



MICRO-XRF ON SEM

Analysis of mineral wool

Application Note # MXRF-SEM-01

Introduction

Mineral wool is mainly used for thermal insulation of building parts and of pipelines, as well as for sound absorbers. Other names for the material are rock wool, stone wool or man-made vitreous fibers (MMVF). As these names indicate, it consists of tiny mineral fibers with typical diameters between 6 and 10 μm . It is produced by spinning molten rock at about 1600°C. In order to obtain the final shape of the mineral wool product, an appropriate organic bonding agent is added to the fiber network.

The investigation of mineral wool using micro-X-ray fluorescence spectroscopy (micro-XRF) in the scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS) is especially promising through the combination of trace element and light element sensitivity with spatial resolution. This is demonstrated in the following example.

Figure 1 shows a photograph of the two investigated mineral wool specimens from different manufacturers.



Figure 1

Photograph of the two analyzed mineral wool specimens

Measurement conditions

Measurements were performed with the micro-focus X-ray source XTrace mounted on a FE-SEM. XTrace is equipped with a Rh-target X-ray tube and with an X-ray optics focussing the X-rays onto a spot of about 50 μm diameter on the specimen. X-ray tube operation conditions were 40 kV high voltage and 600 μA beam current.

Only a few fibers of each sample were fixed on a thin polyester foil. Mounting small and thin specimens on thin foils reduces the spectrum background considerably and improves the detection limit. Figure 2 shows the SEM specimen holder with a few fibers on the foil.

Results

Figure 3 displays the SEM image of fibers taken from the specimen on the left side in Figure 1. The SEM image of the specimen in Figure 1 right is very similar, i.e. both specimens are very difficult to discriminate only by their SEM images.

The XRF spectra clearly reveal the difference in composition of the two investigated mineral wools. Especially significant is the difference in the titanium content. The sulfur line is caused by the glue on the foil.

The spectra in Figure 4 were measured with the X-ray source focused on fiber bundles as shown in Figure 3, with a count rate of approx. 2 kcps.



Figure 2

Mineral wool fibers fixed on polyester foil. The chamber scope image also shows the position of the XTrace's polycapillary optics (right of the microscope pole piece) and the EDS detector (left of the pole piece).

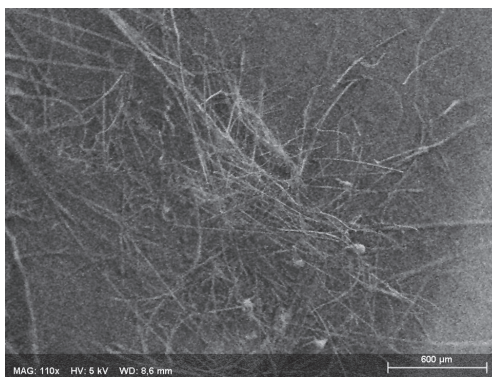


Figure 3

SEM image of the left mineral wool specimen in Figure 1.

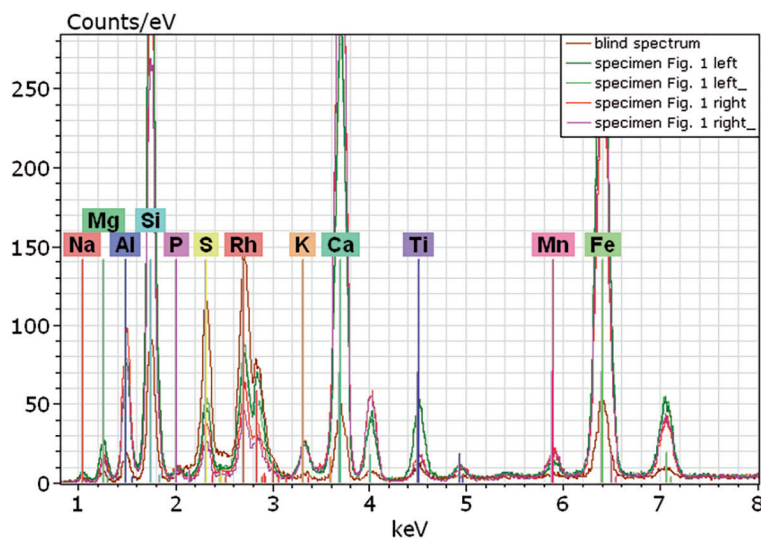


Figure 4

XRF spectra of the two specimens (red and green) together with a blank spectrum from the foil (brown). Rh L is caused by the scattered tube radiation.

Single fibers can also be analyzed. Using the effect that the foil is charged by the X-ray beam, which results in a dark spot in the SEM image, an individual fiber can be selected simply by moving the specimen stage while observing this spot. Figure 5 shows a long fiber on the foil under the dark spot, Figure 6 the SE image of that single fiber at higher magnification and finally Figure 7 the comparison of EDS and XRF spectra of the fiber.

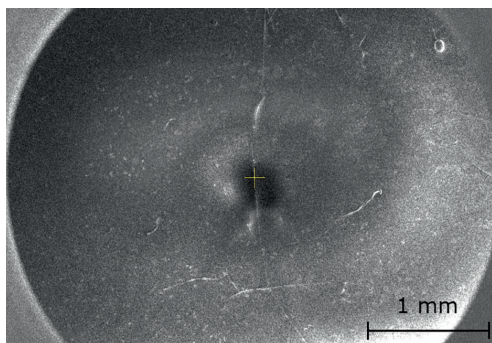


Figure 5

Secondary electron (SE) image at low magnification and low voltage of 2 kV, showing a long fiber. The dark spot in the center of the image is caused by the X-ray beam.

The EDS spectrum in Figure 7 was obtained at 20 kV SEM voltage, for better comparison of higher energy peaks. As it can be seen from the relative peak heights, XRF excitation is more efficient beginning already with the relatively light element Si. Elements K, Ti and Mn are not visible in the EDS spectrum at all, the Ca and Fe peaks are very low.

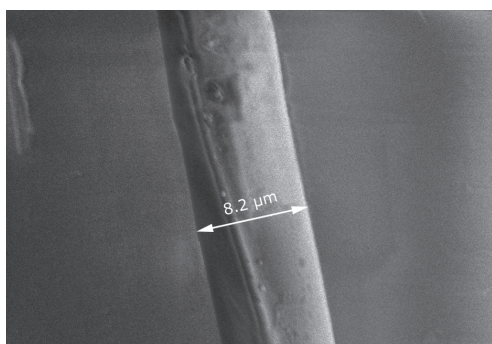


Figure 6

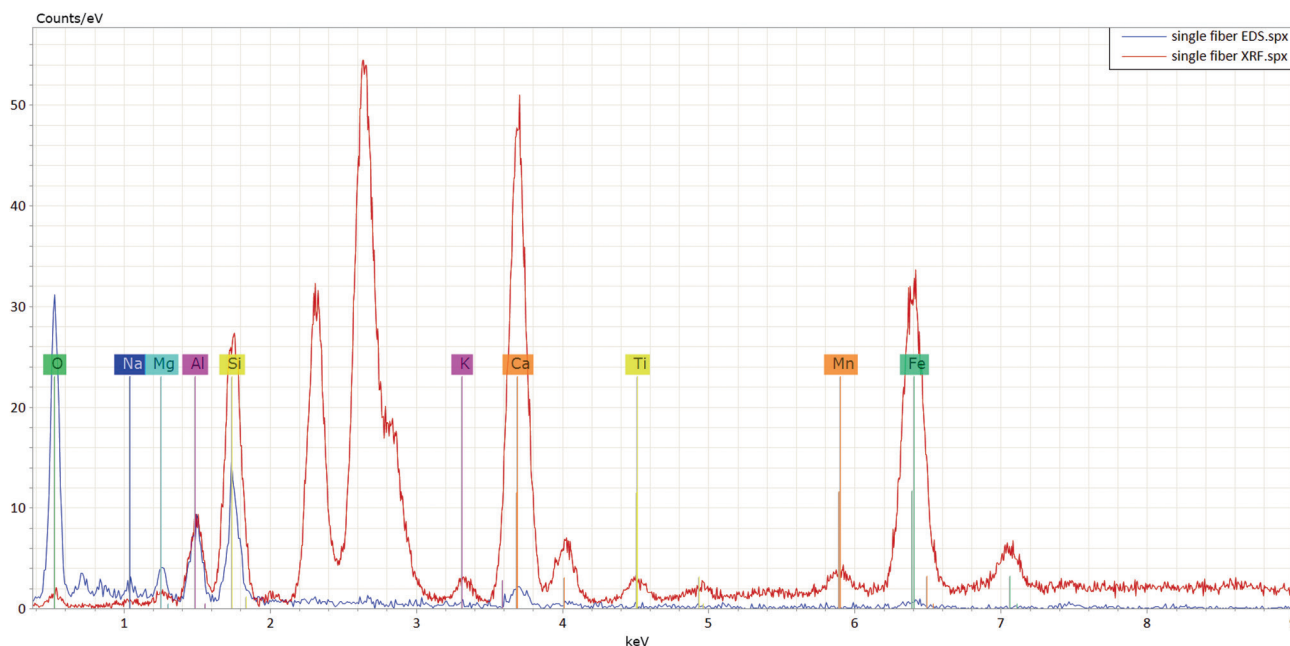
SE image of a single fiber at 2 kV beam voltage and high magnification.

With a 30 mm² XFlash[®] 6 SDD mounted on the SEM the count rates collected from a single fiber were more than sufficient to acquire a line scan across the fiber using the unique stage line scan option of the ESPRIT software.

This line scan is shown in Figure 8. Of course, a considerable broadening is observed giving an impression for the spatial resolution that can be currently achieved with the microfocus X-ray source.

Figure 7

Comparison of XRF (red) and EDS (blue) spectrum of the fiber shown in Figure 5. The peaks not labeled in the red spectrum are spurious peaks, S from the foil and a broad peak caused by scattered tube radiation.



Conclusion

The combination of EDS and micro-XRF to investigate fiber structure and composition of mineral wool has turned out to be a very useful method. Due to the excellent spatial resolution and the sensitivity it was easy to distinguish the mineral wool specimens by their chemistry, although their optical appearance is almost the same. A discriminator is – for instance – the height difference of their Ti peaks.

This discrimination would not have not been possible with EDS alone. A proper specimen mounting, in this case fixing fibers on a few micron thick polyester foils, to attain low background and high detection sensitivity, is very important for such investigations. The same mounting method can be applied as well to acquire individual X-ray spectra of other small particles.

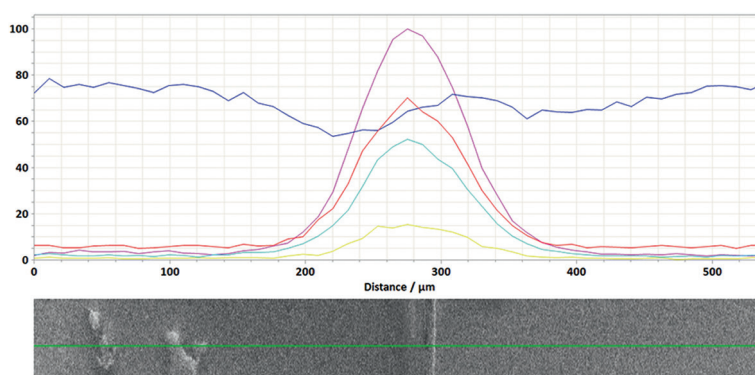
The radiation power (photon flux) of the used X-ray source together with the high collection efficiency of a large area SDD even enable the measurement of an X-ray spectrum of a single fiber, even though the spot size is around 50 μm in this case. A smaller spot

size with the inevitable drawback of reduced photon flux is not necessary to collect spectra of individual or sufficiently separated objects.

Additionally, the superior sensitivity for mid-range to heavy elements of XRF invites to analyze the specimens in detail beyond the scope of this application note. That is, to determine the sum of oxide concentrations Na_2O , K_2O , CaO , MgO and BaO , as prescribed by the European Commission Directive 97/69/EC. his directive determines the maximum content of these oxides for the fibers to be considered nonhazardous.

Figure 8

XRF line scan for Ca (purple), Fe (red), Si (light blue), Ti (yellow) and S (dark blue).



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