Technical Note: 51026

Quantitative X-ray Mapping: WDS Performance, EDS Convenience

Key Words

- EDS
- EPMA
- Microprobe
- Spectral Imaging
- WDS
- X-ray Mapping, Quantitative

Geologists, chemists, materials scientists, and others have used Electron Probe Microanalysis (EPMA) with multiple, automated wavelength dispersive spectrometry (WDS) detectors for many years. WDS analysis in the microprobe predates energy dispersive spectrometry on scanning electron microscopes (SEM/EDS) and still sets the standard for resolution and sensitivity in microanalysis. Traditionally, quantitative analysis in the microprobe has been recognized as more precise than the more common SEM/EDS.

Developments in the Thermo Scientific NORAN™ System 7 X-ray microanalysis system, aimed at improved compositional analysis with EDS, now approach EPMA WDS analytical methods on SEM/EDS systems.

Historical Advantages of WDS Imaging

X-ray imaging with WDS has been used in cases where EDS has historically been unable to show good peak to background ratios, due chiefly to the inherent background in the EDS spectrum. In Figure 1 below, an X-ray map of Fe-Kα was acquired using a late-model Electron Probe MicroAnalyzer. This example shows 50 µm particles of iron in a glass matrix. The high sensitivity of WDS probes allows very clean, and highly resolved features with minimal background. An added benefit is that X-ray emission lines, which overlap in EDS, are normally resolved in WDS, allowing the discrete mapping of elements.

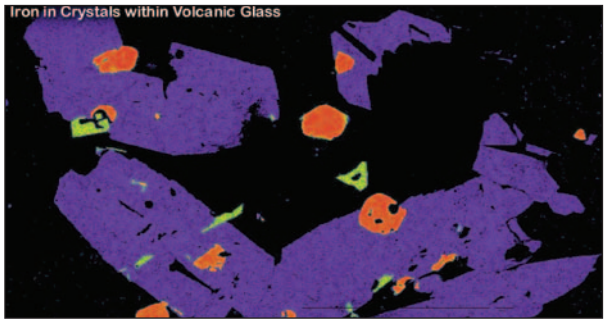

Figure 1: Iron particles in glass matrix, courtesy of the University of Minnesota

Signal-to-Noise in EDS and WDS X-ray Mapping

Figure 2 is characteristic of the older and most common method of EDS X-ray mapping. Here each pixel in the map is representative of the raw number of counts in the element's region of interest in the EDS spectrum. Figure 3 is a much more defined X-ray map using WDS. Here background in not an issue, providing far superior signal-to-noise.

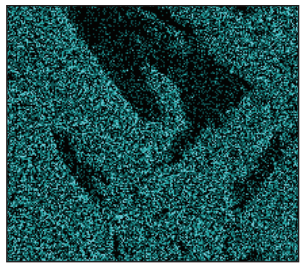

Figure 2: EDS X-ray map of boron

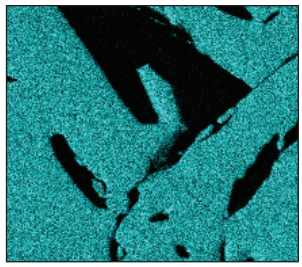

Figure 3: WDS X-ray map of boron

While WDS in the EPMA is clearly the choice for data quality, the analysis is quite time-consuming. The operation is complex, normally requiring a dedicated probe operator, and EPMA equipment is expensive both to purchase and maintain. For these reasons, EDS has become a more popular technique for most non-critical experiments.

Benefits of EPMA WDS X-ray Imaging

- Enhanced signal-to-noise ratio in X-ray mapping
- Discrete mapping of elements with overlapping peaks

Drawbacks to EPMA WDS X-ray Imaging

- Very slow (only one element can be mapped at a time per spectrometer)
- Very expensive (equipment investment more than 400% of EDS)
- Complex operation (typically requiring a dedicated EPMA specialist)

Spectral Imaging — Bringing WDS-type Results to EDS

New to X-ray microanalysis is the Spectral Imaging mode of data collection and data analysis. Spectral images are a combination of x-y pixel location and image intensity, and a complete, deadtime-corrected EDS spectrum. For instance, a 256 x 256 image contains 65,536 individual spectra, each representing the entire energy range and X-ray counts at its location.

More than any other EDS system on the market, our NORAN System 7 is built to collect, store, process and present Spectral Imaging data. Our engineers have optimized NORAN System 7 high-throughput acquisition electronics, pulse processing hardware and software, data storage engine and end-user software to make Spectral Imaging the premier mode of acquisition and analysis.

The NORAN System 7 introduces new techniques hitherto unseen in X-ray microanalysis, such as COMPASS statistical analysis and automatic Xphase. In this Technical Note, we demonstrate Quantitative X-ray Mapping, a new technique that enables new users to achieve microprobe-style results with inexpensive and easy-to-operate EDS.

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Challenges with Traditional EDS Mapping

The images below demonstrate some of the problems typically encountered with traditional EDS mapping, such as overlapping peaks and poor signal-to-noise ratios.

Figure 4 shows a sample consisting of particles embedded in a matrix. Figure 5 shows X-ray maps of sulfur and molybdenum and oxygen. However, with energies of 2.3 for S-K and 2.29 for Mo-L, these maps appear to be identical. All three maps (including oxygen) appear to be of inferior signal-to-noise because of the inherent background in EDS. Using NORAN System 7 we can reduce or eliminate these traditional problems.

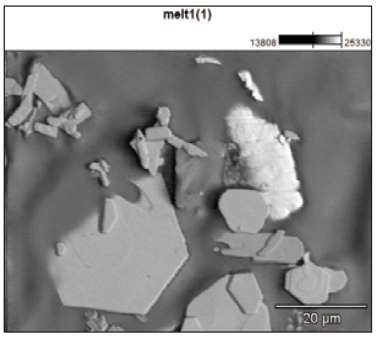

Figure 4: Electron image of particles embedded in a matrix

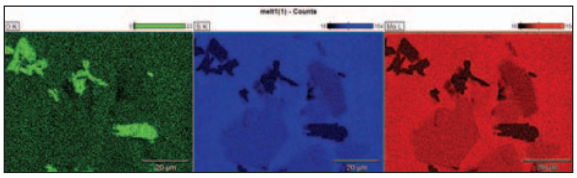

Figure 5: Traditional EDS X-ray maps of O, S and Mo

Quantitative X-ray Maps, a New Solution to Old EDS Problems

The NORAN System 7 offers Quantitative X-ray Mapping as a standard function. Here, a full EDS spectrum is acquired with Spectral Imaging, where each pixel in the image contains a full spectrum. Quantitative X-ray Mapping then calculates the elemental concentrations of all of the elements in the Spectral Image data set. The method, as shown in the following figures, creates much more understandable X-ray maps, due to a better signal-to-noise ratio, and fully separated elemental peaks.

Not only does Quantitative X-ray Mapping greatly increase the signal-to-noise ratio, providing results typical of EPMA WDS acquisitions, the data was collected and presented in only five minutes. Figure 6, a composite image of the elements oxygen, sulfur and molybdenum, shows elemental data that is as perfectly separated spatially as one finds in EPMA WDS.

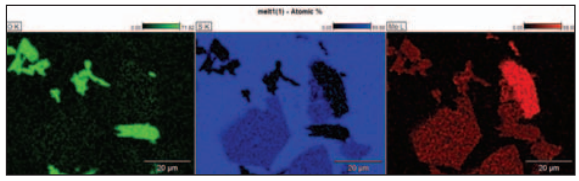

Figure 6: O, S and Mo in a composition image separated by atomic percentage using Quantitative X-ray Mapping

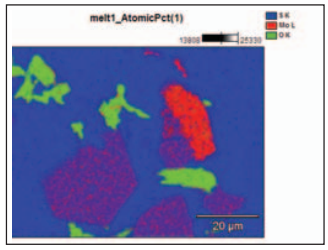

Figure 7: Sulfur and molybdenum spatially separated

Introducing Xphase — Beyond EPMA WDS

Xphase, an optional software module for the NORAN System 7, provides a new method of identifying and visualizing the phase distribution within a sample. Using standard elemental maps or elemental data processed with Spectral Imaging tools, Xphase offers a new level of chemical analysis for X-ray microanalysis.

Figures 8 and 9 below show the same sample as seen in Figures 6 and 7, now in a distribution by phase. Here two compounds (molybdenum oxide and molybdenum disulfide), in additional to the elemental concentrations of sulfur and molybdenum are identified.

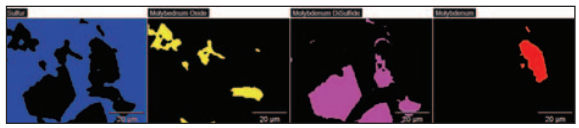

Figure 8: Four phase maps of elemental concentrations and compounds produced by Xphase

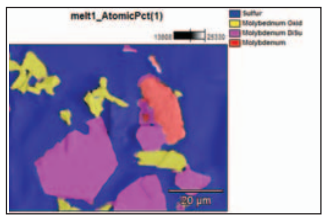

Figure 9: Composite distribution of the four sulfur and molybdenum phases above

Conclusion

In a number of application areas, the NORAN System 7 offers a cost-effective and viable alternative to EPMA WDS for X-ray imaging. Results are obtained with minimal training, and data is often as useful as WDS. Simplicity of operation and low cost of ownership make the NORAN System 7 a perfect complement to the geology, materials science, or failure analysis lab.

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