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The New X-ray Mapping: Applying the Silicon Drift Detector (SDD) for X-ray Spectrometry and Spectrum Imaging with Output Count Rates above 100 kHz

Dale E. Newbury

National Institute of Standards and Technology, Gaithersburg, MD 20899-8370

The silicon drift detector (SDD) is rapidly emerging as the detector of choice for most applications of energy dispersive x-ray spectrometry (EDS) in electron beam instruments, with several manufacturers competing to exploit this detector class [1]. Development of the third generation of SDDs has resulted in an EDS that operates near room temperature with most performance measures equal to or superior to the conventional thick-crystal Si-EDS that operates near liquid nitrogen temperature [2]. Third generation SDDs have demonstrated: (1) resolution performance that is actually superior to Si-EDS for a given detector area, e.g., 134 eV (MnKα) for a 50mm^2 detector; (2) extreme peak stability at maximum resolution, e.g., FWHM broadening of 0.003 eV/percent deadtime at 134 eV; and (3) output count rates above 100 kHz with useful resolution. Figure 1 demonstrates that an output count rate (OCR) above 100 kHz is possible with a resolution of 163 eV, while an OCR above 300 kHz is possible with the resolution degraded to 217 eV. For x-ray mapping, the SDD opens up new possibilities in high speed data acquisition. The SDD makes it attractive to map in the x-ray spectrum image (XSI) mode, in which a complete EDS spectrum, consisting of 2048 channels of 10 eV-width and 2-byte intensity range, is stored at each pixel of a scanned image. A surprising level of data quality can be captured with a short time investment. Figure 2 shows a map of Raney nickel alloy (Ni-Al) captured in 185 seconds (128x128 pixels, 10 ms dwell per pixel with 1.3 ms overhead per pixel for beam positioning and data storage). In addition to achieving data capable of distinguishing the three distinct Ni-Al phases, it is also possible to distinguish a phase containing Fe at the level of 0.042 mass fraction. XSI data collection captures all possible compositional information within the performance limitations of the SDD. A typical XSI data set can be 100 Mbyte or larger, and the prospect is that we will soon be collecting such datasets in Gbyte quantities. This situation severely challenges our software tools to efficiently mine these datasets. Various groups are exploring multivariate data analysis tools for this purpose. Bright and Newbury have pursued the method of "derived x-ray spectra", in which a spectrum-like display is calculated from some or all of the pixels in a channel image [3]. An example is the familiar "sum spectrum", in which all counts in a channel image are added. Peaks in the sum spectrum identify high abundance features in the XSI. The maximum pixel spectrum, in which the maximum value in each channel image is chosen to create the derived x-ray spectrum, has been shown to be highly sensitive to rare, unanticipated features which may occur at a single pixel [3]. SDD XSI mapping and a software toolkit of powerful data mining tools will permit SEM x-ray mapping to enter application areas that previously were impractical, such as evaluating large specimen areas by tiling many high spatial resolution maps, locating rare inclusions that localize trace elements in high purity materials, and detecting contaminants in manufacturing processes.

References

1. L. Struder, et al., Mikrochim. Acta 15, suppl. (1998) 11.2.
2. S. Barkan, et al., Microscopy Today, 12 (2004) 36.
3. D. Bright and D. Newbury, J. Micros. 216 (2004) 186.

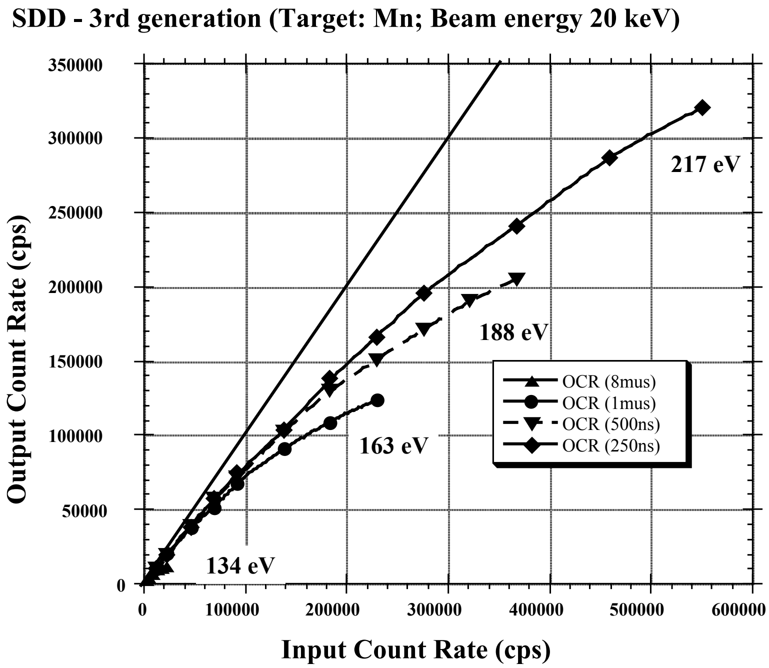
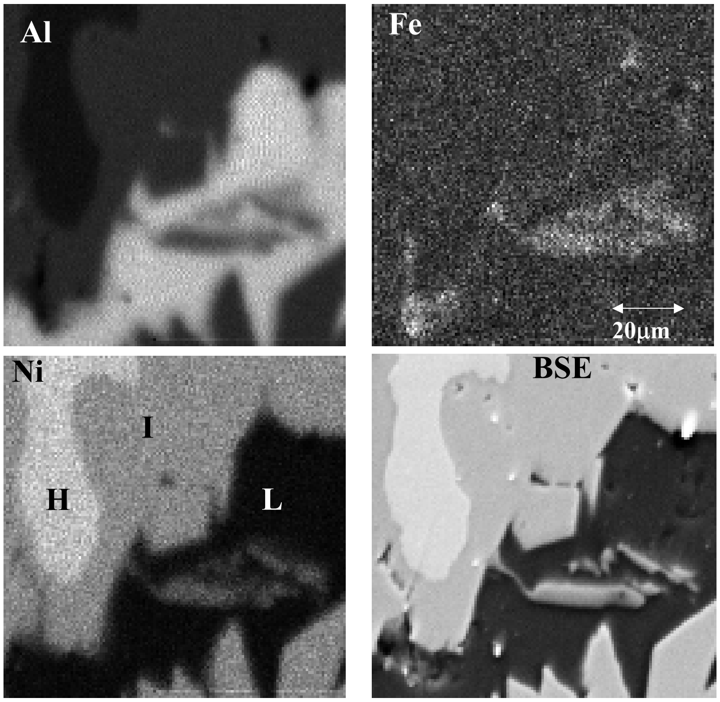


Figure 1. Output count rate vs. input count rate for SDD with different time constants.



Raney Nickel Alloy
E_0 = 20 keV 10 nA
TC = 500 ns (188 eV at MnKα)
128x128
10 ms per pixel
Mapping 185 sec

Phases
Al 99.5 Ni 0.5 "L"
Al 71.2 Ni 24.6 Fe 4.2
Al 60 Ni 40 "I"
Al 46.5 Ni 53.5 "H"

Figure 2. Selected images derived from x-ray spectrum image of Raney nickel collected in 185 seconds (128x128 pixels, 10 ms per pixel plus 1.3 ms overhead) and backscattered electron image.