

Accurate Quantitative EDS Mapping at High Count Rates with a Large Area Silicon Drift Detector

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With the introduction of silicon drift (SDD) detectors, EDS analysis is now possible at acquisition rates in excess of 100,000cps. In particular, rapid qualitative analysis of large areas of a sample is routinely available using spectrum images or X-ray maps.

At high count rates with SDD detectors there is the opportunity for individual photon charge clouds or pulses to overlap causing pile-up problems. Pile-up affects not only the spectrum image but also any spectral data reconstructed from it. Without careful post-collection analysis of the map data or use of specialised pile-up correction software to remove errors [1], these effects can lead to misleading elemental representation errors in the X-ray maps and any spectral data reconstructed from them.

New large area SDDs with active areas up to 80mm² have recently emerged which offer the potential for fast analysis with lower beam currents and with improved accuracy. This enables high count rate mapping and quantitative analysis under conditions which have not previously yielded enough count rate. Software pile-up correction can then be used on the collected data to remove residual sum peaks and pile-up continuum from maps and to correct for losses from peaks so that peak area ratios are preserved for accurate quantitative spectral reconstruction. This work follows a previous study which shows that quantitative results from spectrum image data can be as accurate as results from a live spectrum provided accurate lifetime correction circuitry is used [2].

Figure 1 shows two maps collected under the same beam current, kV and geometry conditions on a sample of cast aluminium alloy. Fig 1a shows an EDS map collected using a conventional 10mm² SDD detector and Fig. 1b shows data collected with an Oxford Instruments X-Max 80mm² SDD detector. Both maps illustrate the phase differences but it is not until the sum spectra from both maps are compared that the real difference in count rate between the two maps becomes apparent. The map collected with the 80mm² detector has over 7 times more counts than the map collected with the 10mm² detector. The sum spectra (Fig. 1c) also clearly highlight the problems seen with pile-up in very fast maps. The strong AlK_α signal at 1.48keV gives a pile up signal at 2.96keV which in maps can be mistaken for AgL_α if no pile-up correction routines are used. Another pile up peak is seen at 2.4keV which is a mixture of the CuL_α + AlK_α peaks and could be mistaken for PbM_α at 2.34keV.

Figures 2a–c show maps of the elemental distributions of Al, Ag and Pb in the sample. Without investigating each point individually, there is no way to identify whether any of the Ag or Pb is actually present in the material. By using a quantitative mapping analysis program which automatically identifies and removes pile-up at each pixel before constructing the map, it is possible to generate quantitative elemental maps which accurately depict the elemental concentrations present in each phase. Figures 2 d-f show these ‘Quant Maps’. These maps

illustrate that the Ag signal is an artefact due to pulse pile up, whereas there are real small precipitates of Pb present in the sample.

Although mapping at high count rates is becoming more accepted, these results clearly demonstrate the care that must be taken when assessing the validity of results. A combination of pile-up correction, quantitative mapping and a large area SDD detector offers the ability to collect large amounts of data in a very short time and reach realistic conclusions about the specimen.

References

- [1] P.J.Statham, *Microchimica Acta*, 155, 289-294, 2006
 [2] S.R.Burgess et al, *Microsc Microanal* 13 (Suppl 2), 2007

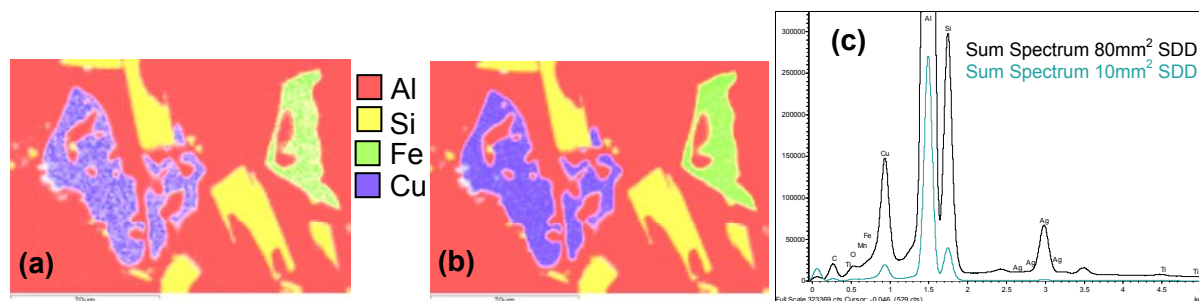


Figure 1. Composite 'mix maps' and associated sum spectrum. (a) Mix map from a conventional 10mm² SDD detector showing the aluminium, silicon, iron and copper EDS spectrum images overlaid (b) Mix map from an 80mm² large area SDD detector (c) Sum spectra from each of the two mix maps overlaid for comparison.

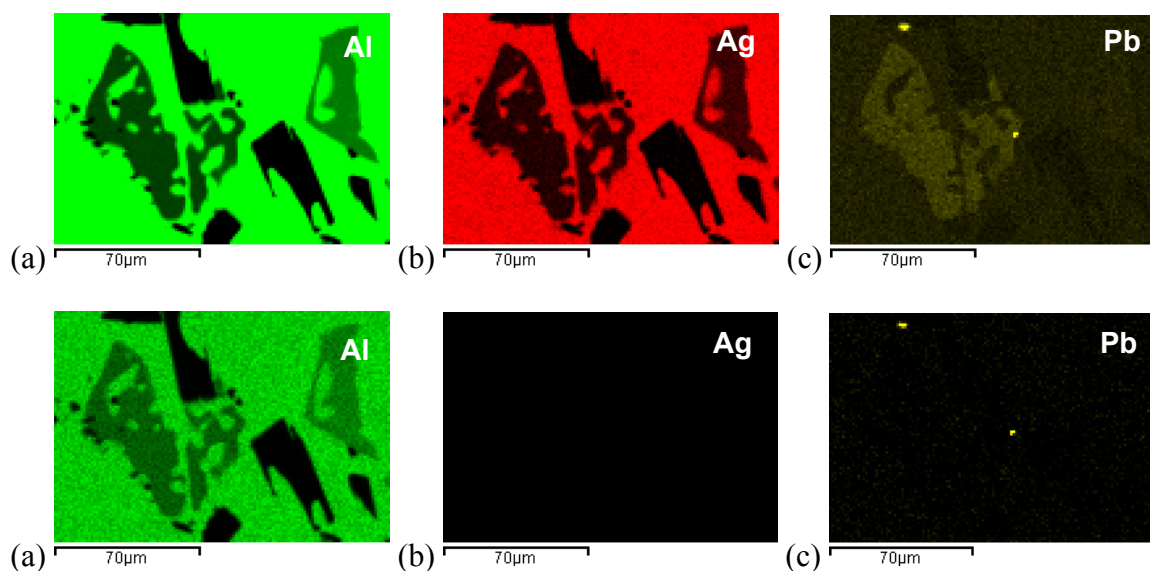


Figure 2. (a-c) Elemental distribution maps displayed without any post-collection analysis. Green = Al, Red = Ag and Yellow = Pb. (d-f) Elemental distribution 'Quant Maps' reconstructed after pile-up correction at each pixel. Green = Al, Red = Ag and Yellow = Pb.