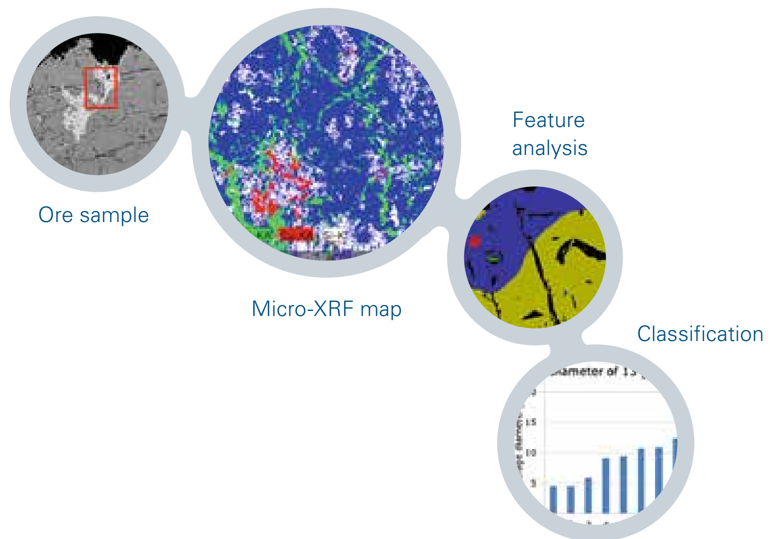


Application Note # EDS-13

Mining industries: Fast ore characterization combining benchtop Micro-XRF and automated SEM-EDS

Energy dispersive spectrometry (EDS) using scanning electron microscopes (SEM) and micro-X-ray fluorescence spectrometry (Micro-XRF) can be performed rapidly since the introduction of silicon drift detector (SDD) technology. The combination of SEM-EDS and benchtop Micro-XRF allows an advanced workflow for the characterization of ore (according to Tab. 1).

Table 1 Workflow for the characterization of ore samples

Task	Method	Option	Reso-lution	Time / min
1. Detect	Benchtop Micro-XRF	Spectrum imaging	~25 µm	30 – 120
2. Locate	High kV SEM-EDS	Automated feature analysis	~3 µm	60 – 120
3. Analyze	Low kV SEM-EDS	Spectrum imaging	~50 nm	~20

Applied to the examples discussed here, ore thick sections from an offset dike of the Sudbury Igneous Complex (SIC), this translates to:

(1) **Detecting** the presence of high demand elements (Co, Ni, Cu, Pt, Pd, As, Te, …) rapidly using Micro-XRF for determining the value of ores.
(2) **Locating** mineral phases of economic interest with SEM-EDS using automated feature analysis.
(3) **Analyzing** them at high spatial resolution using low accelerating voltages to gain insights in arsenide, telluride and sulfide deposit models.

Methods

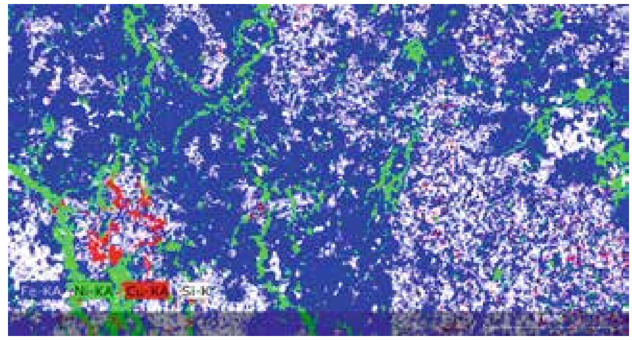
Micro-XRF allows to determine the distribution of elements with Z > 10, also in traces (down to 20 µg/g) with a spatial resolution of >25 µm. Samples with sizes up to 20 x 16 cm^2 can be mapped with the Bruker M4 TORNADO Micro-XRF spectrometer within 4 h to locate regions of interest for subsequent high-resolution SEM studies.

Using the QUANTAX EDS system, minerals in large areas can be classified by automated feature analysis with SEM stage control, a combination of morphological classification with chemical analysis. Analyzing only features of interest by selecting the corresponding threshold in the backscattered electron (BSE) micrograph significantly reduces measurement and evaluation time. Spectra were acquired by point measurements in the center of each grain or by scanning the complete grain. Saving stage coordinates helps to relocate grains to carry out further analyses using spectrum imaging techniques (see Bruker Application Note # EDS-14).

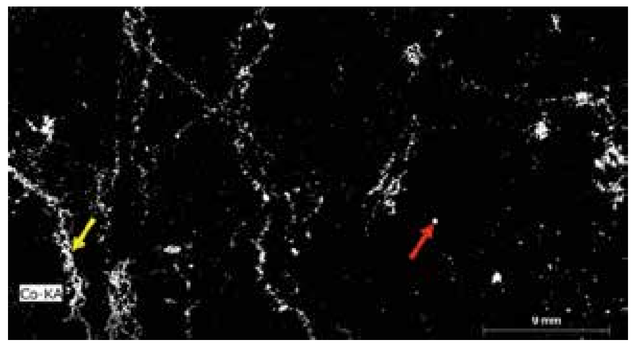
Table 2 Optimal analytical conditions for automated feature analysis using SEM-EDS

Parameter	Conditions	Remarks
BSE threshold	Multiple / single	All / selected grains
BSE pixel resolution	~1 µm	3 times better than smallest feature of interest
HV	25 kV	Spatial resolution in the µm range
Pulse processor setting / dead time	130 kcps / 30%	80 – 160 kcps input count rate, ~90 kcps output count rate
Spectrum acquisition time	0.5 – 1 s	Sufficient count statistics for quantification of major components and classification of minerals

Micro-XRF analysis of a sample from the Parkin dike



(a)



(b)

Fig. 1 Sample from the Sudbury Igneous Complex Parkin dike; (a) Composite Micro-XRF map of iron K, nickel K, copper K, and silicon K of a thick section (44 x 24 mm², M4 TORNADO Micro-XRF spectrometer with 2 x 30mm² XFlash® SDD, 50 kV, 600 µA, 310 kcps, 2h, 20 mbar chamber pressure, 2210 x 1185 pixels², 20 µm pixel size, and 2 ms dwell time, 120 min total measurement time). (b) Micro-XRF map of cobalt K. Note the association of cobalt and nickel with pentlandite (yellow arrow) and arsenide grains (red arrow).

Results

Spectrum imaging using Micro-XRF

The Micro-XRF composite intensity map (Fig. 1a) shows the distribution of minerals, which are mainly iron sulfides but also iron oxides, pentlandite (iron nickel sulfide) and chalcopyrite (copper iron sulfide). Furthermore, minerals of potential economic interest such as cobaltite-gersdorffite (cobalt nickel arsenic sulfide) can be detected (Fig. 1b). Due to the high sensitivity of Micro-XRF it is possible to display an enrichment of cobalt in pentlandite (Fig. 1b). This is difficult for SEM-EDS analysis due to the low concentration of cobalt and the peak overlap of the cobalt K lines with the iron K lines.

Automated feature analysis using SEM-EDS

Minerals were classified (Tab. 4) in the analyzed areas (Fig. 2) according to the parameters as shown in Tab. 3. Using single thresholds by selecting the corresponding threshold in the BSE micrograph, all minerals can be classified (Fig. 3). Minerals of economic interests (arsenides, tellurides) can be located by selecting a bright threshold which significantly reduces the measurement time. Grains of >3 µm in size can be detected using a BSE pixel resolution of ~1 µm (Fig. 4).

Parameter	Worthington dike	Parkin dike	Trill dike
Measured area	1.3 x 0.9 cm²	3.3 x 1.8 cm²	1.5 x 1.1 cm²
Measurement time	292 min	120 min	35 min
BSE threshold	Multiple (all grains)	Bright (As, Te)	Bright (As, Te)

Table 3 Analytical parameters used for the samples discussed here. A multiple threshold was applied to classify all phases, a bright threshold for arsenides and tellurides.

BSE images of the analyzed EDS samples

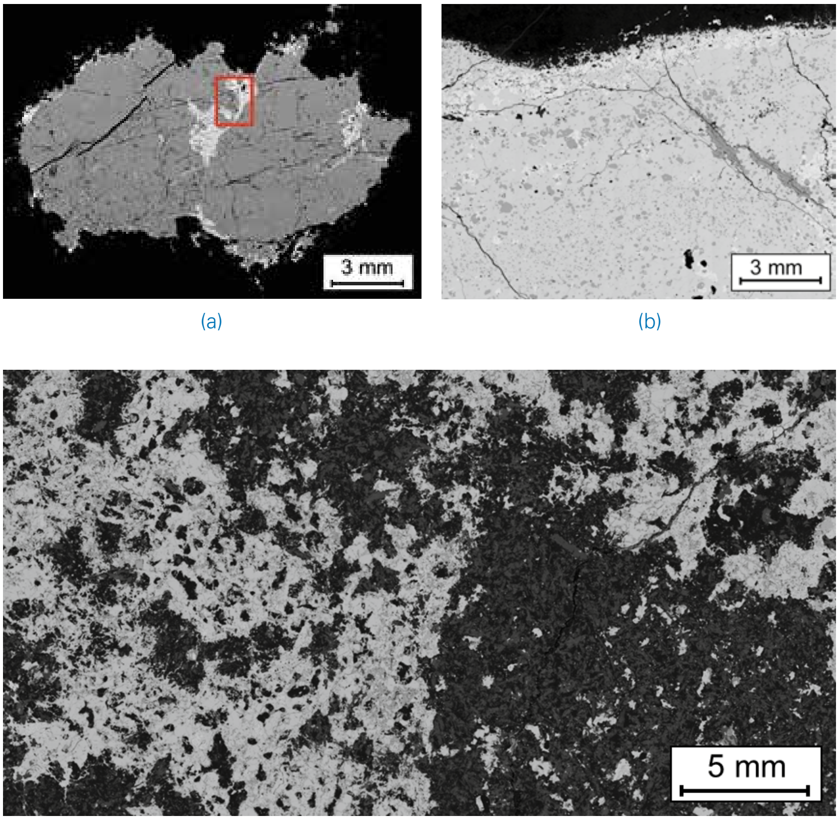


Fig. 2 BSE mosaics of (a) Worthington dike (sample 804014, 15072 x 10061 pixels², 90 fields). The highlighted area is a single field shown in detail below. (b) Trill dike (sample 703351/1, 12560 x 9420 pixels², 256 fields). (c) Parkin dike (Milnet Mine, sample LH09-41, 27456 x 14713 pixels², 875 fields)

Single analysis field of the Worthington dike sample

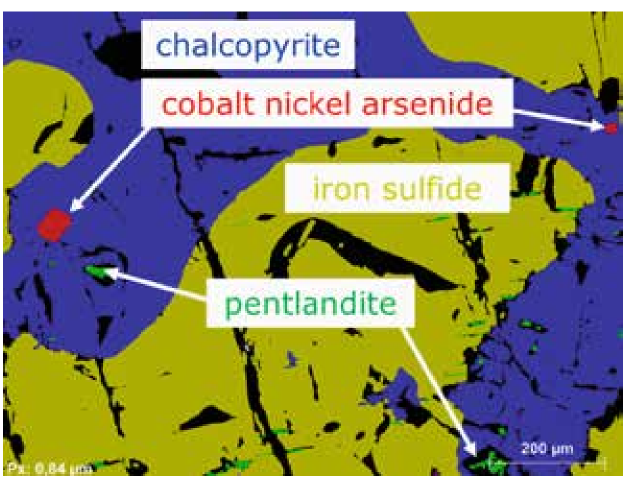


Fig. 3 Example of a single analysis field (highlighted in Fig. 2a) showing the distribution of iron sulfide (yellowish), chalcopyrite (blue), pentlandite (dark green with arrows) and cobalt nickel arsenic sulfide (red with arrows).

Size distribution of telluride particles, Trill dike

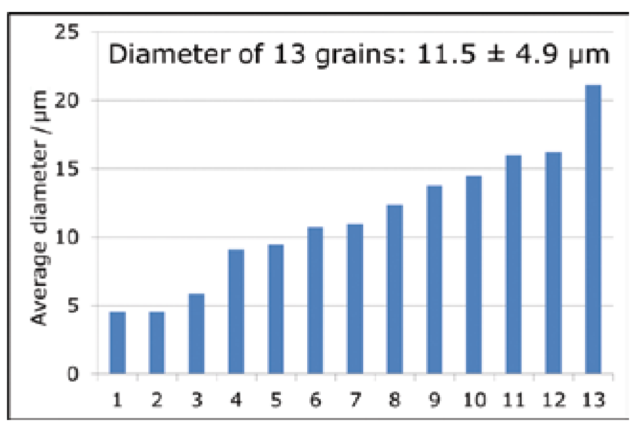


Fig. 4 Size (average diameter) of 13 Pd-Pt-bismuth telluride grains of the Trill dike

	Worthington dike		Parkin dike		Trill dike	
Class	**Count**	**Area /%**	**Count**	**Area /%**	**Count**	**Area /%**
Chalcopyrite	607	3.0460	n.a.	n.a.	n.a.	n.a.
Pentlandite	4126	3.4500	n.a.	n.a.	n.a.	n.a.
Iron sulfide	1058	55.0754	n.a.	n.a.	n.a.	n.a.
Silicates	324	0.0379	1	0.000003	n.a.	n.a.
Co-Ni-arsenide	40	0.1140	9	0.000117	n.d.	n.d.
Pt-arsenide	n.d.	n.d.	5	0.000045	n.d.	n.d.
Arsenide	2	0.0006	n.d.	n.d.	n.d.	n.d.
Pd-Bi-telluride	n.d.	n.d.	8	0.000294	12	0.00065
Pt-Bi-telluride	n.d.	n.d.	n.d.	n.d.	1	0.00008
Bi-telluride	n.d.	n.d.	2	0.000011	n.d.	n.d.
Sb-Pb-phase	n.d.	n.d.	n.d.	n.d.	9	0.00049
Pb-phase	2	0.0020	1	0.000053	30	0.00139
Ag-phase	5	0.0004	1	0.000006	1	0.00001
Monazite	n.d.	n.d.	19	0.000082	n.d.	n.d.
Th-phase	1	0.0004	8	0.000034	n.d.	n.d.
Zr-phase	10	0.0022	50	0.000389	n.d.	n.d.
Baryte	n.d.	n.d.	1	0.000004	n.d.	n.d.
Sphalerite	7	0.0007	n.a.	n.a.	n.a.	n.a.
Unclassified	169	0.0322	n.d.	n.d.	n.d.	n.d.
All	6351	61.7618	105	0.001037	53	0.00261

Table 4 Feature analysis results for all samples, presenting count and area fraction of every class determined (n.a.: not analyzed, n.d.: not detected)

Conclusion

The combination of SEM-EDS and Micro-XRF provides the possibility to improve data and sample understanding. Micro-XRF not only provides the high sensitivity for trace elements but also allows to bridge the gap between the macro sample and the highest spatial resolution of SEM-EDS. Using automated feature analysis it is possible to locate mineral grains of economic interest in a complete thin or thick section within ~2 hours. It can be concluded that improvements in detector and pulse processor technology, software developments have extended the SEM-EDS and Micro-XRF applications turning them into valuable tools for applied and process mineralogy.

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