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Introduction

X-ray mapping (XRM) has become a very powerful technique for understanding the distribution of elements in materials. After performing serious mapping, the value of this technique is realised due to its ability to show variations of elements in any one sample.

X-ray maps are usually collected using raw counts from the elemental peaks of interest, however traditional Region of Interest Mapping (ROIM) does not always provide a completely accurate description of the material phases or elemental distribution. Often from a visual point of view the intensity map (Figure1a), looks better than the quantitative map (Figure1b), and is consequently used for presentation. The reason is that the intensity map has more x-ray counts, giving improved counting statistics and thus making the image look better (smoother).



Figure 1: A barnacle attached to an aluminium plate which has been set in a resin, sectioned and polished. (a=ROI map of Chlorine and b=Quantitative map of Chlorine). Region i is the aluminium plate with 0 wt% Cl, region ii is the barnacle with ~0.03 wt% Cl and region iii is the resin with ~1.3 wt% Cl.

The integrated peak counts are only partially related to the true composition of the material and in fact may not be related at all. Indeed, using ROIM may introduce errors and misleading results. For a true spatial elemental distribution we require background subtraction, overlap correction and inter-element correction.

It is obvious that the ROIM (Fig 1a) is incorrect and actually inverted whereas the quantitative map (Fig 1b) is correct. The ROIM shows the aluminium sum peak (region i) which occurs at the energy for chlorine and region ii is brighter than it should be because the background has not been subtracted.

Quantitative X-Ray Mapping (QXRM)

Quantitative X-Ray Mapping (QXRM) is where the full spectrum at each pixel is read and the background is removed, the elements required are stripped from the spectrum and the results saved for later correction processing. This enables us to achieve true spatial elemental distribution as well as giving us a means to verify our results by post processing to determine actual analyses of spatially distinct areas of the sample from the stored data. Traditional QXRM requires that all the elements of interest be known before the mapping proceeds so that they can be extracted from the full spectrum at each pixel during mapping.

Quantitative mapping enables us to get more reliable results spatially and statistically that are an order of magnitude better than with the usual EDS spot analysis. Another advantage of quantitative x-ray mapping is being able to draw a shape on the image and obtain a fully corrected quantitative result from the area specified (Figure 2).

One of the limitations of QXRM is that there could be other elements present that were initially neglected, and thus a complete new map would be required for any new elements to be ascertained. This is overcome with Full Spectrum X-Ray Mapping (FSXRM), which saves the full spectrum at each pixel of the image. This form of mapping allows the user, at whim, to create new maps (QXRM or ROIM), line scans or line profiles, and analyse spectra from the saved data without having to reload the sample or redo the acquisition.

However, there are a number of requirements and considerations that need to be undertaken when performing x-ray mapping in order to obtain good x-ray maps, whether it be ROIM, QXRM or FSXRM.



ID = 1295 Points.			ID = 2392 Points	s. \	
Livetime = 259. Realtime = . / Bulk STDS.			Livetime = 478. Realtime = . Bulk STDS.		
kV=19.5 Angle=55 /B.C.=1.			kV= 19.5 Angle= 55 B.C.= 1.		
ELEMENT	WEIGHT %	+/- 2 SIGMA	ELEMENT	WEIGHT %	+/- 2 SIGMA
Si	0.03	0.01	Si	0.62	0.04
Cr	0.10	0.02	Cr	16.94	0.22
Mn	10.81	0.27	Mn	2.70	0.11
Fe	3.23	0.15	Fe	73.53	0.59
Ni	2.14	0.15	Ni	1.08	0.08
Cu	81.23	1.06	Cu	0.53	0.07
Mo	0.00	0.00	Mo	1.23	0.09
AI	0.00	0.00	AI	0.00	0.00
TOTAL =	97.54	1.11	TOTAL =	96.63	0.66

Figure 2: QXRM of weld region

Requirements for QXRM

In order to obtain good x-ray maps the following conditions need to be meet:

• *Stable microscope and EDS system*. There has to be low drift in the system.

• Correct calibration of EDS detector parameters. This includes gain, zero, take off angle, count rate, dead time and live time.

• *High resolution detectors*. This reduces affects from overlaps.

• *Dwell time*. A dwell time that facilitates reasonable count statistics.

• *Preferably standards based analysis*. Greater analysis accuracy can be obtained.

• Data processing. You need reasonable processing software.

It is also very important that analysts are careful interpreting their results, especially with ROIM. QXRM works well if set up correctly for accurate analysis. FSXRM is even more powerful, as the data is always available for parameter refinement and extra element inclusion as well as producing ROI maps and QXR maps.