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Elemental Distribution Analysis of a Meteorite Sample from the Rochechouart Structure with the µ-XRF M4 TORNADO

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

Micro X-ray fluorescence (µ-XRF) is well suited for these purposes. It offers great spatial resolution combined with small spot size and high sensitivity for element concentrations. This report describes the elemental examination of a meteorite sample from the Rochechouart structure in France. 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 by 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 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 form the earth's 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 components involved in the formation and the hydrothermal evolution of the impact melt.

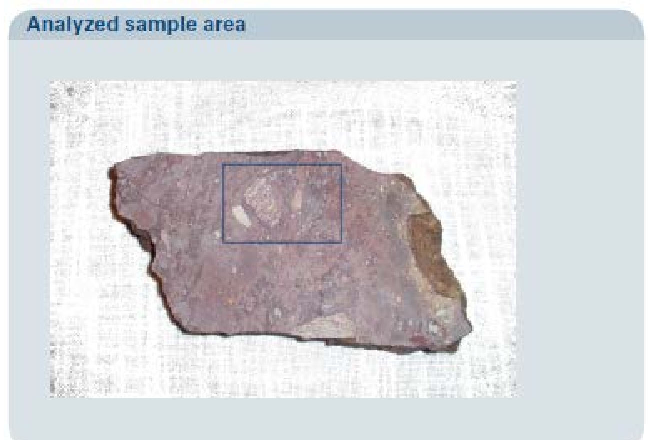

Figure 1: The highlighted sample area was measured with the M4 TORNADO.

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 mm³
- Fast X-Y-Z stage with TurboSpeed for fast mapping and distribution analysis on-the-fly

- Effective excitation of fluorescence through high brilliance X-ray tube together with X-ray optics for concentration of the 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 and HyperMap

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 large amount of time and makes the total measurement time almost identical to the sum of the pixel times. The M4 TORNADO uses Bruker's HyperMap technology in which a complete spectrum is saved for every pixel of the map. This offers a lot of advantages, in particular for the data evaluation.

- **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

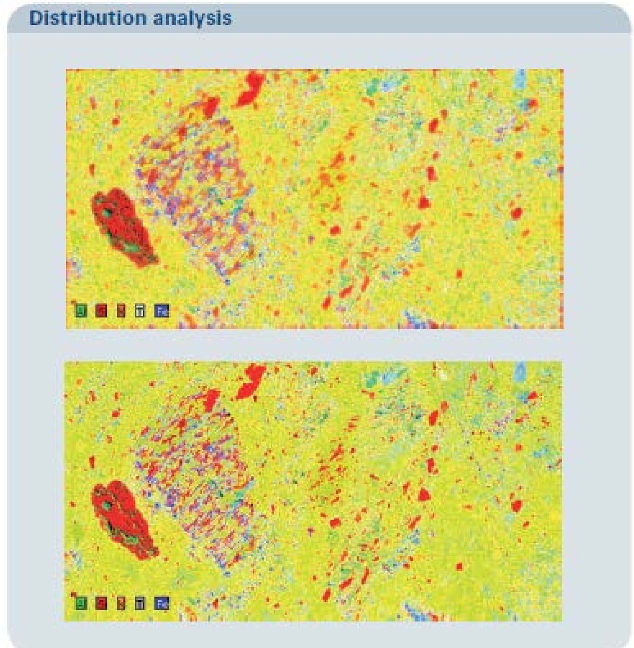

Figure 2: Results of a distribution analysis with an acquisition time per pixel of 5 ms (top) and 200 ms (bottom).

composition of the sample. The distribution of components of the mineral is shown in Figure 2.

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 3 shows the element distribution of Ni as a 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 more blurred and unclear. Element maps for each element present are displayed in Figure 4.

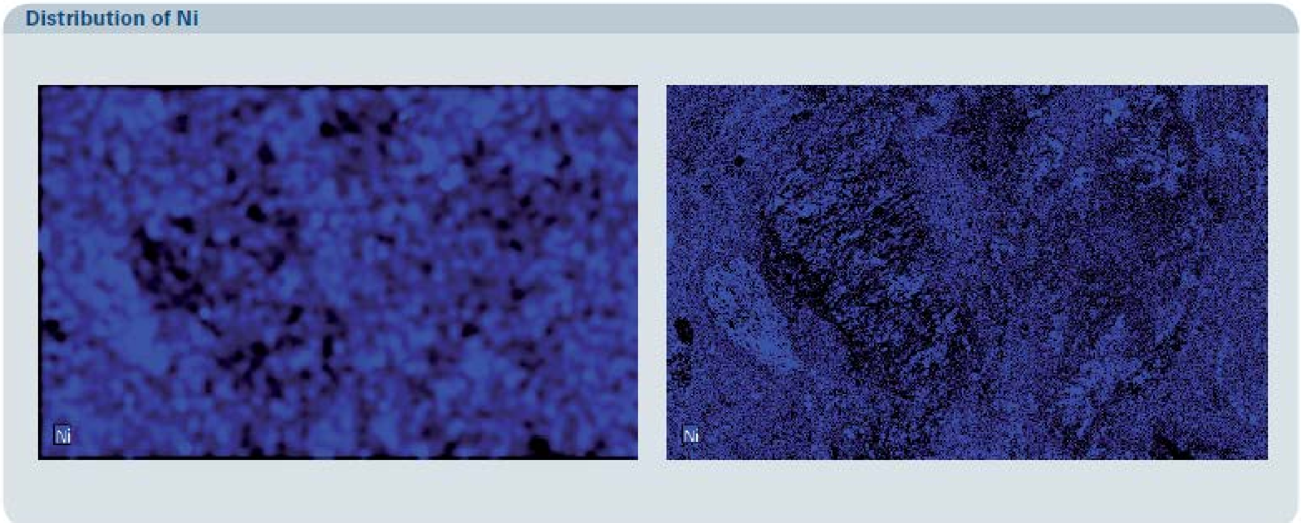

Figure 3: Distribution of Ni for 5 ms (left) and 200 ms (right) acquisition time per pixel.

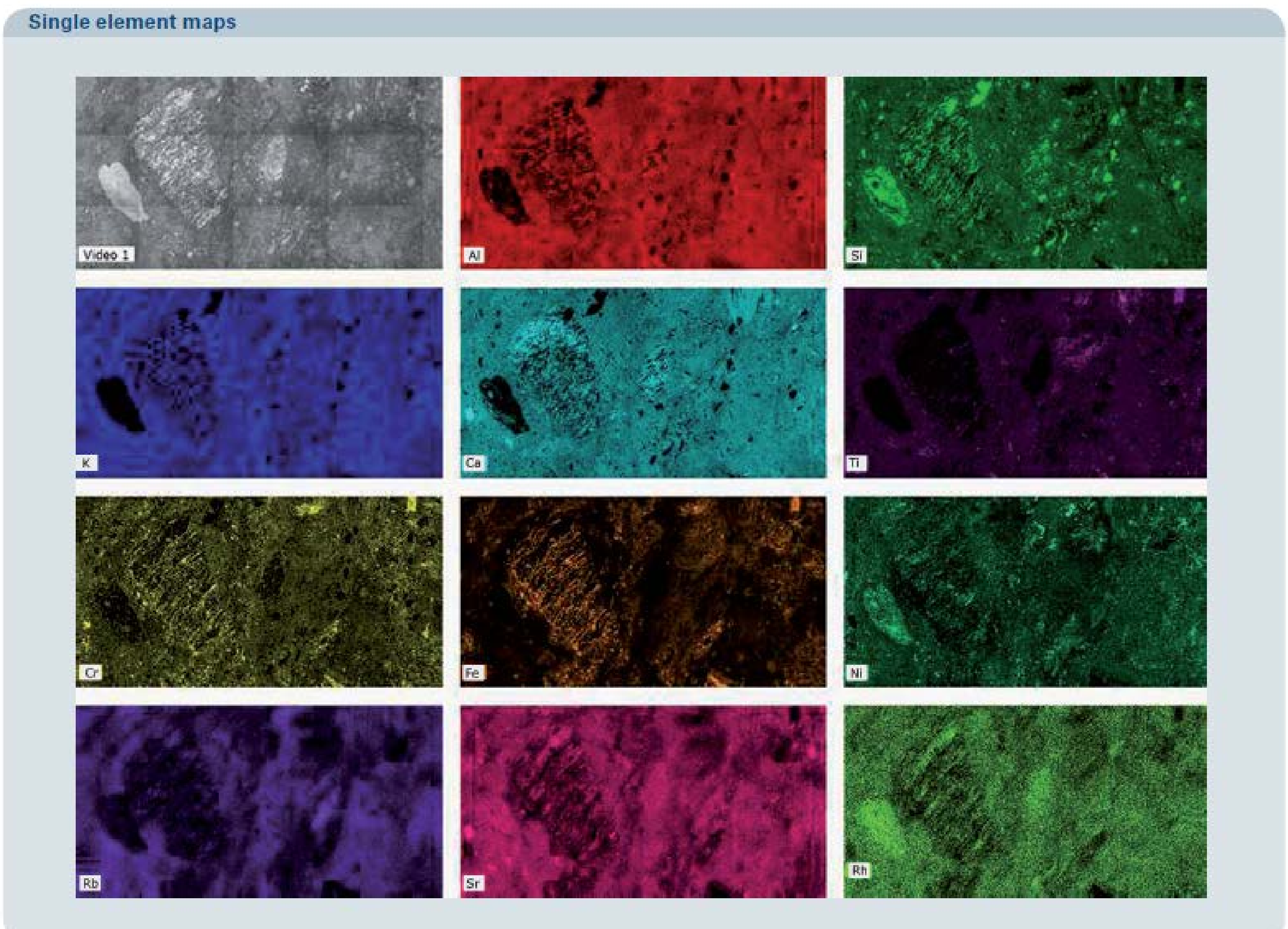

Figure 4: Single element distribution images of a series of elements calculated from the HyperMap at 200 ms acquisition time per pixel.

- **Sum spectra of complete mapping**

HyperMap also offers the possibility to accumulate a spectrum for every pixel and displays the sum spectrum of the complete mapping. Figure 5 shows the sum spectra of both mapping in a log 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 shows the result of a single pixel at 5 ms measurement time. This allows for the identification of main components, but no further evaluation. In Figure 6 the green spectrum is the result of measuring the same pixel for 200 ms. It has better statistics and allows for the identification of minor elements. 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 for quantification.

- **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 7 compares the 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.

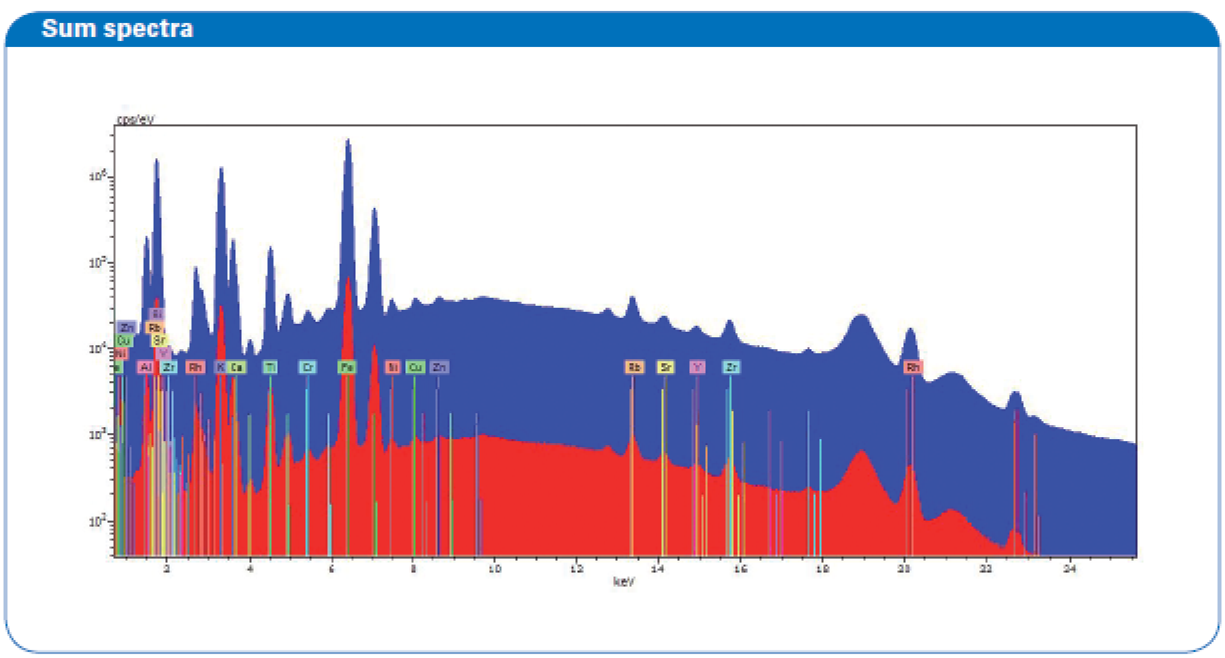

Figure 5: Sum spectrum of both mappings (log scale).

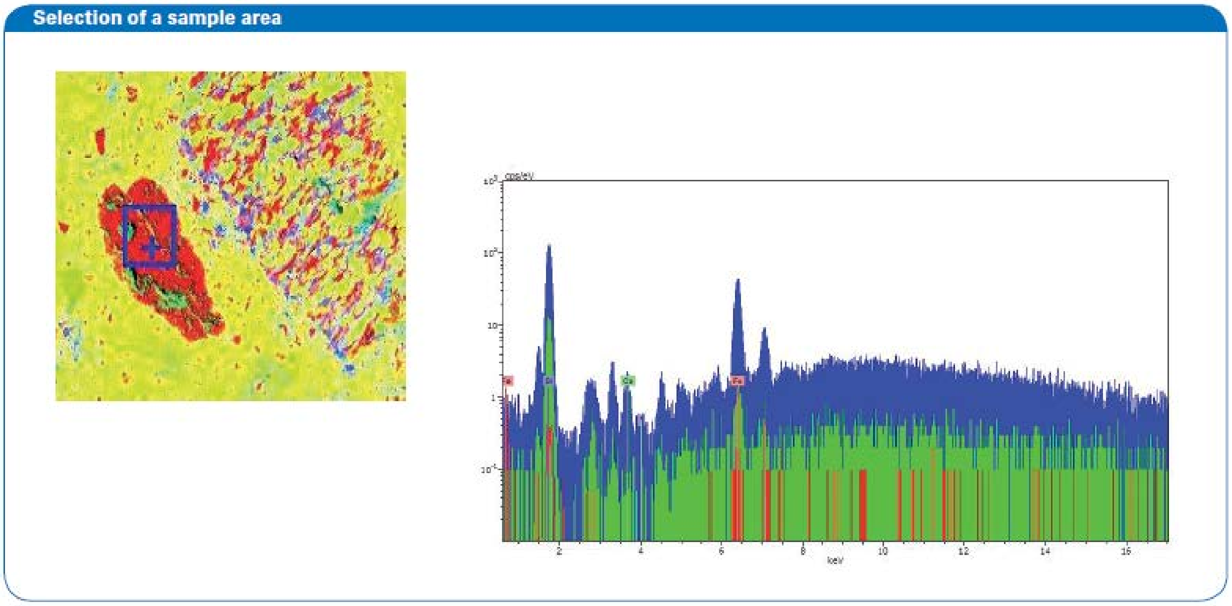

Figure 6: Selected area of the mineral for accumulation of the point and area spectrum. The image on the right shows the spectra of a single point for 5 ms (red) and 200 ms (green) measurement time (absolute counts). The blue one is the accumulated spectrum of the highlighted sample area for 5 ms measurement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

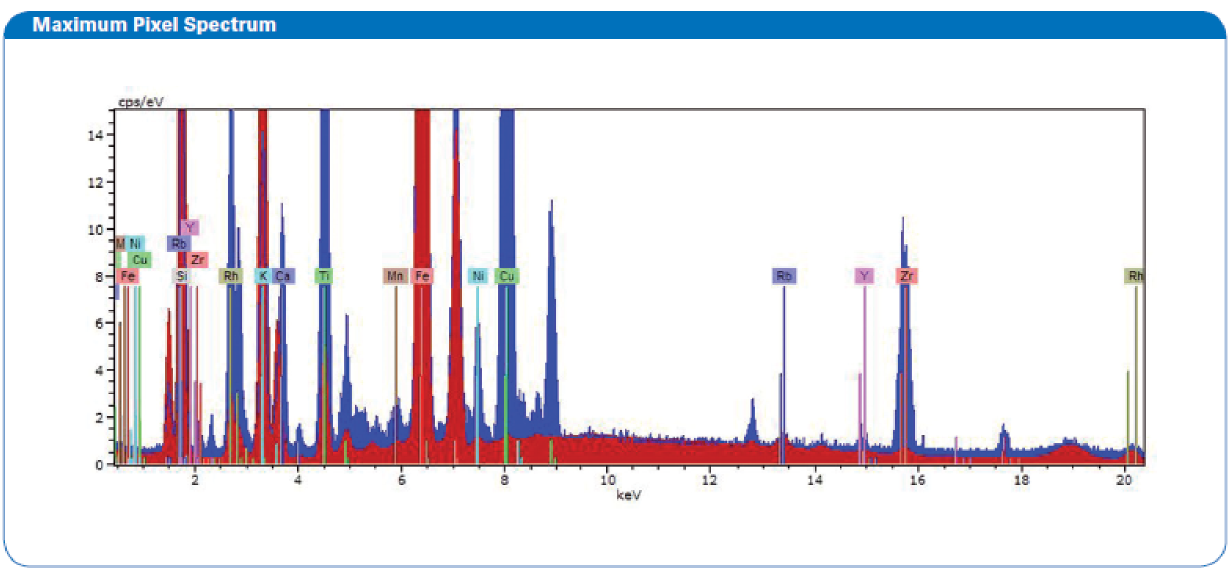

Figure 7: Comparison of the sum spectrum (red) with the Maximum Pixel Spectrum (blue).

Conclusion

The analysis of a geological sample with the M4 TORNADO offers a wide variety of information on the structure and composition of the sample. This includes the complete distribution of several elements and the identification and analysis of single grains. 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 sample. 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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