X-Ray Mapping Considerations and Difficulties

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X-Ray Mapping (XRM) has become a very powerful technique in understanding the distribution of elements in materials. X-ray maps are usually collected using raw counts from the elemental peaks of interest and once you start mapping you realize that for excellent quality results, for most real samples, it is hard not to want to map everything one comes across. This is because, when you start mapping you realise what variations there are to the elements in any sample. However, there are problems with XRM and there are many x-ray mapping considerations required, and difficulties needing to be understood, before you start.

So what is the major problem with x-ray mapping. The two main problems with XRM are the time to map and secondly the peak to background ratio (P:B), which to some extent comes back to time. Analysts are being requested for x-ray mapping as a problem-solving tool in materials science, with the results required as soon as possible. Ideally, we would like to obtain x-ray maps as quickly as obtaining an SE/BSE image on the microscope. A 256x256 map collected over 30 minutes at 20kcps should give a good x-ray map to the 1wt% level, but the image resolution would be on the poor to fair end of the scale. A better resolution of 512x512 is more acceptable, but unfortunately it takes 4 times longer (two hours for the 512 map), which is bordering on the unacceptable. What is really needed is to know the type of count statistics that we would expect to see for this example. In other words, what x-ray count rate are we to obtain. Table 1 shows the quality of map obtained as a function of count rate and mapping time. Furthermore, we need to know what sensitivity in concentration is required.

It has been quoted that recent advances in energy dispersive spectrometry (EDS) have resulted in much higher count rates [1,2,3], which makes it attractive to alter the strategy for x-ray mapping and the dwell time per point can be reduced, consequently obtaining x-ray maps a lot quicker. (High count rate EDS mapping has been around for quite a long time (QEMSEM), it is just that the resolution of the ED spectrum has been relatively poor. Whenever count rates are specified you must also specify spectrum resolution) Unfortunately EDS is not able to work at the very low concentration range (ppm-part per million range). This is best accomplished using wavelength dispersive spectrometry (WDS), as the WDS allows detection limits an order of magnitude lower than EDS. A new technique that will soon appear is x-ray mapping of materials using micro fluorescence, which will yield detection limits lower [4,5] then conventional EDS.

As mentioned earlier, the peak to background ratios are a very significant factor in mapping. It must be emphasized that EDS is really a very poor cousin to WDS when it

comes to mapping not only due to better count statistics per peak but also due to an order of magnitude difference in P:B ratio. However, EDS and WDS analysis obtain very similar results for when moderate beam currents are used and for major elements with no peak overlaps and greater than 10 atomic number (Z > 10). (not strictly true) see above addition. It is always good to remember that WDS will almost always be 10 times better than EDS except if you are input beam current limited.) I am hoping that in future there may be some further development of high solid angle devices.

Another important consideration of EDS analysis is the resolution as the peak to background is dependent on resolution. This is also the same for WDS, but since the resolution is so much better we don't pay much attention to this, but if we are looking at very low levels this can also be important point.

So what is the limiting factor when it comes to detection limits?, what is a good x-ray map?. This is dependent on the observer, but a good map is a good image that differentiates the associated areas in such a fashion so as to allow conclusions to be drawn about these associations. All images have the same quantitative processes. The image should be able to be used for data extraction in the raw and not processed. Processing may be used to aid in the process, but if you cannot see the information in the unprocessed image then it is likely that it is not there. An example of this type of artifact can be seen in Figure 1. Furthermore, often from a visual point of view the intensity map looks better than the quantitative map. The reason for this is that the intensity map has more x-ray counts, giving better counting statistics. However, the intensity x-ray map can have artifacts as shown in Figure 2.

Table 1: Quality of x-ray map obtained as a function of count rate and mapping time.

Image Size	**Dwell Time msec**	**Time to Map**	**@2000cps**	**@20,000cps***	**@60,000cps****
256		10 minutes	Poor	Poor	Fair
256		30 minutes	Poor	Fair	Good
256		1 hour	Fair	Good	Excellent
256		8 hours	Good	Excellent	+
256		64 hours	Excellent	+	++
512		hours			
512		hours			

*High speed PP. **+Beam blanking or second detector.

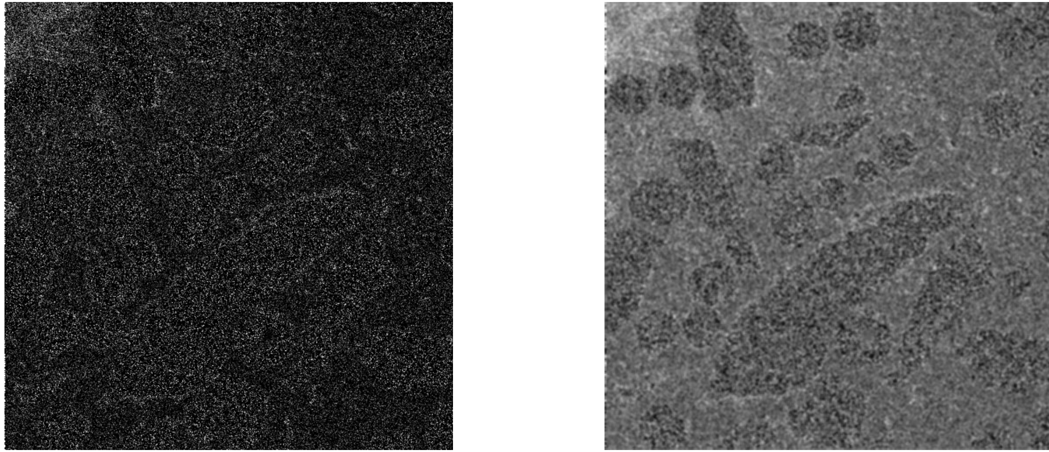

Figure 1: Effect of processing on image. This can also produce a misleading result. Explain!!

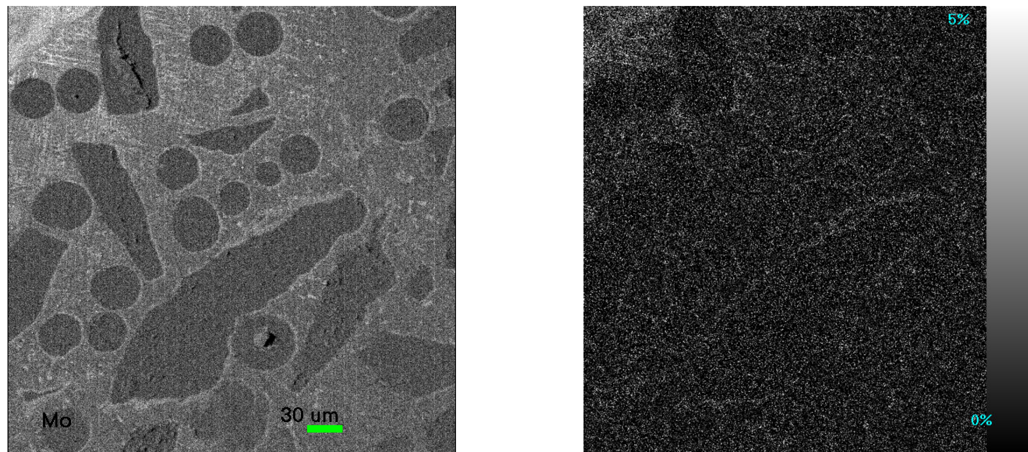

Figure 2: Intensity x-ray map versus quantitative x-ray map.
(A=ROI for Mo, B=Quant and C=WDS for Mo)
Also note that the WD map looks even better than the quant EDS.

[1] Goldstein et al. Scanning electron microscopy and x-ray microanalysis, 3rd ed, Kluwer Academic Plenum, New York, 2003.
[2] Struder, L. et.al., Mikochim. Acta, sppl. (1998) 11.
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