X-Ray Mapping and Post Processing

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Characterisation of materials frequently involves the determination of variation in composition, structure and microstructure, by the use of a variety of imaging and analysis techniques. There is an increasing need to understand materials phenomena and processes and to learn more about exploiting subtle changes in the distribution of elements in materials technology. Scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), wavelength dispersive spectroscopy (WDS) and the combination of these techniques through x-ray mapping (XRM) has become an excellent tool for characterising the distribution of elements and phases in materials. This analytical technique provides a high magnification image related to the distribution and relative abundance of elements within a given specimen and thus makes XRM particularly useful for:

- identifying the location of individual elements and
- mapping the spatial distribution of specific elements and phases within a sample (material surface).

Quantitative x-ray mapping (QXRM) enables reliable quantitative results that can be an order of magnitude better than traditional analysis and is also far superior to regions of interest x-ray maps (ROIM) where low levels of an element or elemental overlaps are present.

Once an x-ray map (XRM) has been collected a number of analytical software methods can be used to process the data and determine further information about the microstructure and properties of the material. To obtain a better understanding of a material's chemical and microstructural properties a number of post-processing methods should be employed, such as:

- Elemental mapping (ROIM and/or QXRM)
- Scatter diagram creation
- Rotational scatter diagrams
- Pseudo colouring
- Filtering techniques
- Ratio mapping and
- Phase mapping

The use of scatter diagrams, which are pixel frequency versus element concentration profiles plotted against each other in two dimensions for selected elements within the sample, is very valuable for observing the clusters which correspond to different chemical phases and boundaries within the material, Figure 1. The contributing pixels to each cluster can be used to reconstruct the spatial distribution of its associated chemical phase or boundary in a chemical image of the specimen.

There are many different types of clusters observed in scatter diagrams, which indicates many properties. For example, observing connection between clusters (linking) indicates the boundaries between chemical phases within a material. The contributing pixels to each cluster can be used to

reconstruct the spatial distribution of its associated chemical phase or boundary in a chemical image of the sample, which is a very important part of the chemical phase identification process. These selected analysis points may then be summed for a more accurate analysis in total or by selecting strategic areas on the image. Selecting areas on the electron or x-ray image and showing where these points plot (retrace) on the scatter diagrams is also important in locating missing clusters.

Through the use of scatter diagrams, the mapping of phases is possible through selecting the clusters and then displaying these similar concentration areas on the image. This is often referred to as phase mapping, but really it should be called chemical phase mapping (CPM), as phase mapping assumes knowledge of atomic positions and requires diffraction analysis. The XRM method that is being described uses chemistry through use of either EDS and/or WDS analysis. It is also important to recognise that all elements need to be mapped, as some chemical phases are determined from very minor elemental variation and even elements that are difficult to analyse. If a full spectrum at each pixel is saved, this makes the process of looking for other elements much easier.

This paper will be discussing elemental mapping and a number of post-processing methods such as pseudo colouring, scatter diagram creation, and chemical phase mapping all of which aid in obtaining a better understanding of a material's chemical and microstructural properties.
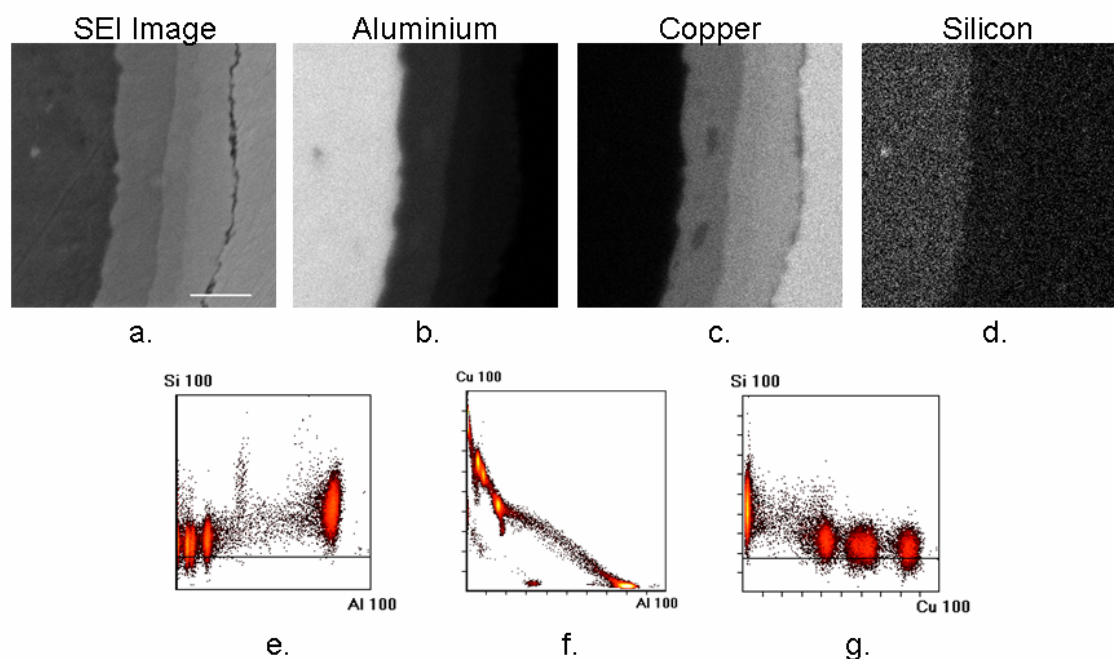

Figure 1: Copper – aluminium roll bonded metal laminate after sintering at 450^0C for 3 hours. a) SE image of the interface between the two metals and elemental x-ray maps of b) aluminium, c) copper and d) silicon, and the scatter diagrams showing different clusters in the bond interface region: e) silicon versus aluminium f) copper versus aluminium g) silicon versus copper. Maps collected at 20keV, 512x512 pixel, 100msec/pixel and 7kcps. Width of field (WOF) = 45μm.