X-Ray Mapping Using Multiple-EDS and WDS Detectors

R. Wuhrer [1], K. Moran [1,2], M. R. Phillips [1] and P. Davey [2]

[1] Microstructural Analysis Unit, University of Technology, Sydney, PO Box 123 Broadway, Australia.
[2] Moran Scientific Pty Ltd, P.O. Box 651Goulburn NSW 2580 Australia.

High quality x-ray mapping (XRM) has been used for over 30 years by experienced wavelength dispersive spectroscopy (WDS) operators. Manufacturers have been developing similar techniques using energy dispersive spectroscopy (EDS) over the last 20 years. This has been generally unsuccessful due to a number of problems such as poor computer specifications, cost, time to map and generally poor peak to background ratios (P:B). With improvements in all the above parameters EDS mapping is now gaining in popularity.

Originally regions of interest mapping (ROIM) has been used but this has now been replaced with the more powerful quantitative x-ray mapping (QXRM). QXRM has become a very powerful technique enabling reliable quantitative results that can be an order of magnitude better than traditional analysis. It is also far superior to ROIM where low levels of an element or elemental overlaps are present.

To take advantage of this increased precision and sensitivity of QXRM requires careful attention to all those parameters necessary for high quality analysis. Important characteristics such as resolution, gains zero calibration, sum and other artifact peaks need to be well characterized for each detector that is being used in the final result. The final image is created from the fully background corrected, overlap corrected and inter-element corrected calculation at each pixel in the image. As there is a very large dynamic concentration range to be considered (5 orders of magnitude) it is important to understand that the precision at each level will depend on how well this characterization is carried out. The low level range requires much more careful work than for major element work. To achieve this, time is no longer an issue but a means to accomplish a more precise result. Correspondingly, if we can introduce more efficient detection then we can achieve this result in a shorter time. This may be achieved by counting more x-rays with the same detector so long as the critical quantitative characteristics of the detectors are not compromised. This can also be currently achieved by adding more detectors.

XRM has been considered a slow technique, claiming many hours of SEM time and often relegated to overnight operation when prolonged runs could be accomplished. This is especially true when trying to obtain high spatial resolution x-ray maps with good statistics at low levels of concentration and a reasonable energy resolution (<145eV). A 512 by 512 EDS map collected at 10kcps mapping to the 1% level takes around 10 hours to collect, which is reasonably long. Furthermore, an SEM with accurate beam stability for that period is required. Resorting to increased beam current or higher count rates does not always obtain the required result although this may be an option under certain circumstances.

The introduction of a second EDS x-ray detector can reduce the map time accordingly, for example from 10 hours to 5 hours. This is now becoming a more acceptable time frame for x-ray mapping, but still is to long. It is important to realise that each time you double the number of detectors you halve the time to collect a map. The biggest time advantage is in adding a second detector. However, a two time statistical advantage requires four times the number of detectors.

A more acceptable time frame would require at least ten EDS or other x-ray detectors thus reducing the time ten fold. Therefore, the 512x512 EDS map collected at 10kcps could be reduced to a one hour map. Furthermore, the incorporation of WDS detectors is also very useful for trace elements, light elements, elements with strong EDS overlaps or where high count rates are required. Our current work involves a multi-detector energy dispersive spectrometer (EDS) and wavelength dispersive spectrometer (WDS) x-ray mapping system incorporated on a Jeol 733 microprobe (Figure 1a) and a Jeol 35CF with two EDS detectors (Figure1b). The Jeol 733 is currently operating with three EDS x-ray detectors and three WDS detectors simultaneously.

For QXRM to work properly for multi-detector systems, the characteristics of each detector must be accurately determined so that the final quantification of the individual detectors can be summed. To accomplish this effectively, the full spectrum at each pixel for each EDS detector should be saved. As a final check for consistency between detectors, a technique was developed that involves assigning a different RGB colour for each detector for the same element. By doing this, when we combine the three maps of the same element, we should obtain a grey scale map, not a colour map. This indicates total correlation between the three detectors at the most critical final stage of quantification.

In this paper we will be discussing the advantages and necessities of mapping using multiple EDS detectors with special emphasis on the effect of EDS parameters such as resolution, dead time, pulse pileup, take off angle, window thickness, solid angle and gain zero calibration as well as techniques developed to make sure accurate x-ray mapping can be done.

a. b.

Figure 1: a) JEOL 733 Microprobe with three EDS X-Ray detectors and three WDS detectors and b) JEOL 35CF with two EDS X-Ray detectors.