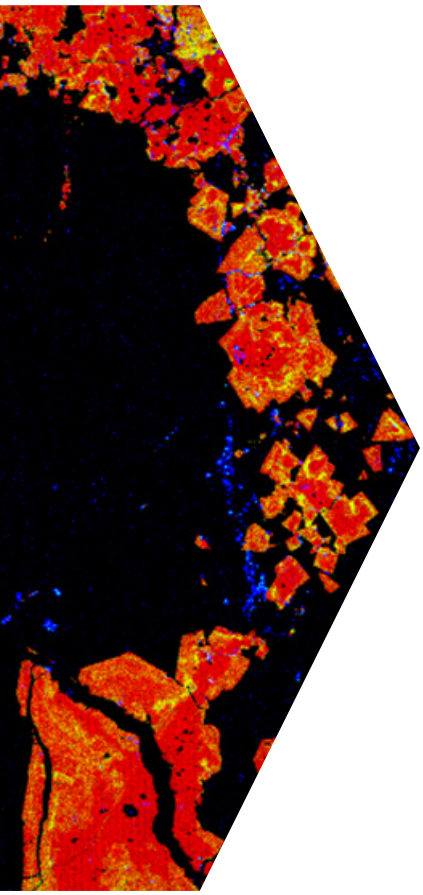
Applications Note

AttoMap MicroXRF



Quantification of Mineralogy with XRF Microscopy

Efficient trace element detection in materials has become important as the global demand for mineral resources increases. Sigray AttoMap™ series provides mineralogical quantification of minerals from major concentrations to trace-level (ppm-level) quantities and at microns-scale resolution and acquisition speeds of down to 5ms/point.

This white paper will review mineralogical applications of the AttoMap XRF microscope

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Quantification of Mineralogy with the **AttoMap™ Series XRF Microscopes**

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Introduction: Elemental spatial distribution and relative elemental quantification techniques are essential for mineralogy research in the natural resource industries (oil & gas and mining). This information is often required at trace levels to maximize valuable material extraction and optimize ore processing[1]. The AttoMap's automated mineralogy capabilities complement existing approaches, including: scanning electron microscope (SEM) based systems, laser ablation inductively coupled mass spectrometry (LA-ICP-MS), and nanoscale secondary ion mass spectrometry (nanoSIMS). AttoMap provides down to 3-5 µm resolution, sub-ppm sensitivities, and machine learning-powered software for quantitative grain segmentation with open-box extensibility.

Current Mineralogical Approaches: MLA and QEMSCAN

Scanning electron microscopes (SEM) based Automated Mineralogy systems[2] have become a key method for characterizing mineralogy. In these systems, an electron beam is stepped across a polished sample surface, exciting x-ray fluorescence (XRF), which is recorded based on beam position. This process produces a mineralogical map at microns-scale resolution, as shown on the left-hand side of Figure 1.

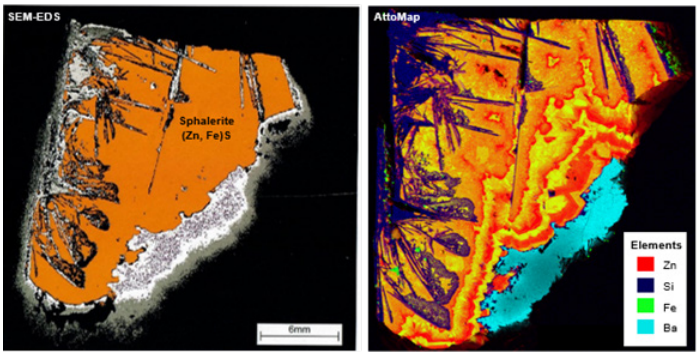


Figure 1: *Mapped mineral sample. Left: SEM-EDS result showing sphalerite ((Zn, Fe)S) in orange. Right: AttoMap result showing Zn (red), Si (navy), Fe (green), and Ba (cyan). Note that AttoMap provides an abundance of textural information, showing Zn-rich regions (yellow from green and red overlap) within the region broadly identified as spheralite by SEM-EDS. Courtesy Dr. Dieter Rammlmair, BGR*

However, SEM-based techniques have a major limitation: their sensitivity to trace elements (e.g. <0.1%) is restricted due to the large bremsstrahlung background inherent in electron excitation. As a result, complementary techniques—such as laser ablation inductively coupled mass spectrometry (LA-ICP-MS) and nanoscale secondary ion mass spectrometry (nanoSIMS)—are often used alongside SEM-EDS to obtain trace mineral information. There are some additional challenges to these techniques, including: interference artifacts, matrix dependency, and variability due to conditions (ablation and analytical count times), which can mask or mimic trace element distributions[3].

Novel Approach with Sigray AttoMap XRF Microscope

AttoMap microXRF was developed using patented x-ray source and optics technologies to enable synchrotron-like microXRF performance in a laboratory system. The system features several key advantages for mineralogical investigations:

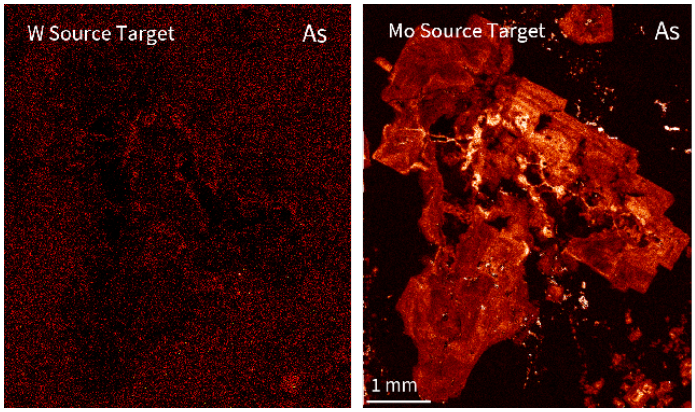


Figure 2: *Selection of x-ray source target can dramatically XRF sensitivity. Sigray's patented multi-target source enables optimizing for almost all elements in the periodic table.*

1. A patented **multi-target x-ray source** allows users to optimize fluorescence signals of interest and detect trace elements at the sub-ppm level (see Fig. 2).
2. A **large sample stage**, allowing large, intact specimens to be scanned (up to 300mm travel on the ambient 200 series and 100mm travel on the vacuum 310 series)
3. **Straightforward sample preparation**: The large working distance of Sigray's proprietary optics allows for imaging samples even having some topography, such as powders and particles, and does not require the polished surfaces required by SEM-EDS. No additional preparation is required

(e.g., resin embedding, polishing, or carbon coating).

4. Variable **spatial resolutions from 5 to 100 µm**, providing flexibility in trading off resolution with FOV/throughput.
5. **Mineralogical software analysis tools**: Sigray's software implements k-means clustering (an unsupervised machine learning algorithm) to segment grain boundaries by mineralogy. Grains can be segmented by XRF data alone or by additionally incorporating the correlative optical microscopy images acquired inside the AttoMap. The suite of tools includes an easy-to-use GUI interface and Jupyter python notebooks, allowing easy extension of algorithms and open source collaboration.

In this applications note, we used an AttoMap-200 ambient system to analyze the mineralogical composition of a rock sample courtesy of Dr. Sakthi Chinnasamy, IIT Bombay.

Results and Discussion

A polished thin section of rock containing pyrite and arsenopyrite (sulfides), silicates, and carbonates was imaged. Given the minerals of interest, a Mo target and optic were selected. A large-field-of-view (LFOV) overview scan was first acquired at 30 µm dwell width and 0.1s dwell time (Fig. 3).

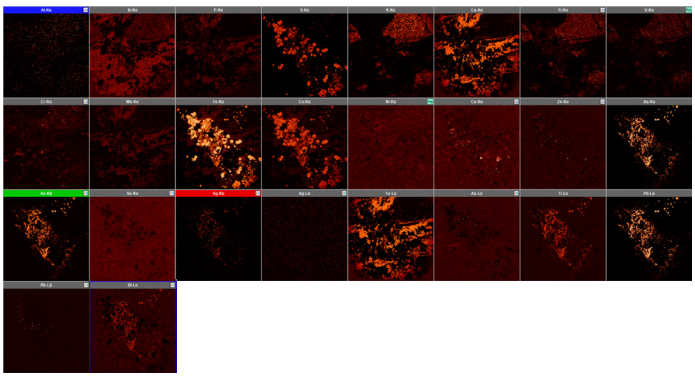
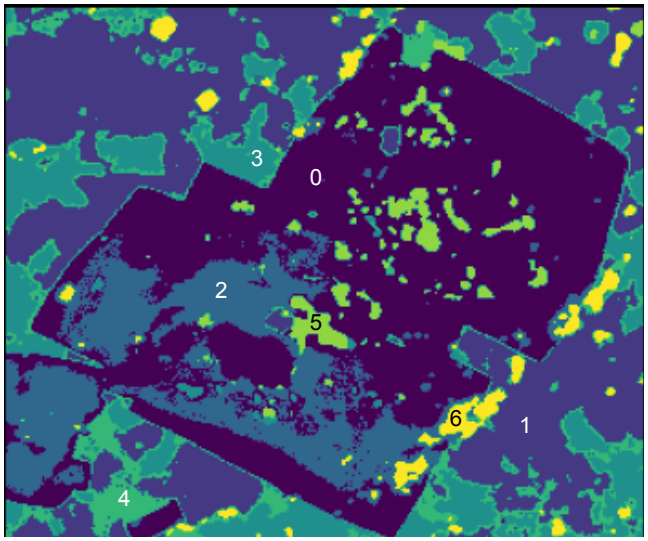


Figure 3: *Software view of LFOV at 30 µm dwell width. Multiple elements of interest are acquired simultaneously and displayed as "heat maps".*

Clicking on specific subregions of interest queued the ROIs for higher resolution scans performed at 10 µm dwell width and 0.1s dwell time.

For each subregion, machine learning-based spectral deconvolution identified key spectral features (Fig. 4) and successfully distinguished elements with overlapping energy lines (e.g., As K-α from air and Pb L-α from the sample). Seven regions were identified, and relative weight percentages of minerals were calculated for each region using a fundamental parameters (FP) model.



	Region 0	Region 1	Region 2	Region 3	Region 4	Region 5	Region 6
Arsenic	0.1275	0.1277	1.3008	0.2519	0.2413	0.2083	15.2562
Calcium	0.567	5.8313	0.5578	51.4627	21.127	1.1187	3.8056
Chromium	0.0066	0.1032	0.0182	0.0393	0.0696	0.0265	0.0563
Copper	0.1226	0.0671	0.0703	0.0796	0.0793	9.3215	0.0302
Iron	43.4856	2.0032	42.6952	20.5661	9.9628	37.2082	28.4973
Lead	0.378		0.1226		0.5066	0.2846	
Manganese		0.0516		0.8785	0.2312	0.0522	0.044
Nickel	0.0187	0.0352	0.0279	0.03	0.0286	0.0194	0.0587
Phosphorus	0	0.7844	0.1092	1.4945	0.4294		
Potassium	0.0095	0.3774		0.4249	1.1677		0.2035
Silicon	3.3172	85.8527	2.7076	19.7786	55.2531	4.1562	26.3397
Sulfur	51.9456	4.6494	52.3669	4.8221	10.7366	47.545	25.5782
Titanium		0.0905		0.0615	0.1126		0.1011
Vanadium			0.011	0.0313			
Zinc	0.0215	0.026	0.0126	0.079	0.0542	0.0592	0.0294

Figure 4: *K-means clustering with deconvoluted spectra used to pull out the most prominent 7 regions of a selected subregion of interest. Peaks were then fit and a fundamental parameters model was used to approximate the composition of the 7 regions.*

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2. Zhou, W., et al. (2006). Fundamentals of scanning electron microscopy (SEM). In Scanning Microscopy for Nanotechnology (pp. 1-40). Springer, New York, NY.
3. Prencipe, I., et al. (2015). Energy dispersive x-ray spectroscopy for nanostructured thin film density evaluation. Science and Technology of Advanced Materials, 16(2), 025007.
4. Nikonow, W. and Rammlmair, D. (2017). Automated mineralogy based on micro-energy-dispersive X-ray fluorescence microscopy (µ-EDXRF) [...]. Geoscientific Instrumentation, Methods and Data Systems, 6(2), 429-437.

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