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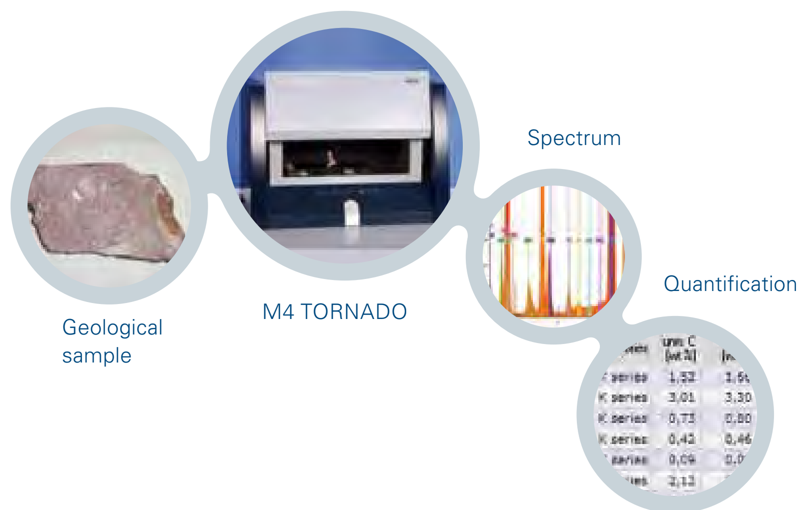


Lab Report XRF 441

Elemental distribution analysis on geological samples with the M4 TORNADO

Geological samples are inhomogeneous. The distribution analysis of their different compounds is important for understanding geological processes, exploration of mineral resources, and other applications.

Micro X-ray fluorescence (µ-XRF) is well suited for these purposes. It offers very good spatial resolution combined with high sensitivity for small element concentrations. This report describes the examination of a geological sample to discuss the influence of different measurement parameters on the analytical results.

Sample

The investigated sample is an impact melt breccia from the Rochechouart structure in France (25 km diameter). This meteorite impact structure was dated and revealed an age of 214 ± 8 million years. The Rochechouart is the first impact structure on earth in which traces of the impacting asteroid were found. Previously they had only been found in Apollo samples from the moon. The traces of the projectile were partially vaporized, melted or highly fragmented during the impact event and mixed with the instantaneously molten and shocked rocks from the target of the Massif Central. The presence of a projectile in an impact melt rock can be recognized by the enrichment in certain elements more abundant in meteorites than in rock from the earth crust. These elements are mainly platinum group elements (PGE = Os, Ru, Ir, Pd, Pt and Rh), but also other siderophile elements such as Ni. Whereas the found PGE are in the low ppb concentration range, the concentration of Ni is in the medium ppm range and therefore detectable with X-ray fluorescence.

The analyzed sample, shown in figure 1, was a roughly polished slab. The multi-element imaging of the M4 TORNADO with a detection limit in the low ppm range for many elements allows a better understanding of the distribution of the components involved in the formation and the hydrothermal evolution of the impact melt.

Instrument

The measurements were performed with the Bruker M4 TORNADO. This instrument is characterized by the following features:

- large vacuum chamber for samples up to 200 x 300 x 150 mm^3
- fast X-Y-Z stage with TurboSpeed for fast mapping and distribution analysis on-the-fly

Analyzed sample area

Fig. 1 The highlighted sample area was analyzed with μ-XRF

- effective excitation of fluorescence through high brilliance X-ray tube together with X-ray optics for concentration of tube radiation to spot sizes down to 25 μm
- detection of fluorescence radiation with silicon drift detectors (SDD) with high count rate capability
- quantification with standardless models using full pattern fitting.

The measurement was performed under the following conditions:

- Excitation: 50 kV, 600 μA
- Analyzed area: approx. 48 x 25 mm
- Number of pixels: 600 x 313
- Acquisition time per pixel: 5 ms and 200 ms, respectively
- Total measurement time: 20 min and 10.5 h, respectively

Total measurement time

The total time for the measurement is not only determined by the acquisition time per pixel but also by the time required for stage movement. The M4 TORNADO performs the measurement on-the-fly, which means that the stage moves constantly and does not stop at every pixel. This saves a lot of time and makes the total measurement time almost identical to the sum of pixel times.

The measurement can be performed unidirectionally. In this case, the time for the return travel of the stage after every line must be added to the time for actual scanning. This may add up to several minutes for large scans. Another possibility is meandering. This saves the time for the return travel, but requires a stage with very small backlash. The M4 TORNADO can measure in both modes.

Distribution analysis results

Figure 2 shows the results of distribution analysis at two different acquisition times per pixel for some selected elements. The upper image shows that a good elemental distribution analysis can already be achieved after an extremely short acquisition time of only 5 ms per pixel. The main structure of the mineral can be recognized, but the finer detail is blurred due to the high noise that results from the large statistical error. If the measurement time is increased to 200 ms (bottom), the image becomes smoother and allows better identification of different phases in the mineral and the separation of even small inclusions and grains.

Distribution analysis

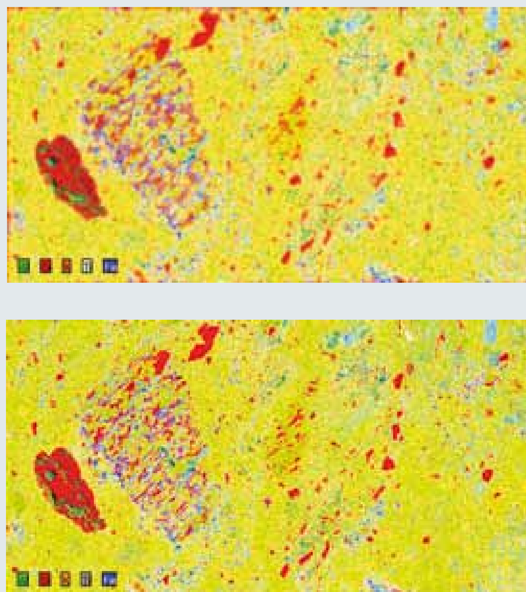


Fig. 2 Results of a distribution analysis with an acquisition time per pixel of 5 ms (top) and 200 ms (bottom)

HyperMap performance

The M4 TORNADO uses Bruker's HyperMap technology; i.e. a complete spectrum is saved for every pixel of the map. This offers a lot of advantages, in particular for the data evaluation. A few of them will be demonstrated in the next sections using the data of this measurement.

- Distribution of single elements

The availability of data for every pixel permits the calculation of the distribution of single elements. This provides additional information on the composition of the sample. The distribution of the main components of the mineral is shown in figure 3.

Single element maps

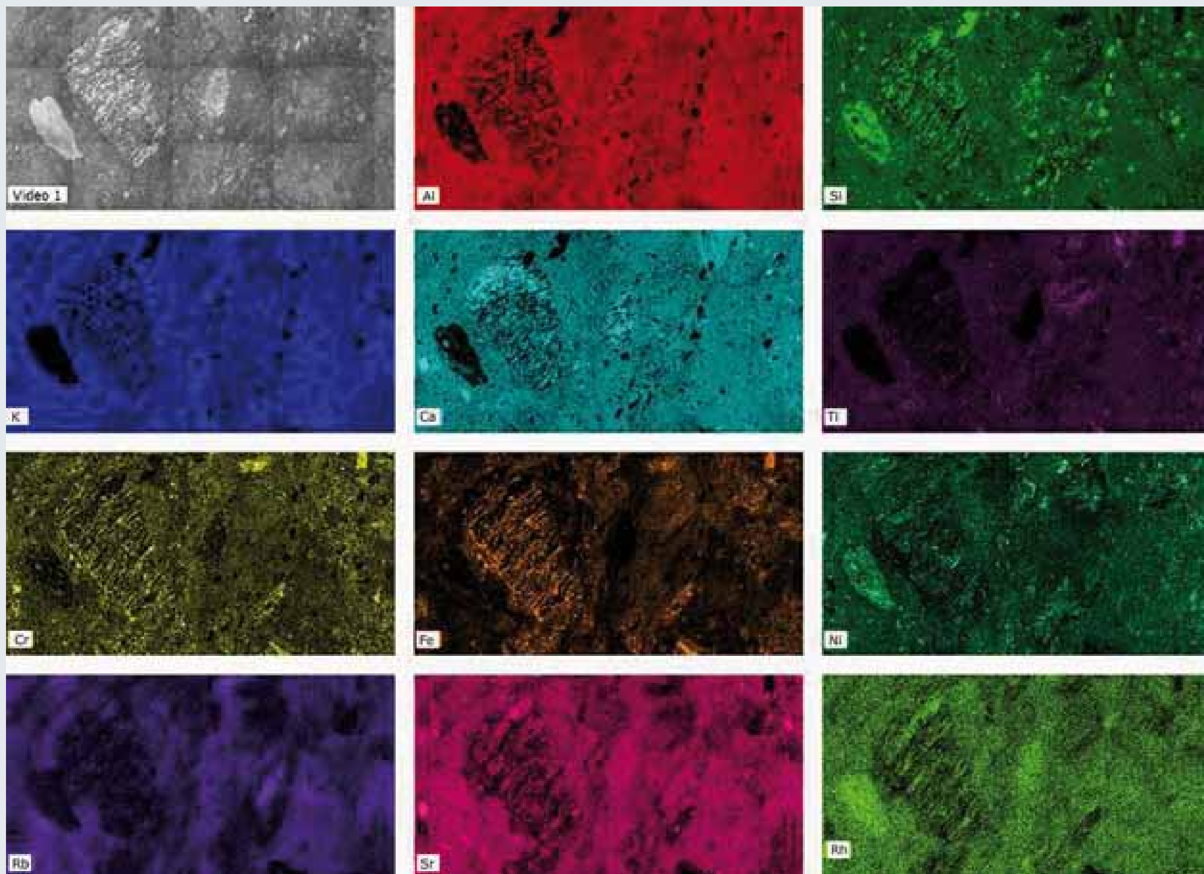


Fig. 3 Single element distribution images of a series of elements calculated from the HyperMap at 200 ms acquisition time per pixel

The resolution of the element distribution images is influenced by the measurement time. This can be concluded by comparing the distribution images of the same element at two different measurement times. Figure 4 shows the element distribution of Ni as trace element, which is interesting for determining the base of the impact crater. The differences in spatial resolution at both measurement times can be clearly observed.

The image resulting from the analysis with the longer measurement time shows clear element distribution with sharp structures. As for the analysis at the short measurement time, the Ni image is blurred and unclear.

- Sum spectra of complete mapping

HyperMap also offers the possibility to accumulate a spectrum for every pixel and display the sum spectrum of the complete mapping. Figure 5 shows the sum spectra of both mappings in a logarithmic scale. The intensity of one of the spectra is significantly higher (factor 40), due to the longer measurement time. However, other differences between the spectra are negligible, since the measurement time was long enough in both cases.

- Single point and accumulated spectra

The acquisition of a spectrum for a single point or a selected sample area is also possible. This is shown in figure 6. In this case significant differences between the measurement results can be detected in dependence of the analyzed area and the measurement time.

The red spectrum shows the result of a single pixel at 5 ms measurement time. This only allows the identification of main components, but no further evaluation. The green spectrum is the result of measuring the same pixel for 200 ms. It has better statistics and allows the identification of minor elements too. Finally, the blue spectrum results from the measurement of a large area with similar composition. It shows good statistics and traces even at a measurement time of only 5 ms and allows quantification.

Distribution of Ni

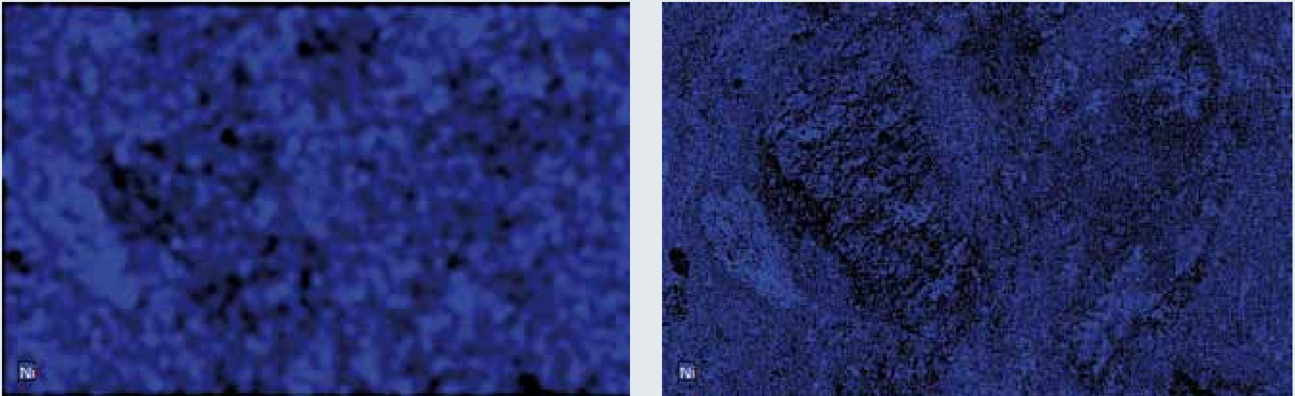


Fig. 4 Distribution of Ni for 5 ms (left) and 200 ms (right) acquisition time per pixel

Sum spectra

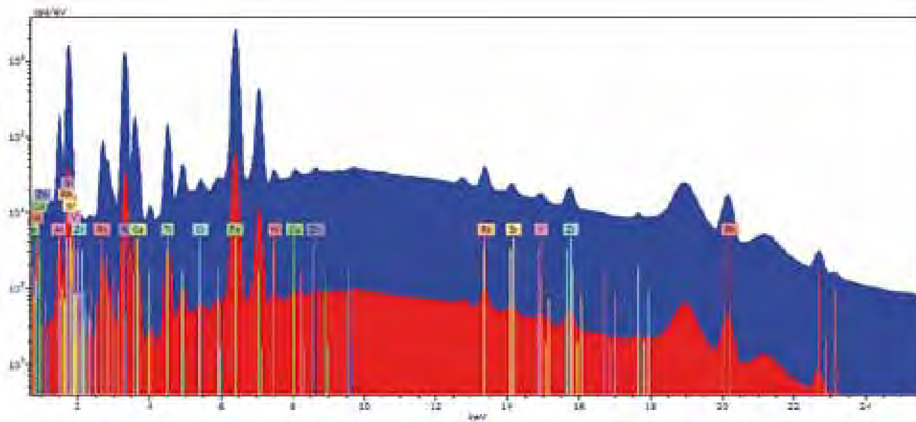


Fig. 5 Sum spectra of both mappings (logarithmic scaling)

- Trace analysis

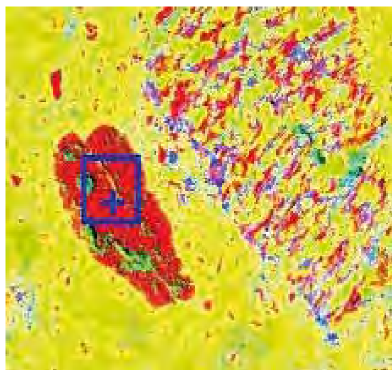
HyperMap also offers the function Maximum Pixel Spectrum, which generates a spectrum out of the highest channel content for every pixel of the complete map. Figure 11 compares this spectrum with the sum spectrum of the entire map. The Maximum Pixel Spectrum provides more information on trace elements, which can be used for the calculation of their distribution. This is valid for Ni, Cu and Zr in this case. The Rh-peak results from the scattering of tube radiation on light sample areas (in the example, the SiO_2 areas).

- Line Scan

The availability of data for every pixel also permits the calculation of the element distribution along a line - a line scan. This is demonstrated in figure 8.

The line scan can be performed on a single line (the thicker one in the middle) or it can be broadened to include neighboring pixels (the complete highlighted range). The analysis of a broader area (bottom) shows the element distribution more clearly due to the better count statistics.

Selection of a sample area



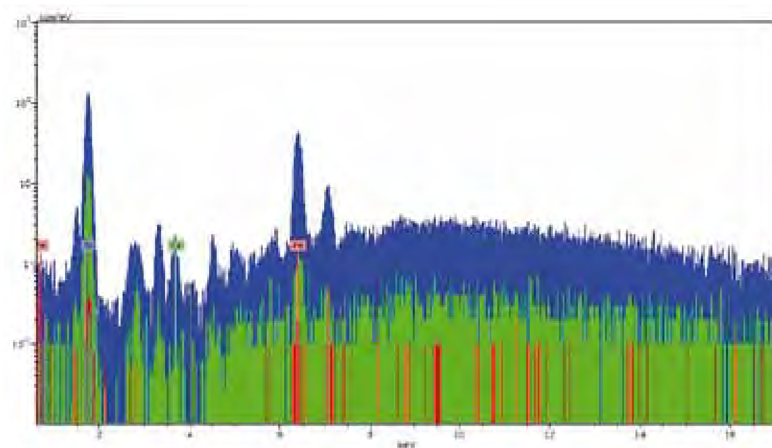


Fig. 6 Selected area of the mineral for accumulation of the point and area spectrum. The image on the right shows the spectra of a single point for 5 ms (red) and 200 ms (green) measurement time (absolute counts). The blue one is the accumulated spectrum of the highlighted sample area for 5 ms measurement time (counts per seconds).

Maximum Pixel Spectrum

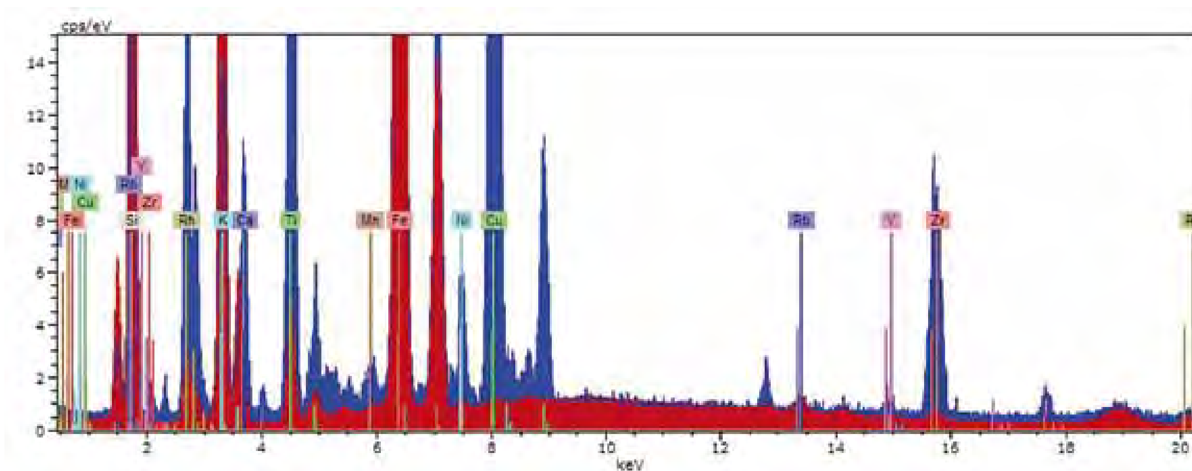


Fig. 7 Comparison of the sum spectrum (red) with the Maximum Pixel Spectrum (blue)

Conclusions

The analysis of a geological sample with μ-XRF offers a wide variety of information on the structure and composition of the sample. This includes the complete distribution of several elements, the identification and analysis of single grains and the execution of line scans. The high count rate capability and the excellent detector performance of the M4 TORNADO permit reliable data collection within a very short measurement time. The TurboSpeed stage allows fast mapping and measurement on-the-fly.

Even acquisition times in the low millisecond range can deliver first impressions of the structure of the material. Longer measurement times ensure better spatial resolution due to better statistics and the enhanced contrast between pixels. Longer measurement times also offer the possibility to examine traces.

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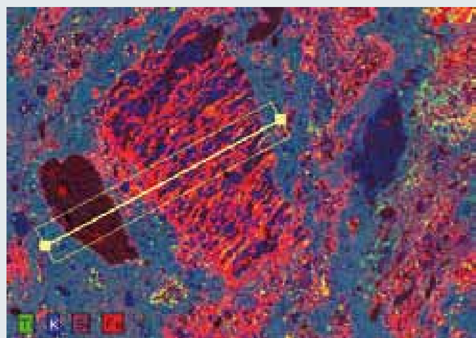
Calculation of the element distribution along a line



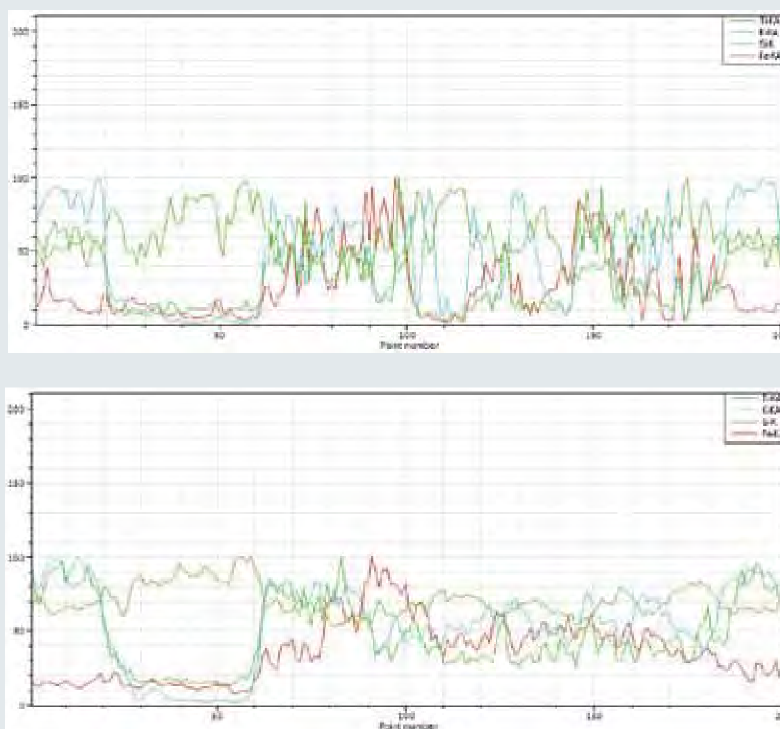


Fig. 8 Comparison of the line scan results when measuring only the center line (top) and the complete highlighted area (bottom)

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