Lab Report XRF 146

S8 TIGER Series 2

- Analysis of the Elemental Distribution in the metallic Coarse Octahedrite Meteorite Canyon Diablo with the XRF^2 mapping of the S8 TIGER Series 2

The Barringer crater in Arizona (USA) was created about 50,000 years ago by the impact of the meteorite Canyon Diablo. Experts are estimating the weight of the meteorite to be about 300,000 t and with the diameter to be about 45 m. It has hit the earth with a speed of 15 – 30 km/s. This meteorite belongs to the class of iron-nickel objects originated from the asteroid belt. About 30 t of the initial mass have been found so far. The Canyon Diablo is scientifically one of the most important meteorites. Based on its material C.C. Patterson and F.G. Houtermans have dated the age of the Earth to be about 4.55 billion years.

Additionally H. Moissan has identified for the first time silicon carbide (Moissanite) for the first time in this meteorite, the second hardest material after diamond. Consisting mainly of Fe, Ni and traces of C, S, P, Ga and Ge the meteorite contains particular iron-nickel phases being present in this meteorite, which do not occur on earth. These phases are not distributed homogenously in this meteorite. With the XRF^2 elemental mapping of the S8 TIGER Series 2 the element distribution in the meteorite fragment and the distribution of these rare phases can be analyzed and visualized.

XRF

Instrumentation

The S8 TIGER Series 2 is a high power 4 kW wavelength dispersive X-ray fluorescence spectrometer with unrivalled analytical flexibility. The excitation of elements is done with a voltage ranging from 20 kV for the light elements up to 60 kV for the heaviest elements. The current can instantaneously be adjusted between 5 – 170 mA to detect trace elements and keep the major elements the count rate in the linear range of the detector. This DynaMatch technology is unique among this instrumentation. With up to four collimators and up to eight analyzer crystals the S8 TIGER Series 2 can provide either optimal resolution and/or high intensity for the analysis of fluorescence lines. With the new HighSense detector technology the intensities of element lines are detected; for light elements with the flow counter and for the heavy elements with the scintillation counter. Based on this advanced WDXRF technology the S8 TIGER Series 2 detects all elements from Be to U, covering the trace region up to 100%.

Figure 1: S8 TIGER Series 2 with the XRF^2 functionality for elemental mapping analysis

To analyze the element distribution in samples or very small sample amounts down to a single particle the S8 TIGER Series 2 can be equipped with the XRF^2 mapping tool . The beam is collimated with the HighSense mask down to either 1.2 mm or even down to 300 µm spot size (FWHM). The surface is than scanned with a step size of 100 µm, providing the best available spatial resolution for this kind of instrumentation. The optimal detection of light and heavy elements is done based on WDXRF technology providing a much better intensity compared to EDXRF for light and heavy elements and maintaining the excellent spectral resolution of WDXRF. This is especially important for the investigation of the very thin iron-nickel phases in this meteorite. Based on the high intensity setup of the S8 TIGER Series 2 even the detection of traces and rare elements is possible.

Sample and Measurement

When loading the sample the S8 TIGER automatically takes a high definition picture of the sample mounted in the holder. The meteorite fragment is shown in figure 2, the high resolution picture in figure 3. For the measurements now the spots, lines, areas or the entire sample surface can be selected by the mouse "drag and drop". After the definition of spot size, step size and measurement parameters the instrument autonomously scans the sample.

Figure 2: Meteorite samples "Canyon Diablo" with inclusion

Figure 3: High Res picture taken from the sample in the S8 TIGER Series 2

Results

This meteorite sample from Canyon Diablo is very interesting when looking at the elemental distribution. We have found a grey semi-metallic inclusion with a diameter of about 8 mm. The element maps of Fe, Ni, Cr and S shows the absence of iron and nickel while chromium and sulphur are only occurring in this inclusion, see figure 4 and 5. When the liquid metal system Ni – Fe – Cr is cooling down the presence of S leads to an eutectic Cr – S compound which is no longer soluble in the metal. The Sulphur led for the meteorite to the reduction of the chromium concentration in the metallic part. The same process is found in welding, where the presence of sulphur leads to segregation of the molten metal. The non-metallic sulphur inclusions will finally lead to micro cracks along the welding joint. This would be an example of daily use beside cosmic science: The analysis of similar samples from metal parts with the S8 TIGER Series 2 and XRF^2 to evaluate the performance of the material in case of welding.

Another element map is very interesting: The element distribution of nickel in the metallic part of the meteorite shows fine lines of higher concentrations next to fine structures of lines with lower amounts of nickel, shown in figure 6. This phenomena has been found in meteorites the first time by A. Widmannstätten when doing leaching experiments with meteorites (see insert on picture 6) The structure of these fine lines is caused by the difference in resistance to edging of the iron-nickel phases kamacite and taenite. While the nickel rich taenite is less affected by the acid, the kamacite is slightly dissolved by the acid. In this kind of material these structures are only created under cosmic conditions and cannot be reproduced on Earth. Similar structures on Earth are seen for low carbon steels under specific conditions e.g. by welding. With the S8 TIGER Series 2 and XRF^2 mapping this fine structure can be identified even without leaching and destroying the sample based on the very small spot size of 300 µm.

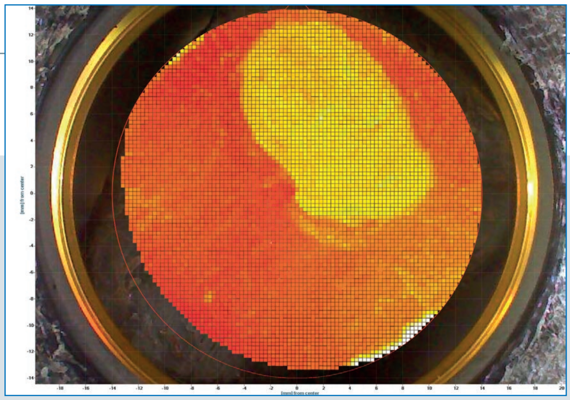


Figure 3: Element map of iron analyzed with the S8 TIGER Series 2

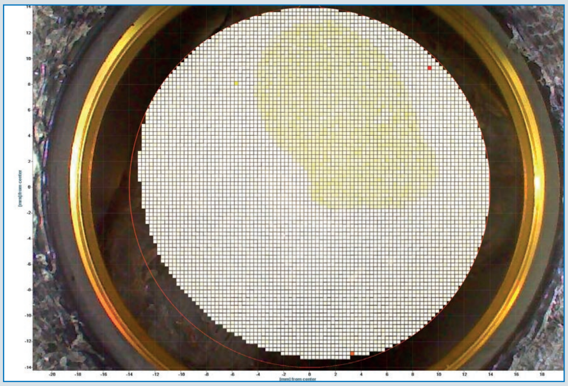


Figure 4: Element map of chromium analyzed with the S8 TIGER Series 2

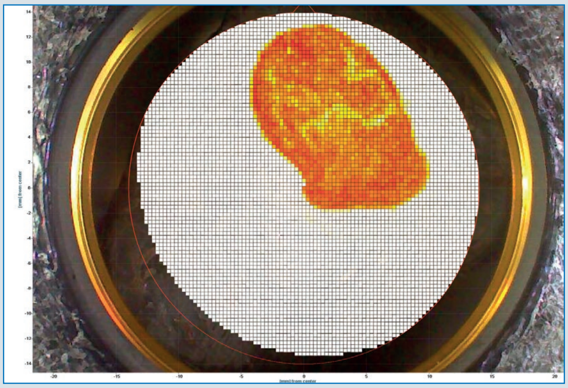
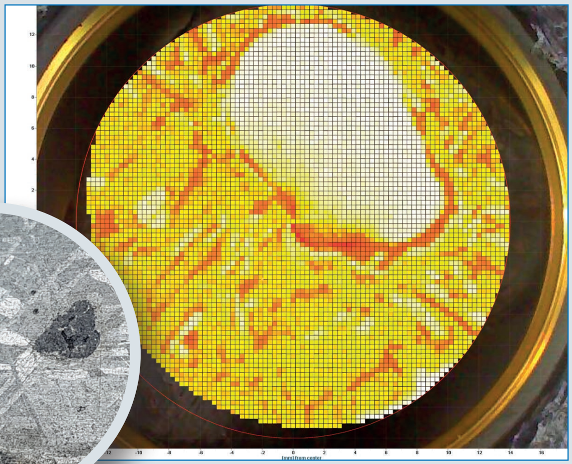


Figure 5: Element map of sulphur analyzed with the S8 TIGER Series 2



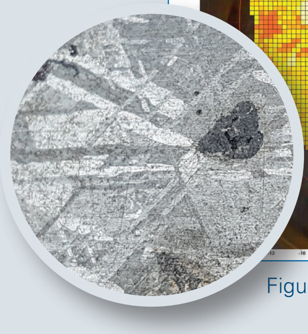


Figure 6: Element map of nickel analyzed with the S8 TIGER Series 2

The bulk analysis of the metallic part of the meteorite sample has shown a concentration of about 300 ppm germanium. In contrast to all other elements germanium seems to be homogeneously distributed in a 'spotted' kind of pattern across the whole meteorite, even in the non-metallic chromium-sulphur inclusion, see figure 7. The S8 TIGER Series 2 is able to provide even distribution information on traces with low intensities.

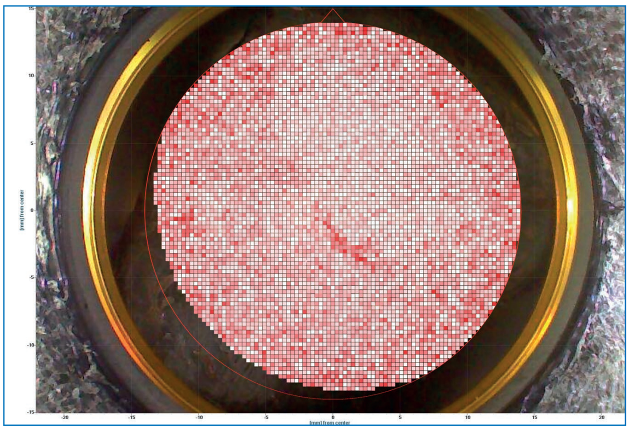


Figure 7: Element map of germanium analyzed with the S8 TIGER Series 2

Conclusion

The analysis of the meteorite sample "Canyon Diablo" with the S8 TIGER Series 2 and XRF^2 has shown very interesting results. With the very high local resolution of 300 µm (Spotsize FWHM) and the accurate step size of 100 µm even very fine structures could be shown. This was only possible due to the high sensitivity of the HighSense beam path of the S8 TIGER Series 2 making even small differences in element concentration visible. The high sensitivity for light elements helped to recognize specific material properties, while the high spectral resolution in combination with the high power excitation of the WDXRF based mapping enabled the analysis of traces of germanium in these small spots. The power and outstanding performance of the XRF^2 mapping of the S8 TIGER Series 2 is demonstrated with the analysis of traces of germanium with a spot size of 300 µm in the presence of high iron and nickel concentrations: The HighSense beam path is providing significantly higher sensitivity than any other WDXRF system while maintaining the high resolution of WDXRF.

The results of the meteorite sample shows the value of the S8 TIGER Series 2 as vital analytical tool not only for astrophysics but also for material research. Material properties can be analyzed based on element mapping with almost no sample preparation. This makes the S8 TIGER Series 2 with XRF^2 mapping an interesting addition to any material science department or in industrial R&D.

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