MICRO-XRF

Analysis of geological thin sections

Application Note # XRF 456

Introduction

The element distribution and the mineralogical composition of a geological sample provide key information for the understanding of geological structures and processes. To obtain this information is useful in many fields including geological research topics as well as mining and prospection of natural resources.

Thin sections are routinely studied with polarizing petrographic microscopy, and/or electron microscopy in combination with energy dispersive X-ray spectroscopy (SEM-EDS). These two approaches are standard in geological work. For the SEM analysis samples usually have to be coated with an electrically conductive material and measured in high vacuum. The SEM allows resolving compositional variations in a sample down to the nanometer range.

Micro X-ray fluorescence (micro-XRF) analysis uses direct excitation of the sample with focused X-rays. This allows the combination of micrometer-scaled spatial resolution of a polycapillary lens with the trace element sensitivity of XRF, where the detection limits for most elements are a few tens to hundreds of ppm. No additional sample preparation is required for micro-XRF. The high speed of the data acquisition and evaluation allows scanning multiple thin sections in less than one hour.

The samples

A thin section is a thin sample slice prepared from a rock, mineral, soil, bone, or even metal sample. The samples are mounted on a glass slide and ground and polished smooth using progressively finer abrasive grit until the sample is around 30 µm thick. The standard size of a thin section is 28 x 46 mm². 18 thin sections can be placed on the stage of the M4 TORNADO and measured in a single run. Figure 1 shows a mosaic image of this arrangement, the total measurement area was 166 x 140 mm².

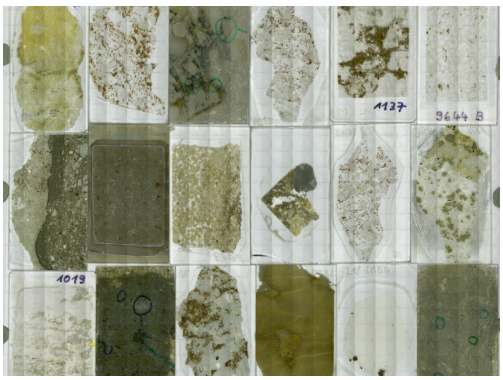

Figure 1
Mosaic image of the complete sample stage (close to the maximum area that can be analyzed in one run) with 18 geological thin sections

Instrumentation

The measurements were performed with a Bruker M4 TORNADO. This micro-XRF spectrometer uses a focused X-ray beam (spot size <20 µm) to induce fluorescence in the sample. This signal is analyzed with an energy dispersive detector. The M4 TORNADO combines high spatial resolution with fast data processing and a high speed motorized XYZ-stage for sample positioning.materials can be generated. This method is very fast and does neither require peak identification nor the calculation of quantification.

Measurement conditions

The X-ray tube settings used for the analysis were 50 kV and 600 µA. The samples were measured at a pressure of 20 mbar. Step size and integration time per pixel were set to 50 µm and 1 ms, respectively. For comparison additional measurements were done with a lower resolution (larger step size of 200 µm). This influences the total measurement time and the size of the data set as shown in Table 1.

When operating in the continuous data acquisition mode (on-the-fly), the instrument can scan a sample at very high speed with dwell times per pixel even below 1 ms. Further improvement in the data acquisition can be achieved by using an upgraded system with a second detector for parallel data collection. During the measurement the spectrum of every single pixel was saved using the position tagged spectroscopy (HyperMap).

Element distribution

Figure 2 displays the distribution of the main components for the thin sections in Figure 1. The data was collected under an optimal speed/resolution regime for this task (total measurement time of 5.2 h, approx. 17 min for a single sample). The resolution is more than sufficient to classify the main mineralogical features in each thin section and to identify regions of particular interest for analysis in detail.

A comparison of the data extracted from the large scan is shown in Figure 3. If the step size is reduced by a factor of 4, the number of pixels increases by a factor of 16. The images illustrate the gain in resolution due to the smaller step size used in the high resolution scan.

The element distribution data can be evaluated within this consistent dataset or the information for each sample can be "cut out" and saved independently in separate files (Figure 4).

Measurement conditions		
Step size	200 µm	50 µm
Number of pixels	830 x 700 5.8 10^5	3320 x 2800 9.3 10^6
Pixel time	3 ms	1 ms
Total time for: **complete set** **single sample**	 1.2 h 4 min	 5.2 h 17 min
Size of data set	350 MB	2300 MB

Table 1
Measurement conditions for different resolutions

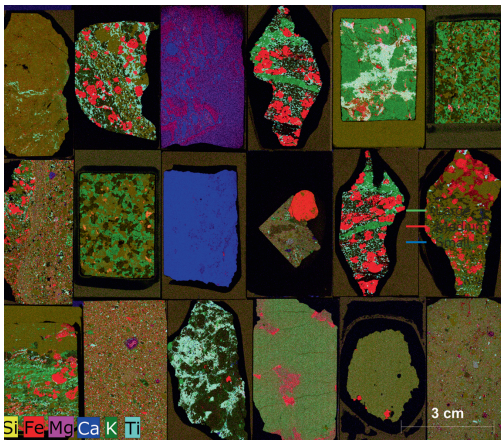

Figure 2
Distribution of the main components in the thin sections. Each color represents an individual element.

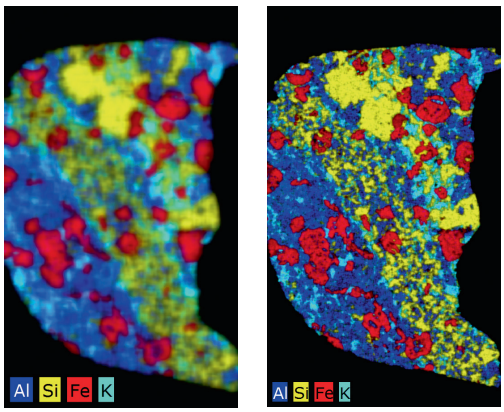

Figure 3
Distribution of major elements of the gneiss sample (second from left in the top row in Figure 2) for step sizes of 200 µm (left) and 50 µm (right).

Data mining tools

Besides offering the possibility to digitize thin sections in short time and to store the data with the full spectrum for each pixel, the M4 TORNADO software offers a large number of options to extract the data.

The Objects tool provides means for a detailed analysis of specific areas within an elemental distribution (HyperMap).

Figure 5 shows the Objects tool and the corresponding points, lines, rectangles, circles, and irregular shaped areas drawn over distinctive regions of a HyperMap.

These objects help to calculate the sum spectra of the defined regions in the measured HyperMap and allow to identify the present mineral phases. Figure 6 shows the sum spectra of the phases ilmenite, garnet and quartz determined in the sample in Figure 5.

By drawing a line into a measured HyperMap the corresponding element intensity profile can be displayed. To reduce statistic effects this line can be widened by summing up more pixel columns perpendicular to the line. Figure 7 shows the profile data extracted from the line plotted in the HyperMap in Figure 5. Each element is normalized to its maximum intensity, allowing to visualize the correlation of major and minor elements within one mineral (Fe garnet with Cr and Mn).

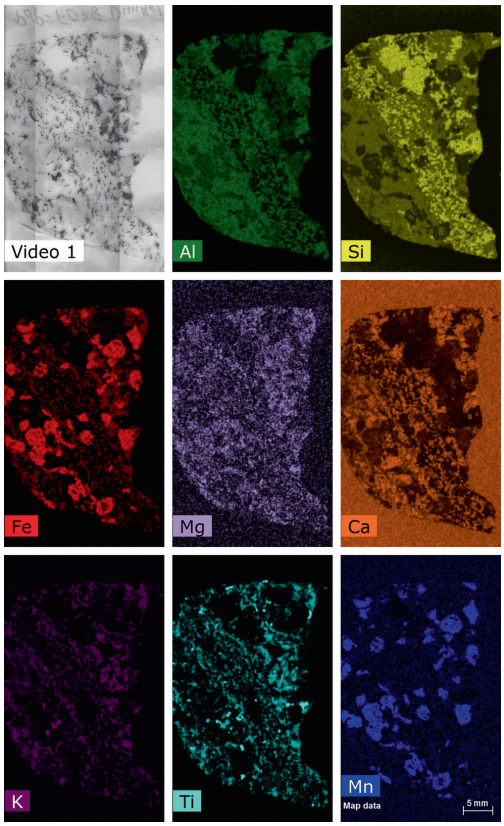

Figure 4
Distributions of major elements of the gneiss, displayed in individual frames.

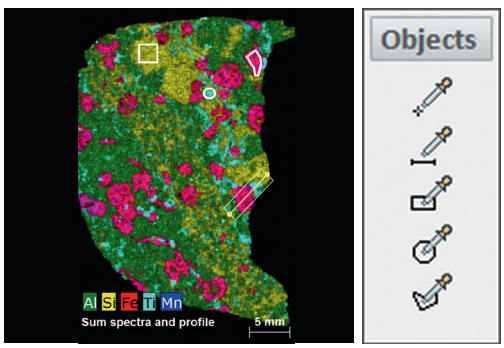

Figure 5
HyperMap of the gneiss sample with defined objects for sum spectra and element profiles.

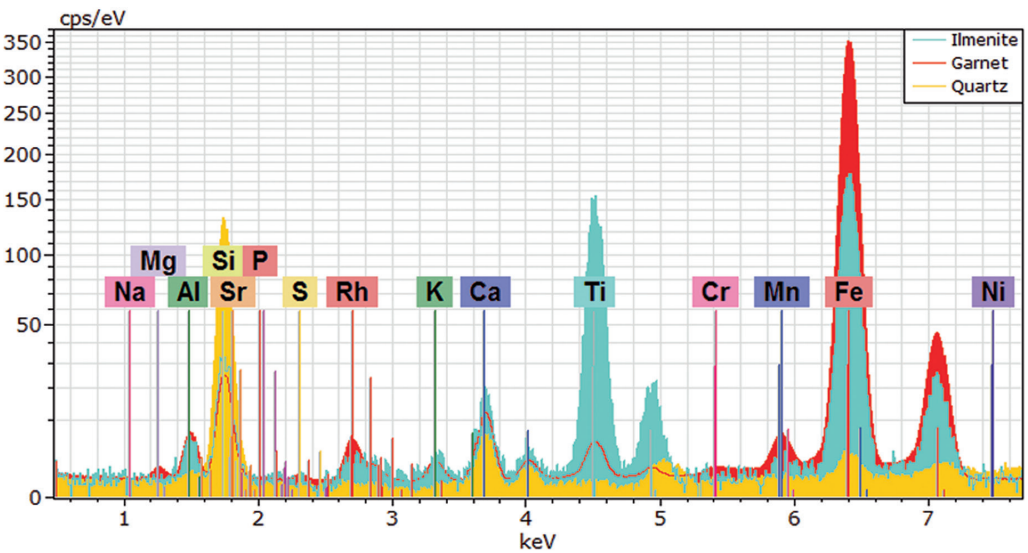

Figure 6
Sum spectra of the phases ilmenite, garnet and quartz in the gneiss sample.

Phase analysis

Phases are determined by finding areas with similar spectra within the data set. The relative ratios of distinct chemical phases are determined. Figure 8 displays the mineralogical phases found in the gneiss sample in Figure 5.

The results given in Table 2 show the ratio of garnet to quartz to be ~ 2:1, whereas feldspar and quartz occur in quite similar proportions.

Summary

The M4 TORNADO is a powerful micro-XRF spectrometer for the fast analysis of geological samples, not only especially for thin sections but also for bulk and powder samples.
The M4 TORNADO is used in petrology for a quick compositional pre-screening of thin sections and for the digitization of thin sections before optical or electron microscope studies. Large scan areas of up to 170 x 140 mm² allow to analyze up to 18 thin sections in a single run.

The M4 TORNADO software supports comprehensive data presentation and evaluation. The visualization of the chemical map and the selection of areas of interest enable advanced post processing of the HyperMap data as phase analysis for fast identification of elements and mineral phases, quantitative analysis and the finding of trace elements.

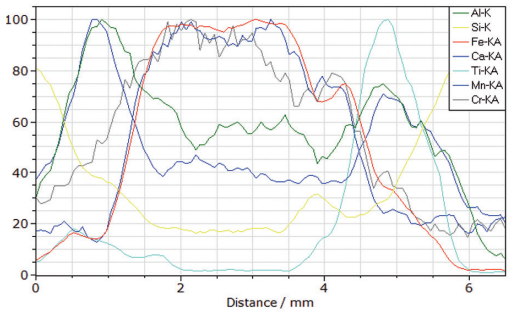

Figure 7
Distribution of mineral phases including selected elements along a line profile.

Table 2
Results of the phase analysis in wt.%

Results of the phase analysis

Phase number	Phase	Content
Phase 1	Glass	35
Phase 2	Feldspar	14.7
Phase 3	Quarz	13.6
Phase 4	Garnet	22.5
Phase 5	Biotite	13.9

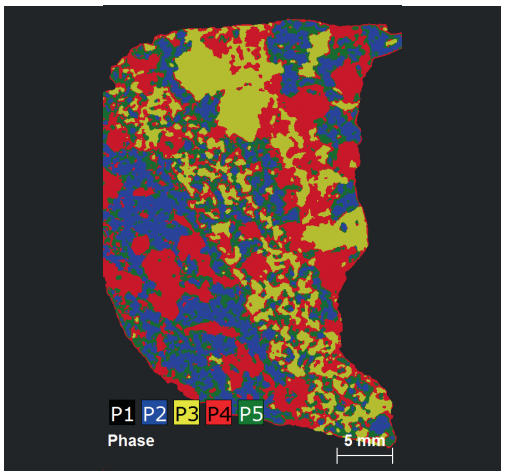

Figure 8
Distribution of mineralogical phases found in the gneiss sample.

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