Spectral Imaging in X-Ray Microanalysis: Development and Applications

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INTRODUCTION
Chemical analysis of materials has been performed on electron beam instruments since the early history of these devices. The microprobe was developed by Castaing [1] using wavelength-dispersive spectrometry (WDS) for the express purpose of the chemical analysis of materials.

These electron beam instruments evolved into scanning electron microscopes (SEMs) for the production of high-resolution, high depth-of-field images. The WD spectrometer was the first to be mounted on an SEM, but it has been slowly supplanted by the energy-dispersive spectrometer (EDS) when it was developed in the late 1960s. Both types of spectrometers are also used on transmission electron microscopes (TEMs), with EDS being the dominant type because of its higher efficiency. EDS has one highly desirable characteristic: the ability to collect the whole energy spectrum simultaneously.

THE EARLY METHODS OF X-RAY MAPPING
The early users of microprobes with scanning beams found that they could produce an X-ray image, that is an image of a specific X-ray energy, by routing a signal from the spectrometer back to the cathode ray tube (CRT) display normally used to show a secondary or backscattered electron image. These images were formed by bright dots which occurred at a position where an X-ray event of a particular energy was detected by the WD or ED detector. Such images, or X-ray dot maps as they were called, were produced using one energy region at a time. Once microprocessors and computers were applied to EDS systems, multi-element maps could be collected and stored digitally in the computer memory and storage media. These were then referred to as digital X-ray maps. Such maps were much more useful than dot maps as they showed higher concentrations of an element at brighter areas of the image. The data were in digital form, so it could be processed after the experiment was complete. Therefore many elements could be collected simultaneously with an EDS system, saving large amounts of time.

DEVELOPMENT OF SPECTRAL MAPPING
Even before large-memory storage devices were available, the idea of saving the whole EDS spectrum at each pixel of an image was seen as being desirable.[2] With digital X-ray maps, the user specified in advance the elements or energy regions that were to be stored in memory. If the user discovered that an additional element was present, but had not been selected originally, the whole experiment would have to be repeated. Also, many energy regions off the elemental peaks would have to be stored and considered for possible changes.

Figure 1: Spectrum derived by summing the spectra from the brightest pixels in the secondary electron image. The sample is a multiphase Fe-Cr steel.
have to be collected if background correction and/or quantitative information were needed. Since there was usually a limit of the number of regions to be collected, perhaps 32 or less, this may have restricted the number of elements that could be collected. When this idea originated, a spectral image represented a large amount of data to be collected; the only memory device that could handle it was magnetic tape. This was not very practical, as the data could only be accessed serially, not randomly, so the idea had to wait until large randomly accessible memory was available.

A spectral image can be thought of as a 3D datacube with the X-Y dimensions of the image being two sides of the cube, and an X-ray spectrum at each pixel being the third dimension. This datacube then represents all the data from the experiment, so virtually any information available from the experiment can be extracted from it.

**EXTRACTING INFORMATION FROM SPECTRAL IMAGES**

**Spectra**

Spectra can be recovered from the data set in a large number of ways. The simplest is to click on a pixel of interest. The resulting spectrum will have few counts in it, depending on the length of time used to collect the spectral image. However, by summing together nearest-neighbor pixels, a more statistically valid spectrum can be obtained. Various drawing tools can be used to sum together pixels in an area to obtain high quality spectra, which may represent some feature of the sample. Image histograms can also be used so that spectra can be summed from areas of certain grey levels in the image or from count levels in any of the X-ray images, as shown in Figure 1.

NIST has developed an interesting method for finding small amounts of an element which does not show up easily in the total spectrum from the sample [3]. Called a ‘maximum pixel spectrum’, it creates a single spectrum by finding in the datacube the highest count for all channels in the spectral image. The maximum pixel spectrum is obtained by choosing the maximum value of each pixel within an energy plane to represent that channel intensity in the derived spectrum. This method finds elements which exist in only a small portion of the spectral image.

**Images and line scans**

X-ray images and line scans can be extracted from the datacube merely by identifying elements or energy ranges, and in the case of a line scan, the co-ordinates of the line. Since the whole spectrum is available, other operations can be performed on each spectrum. These include background correction, overlap correction and matrix correction, to achieve qualitative chemical analysis. If all these corrections are performed, then true chemical images can be produced.

**Phases**

Phase analysis can be performed on spectral images in a number of ways. Some of them depend on multivariate statistical analysis (MSA) techniques. Primary examples of these are principal component analysis (PCA) and cluster analysis. PCA is a largely unsupervised technique, although the user must decide how many components found are significant. Cluster analysis can be performed in several ways, one of which forces a guess at the number of phases present. The other works unsupervised but requires a user review of the results.

Other methods involve matching of spectra to those of known phases, or user selected spectra from the data set. The result is the number of phases, their area fractions, and the chemical composition of each phase.

**Hyperspectral imaging**

When spectra of more than one type are collected at each pixel, this is referred to as hyperspectral imaging. This type of spectral imaging is not common at present but could become important in the future. On TEMs, for example, it is feasible to collect EDS and EELS spectra simultaneously. The topic will become featured at conferences in the coming years.

**CONCLUSIONS**

Spectral imaging has become a powerful means of collecting data on electron beam instruments. It is becoming a more routine way of examining samples in the SEM with an energy-dispersive spectrometer. The user can collect much more data over any area of a sample by spectral imaging than is possible by probing around on the sample in a few selected areas and then deciding to do X-ray maps in what was formerly considered to be the conventional way.

**REFERENCES**