EDS

Low energy X-ray EDS analysis of ore mineralization at high spatial resolution

Application Note # EDS-14

Introduction

Developments in energy-dispersive spectrometry (EDS) offer advanced analysis of low energy X-ray lines at high spatial resolution. This will be demonstrated for cobalt nickel arsenic sulfide grains in samples from the Offset Dike of the Sudbury Igneous Complex (SIC) (Hecht et al. 2010).

The minerals of economic interest were detected using a Bruker M4 TORNADO micro-XRF spectrometer and located by automated feature analysis using SEM-EDS (Bruker Application Note # EDS-13).

Method

A FE-SEM equipped with a QUANTAX EDS system including an XFlash® 6 | 10 silicon drift detector (SDD) was used to study the element composition in bulk samples at high spatial resolution (pixel size < 50 nm). Spectrum images (HyperMap) were acquired with parameters as listed in Table 1.

Table 1
Analytical conditions for spectrum imaging (HyperMap)

	Worthington dike	Parkin dike
Accelerating voltage / kV	7	7
Beam current / pA	22	22
Input count rate / kcps	~97	~97
Map resolution / pixels^2	640 x 360	1024 x 768
Pixel resolution / nm	45	50
Acquisition time / min	20	187

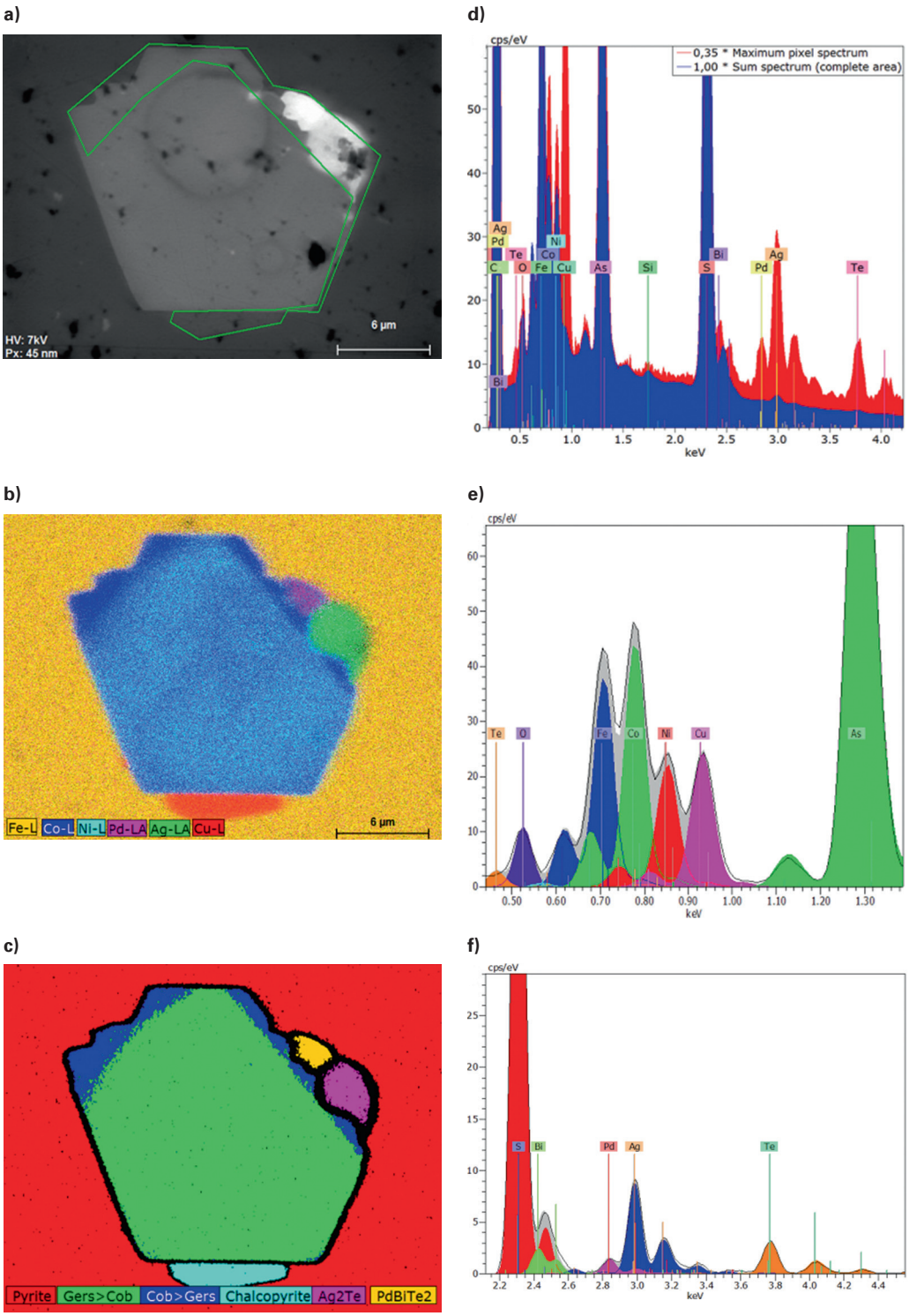

Figure 1
(a) BSE micrograph of sulfide mineralization in a sample from the Worthington dike.
(b) Composite net intensity map showing the distribution of Fe L, Co L, Pd L, Ag L, and Cu L.
(c) Chemical phase map showing the distribution of pyrite (iron sulfide), chalcopyrite (copper iron sulfide), cobaltite/gersdorffite (cobalt nickel arsenic sulfide), and telluride phases.
The 2 x 2 binning net intensity of Fe L, Co L, Ni L, As L, Pd L, Ag L, Cu L, Bi M, Te L and S K was considered.
(d) Comparison of the sum spectrum (blue) of the complete analyzed area and the Maximum Pixel Spectrum (red).
(e, f) Deconvolution results of the selected area shown in (a)

The EDS databases contain complete spectra for each pixel which supports data mining. The element identification can be improved by using the Maximum Pixel Spectrum function (Bright and Newbury, 2004). This function synthesizes a spectrum consisting of the highest count level found in each spectrum energy channel. Even elements which occur in only a few or just one pixel of an element map can be easily identified.

Chemical phase mapping (Autophase) detects similarly composed spectra with the help of mathematical methods or user defined areas.

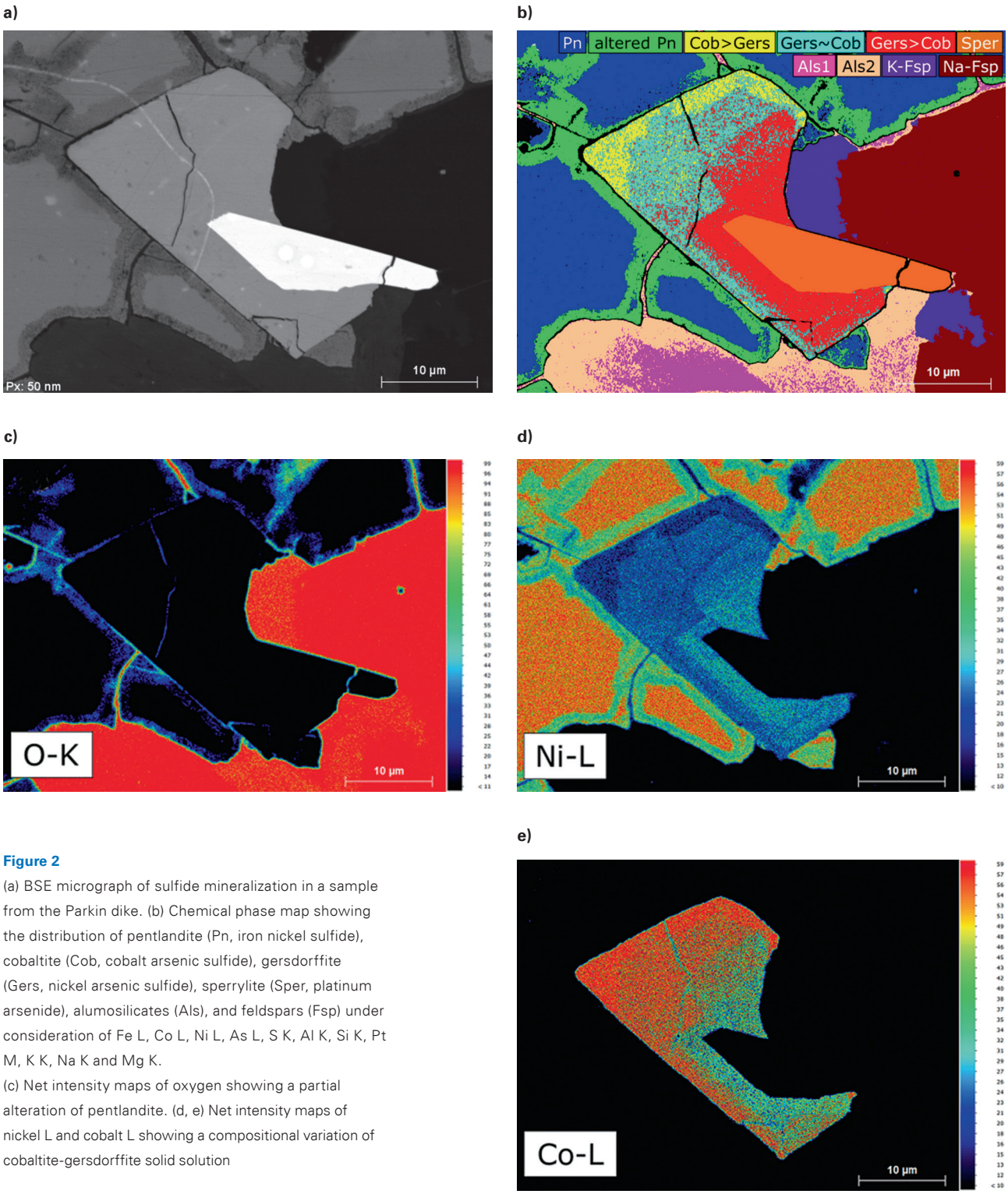

Figure 2

(a) BSE micrograph of sulfide mineralization in a sample from the Parkin dike. (b) Chemical phase map showing the distribution of pentlandite (Pn, iron nickel sulfide), cobaltite (Cob, cobalt arsenic sulfide), gersdorffite (Gers, nickel arsenic sulfide), sperrylite (Sper, platinum arsenide), alumosilicates (Als), and feldspars (Fsp) under consideration of Fe L, Co L, Ni L, As L, S K, Al K, Si K, Pt M, K K, Na K and Mg K.
(c) Net intensity maps of oxygen showing a partial alteration of pentlandite. (d, e) Net intensity maps of nickel L and cobalt L showing a compositional variation of cobaltite-gersdorffite solid solution

To separate overlapping peaks, the line deconvolution algorithms incorporated in the analysis software are of crucial importance. By using an extended atomic database with more than 300 additional L, M and N lines in the low energy range of 0 – 6 keV (Aßmann and Wendt, 2003), the element identification can be improved considerably. The distribution of elements with strong overlapping peaks can be displayed by net intensity maps during or after map acquisition.

Results

The first example of a cobalt nickel arsenic sulfide grain (Figure 1a) shows the element distribution of low energy X-ray lines (Figure 1b) and mineral phases (Figure 1c). Some elements like palladium, tellurium and bismuth were not identified by the sum spectrum of the analyzed area, but in the Maximum Pixel Spectrum function (Figure 1d). Strong overlapping peaks of element lines at the lowermost energy region (iron L, nickel L, cobalt L and copper L, Figure 1e) and at the intermediate energy region (sulfur K, bismuth M, palladium L, and silver L, Figure 1f) can be separated by peak deconvolution.

The second example (Figure 2) reveals a partial alteration of pentlandite (iron nickel sulfide). Compositional variation of a cobaltite-gersdorffite solid solution with substitution of cobalt with nickel at the rim can be recognized.

Conclusion

The discussed element analysis at high spatial resolution shows that the use of low energy X-ray lines for qualitative composition determination is now possible. This is a result of technological developments in silicon drift detectors, research concerning atomic data and modern data proces- sing. The analysis of features at the sub-µm scale using SEM-EDS can provide new insights for sulfide, telluride and arsenide deposit models, because a vast amount of detailed information can be collected in a short period of time.

References

Aßmann, A., Wendt, M., (2003): The identification of soft X-ray lines, Spectrochimica Acta Part B, Vol. 58, 711-716.

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Hecht, L., Siegert, S., Al Barazi, S., and Tichomirowa, M., (2010): Genesis of PGE-Rich Sulfide Mineralization in Offset Dikes of the Sudbury Impact Structure: Abstracts, 11th International Platinum Symposium, 21-24 June 2010, Sudbury, Ontario, Canada.

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