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DOI: 10.1017/S1431927606065792

Microsc Microanal 12(Supp 2), 2006

High Speed Particle Analysis with a Silicon Drift Detector

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One of the principal reasons that automated particle analysis by scanned electron beam has not been adopted more widely by industrial quality assurance and quality control (QA/QC) is the analysis duration. Many industrial users could use the diagnostic information from electron beam studies of particle populations but are unwilling to spend hours per specimen to sample enough particles for meaningful population statistics. An optimized e-beam particle analysis systems with standard lithium-drifted silicon (Si(Li)) energy dispersive x-ray detectors (EDS) can analyze about 1000 particles in an hour on a moderately heavily laden sample assuming an acquisition time of about 3 seconds per particle. The majority of this time is spent collecting EDS spectra with particle search and stage movement time as substantial second-tier contributors.

Silicon drift detectors (SDD) potentially represent a way to reduce the time spent on x-ray analysis. SDD offer higher maximum output count rates at equivalent resolution. A 50 mm^2 SDD detector can measure over 150,000 output x-ray events per second at a time constant of 1 µs and a resolution of 163 eV[1]. At a time constant of 1 µs the resolution of an Si(Li) detector would be well over 200 eV. At 4 µs process time, a Si(Li) detector is capable of less than 25,000 output x-ray events per second at 178 eV resolution.

To demonstrate the practical difference between a 50 mm^2 Gresham Si(Li) and 50 mm^2 Radiant SDD detector, one of each was installed on an ASPEX Personal SEM[2]. The detectors were installed at rotation symmetric locations with respect to the beam axis. A particulate sample was created from K3189, a glass of known composition, by cryomilling a bulk sample. The system was both configured for high throughput at roughly the same resolution and dead time (30%) on each detector. The SDD detector was configured with a 1 µs process time and a 10 second acquisition time and the Si(Li) detector was configured with a 6.4 µs process time and a 30 second acquisition time. Over 1000 particles with average diameters of 1 to 10 µm were analyzed with each detector using ASPEX Automated Feature Analysis (AFA) software. The resulting spectra were sub-sampled (taking into account counting statistics) to simulate acquisitions of shorter duration. Figures 1A)-1D) are ternary diagrams showing the relative variation in the Fe, Si and Ca k-ratios as a function of detector and acquisition time. 1A) and 1C) show spectra collected on the SDD at 0.4 and 6.4 second acquisition times. 1B) and 1D) show spectra with similar variation collected on the Si(Li) detector at 1.6 and 25.6 second acquisition times. These plots demonstrate that the SDD is capable of collecting similarly precise data in ¼ the acquisition time of the Si(Li) detector. Increasing the acquisition time beyond 6.4 seconds for the SDD and 25.6 seconds for the Si(Li) detector gave little improvement in precision.

The enhanced performance characteristics of the SDD detector should initiate a rethink of the design of the optimal electron beam system for high speed particle analysis. The potential for higher throughput requires higher beam brightnesses to make the optimal use of the SDD while retaining equivalent image resolution and size measurement precision. While a tungsten filament SEM is well matched to a Si(Li) detector, a LaB_6 or thermal field emitter may be a better match for the SDD. In addition, increased beam brightnesses will improve the imaging detector (BSED or SED) performance and further speed up the search and measurement process. All of these improvements will make a powerful tool more attractive to QA/QC laboratories potentially improving our nation's industrial competitiveness.

[1] D. Newbury, Scanning **27**, pp 227-239 (2005)
[2] Disclaimer: Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

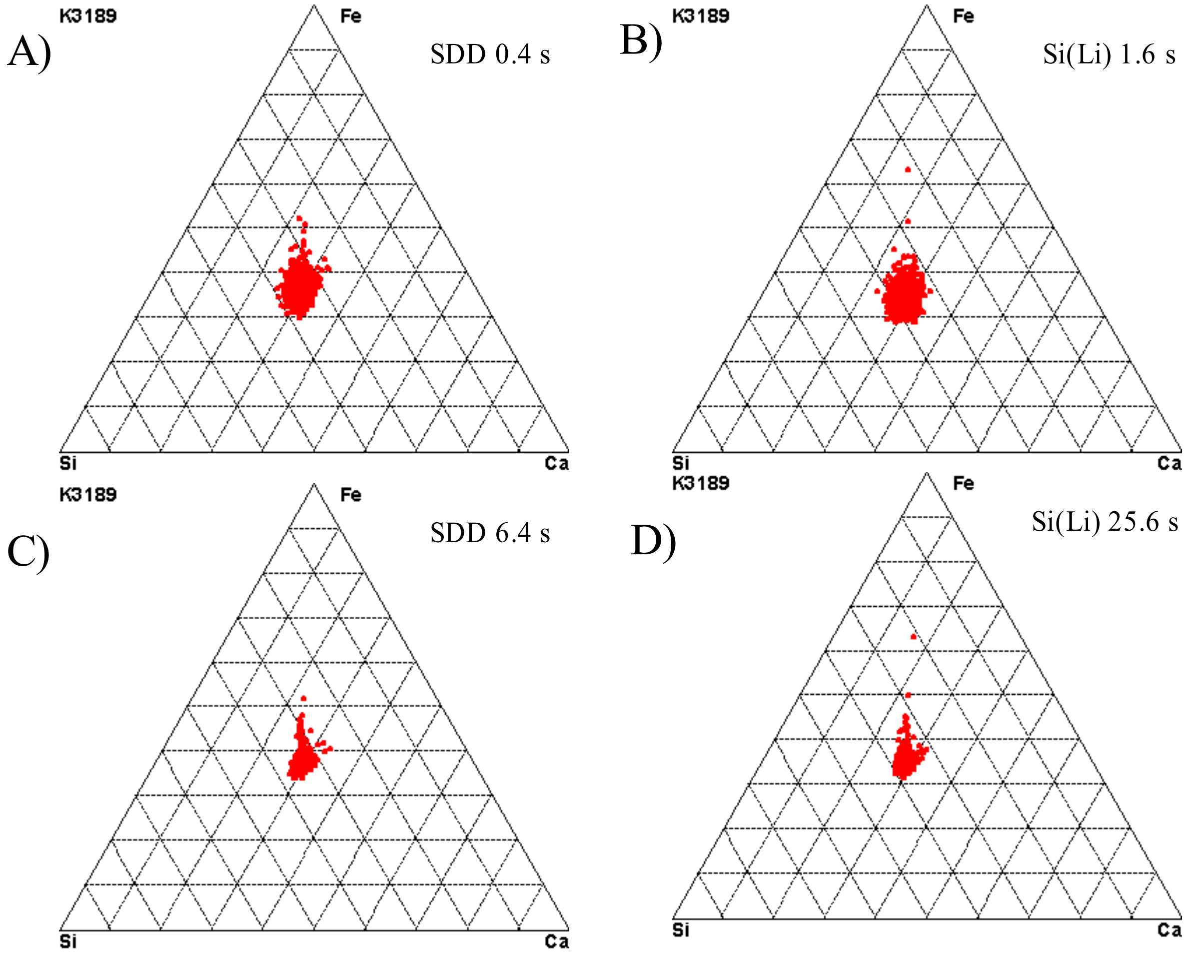

Figure 1: Four ternary diagrams visualizing the spread in Si, Ca & Fe k-ratios as measured from K3189 glass particles using a Gresham Si(Li) and a Radiant SDD detector. The acquisition times in A & B (0.4 and 1.6 seconds) and C & D (6.4 and 25.6 seconds) were chosen to give similarly precise results.