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Artifacts in Qualititative and Quantitative X-Ray Mapping

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The introduction of silicon drift detectors (SDD's) into the world of SEM microanlysis has allowed microscopy users to be able to produce 1-2 hour x-ray maps (XRM) instead of the usual overnight x-ray maps. Excellent quantitative x-ray maps (QXM) can now be obtained in around two hours for major elements (>10wt% evenly distributed) and minor elements (>1wt% localised) within samples. There are SDD's now available with 123eV resolution and 80mm^2 active area that can handle 100,000cps. There are also smaller and cheaper SDD's available with high count rates of 100,000cps at 134eV resolution. These SDD's are housed inside a very small package, which allows the use of multiple detectors around the SEM column and makes the affordability of multiple detectors mapping a reality.

Rapid x-ray mapping can produce some very appealing images of the bulk chemistry of a sample. However, it is not good enough just to show appealing x-ray images of the major elements. Faster does not necessarily mean better as it is important to be addressing the problems of overlaps, pulse pile up and other spectrum and sample artifacts. There are still all of the pitfalls that have been described in many papers [1, 2]. For quantitative work the count rate for these detectors needs to be not more than 100,000cps, as pulse pileup will cause problems for low concentration work. For qualitative work and rough surface mapping the count rate will be limited to the available beam current of the microscope. Using a conventional tungsten SEM, it is quite difficult to obtain 100,000cps with good image resolution.

There are many artifacts, whether qualitative or quantitative, that may occur in the x-ray mapping process. Most of the artifacts seen occur in the normal process of analysing samples. Some of these artifacts include:

- Effect of absorption edges on maps (Figure 1) and sum peaks.
- Normalised maps (Figure 2).
- Background and overlap removal.
- Spectrum resolution and peak shifts with their respective affect on overlaps.
- High and low accelerating voltage. Inappropriate accelerating voltage can cause incorrect and even no detection of specific elements in certain areas.
- Low resolution mapping, which can give the wrong impression about where the elements are distributed within the sample.
- Spatial information versus counting statistics.
- Specimen drift and beam wander.
- Phase interface problems.
- Beam size and shape effects.
- Anomalies caused by filtering.

This paper will present the above artifacts observed through x-ray mapping and will show specific examples where these types of artifacts occur.

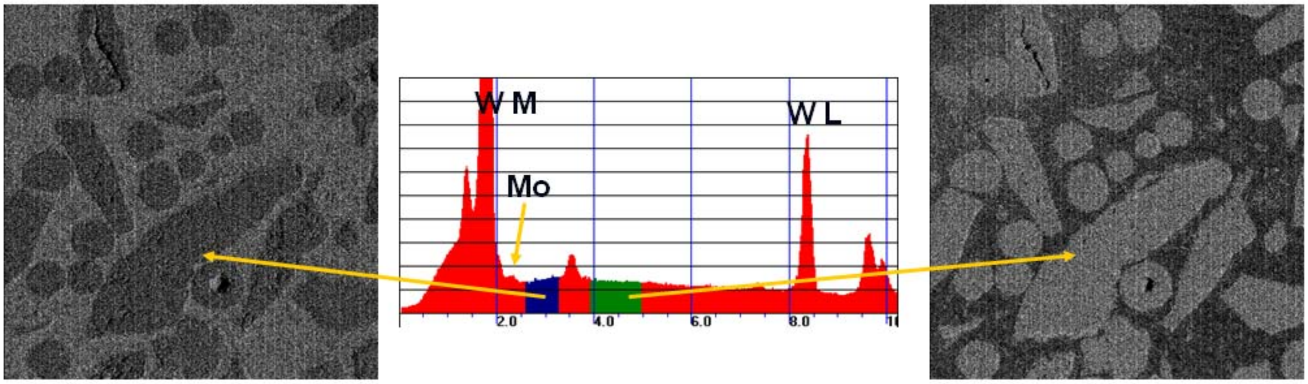

Fig. 1: Regions of interest (ROI) x-ray map of two different background regions. Sample is tungsten carbide in a nickel-copper matrix. Client wants to map molydenum, which is in very low concentrations and Mo exists above the absorption edge of W (M line). The two grey levels should be similar and images reveal the affect of absorption edge on the two backgrounds. The image on the right is correct, but the left image shows the background is absorbed by the tungsten M line. This type of work has to be done quantitatively.

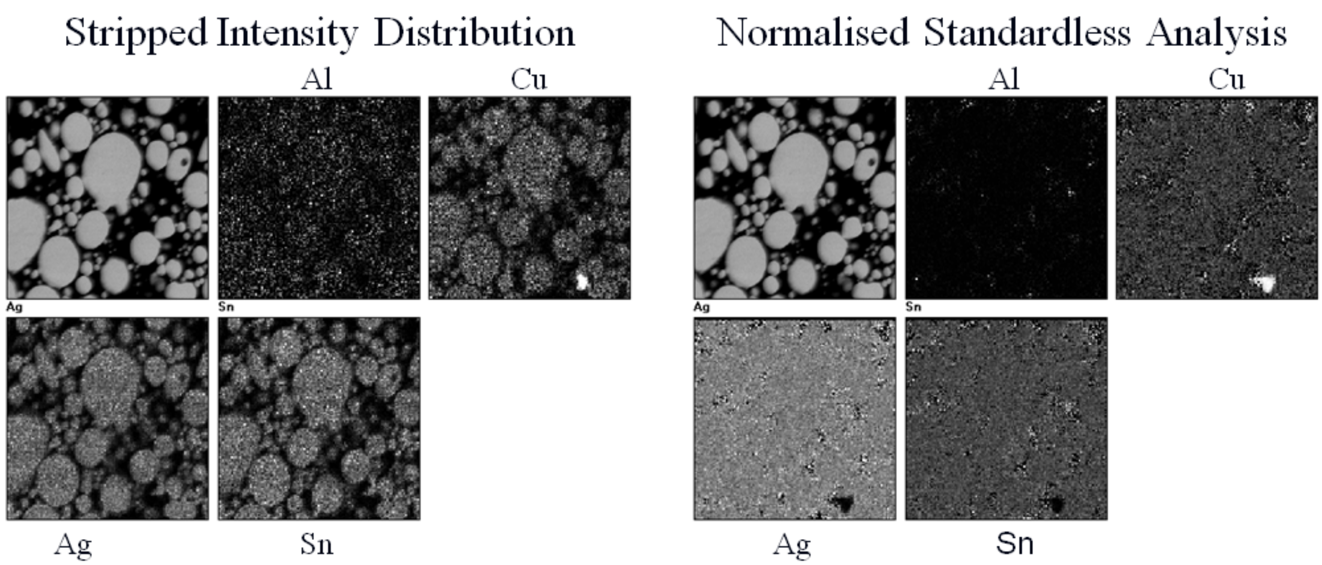

Fig. 2: X-ray maps of Al, Cu, Ag and Sn dental product embedded in polymeric resin using a Be window EDS detector. a. The x-ray maps of the stripped intensity distribution are background subtracted, overlap corrected only (There is also no normalisation) and b. The x-ray maps are normalised standardless x-ray maps. The x-ray maps were normalised with no chlorine, carbon and oxygen (which is in the resin) and have not been included in the analysis correction. The spectrum from the resin shows scattered x-rays proportional to those elements in the particles.

1. K. Moran and R. Wuhrer, *"The importance of knowing detector parameters when performing multi-EDS x-ray mapping"*, AMAS VIII-The eight biennial symposium, University of Melbourne (2005) 29.
2. K. Moran and R. Wuhrer, *"Region of interest mapping (ROIM), quantitative x-ray mapping (QXRM) and speckle"*, AMAS VIII-The eight biennial symposium, University of Melbourne (2005) 53.

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