, 6] tool to detect an unexpected contaminant, Ca, that occurs at a single pixel. (2) Detection of rare inclusions in high purity materials: SDD mapping permits rapid examination of many fields of view, most of which may be empty of features of interest in such pure materials. The MAXIMUM PIXEL spectrum enables rapid evaluation of XSI databases, revealing those image databases worthy of further examination. (3) Particle mapping: Conventional particle analysis usually proceeds by overscanning a particle while collecting the EDS spectrum to obtain a representative spectrum. This procedure obviously ignores any particle heterogeneity, which is often observed, especially in particle aggregates. XSI mapping enables the analyst to capture the true distribution of elements in heterogeneous particles. The SUM derived spectrum can then be calculated from the contiguous pixels that define each distinct region within the heterogeneous particle to determine chemical phases. Other important factors that affect the accuracy of particle analysis, such as particle geometry effects, can be directly detected in the shape of the x-ray continuum observed in the individual pixel or pixel group spectra and subsequently corrected by peak-to-background methods.

References:

- [1] L. Struder, et al, *Mikrochim. Acta, Suppl*, **15** (1998) 11.
- [2] S. Barkan, et al, *Microscopy Today*, **12**, (2004) 36.
- [3] D. Newbury, *SCANNING*, **27** (2005) 227.
- [4] P. Kotula et al, *Microsc. Microanal.*, **9** (2003) 1.
- [5] D. Newbury and D. Bright *SCANNING*, **27** (2005) 15.
- [6] D. Bright, LISPIX, a pc-based image processing engine; <http://www.nist.gov/lispix/>

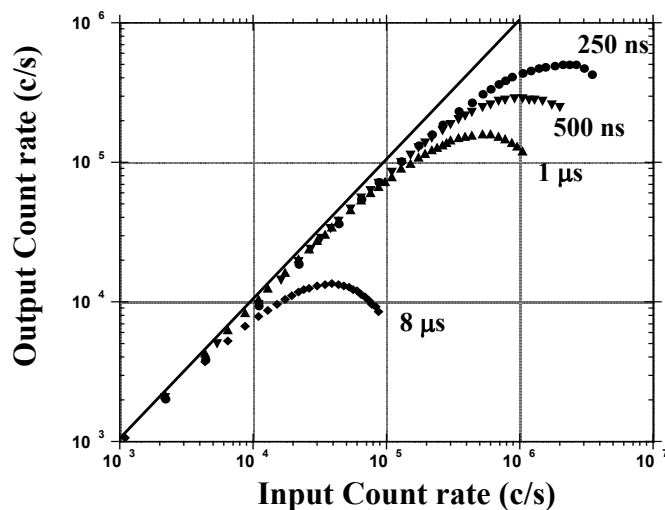


Figure 1. OCR vs ICR performance of a Radiant Detectors "Vortex" SDD with a peaking time constant (resolution) of 8 μs (134 eV), 1 μs (163 eV), 500 ns (188 eV) and 250 ns (217 eV).

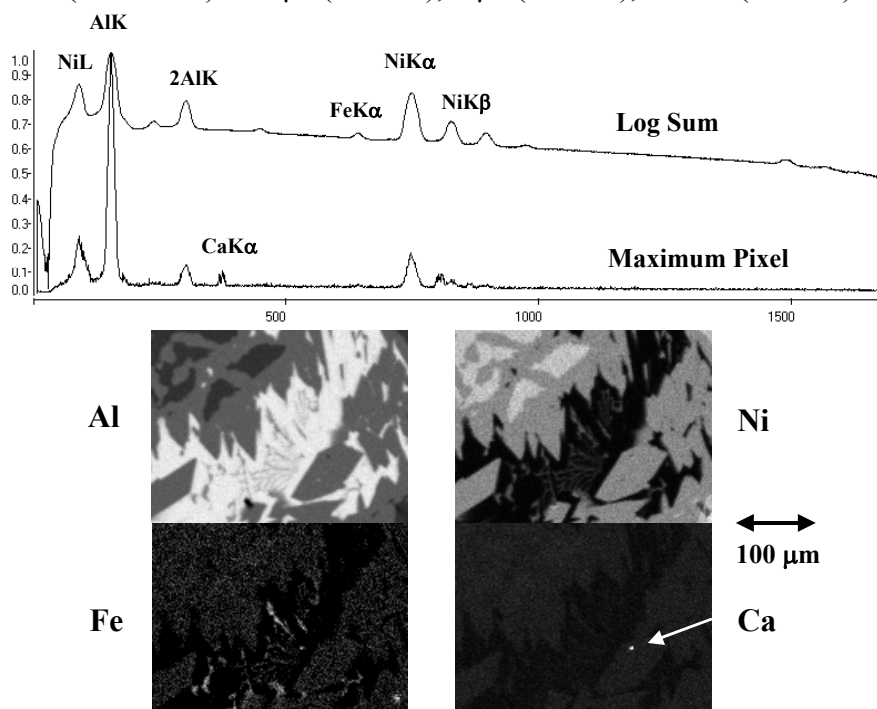


Figure 2. X-ray maps derived from XSI of Raney nickel, showing Fe-phase and Ca-rich inclusion.