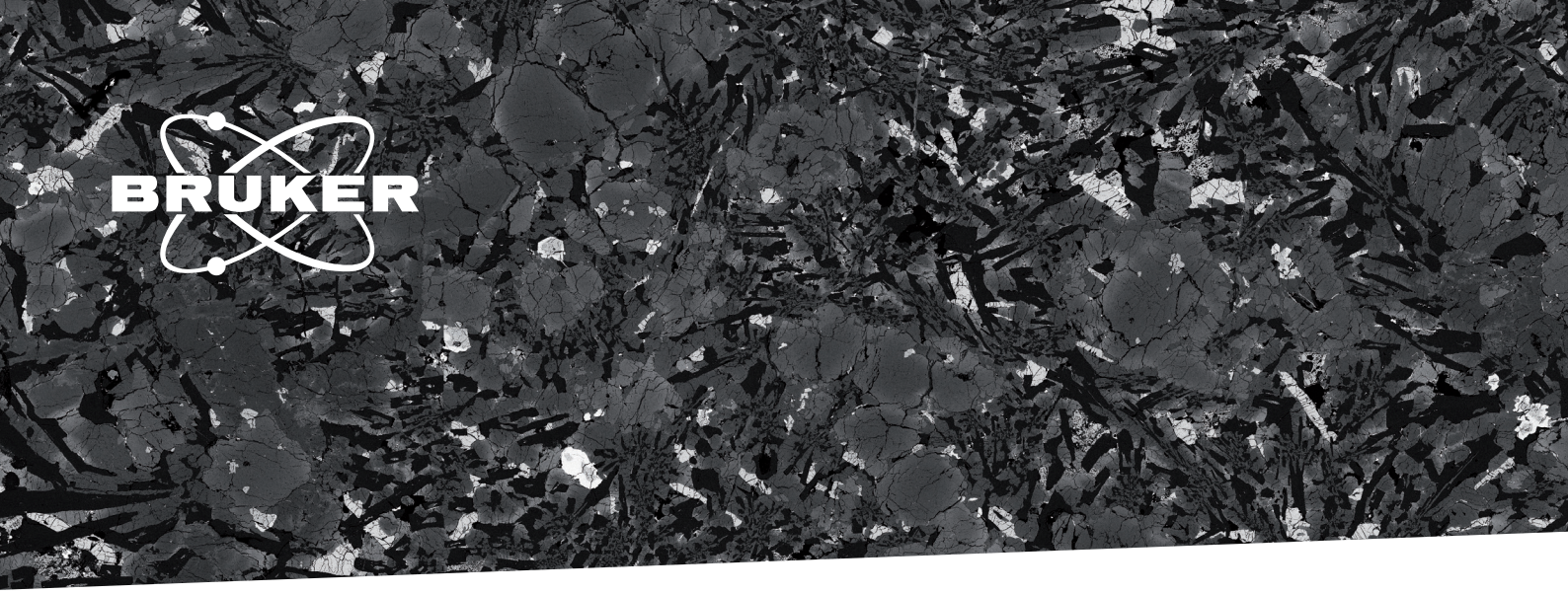

EDS

Specific small particle search by mineral grain feature analysis and chemical evaluation using QUANTAX

Application Note # EDS-05

Introduction

It is a frequent requirement for a small and rare mineral grain containing a specific element to be found inside a large sample. The challenge here is to find as many as possible (if not all) of these grains, and do it in a reasonable time frame. Manual methods are tedious and time consuming, while an automated feature analysis program can be very fast.

Feature analysis is the study of shapes, the calculation of its morphological parameters and positions and the tabulation of these results. For further information depth, the chemical composition can also be measured and particles can be classified subsequently by applying user defined rules to the obtained data (such as: must contain Zr). With respect to analysis speed, the compromise lays in the resolution and magnification of the recorded and analyzed images. The number of phases that are analyzed by the system as well as the counts per spectrum and count rate also affect speed.

In this case the analyzed sample was a lunar basaltic meteorite Dhofar287-A thick section hosting small grains containing baddeleyite (zirconium oxide mineral ZrO_2 or zirconia with a stoichiometric ratio of Zr: 74.03 wt. % to O: 25.97 wt. %).

It was mounted on a glass slide, measured 9000 x 5000 x >100 µm and weighed about 200 mg. The glass slide was carbon coated, placed on a thin-section holder and mounted on the stage of the scanning electron microscope (SEM).

The expected size of these grains can be as small as 3 µm or as big as more than 60 µm. All these grains needed to be found so that they could be measured with radiometric dating methods such as a U-Pb using an ion probe. Grains under 3-6 µm would not be measurable with this method, so size filtering was also a necessity.

Methods

To perform this search in an automated manner and achieve reliable results, a SEM with a backscattered electron (BSE) detector was used in combination with the QUANTAX EDS system together with a Bruker particle analysis solution. The SEM used, a JEOL JSM6490LV, was set to an accelerating voltage of 15 kV and sufficient beam current to obtain 40,000 cps on Zirconium with an XFlash® 5010 EDS detector (FWHM of 123 eV at Mn Kα). The microscope was also set up to obtain a good resolution at 140x magnification using a solid state BSE detector. The actual settings for the measurement were:

- Magnification: 140x
- Resolution: 1024 x768 pixels
- Calibration/sizing error: 0.637 µm/pixel
- Spectrum statistics: 100,000 counts

Binarization is a method for differentiating regions of disinterest (matrix) from those of interest (particle) by creating a binary image (black and white) via thresholds. These thresholds are set on a greyscale image (0 to 255 black to white) and provide a way of simplifying the image to two states: interest (e.g. white) and non-interest (e.g. black).

The binarization thresholds were set to exclude all phases that were not of interest. It is important to know in which region of atomic number contrast the phase of interest lays in, in order to get the binarization as tight as possible. This reduces the number of false positive analyses. Making it too tight causes the loss of some of the particles that might be brighter or darker than the average.

The binarization thresholds for this particle detection were set between 180 and 255 (the brightest 30%). In this way, the zirconium standard was included as well. Once the particle in the BSE greyscale region was found, a morphological analysis was performed. Particles which were smaller than 3 µm length and 2 µm width were filtered by a pre-classifier. The chemical composition of the remaining particles was then determined by EDS analysis at their innermost point. Quantification was performed via Bruker´s standardless automatic P/B ZAF routine.

At the end of the measurement, the false positives were filtered according to their chemical composition. Only the phases of interest were then used for further analysis. This included returning to the position of the grain and acquiring new images and spectra for each one.

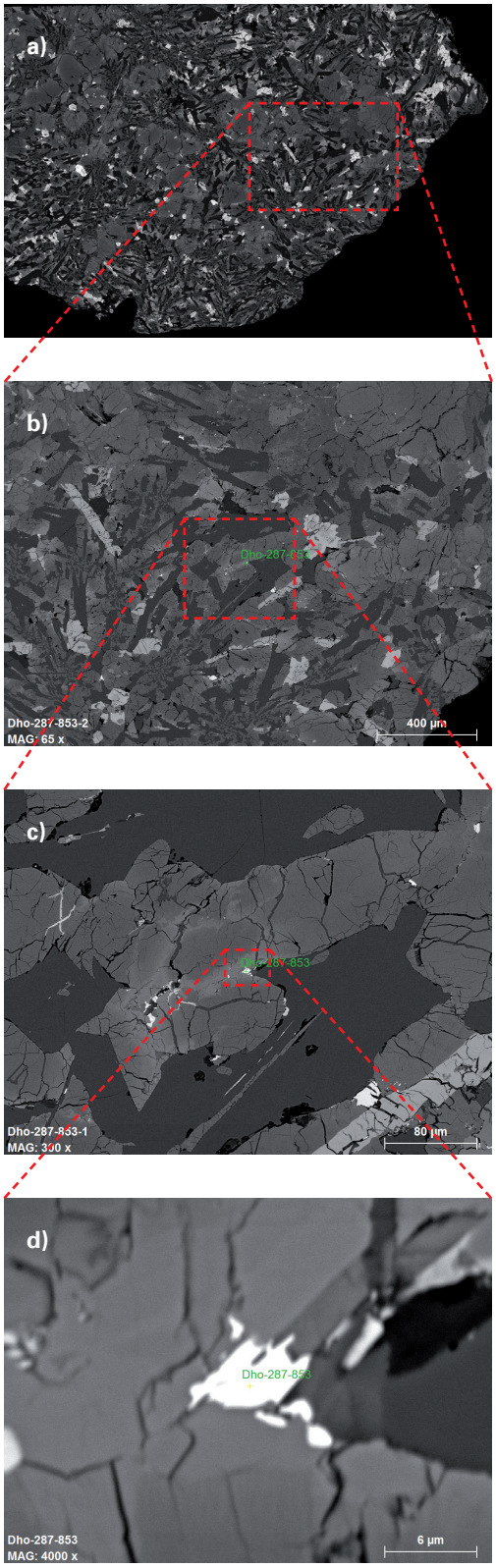

Figure 1
a) Composite image of sample, 90 megapixel resolution
b) Low magnification (65x), c) medium magnification (300x) and
d) high magnification (4000x) images of particle Dho-287-853

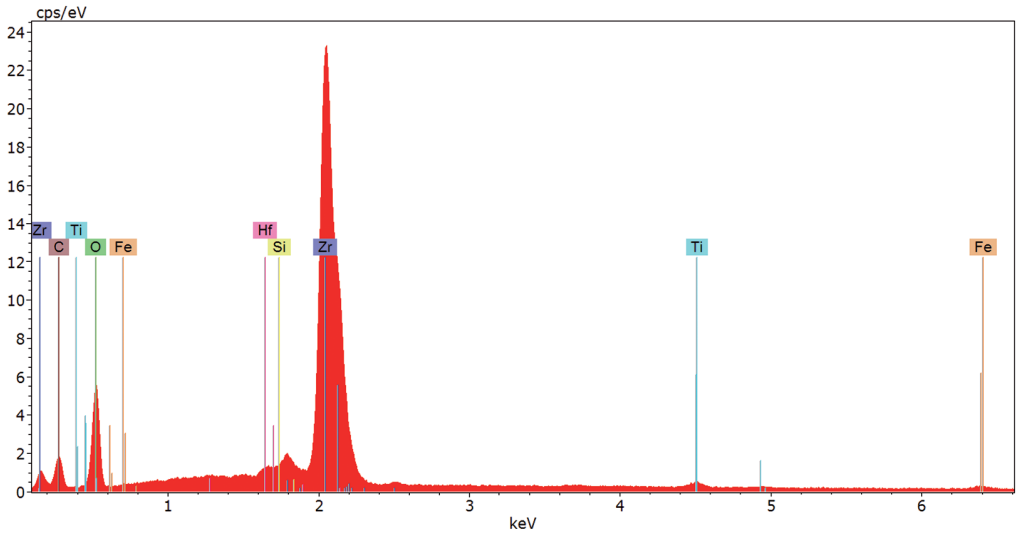

Figure 2
Spectrum of particle Dho-287-853

Results

Acquiring 90 images, the measurement took 86 minutes and found 997 particles that were in the binarization range. Furthermore, a tiled composite image of the entire measured area was generated and saved as shown in Figure 1. Utilizing the saved uncompromised data, a high resolution composite image was generated by tiling or stitching the images together, which was used for manual offline inspection.

For each found particle a spectrum was acquired and quantified (see Figure 2 for a spectrum of particle Dho-287-853). The results were filtered according to the Zirconium content of 55% or more, resulting in a list of 11 particles. Some of the more important morphological data for these particles is shown in Table 1.

Using the software, these particles were driven back to via the "drive stage to particle" operation, and new images were acquired to help finding these small particles at a later date. The new images were acquired at magnifications of 4000x, 300x and 65x.

New spectra were also acquired to confirm the zirconium concentration, this time at a lower count rate of 6,000 cps with 500,000 counts per spectrum. These spectra were then re-quantified using the stoichiometric relationship of ZrO_2. The results are shown in Table 2. Some of the concentrations have contributions from other minerals due to the small size/depth of the particle.

ID	Area/µm²	Max. length /µm	Width/µm	Perimet./µm	Zr Wt.%
01-00073	8.1	4.9	4.0	16.5	63.2
01-00143	5.3	3.4	2.6	9.5	60.3
01-00365	12.2	6.3	3.8	16.4	62.0
01-00381	13.4	6.7	3.2	16.0	62.7
01-00399	14.2	9.4	3.9	28.0	59.5
01-00571	6.1	4.3	2.6	10.7	67.9
01-00620	5.7	4.3	2.3	10.3	63.5
01-00815	13.0	6.4	4.3	17.5	66.2
01-00853	19.1	8.1	4.5	19.9	66.5
01-00870	9.8	4.9	3.2	12.2	66.9
01-00951	11.4	5.1	4.4	17.0	60.6

Table 1
Particle list for Zr > 55% (other elements and morphological parameters excluded)

Spectrum	SiO_2	TiO_2	HfO_2	ZrO_2	Al_2O_3	FeO	CaO	Sum
Dho-287-073	1.67	3.48	1.24	90.51		2.61	0.49	100.0
Dho-287-143	2.02	3.93	1.28	90.56		1.82	0.40	100.0
Dho-287-365	0.91	4.38	1.16	90.41		2.75	0.39	100.0
Dho-287-381	0.66	3.21	1.06	93.02		1.75	0.31	100.0
Dho-287-399	0.70	3.20	0.83	92.76		2.22	0.30	100.0
Dho-287-571	0.75	1.09	0.74	95.37		1.75	0.31	100.0
Dho-287-620	1.21	1.60	1.40	93.15		2.19	0.45	100.0
Dho-287-815	1.98	2.87	0.74	91.06		2.91	0.45	100.0
Dho-287-853	0.72	1.90	1.40	94.23		1.75		100.0
Dho-287-870	0.70	1.12	1.47	94.83		1.55	0.33	100.0
Dho-287-951	1.02	1.24	0.94	94.76	0.34	1.40	0.29	100.0

Table 2
Quantitative results based on P/B ZAF method in stoichiometric weight percent

Conclusions

The measurement was successful in finding small and rare baddeleyite particles via the material contrast of the BSE image and provided the chemical concentrations of each particle. Furthermore, this was done in a short period of time due to the fully automatic image acquisition and analysis, spectrum acquisition and stage movement as well as the speed of the XFlash® detector.

Performing this operation manually could have taken many hours, while performing it with a QUANTAX particle analysis solution the measurement took less than 1 hour 30 minutes. Additionally, using an automated feature analysis tool provides a complete set of morphological measurements together with the chemical makeup.

The ability to create high resolution composite images (see Figure 3) provides an overview image that can be used to help find specific particles at later dates or to explore the sample offline.

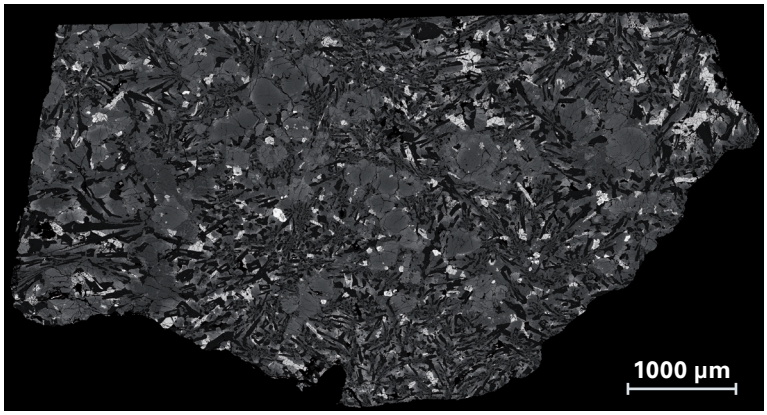

Figure 3
Entire composite image of sample

Acknowledgement
Sample and search criteria courtesy of Vera Fernandes from the Berkeley Geochronology Center.

Author
Samuel Scheller, Global Product Manager EDS / SEM, Bruker Nano GmbH

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Electron Microscope Analyzers
Bruker Nano GmbH
Berlin · Germany
info.ema@bruker.com

www.bruker.com/quantax-eds-for-sem