Introduction

Developments in energy-dispersive spectroscopy (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>
<thead>
<tr>
<th></th>
<th>Worthington dike</th>
<th>Parkin dike</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accelerating voltage / kV</td>
<td>7</td>
<td>7</td>
</tr>
<tr>
<td>Beam current / pA</td>
<td>22</td>
<td>22</td>
</tr>
<tr>
<td>Input count rate / kcps</td>
<td>–97</td>
<td>–97</td>
</tr>
<tr>
<td>Map resolution / pixels²</td>
<td>640 x 360</td>
<td>1024 x 768</td>
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<tr>
<td>Pixel resolution / nm</td>
<td>45</td>
<td>50</td>
</tr>
<tr>
<td>Acquisition time / min</td>
<td>20</td>
<td>187</td>
</tr>
</tbody>
</table>

Table 1
Analytical conditions for spectrum imaging (HyperMap)
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 processing. 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


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