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Throughput in Quantitative Analysis—from 23 Elements per Point to 50,000 Points per Element

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Most research on improving quantitative analysis has been directed to increasing accuracy by understanding precision and reducing bias. However, another aspect of quantitative analysis receiving less effort is increased throughput. In the early days of automated EPMAs, we were impressed with the throughput increase provided by the addition of a computer. In 1977, we proudly reported analyzing 23 elements in a lunar sample in 37 mins., 25 of which were devoted to the matrix correction [1, 2]. Ways to improve throughput often center on the detector and its associated electronics. The digital pulse processor reduces dead time [3] and the silicon drift detector can inherently detect and process more photons than traditional Si(Li) and HpGe detectors [4]. In the special case of quantitative analysis on x-ray maps, throughput is the overriding consideration. Although accuracy is always important, the reduced precision associated with maps limits the possible accuracy, even if there were no bias. But information lies in the multitude of analytical points that constitute a map; thus, the faster we can collect and process the pixels, the more information we obtain. Newbury and colleagues did pioneering work on quantitative EPMA, and they reviewed the many issues involved in quantifying x-ray maps [5].

Hardware is now fast enough to do background filtering and peak deconvolution on every pixel in a spectrum image. On a 3GHz processor, it takes about 15 mins to quantify 51,200 pixels in a 256x200 map. An alternative to quantifying every pixel is to sort the data into phases, sum the pixels of a phase into one high-precision spectrum, and quantify only one spectrum/phase. This approach assumes the data are sorted correctly, and the phases are homogeneous within the precision of the analysis. The benefit is that the number of analyses equals the number of phases, not the number of pixels, greatly reducing the time for post-collection processing. These two approaches have been compared previously [6].

In maps, there is a clear tradeoff between resolution and contrast. Fewer pixels means more contrast, but matching the pixel resolution with x-ray range is also a consideration. At a typical magnification of a few thousand times, 128x100 pixels produces a discrete analytical point at each pixel. Fig. 1 shows a comparison of a 64x50 map with one at 256x200, each with a spectrum from a single pixel for the same live time. Clearly, the former would yield a better quantitative analysis.

If the EDS system permits, one can collect the electron image at high resolution and the map at lower and overlay them. Fig. 2 shows quantitative maps of Mg, Si, Ca, Fe, and Ce each in a different color at 128x100 pixels overlaid on a 2048x1536 SE image. The maps were collected with a silicon drift detector at 100 kcps for 10 min. The composite image is shown in the lower right. In the zoomed region, one can see individual rectangular blue pixels from the Ce image overlying the electron image, whose pixels are barely resolved. With this method, the analyst can collect high-contrast, low-resolution maps suitable for quantification, while preserving the high-resolution electron image.

References

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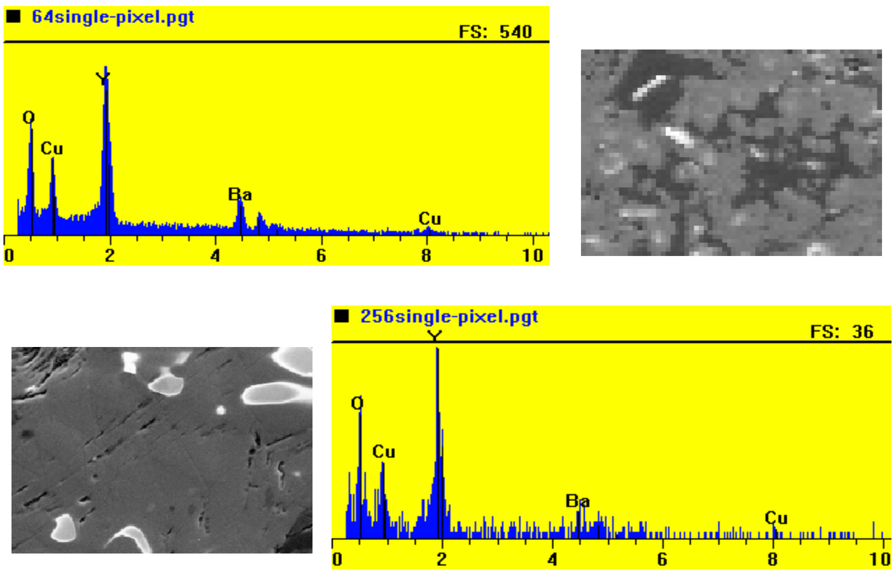

Fig. 1 Comparison of low-resolution (64x50 upper) with higher-resolution (256x200 lower) spectrum images with single-pixel spectra extracted from each. Note the Counts/FS in the spectra.

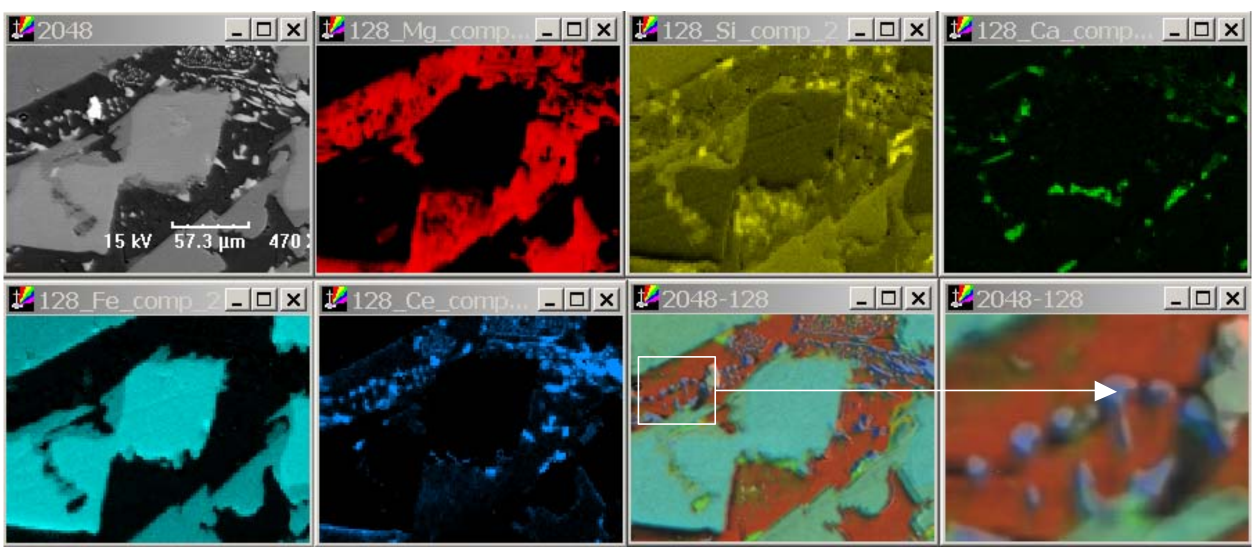

Fig 2. 2048x1536 SE image with quantitative 128x100 maps for: Mg, Si, Ca, Fe, and Ce. Low resolution, high-contrast maps are overlaid on the high-resolution electron image; n.b., the square blue pixels representing cerium intensity on the nearly continuous underlying image.

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