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AMETEK
MATERIALS ANALYSIS DIVISION

Eagle III Micro-XRF: Elemental Imaging Analysis

EDAX, Inc.

The Eagle III micro-XRF elemental analyzer marries an optical microscope to a micro-beam XRF analyzer. Samples are mounted to a motorized XYZ stage, which allows for multiple samples to be analyzed or for a single sample to be measured at different points. This allows for a sample to be studied for compositional homogeneity. One type of analysis mode for homogeneity is known as elemental imaging.

To create an elemental image, a sample is analyzed multiple times, typically as a matrix of points. The individual spectra are incorporated into a database and elemental images are created with pixels calculated based on some property such as XRF intensity or wt%. Each image pixel is related to the parent spectrum and a position on the sample. As long as the sample is not removed from the stage, the sample can be repositioned via the elemental image for further analysis. Elemental imaging is typically done where relatively short dwell times can be used and the density of analysis positions is relatively high. In this article, we shall focus on image processing and data mining to improve the yield of valuable results from the large databases generated by elemental imaging.

A typical use for elemental imaging is to create overlays highlighting regions of the sample that are rich in various elements. In Figure 1a, the elemental image overlay of a fossilized bone is shown overlaying elements associated with mineral (Na-green, Si-yellow, K-blue, Fe-red) and bone (P-grey, Fe-red). One can see the process of prehistoric bone fossilization from the exterior to the interior of the bone. However, this type of overlay leads to poor results when elements compete with nearly equal dominance within the same phase. Selecting Ca and P, both found in the bone, results in a speckled appearance (see Figure 1b). However, by mixing Ca, P, and Si through an RGB mixer, it is possible to provide additional phase contrast with a larger color palette (see Figure 1c). The bone phase, a combination of Ca and P, is now shown in a uniform purple hue and a previously overlooked mineral phase is clearly identified in orange as a combination of Ca and Si intensities.

Often, elemental imaging is employed to look for patterns or qualify homogeneity. This is the case for silicone coating of paper products in the Si grey scale map, Figure 2a. At first glance, it would appear that the silicone has aggregated into clumps on the paper. A composite spectrum was mined from three equal areas within the Si map to further probe the data. As seen in Figure 2b,

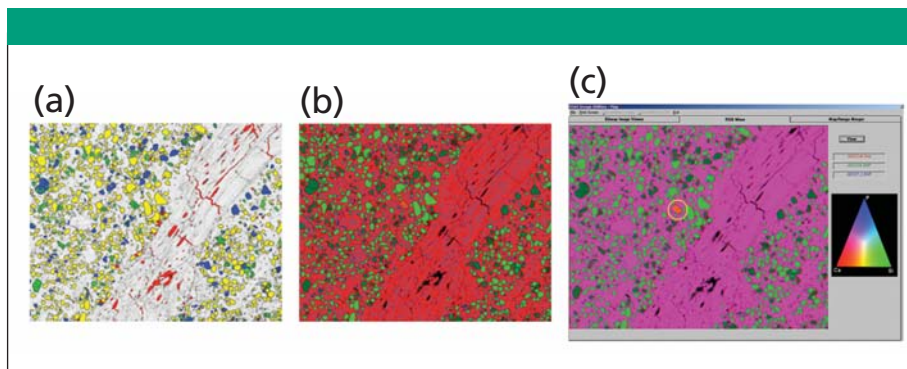


Figure 1: (a) Fossilization of prehistoric bone. (b) Elemental image overlay of Ca - red; P - blue; Si - green. (c) RGB mix of Ca, P, and Si.

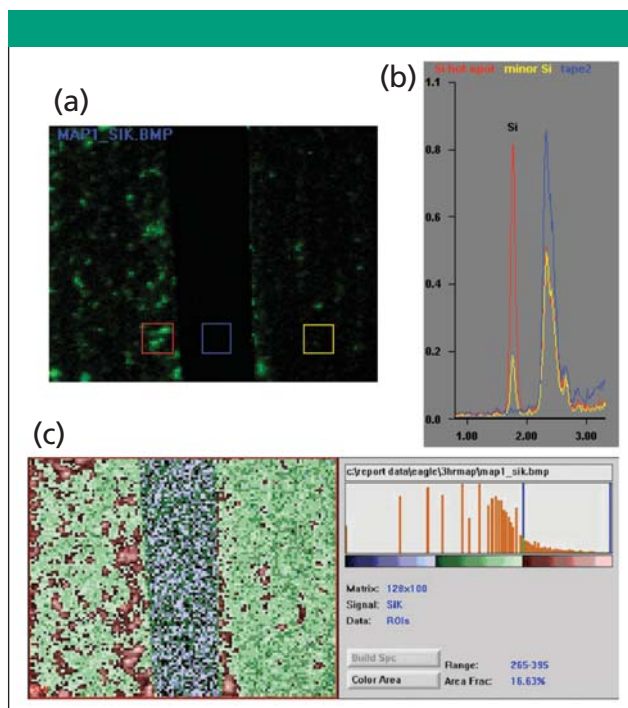


Figure 2: (a) Si(K) map of facial tissue. Carbon tape was used to mask the central strip to create a signal free zone providing a check on the map data. (b) Composite spectra mined from three equal zones within the map: Si hot spot – red; unmasked area avoiding Si hot spots – yellow; taped area – blue. (c) Si map rescaled using a 3-log band scale.

the yellow zone, which appears to have little if any Si intensity, does indeed have significant elemental intensity, which has been hidden by the high intensity of the Si hot spots in the grey scale map. Rescaling the Si map using a 3-log band scale clearly



Figure 3: Granite - Al-cyan; Si-yellow; K-red; Ca-green; Mn-blue; Fe-magenta.

demonstrates the hot spots as well as the background silicone coating on the remainder of the paper (see Figure 2c). It is important to know that the tissue has some silicone coverage over the majority of its area. The map's histogram can yield information about the fractional coverage of a specific region of the histogram. For example, selecting that portion of the histogram representing the Si hot spots shows that these spots represent about 17% of the map's total area, including the masked region.

Of course, one of the advantages of elemental imaging is that each image pixel can be correlated to a full XRF spectrum. Elemental images can be reconstructed without having to specify the elements at the time of the data collection. However, it can be a challenge to determine all of the elements to look for in the spectral map database unless one has knowledge about how the sample was made. This is particularly true for elements found only in trace material phases or small grains and inclusions. Another tool that assists the search of the spectral map database is the maximum channel spectrum. The maximum channel spectrum is constructed by using the maximum intensity found in the map database for each spectral energy channel. The goal is not to create a low-noise spectrum, but rather to generate a spectrum where the significance of the trace elements is magnified.

In Figure 3, we show the elemental image overlay of a slab of granite where elemental map intensities have been generated after background subtraction and peak deconvolution. Being a natural geological sample, there is no manufacturer to consult and the origin was unknown. The elemental image demonstrates the complexity of the sample, which has numerous phases.

In Figure 4, we have generated the maximum channel spectrum (solid red) and compared this to the total map spectrum (blue outline) highlighting the spectral region from 14 kV to 19

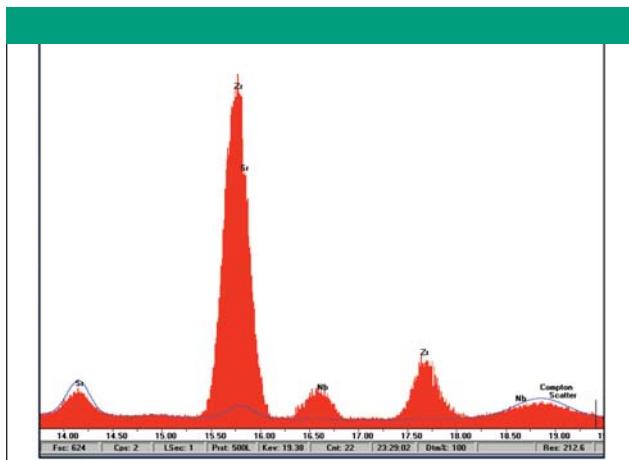


Figure 4: Comparison of maximum channel spectrum to total map spectrum.

kV. The total map spectrum, which is also used to look for elements in the sample, is a sum of all the spectra in the map. In the maximum channel spectrum, one can easily identify the Zr(K) series as well as the peak for NbKa. In the total map spectrum, the ZrKa is obscured by the SrKb while the ZrKb and NbKa are almost lost in the background. Additional elemental images for Zr and Nb can now be built with confidence. Peak deconvolution is important in this situation to deconvolute the ZrKa from the SrKb to generate a high quality Zr map (see Figure 5) without artifacts generated by the SrKb peak. Zr and Nb are found in small grains throughout the slab.

Conclusion

Micro-XRF elemental imaging is a powerful tool for sample analysis which generates a large dataset. It is important to provide software tools that allow the user to highlight features of the elemental images and more easily search the spectral map database for elemental information. The features described here are a small representation of the many efforts that EDAX makes to provide more powerful analytical tools to the worldwide scientific community.

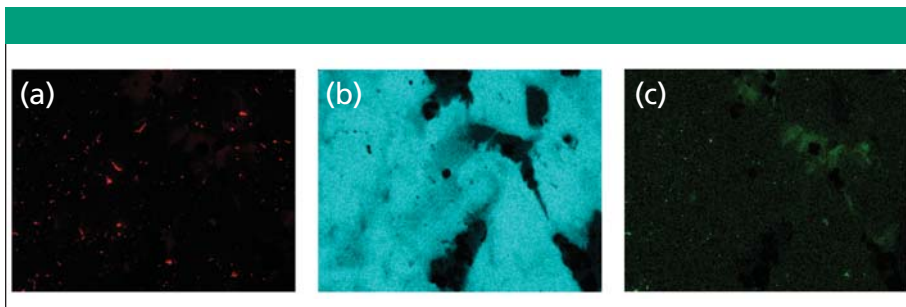


Figure 5: (a) Zr(K) map, (b) Sr(K) map, and (c) Nb(K) map.

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