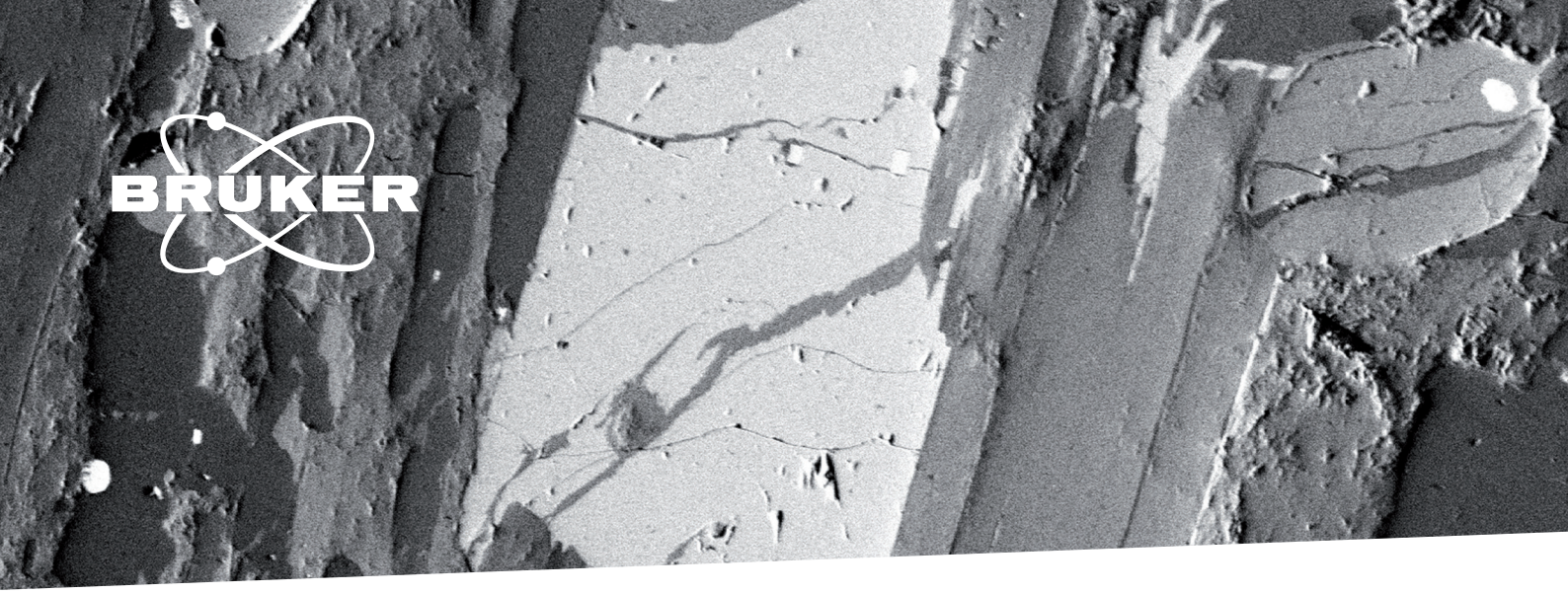


EDS

Identification of trace elements and corresponding phases with hyperspectral imaging (QUANTAX HyperMap)

Application Note # EDS-04

Introduction

The determination of trace elements and the phases that contain them is a common task in microanalysis. Elements are normally identified by acquiring a sum spectrum over a large region of interest. However, elements that are present only in a very small part of the analyzed region cannot be detected in the sum spectrum due to their low overall concentration.

In this report, QUANTAX HyperMap is used to overcome this shortfall.

Methods used

A Bruker QUANTAX EDS system equipped with a liquid nitrogen free XFlash® 5010 Detector (energy resolution of 123 eV for MnKα at 100,000 cps) was used to acquire a HyperMap database. Acquisition was performed under the following conditions:

- Accelerating voltage: 15 kV
- Acquisition time: 20 h
- Input count rate: 16,300 cps

The HyperMap database contains, in addition to the image, an EDS spectrum for each pixel (in the discussed example 640 x 480 pixel = 307,200 spectra). This supports offline evaluation during or any time after the mapping acquisition.

The Maximum Pixel Spectrum function (Bright and Newbury, 2004) synthesizes a spectrum out of the HyperMap data that contains the highest count level found in each spectrum channel. With this routine, trace elements which occur in only one pixel can be detected

qualitatively. In order to identify the phases that contain detected elements, two different element map methods were used: the region of interest (ROI) intensity map and the quantitative map (QMap). The ROI intensity map displays the intensity of a distinct region in the spectrum related to the element line. The QMap displays the element concentration based on the quantification result of each pixel. The QMap and a relevant area were quantified using the standardless peak-to-background (P/B) model (Wendt and Schmidt, 1978) and subsequent ZAF-correction.

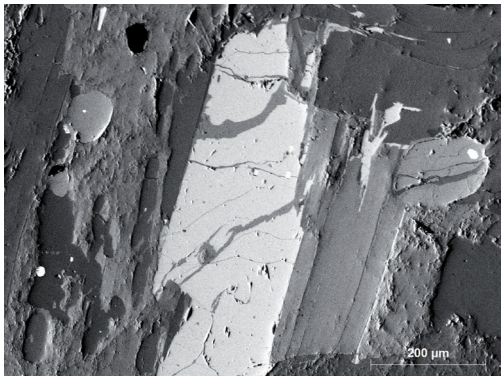


Figure 1
BSE micograph of a granite sample showing the region analyzed by HyperMap. The bright region in the center represents ilmenite.

Results

The analyzed sample is a polished granite sample, which consists of many different minerals (Figure 1). In Figure 2 the sum spectrum is compared to the Maximum Pixel Spectrum; both were obtained out of the HyperMap database. The Maximum Pixel Spectrum clearly shows the presence of thorium, lanthanum and cerium, which cannot be detected in the sum spectrum due to their low overall concentration.

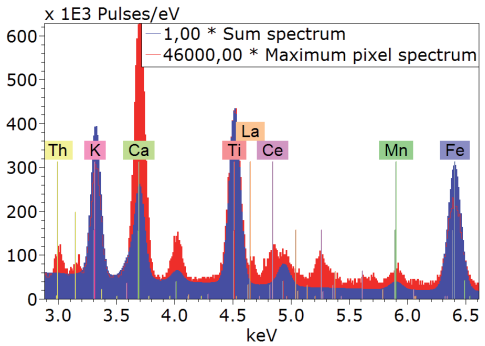
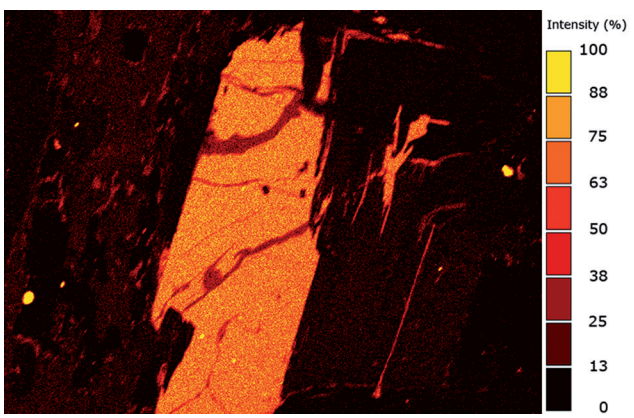


Figure 2
Comparison of the sum spectrum of the region shown in Figure 1 with the Maximum Pixel Spectrum. Spectra are normalized for better presentation.

In order to identify the mineral phase which contains these elements, a ROI intensity map was performed for the element line Ce-Lα at 4.837 keV (Figure 3 left). Small round phases with a high intensity are potential candidates for the mineral containing cerium. In addition, other phases such as ilmenite display an intermediate intensity. However, this is a false element distribution that results from background modulation, as ilmenite has a significantly higher background than silicate minerals.

To verify the presence of the elements in the identified phase, one region of the HyperMap with high cerium concentration was marked and its spectrum was evaluated. The deconvolution result considering only lanthanum and cerium (Figure 4 left) shows a significant difference between the sum of the deconvoluted peaks and the measured spectrum. This indicates that some elements were not identified. The deconvolution considering praseodymium, neodymium and samarium (Figure 4 right) results in a perfect match. In addition, the quantification results (Table 1 and 2) show that these and other detected elements are present in the identified phase. This phase is monazite, which is an important mineral for radiometric age dating.



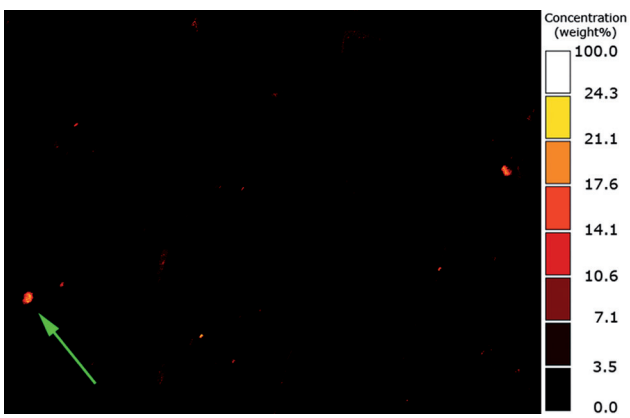
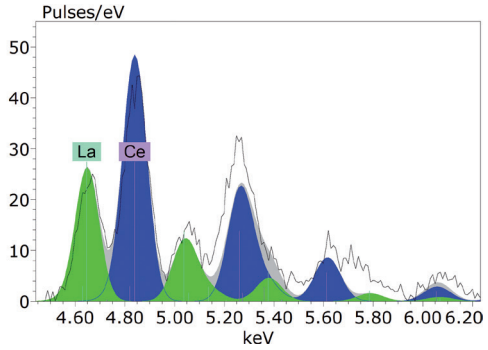


Figure 3
Comparison of the element distribution of cerium. Left: ROI map showing intensity of Ce-Lα (%). Right: background corrected QMap showing Ce concentration (weight %); the arrow points to the phase containing cerium (see Table 1).



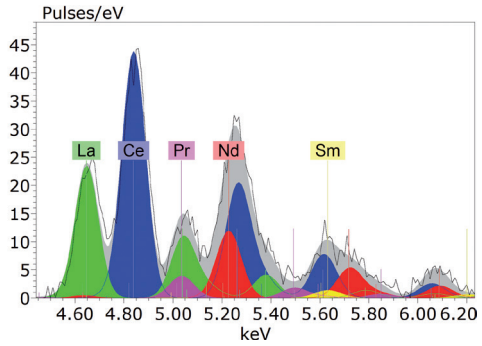


Figure 4
Deconvolution of overlapped, background corrected peaks without (left) and with (right) consideration of praseodymium, neodymium and samarium. Each color represents the deconvoluted peak for one element; the grey peak in the background is the sum of the deconvoluted peaks. The black line is the measured spectrum.

Element	wt.%	at.%
P	14.89	17.87
Ca	1.24	1.15
La	12.18	3.26
Ce	23.63	6.27
Pr	2.89	0.76
Nd	9.49	2.45
Sm	0.55	0.14
Th	6.23	1.00
O	28.90	67.12
Total	**100.00**	**100.00**

Table 1 (left)
Element concentration in monazite. The results were obtained from a 12-pixel region of the HyperMap. The spectra contained ~60,000 counts.

Oxide	wt.%
P_2O_5	34.13
CaO	1.73
La_2O_3	14.28
Ce_2O_3	27.68
Pr_2O_3	3.38
Nd_2O_3	11.07
Sm_2O_3	0.64
ThO_2	7.09
Total	**100.00**

Table 2 (right)
Oxide concentration in monazite using the same spectra as in Table 1.

Conclusions

Using HyperMap and Maximum Pixel Spectrum, we were able to prove the existence of trace elements that were not visible in the sample´s sum spectrum. Element identification results were improved through peak deconvolution, as the peaks of further elements were interfered by other higher peaks. It can be concluded that HyperMap in combination with Maxium Pixel Spectrum is an invaluable tool for the analysis of complex multi-element samples.

References

Bright, D.S. and Newbury, D.E. (2004). Maximum pixel spectrum: an new tool for detecting and recovering rare, unanticipated features from spectrum image data cubes, Journal of Microscopy, 216, 186-193.

Wendt, M. and Schmidt, A. (1978). Improved reproducibility of energy-dispersive X-ray micronanalysis by normalization to the background, Physica status solidi (a) 46, 179-183.

Acknowledgement

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All configurations and specifications are subject to change without notice.
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