Forty Years of X-ray Mapping, from the Beginning to Position-Tagged Spectrometry

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The ability of scanning beam x-ray microanalysis to show the spatial distribution of elements was recognized quite early[1]. At first it was done one element at a time by wavelength dispersive spectroscopy (WDS), but when energy dispersive spectroscopy was developed, that became the technique of choice. Because there have been several significant improvements in the technology, and also because maps are used so universally, I thought a review of the technology from the early days to the latest technology might be interesting. Though modern maps are all digital, the original analog "dot" maps are used by a few even to this day. Other names for the technique are sometimes used such as "x-ray image", which is correct but can be confused with radiography. Compositional image has also been used, but the term is broad and includes more than x-ray maps. Consequently, the universally recognized term "maps" will be used.

When an electron beam of sufficient voltage scans over a specimen, it generates both characteristic and continuum X-rays. If one tunes a wavelength spectrometer to a particular wavelength or selects a region of interest in an EDS spectrum, the photons detected can be displayed on a screen in sync with the beam. Ironically, analog maps can only display binary information, i.e., either a dot or no dot. Digital maps contain an intensity value at each pixel, and are displayed as a continuous range of intensity. And the latest technology stores an entire spectrum at each pixel. In analog maps, one discerns variations in composition by observing clusters of dots. The human eye is quite good at perceiving such variations, but the inherent contrast of the maps is low. Several hundred thousand counts must be collected in the entire map, if one is to discriminate phases containing the same element in different concentrations. There is one area in which analog maps are superior to most digital ones, and that is a rapid scan of the specimen to see if the area chosen is suitable for further analysis. But even in the case of a short survey scan, it must be recorded on film.

It is interesting to note that the tradition of displaying each element in a different color goes back to the beginning[2]. And creating such displays took what now seems like heroic efforts. Commonly, the monochrome CRT or black and white prints were photographed on color film through an appropriate filter. To create a map of more than one element, one had to multiple expose the film through a different color filter for each element.

The first digital maps were introduced in the late 1970s. In a digital map, a numerical value for x-ray intensity is stored in each pixel. Because the x-ray range is typically much greater than that of secondary electrons, the spatial resolution of the map is chosen to be much less than that of the image. This saves collection time, because only about 10,000 points need to be sampled, compared with about a quarter of a million for the 512 x 512 image. In the early digital systems, the computer took control of the electron beam and scanned it in steps across the specimen's surface, dwelling at each point to accumulate counts. Consequently, the x-ray map developed in one direction over time, e.g., left to right. Although the very first digital maps were monochrome, they soon made use of color CRTs. Thus it was easy to pseudocolor each element differently, and the x-ray intensity was represented by the intensity of the color on the screen.

The great advantage of digital maps has not so much to do with their display as what the computer can do with the data. Many elements can be mapped simultaneously by EDS, and by a process of image comparison, the computer can find associations and correlations. All pixels of all chosen elements can be screened in seconds, displaying only those areas that match the screening criteria. Such a criterion could be simply that the intensity of some element must be greater than some value. Or it could be a complex Boolean expression. In either case, in seconds one can find only those parts of the specimen that match the selection. At this point a quantitative analysis of the selected areas could be performed, or image analysis could be used to quantify the microstructure.

Inasmuch as the maps are digital, the tools of image processing are available. The value of image processing is not to make the images look better, but to enhance certain areas for thresholding and image analysis. Image processing does not necessarily improve an image, but it does change it, and in the case of x-ray maps the raw intensity data is changed. The tools for making x-ray maps look good are proper collection conditions and choice of color table. If multiple resolutions are available, the number of pixels should be chosen to match the x-ray range or be larger than that if undersampling is not a problem. Undersampling is indicated if one is not looking for concentration differences on the scale of the x-ray range. By using a coarser digital spatial resolution, more counts fall in each pixel and contrast is enhanced. In some systems the appearance of each map degrades as more maps are displayed. This is because if only a total of 8 bits are available, each element must divide up the number of gray levels. On a 16 bit system, this is not a problem, but the 16 bit x-ray data must still be scaled to an 8 bit screen, and choice of color representation has a strong effect on the perception of the display.

There have been attempts over the years to do a quantitative analysis on each pixel, Newbury[3] and his coworkers did a great deal of work in this area. The argument here is that if the microscope is doing nothing overnight, why not collect and process a quantitative map. However, the manufacturers at the time instead chose to pursue fast mapping, which is hardware to process high count rates. Lately, there has been some interest in quantitative maps, though the time required to collect adequate statistics is still an impediment.

The latest development in x-ray mapping is a new technology that integrates the processes of collecting a digital image, x-ray maps, and spectra into one live operation. The technology is called position-tagged spectrometry (PST), and the principle is as follows: With the electron beam scanning the sample, the EDS detector measures the energy of each incoming x-ray photon, which is tagged with the coordinates of the position from which it was generated. This technology is made possible by uniquely designed hardware that integrates the functions of beam scanning, image collection, and x-ray measurement. Inasmuch as it is possible to collect a spectrum at each pixel, it is similar to the "Spectrum Imaging" technique for EELS described by Jeanguillaume et al.[4] and further refined by Hunt and Williams.[5] However the approaches are fundamentally different. In spectrum imaging, the beam dwells at each point for a short time while a spectrum is collected. In PTS, the beam scans continuously while simultaneously collecting a live digital image, entire x-ray spectra from each point, and displaying x-ray maps - all in real time.

Because the entire EDS spectrum is available for every point, maps for any energy range (element) can be added to the display either during or after collection. Elements added later during collection are comparable to those displayed from the beginning. Once satisfied with the maps, one can have the computer search through the dateset and sort the microstructure into phases based on x-ray intensity. Whether an autophase program is used or some combination of computer/operator interaction, a spectrum can be constructed from every part of the map belonging to that phase. Spectra obtained in this manner have high counts/full scale values and are suitable for quantitative analysis. Rather than try to quantify the composition at every pixel, quantitative analysis can be run on each phase. In addition to the spectrum-from-phase capability, spectra can be constructed from any region of the image that can be defined by gray level thresholding, outlining, or image processing.

In summary, x-ray mapping has been around for 40 years. Its value was discovered in the first scanning beam instruments, and its versatility has increased steadily with advances in computer power. We have gone from analog maps to digital maps to digital image comparison. Now with PTS, we can thoroughly characterize a microstructure. We can determine 1) what phases are present, 2) how much of each is present, 3) where they are, and 4) what is their composition. ■

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