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Improving Sensitivity and Productivity with High count rate X-ray spectrum images

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The headline performance advantage of SDD technology and a major reason for its rapid adoption for EDS analysis is its significantly improved count rate handling capability. With older Si(Li) detectors, count rates of few 10s of thousands of counts per second were practical. With SDD technology 100s of thousands of counts per second can be used, and with larger area SDD sensors such rates can be achieved at relatively low beam currents. These count rate improvements have made it possible to use X-ray maps for routine sample investigation and collect complete spectra at every pixel, where previously only spectra were acquired by the user selecting positions on an electron image.

There are two main motivations for collecting higher count rate X-ray maps: improving productivity by faster visualization of the spatial distribution of major elements, and improving sensitivity by collecting more X-ray counts to detect low concentration elements or low abundance phases. It is well known that there is a trade off in accuracy at high count rates caused by worsening peak resolution and an increased chance of peak overlap. This is partly mitigated by the much better count handling characteristics of SDD and improved algorithms to address peak overlaps in X-ray mapping data.

In this study we have looked at the usefulness and potential pitfalls in pushing for higher count rates by acquiring spectrum images over the same sample area at high count rate in less than 1 s (to study productivity) and 2.5 minutes (to study sensitivity), at 20 kV with a 150 mm^2 detector. The sample is a mineral grain mount used in the study of ore liberation, containing different phases including silicates, sulfides, sulfates and phosphates. The results of the two datasets for sulfur, plotted as windows integral X-ray maps, are shown in Fig. 1. The 1 s acquisition clearly shows the location of sulfur where present in high concentration. The 2.5 minute acquisition shows greater sensitivity where a number of different contrast levels are now visible in some grains and a large number of additional grains are seen with an apparent lower sulfur concentration.

As a benchmark we have also collected datasets with the same number of counts, but using a low count rate and an extended acquisition time (45 minutes), but under otherwise identical conditions. If there is no count rate dependence, the 45 minute X-ray maps (Fig. 2a) for aluminium (Al Kα), lead (Pb Mα) and sulfur (S Kα), should show the same results as the equivalent high count rate maps (2.5 min. - Fig. 2b). This is the case for Al, however for Pb and particularly S there are differences in phases with visually lower concentrations. One benefit of collecting a spectrum at every pixel is that we can process pixel by pixel to remove peak overlap and background variation artifacts. The results of this processing (Fig. 2c) show that much of the apparent information in the 2.5 minute acquisition is actually caused by variations in X-ray background intensity between phases. There are sufficient counts to allow effective separation of the PbM and SK signals to show that Pb is present in only one small phase (PbS). This result shows that despite degradation of spectrum resolution, modern algorithms can do useful spectrum processing on high count rate data. However, problems occur if the shape of the spectrum peaks are distorted due to the short processing time of each X-ray signal and the short time between incoming X-ray events experienced at high count rates. The most commonly-seen example of such pile-up effects is “sum” or “coincidence” peaks. In Fig. 2d, the 2.5 minute maps have been further processed to include a pixel by

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pixel pulse pile-up correction. This removes the last of the low intensity regions seen in grains of the sulfur map, which are actually Si + O pile-up from quartz (SiO_2) grains. The final S map highlights 4 different sulfur concentration levels ($BaSO_4$, FeS_2, ZnS and PbS).

The 1s acquisition shows collecting at high count rates can provide a near real-time picture of the distribution of major elements in a sample provided these elements can be identified. We have therefore been investigating new approaches to identify the major elements present, particularly in minor phases like the Pb in PbS, where overall statistics are very low. The 2.5 minute acquisition shows that minor and major elements can correctly be identified with suitable processing and we have been studying the use of the full spectrum image for automated particle analysis where grains cannot be easily distinguished by backscattered electron intensity but can be distinguished by the X-ray signal.

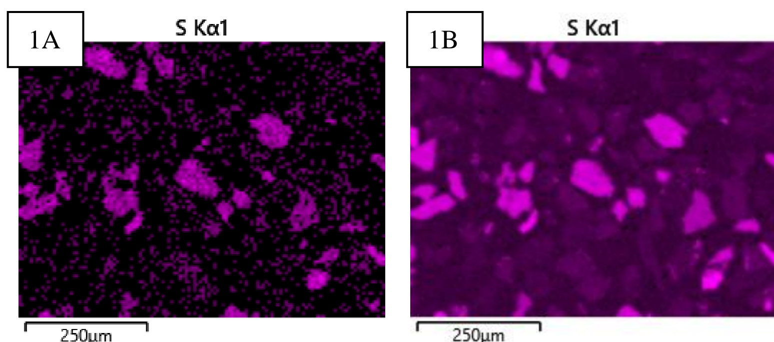

Figure 1. Sulfur X-ray maps collected for (A) 0.9 s (350,000 cps) and (B) 154 s (220,000 cps) from a mineral grain mount sample.

Figure 2. (A) Al, Pb and S X-ray maps collected at low count rate for 45 minutes (4,500 cps). (B) X-ray maps collected for 154 s (200,000 cps) for the same total counts. (C) 154 s data processed to separate peak overlaps and remove X-ray background (TruMap). (D) 154 s data processed as quantitative maps also corrected for pulse pile-up at each pixel

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