Methods of Maximizing X-ray Detection in SEMs

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Sample throughput is a significant issue in the microscopy lab. It is possible to improve the efficiency of analysis by changing the way we operate the microscope or upgrading the analytical system. One method to increase the efficiency of the analyst is to automate as many procedures as possible, including acquisitions. However, the most effective way to increase efficiency is to reduce the time that the analyst spends acquiring data. This is fine, but statistical confidence must not be reduced by the use of shorter acquisition times and the quality of the data or shape of the peaks must not be compromised as well (Figure 1, Table 1). To maintain the same level of statistical confidence of a longer analysis, an increase of the x-ray detection rate is needed.

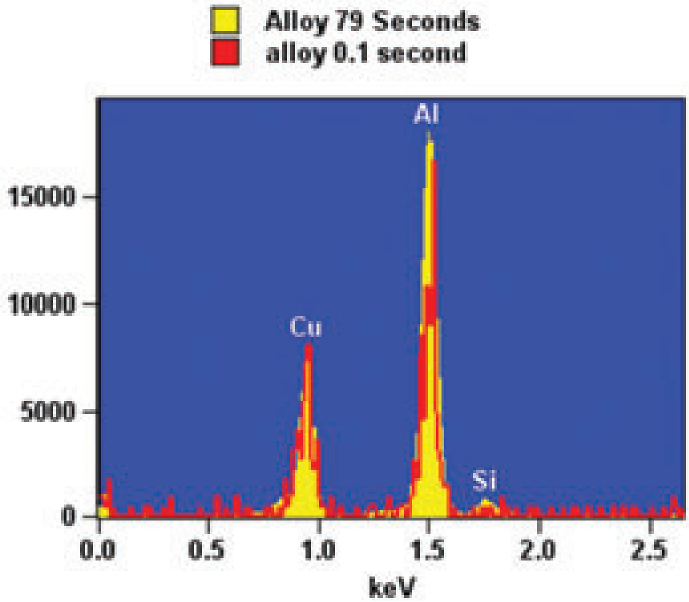

FIGURE 1: Comparison of acquisitions of 0.1 second versus 79 seconds for a typical Si(Li) detector. Note the increased Relative Errors at the shorter counting time.

Table 1: Integrated peak net counts and associated errors for short and long acquisition times

Element Line	Net Counts	Net Error	Relative Error
	0.1 Seconds		
Al *K*	204	+/-11	5 %
Si *K*	1	+/-3	300 %
Cu *L*	85	+/-13	15 %
	79 Seconds		
Al *K*	161006	+/-702	0.4 %
Si *K*	7232	+/-199	2.8 %
Cu *L*	43648	+/-419	1.0 %

One of the methods to increase the detection rate of an EDS analysis in an SEM is to increase the beam current of the SEM. This is beneficial, until the physics of the electron beam and incoming x-ray flux swamps the detection ability of the sensor and electronics. For lithium-drifted silicon diode (Si(Li)) detectors, this is usually not a problem until the x-ray storage rate exceeds ~50k cps. At these rates, the spectral performance of Si(Li) detectors degrades significantly. For these rates, silicon drift detectors (SDD) are preferred because their spectral resolution does not degrade as quickly, and more beam current can be deposited into the sample for increased x-ray generation and detection (Figure 2).

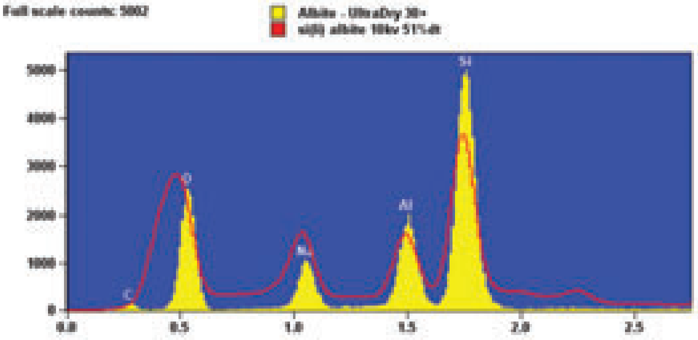

FIGURE 2. Spectra collected with 30 mm^2 UltraDry SDD and 10 mm^2 Si(Li) detectors at identical SEM operating conditions. The incoming x-ray rate is beyond the usable operating range of the Si(Li) detector.

To further increase the x-ray storage rate, an increase in the detector sensor size is extremely beneficial. Using a 30 mm^2 sensor provides an instant 3x increase in the potential storage rate (Figure 3). This is used to great effect for low beam current applications like TEM and cold FESEM where large area Si(Li) sensors with 30 or 40mm^2 active area have been in use for many years.

One way to avoid overloading the electronics with this increased detection rate is to increase the number of parallel electronics circuits. This increases the storage rate in a linear fashion based on the number of electronic front-ends. The sensors for these parallel circuits could come from a single detector or from multiple detectors. It would appear that having multiple sensors in a single detector would be the most practical solution. However, the physical size of the detector vacuum envelope causes secondary issues to be considered. Quadrupling the area of a detector will necessarily increase the diameter by a factor of 2. A detector container of this size may have to be mounted at a larger distance from the sample compared to the smaller detector. In this configuration, the x-ray detection and storage rate will not increase by the expected factor of 4 because of the reduction of the solid-angle of the detector at this long distance. A smaller diameter 30mm^2 detector at a closer distance may actually have more solid angle (and therefore x-ray storage rate) than the larger diameter 40mm^2 at a greater sample to detector distance (Figure 3). This indicates that the detector sensor size is an important consideration but is not the most important. The solid-angle of the detector is a much more important consideration

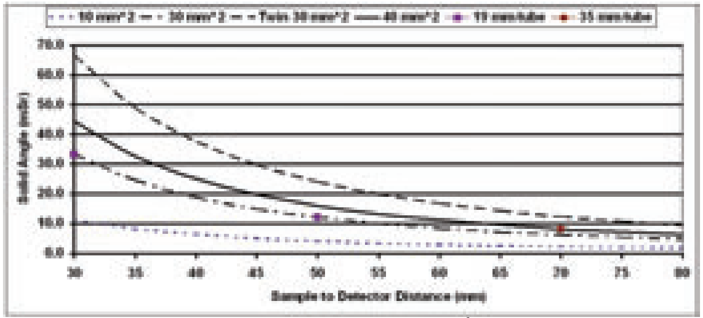

FIGURE 3. Detector geometry comparisons for various sensor sizes, sample-to-detector distances, and tube diameters.

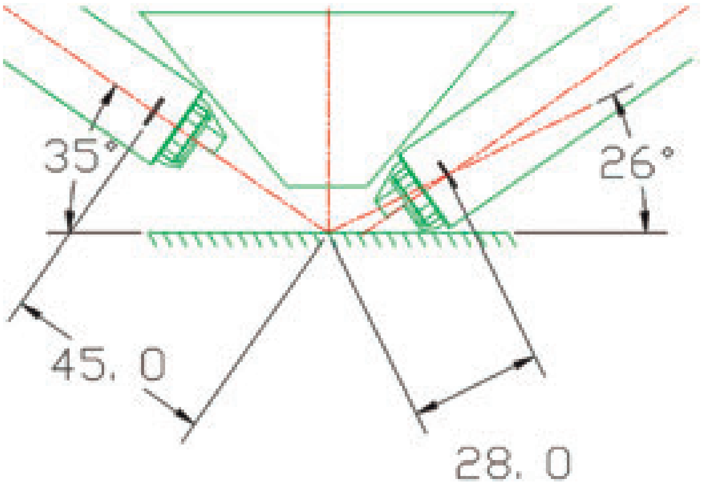

FIGURE 4. Schematic chamber drawing with 30mm^2 detector in a 19 mm tube. Note that a larger tube would require the need of a larger sample-to-detector distance (see points in Figure 3).

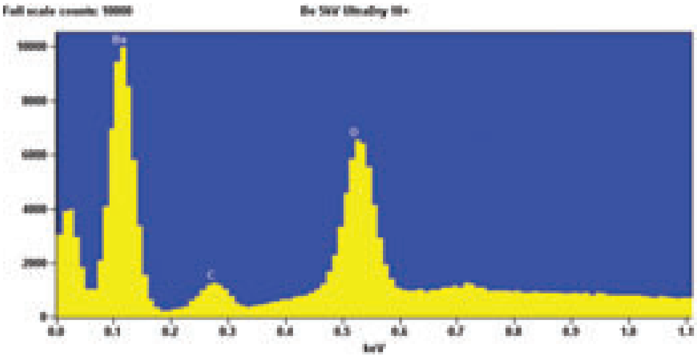

FIGURE 5. New Silicon Drift Detectors with 10mm^2 and 30mm^2 sensors have sensitivity down to Beryllium increasing the confidence in low energy analysis.

for the efficiency of the analyst.

An increase of solid-angle is thus shown to be beneficial and is the easiest method to accomplish: simply place the detector closer to the specimen. In practice, obstructions may be encountered, so a different geometry may be needed (Figure 4). If the detector is designed from the outset for optimal microanalysis, extremely high solid-angle values may be realized (Figure 3).

Confidence in the resulting data requires not only good counting statistics but also good spectrum quality. Recent improvements to the silicon drift detector sensor and supporting electronics have allowed resolution of low energy peaks while still maintaining the high throughput that silicon drift detectors are known for. This improvement now allows for detection of Gaussian peaks down to 110eV (Beryllium) and in some cases below this energy (Figure 5). This allows the silicon drift detector to be routinely used in applications that require low beam energy such as polymers and non-conductive samples.

Conclusions

High laboratory efficiency requires fast data acquisition while maintaining data integrity. Optimization of x-ray collection can include larger x-ray detectors, multiple detectors, and optimized detector mounting. The latest generation of large-area, silicon drift detectors and dedicated electronics are making significant progress in helping analysts. Future developments will require that silicon drift detectors perform well in all types of electron microscopes. ■