MICRO-XRF

Combining Micro-XRF and Micro-CT: Mineral Measurements of Drill Cores

Application Note # XRF 467

Introduction

In mining as well as oil and gas industry, drilling is one of the most important steps in the exploration phase. The analysis of drill cores during exploration helps understanding the geology, mineralogy or petrology of the host rock. There are many methods and procedures to do this, each with their own (sample preparation) requirements and limitations.

Two such methods used in geology, which happen to require limited or no preparation, are micro X-ray fluorescence spectrometry (micro-XRF) and micro computed tomography (micro-CT). Micro-XRF produces 2D element or even mineral distribution maps down to the tens of micron scale, while micro-CT provides 3D reconstruction of material densities down to sub-micrometer scales. 3D element or mineral representation cannot be achieved by any of the methods alone, but with the combination of both.

This lab report describes the analysis of a sectioned drill core to determine the information that both methods combined can generate.

Functional principle

In micro-XRF, the focused X-ray beam penetrates the material and excites a small volume of material causing secondary characteristic X-rays to be produced. This fluorescence in turn can leave the sample and can be detected by an energy dispersive detector.

With micro-CT, the sample is irradiated with high energy X-rays. With a digital image plate, which is sensitive to X-rays and placed on the opposing side of the sample, a transmission image or radiograph is created. Its magnification can be controlled by changing the sourcesample-detector distance, i.e. moving the sample closer to the source magnifies the image but also reduces the field of view. The sample is then rotated over many hundreds or thousands of steps, and the resulting images are combined to reconstruct a 3D image of the sample.

Sample

The sample, shown in Figure 1, was a piece of a sectioned drill core (100 mm x 35 mm), containing iron oxides and iron sulfides as well as quartz and other minerals such as olivine and albite. The sectioned surface was polished previously for light microscopy analysis, but no further sample preparation was required.

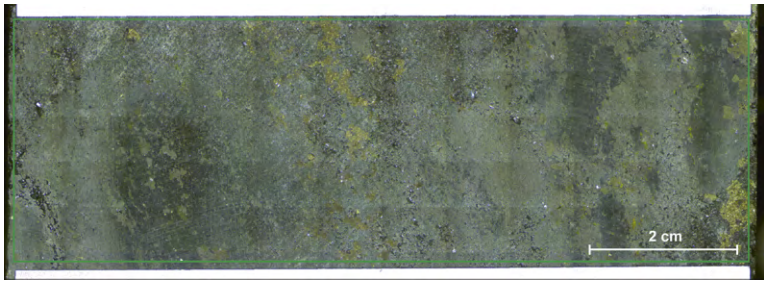

Figure 1
M4 TORNADO mosaic image of a drill core sample with defined map area.

Micro-XRF measurement conditions

The measurement was initially performed with a Bruker M4 TORNADO^{AMICS} equipped with a Rh tube, a polycapillary lens and two 30 mm² XFlash® silicon drift detectors (SDD). The following standard measurement conditions were used:

- tube voltage of 50 kV
- current of 200 µA
- no primary beam filter
- chamber pressure of 20 mbar.

The measurement was carried out with a step size of 50 µm and dwell time of 30 ms per pixel over an area of 2000x660 pixels. The total measurement time was 10 hours and 55 minutes.

Micro-CT measurement conditions

The large sample chamber and the long vertical travel of Bruker's SKYSCAN 2211 allows scanning of the entire length of the drill core at a relatively high resolution. This ensures optimal matching with the micro-XRF measurements, as the same area or volume is analyzed using both technologies.

The micro-CT analyses were performed using the micro-CT system SKYSCAN 2211 equipped with an open transmission X-ray tube with a tungsten target. The 1920x1536 pixel flat panel detector was used for this measurement under these conditions:

- tube voltage of 180 kV
- exposure time of 775 ms (4 frames)
- rotation step of 0.1 degrees (3600 projections)
- 0.5 mm Mo filter.

Due to the size of the drill core, four separate scans were vertically stitched together. The resulting pixel size was 27 µm with a total acquisition time of 12 hours.

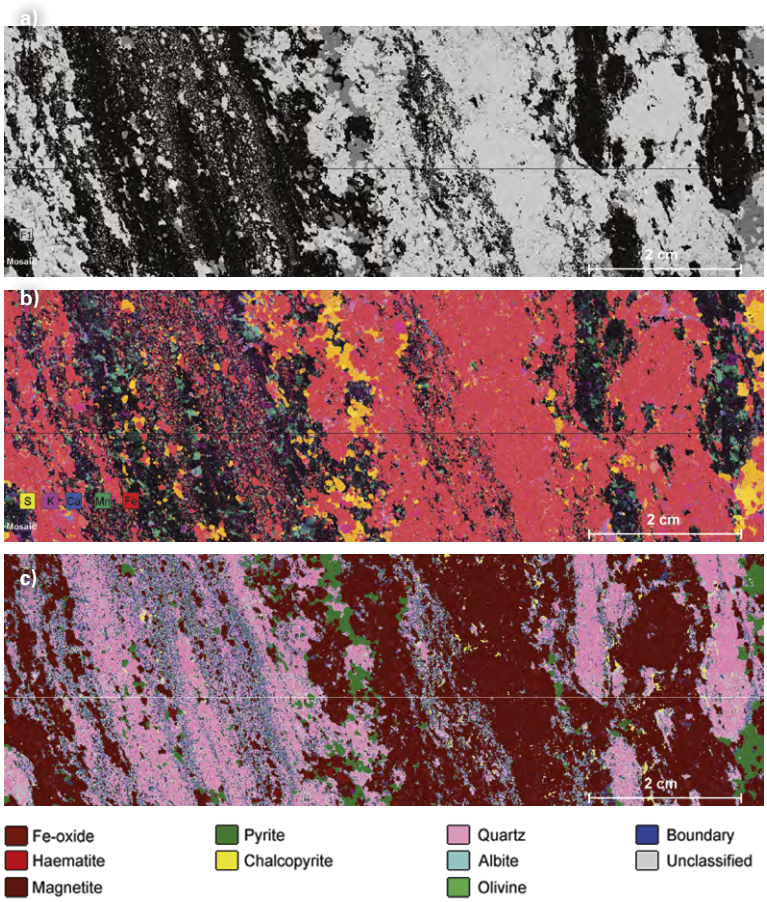

Figure 2
a) M4 TORNADO X-ray intensity map showing a material contrast similar to BSE in SEM or micro-CT,
b) element maps of sulfur, potassium, calcium, manganese and iron,
c) AMICS mineral map showing iron oxides, iron sulfides and silicate minerals.

Results

Micro-XRF results

Figure 2a shows the X-ray intensity maps and Figure 2b displays the element map for sulfur, potassium, calcium, manganese, and iron.

Mineral phases can be inferred but are not definite, hence, making many minerals such as sulfides indistinguishable.

The mineral map in Figure 2c was acquired with the AMICS software and reveals that a large part of the sample is composed of iron-rich minerals (red, green and yellow colors) including iron oxides such as hematite (Fe_2O_3) and magnetite (Fe_3O_4) and iron sulfides such as pyrite (FeS_2) and chalcopyrite ($CuFeS_2$). In pink and light blue, silicate minerals such as quartz (SiO_2), olivine $(Mg,Fe)_2SiO_4$ and albite ($NaAlSi_3O_8$) are identified. False positives such as garnet (mainly almandine $Fe_3Al_2(SiO_4)_3$ are an artefact due to phase boundaries.

Micro-CT results

The resulting micro-CT data are gray value images (Figure 3) where the entire gray value range is a relative distribution between the linear attenuation coefficients of the sample's content (Figure 4). This linear attenuation coefficient mainly depends on the atomic number of the elements present in the sample.

Low gray values (dark in the image) represent a low atomic number while high gray values (bright in the image) represent high atomic numbers. It is important to know that this gray value distribution is relative and by no means absolute. No mineral identification can be done without prior knowledge on the sample's composition. In the case of the analyzed drill core, the two groups of material phases can be clearly identified in Figure 3. Based on the known composition of the minerals from the micro-XRF analyses, it can be deduced that the dark areas are silicate minerals, while the bright areas are iron-rich or sulfide minerals.

From this information the theoretical attenuation coefficient was calculated using the *muCalcTool* (developed by the University of Texas, Austin, USA; online available). As the sample was scanned using an accelerating voltage of 180 keV, the average energy of the X-ray beam lies around 80–100 keV. Figure 4 shows the distribution of the attenuation coefficients of the identified minerals in the sample in the range from 65 to 115 keV.

The iron oxides have the highest attenuation coefficients and can be seen as the brightest phase in Figure 5. As the attenuation lines of hematite and magnetite are overlapping, no contrast between the two iron oxides could be seen using micro-CT.

The iron sulfides pyrite and chalcopyrite have slightly lower attenuation coefficients than the iron oxides and appear in a darker gray. Due to the general overlap of their attenuation lines no contrast between the two iron sulfide phases is visible.

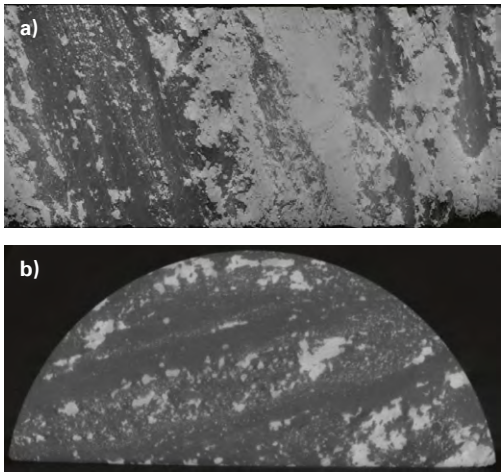

Figure 3
Scans with the SKYSCAN 2211:
a) Vertical cross-section through the micro-CT dataset close to the drill core's surface;
b) horizontal cross-section through the drill core.

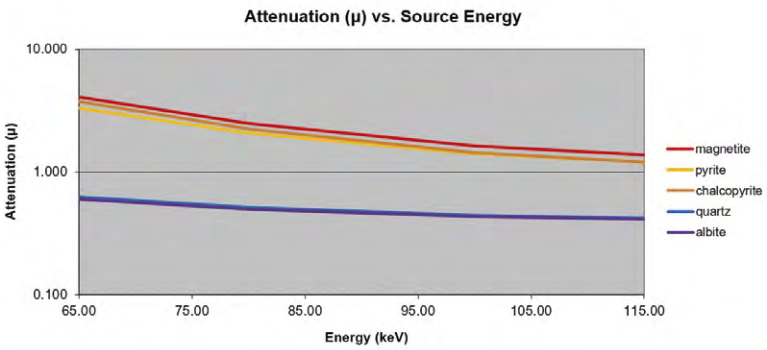

Figure 4
Calculation of the attenuation coefficients of the identified minerals. Albite and quartz have low attenuation coefficients, the coefficients of the iron-rich minerals are significantly higher.

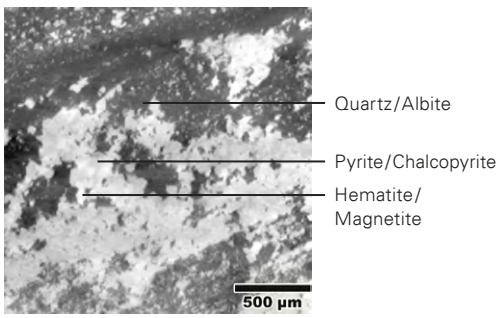

Figure 5
Detail of the micro-CT dataset after post-processing showing the very slight contrast within the iron-rich and the silicate minerals.

Finally, the silicate minerals quartz and albite have the lowest attenuation coefficients of the identified minerals and appear dark as can be seen in Figure 5.

Knowing the attenuation coefficients it is possible to draw some conclusions about the 3D distribution of minerals using only the CT images. Silicate minerals (blue) and iron minerals (green) can be identified as two separate groups as segmented and visualized in 3D (Figure 6a). Parts of the silicate phase have been virtually cut away to show the iron structure. Within the iron-rich minerals, there is some contrast present between hematite/magnetite and pyrite/chalcopyrite as can be seen in Figure 5, but this contrast is too low for a reliable quantitative 3D analysis.

Figure 6b shows the overlay and correlation of the micro-XRF and micro-CT data for the most important mineral phases in the drill core. The micro-XRF mineral contrast is far more detailed than micro-CT. The 3D combination of a 2D micro- XRF map and 3D micro-CT volume render of iron-rich and silicate minerals as depicted in Figure 6c displays the good correlation between the mineral groups, showing that the two analysis methods can be used to transpose mineral data from 2D to 3D.

The same analysis procedure on a slightly smaller sample would probably provide better results, as this would allow for better transmission through the sample and subsequently better image quality.

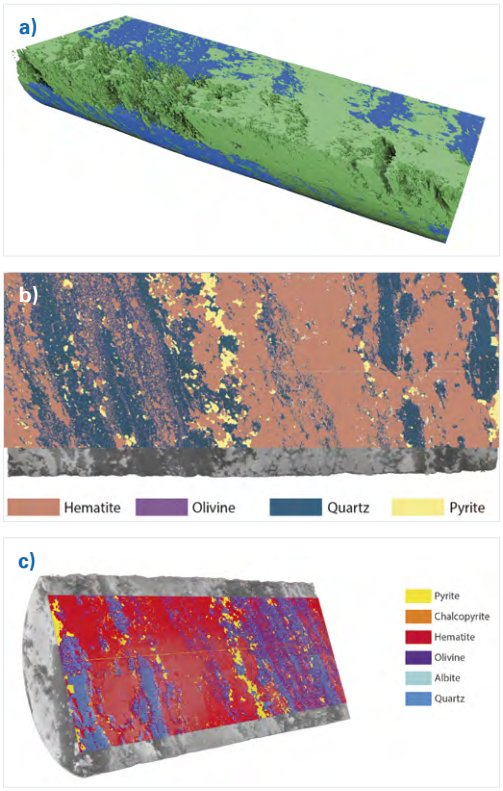

Figure 6
a) Volume render of the two mineral phases inside the sample, (blue = silicates, green = iron-rich minerals), b) Overlay of the micro-XRF dataset on a gray value rendering with the most important minerals, c) 3D combination of a 2D micro-XRF map and a 3D CT volume render of iron-rich minerals and silicate minerals.

Conclusion

Combining micro-CT and micro-XRF provides additional information to both imaging techniques. Micro-CT is very good at analyzing large samples and providing 3D structural information, but lacks the ability of exact identification of elements and minerals. Micro-XRF can provide mineral maps with high accuracy, but is a snapshot of the sample's surface. A combination of both methods gives a much greater insight in a sample's composition and the distribution of different minerals.

In cases where the X-ray attenuation contrast between different minerals is very low, micro-XRF can determine the exact composition of a certain X-ray CT gray value range. The micro-CT dataset can then be used to evaluate the consistency of different layers throughout the sample, and evaluate the content of different minerals.

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