Lab Report XRF 147

S8 TIGER Series 2

- Analysis of the Elemental Distribution in Fossilized Ammonite shell for Paleontology with the XRF^2 Mapping of the S8 TIGER Series 2

Ammonoids are an extinct group of marine lifeforms related to living animals, such as octopuses, squids, cuttlefish and even closer to living nautiloids. Starting from the Devonian period ammonoids were widely distributed until their extinction. Ammonite fossils are perfect indicators for indexing geological time periods, since the group is accurately characterized and the evolution of this species over time is well understood.

In most of the fossils only inorganic parts are preserved. In animal shells and bones the elemental distribution indicates the metabolism and lets researchers closely understand how the lifeform has developed and how environmental conditions have influenced the development. To demonstrate the quality of the data achieved from the S8 TIGER Series 2 XRF^2 mapping tool we have analyzed an early form of ammonite; the analyzed surface was a polished cross section.

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 for major elements the count rate in the linear range of the detector. Uniquely for this kind of instrumentation this switch happens momentarily with our DynaMatch technology. With up to four collimators and up to eight analyzer crystals the S8 TIGER Series 2 provides optimal resolution and 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 Am, covering the trace region up to 100%.

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

To map the element distribution in samples or analyzing 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 this investigation to map the element distribution in the minute shells of this fossil. Based on the high intensity setup of the S8 TIGER Series 2 even the detection of very light elements 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 ammonite is shown in figure 2, the high resolution picture in figure 3. For the measurements spots, lines, areas or the entire sample surface can be selected with the mouse by "drag and drop". After the definition of spot size, step size and measurement parameters the instrument autonomously scans the sample.

Figure 2: Ammonite fossil sample

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

Results

This ammonite sample shows an interesting element distribution which gives an indication about the body structure and what has happened during the fossilization. The calcium concentration correlates with the brown chamber fillings, while silicon shows a correlation with the dark grey filling at the center and entrance of the ammonite shell. Here we also find iron with its highest concentration at the shell entrance – a possible indicator for later influence from sediments. More interesting is that strontium is only found in the shell – enriched during growth of the animal due to its metabolism.

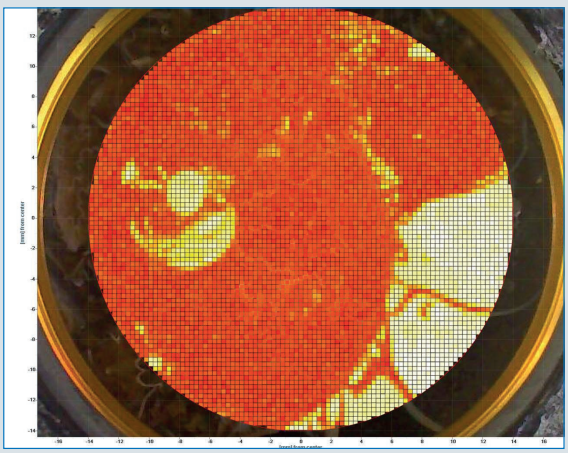

Figure 4: Element map of calcium analyzed with the S8 TIGER Series 2

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

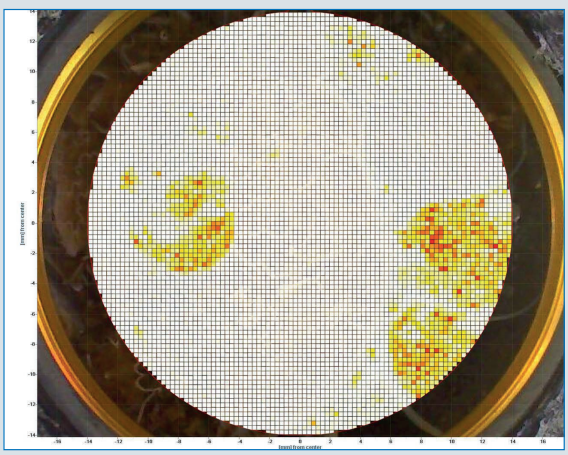

Figure 5: Element map of silicon analyzed with the S8 TIGER Series 2

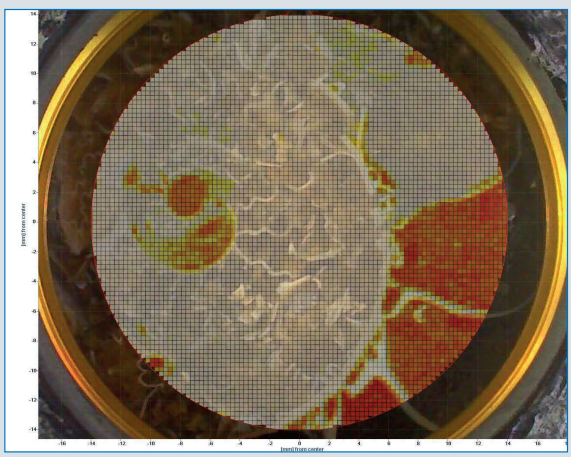

Figure 7: Element map of iron analyzed with the S8 TIGER Series 2

Conclusion

The elemental mapping of the ammonite fossil with the S8 TIGER Series 2 and XRF^2 has helped to investigate the influence of metabolism on shell biochemistry. With the very high local resolution of 300 µm (Spotsize FWHM) and the high accurate step size of 100 µm even the finest 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 concentrations visible. The high sensitivity for light elements gives clear indications about the mineralization process, while the high spectral resolution in combination with the high power excitation of the WDXRF based mapping enabled the analysis of traces of strontium in the thin shell. The power and outstanding performance of the XRF2 mapping of the S8 TIGER Series 2 is demonstrated with the analysis of traces of strontium with a spot size of 300 µm: The HighSense beam path are providing significantly high sensitivity than any other WDXRF system and maintaining the high resolution of WDXRF.

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