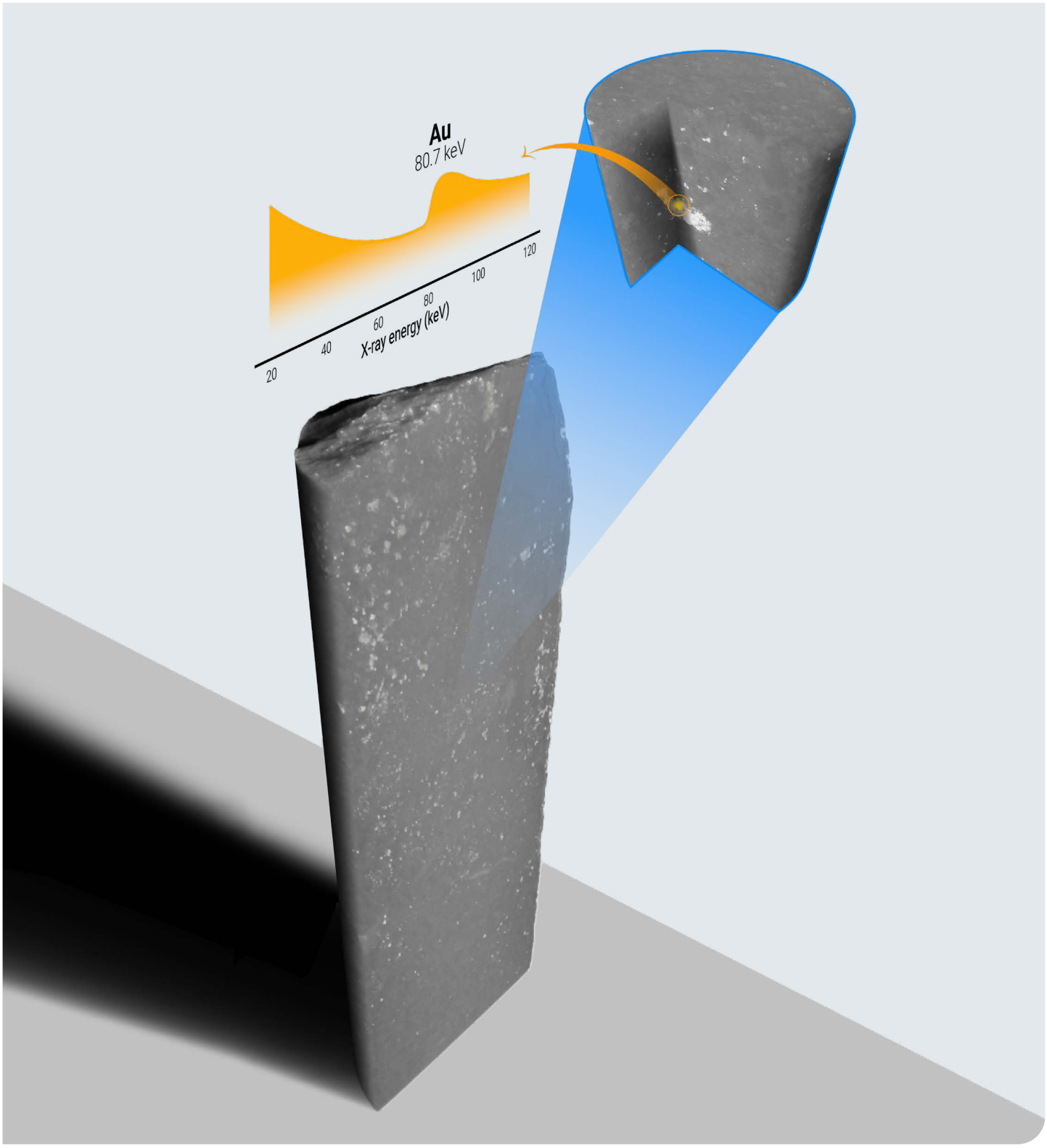
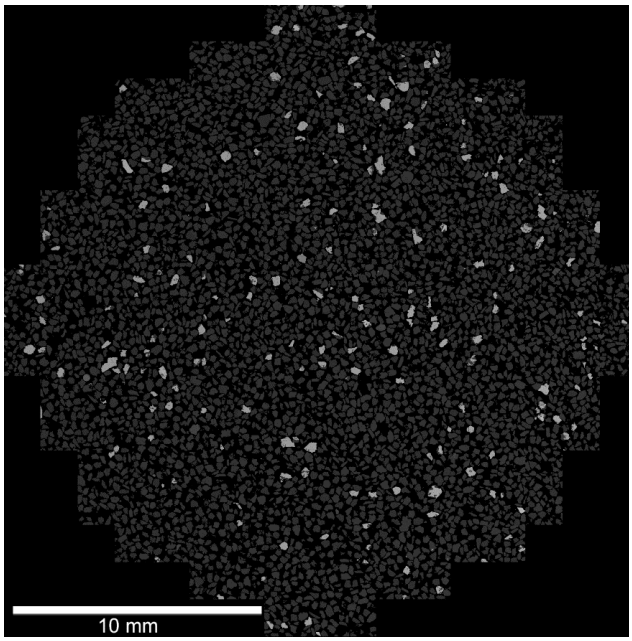
3D elemental analysis inside geological samples using spectral-CT



Introduction

SEM-EDS analysis, which is often paired with automated mineralogical analysis, is currently the gold standard for quantifying textural properties and the amount of important ore minerals in geological samples. Using a standard approach, representative samples of the ore are ground and screened to specific size fractions and embedded in an epoxy resin. After curing of the resin, these cylindrical mounts are polished to achieve a flat surface ideal for analysis in the electron microscope. Using the SEM, detailed analysis of this surface can be performed by combining different imaging and analysis techniques. The backscattered electron signal is used to produce a high-resolution overview image of the sample. Using atomic number-based contrast, minerals containing heavier elements appear brighter than those with elements of lower atomic numbers.

▲ **Figure 1:** Polished resin block containing ore particles.



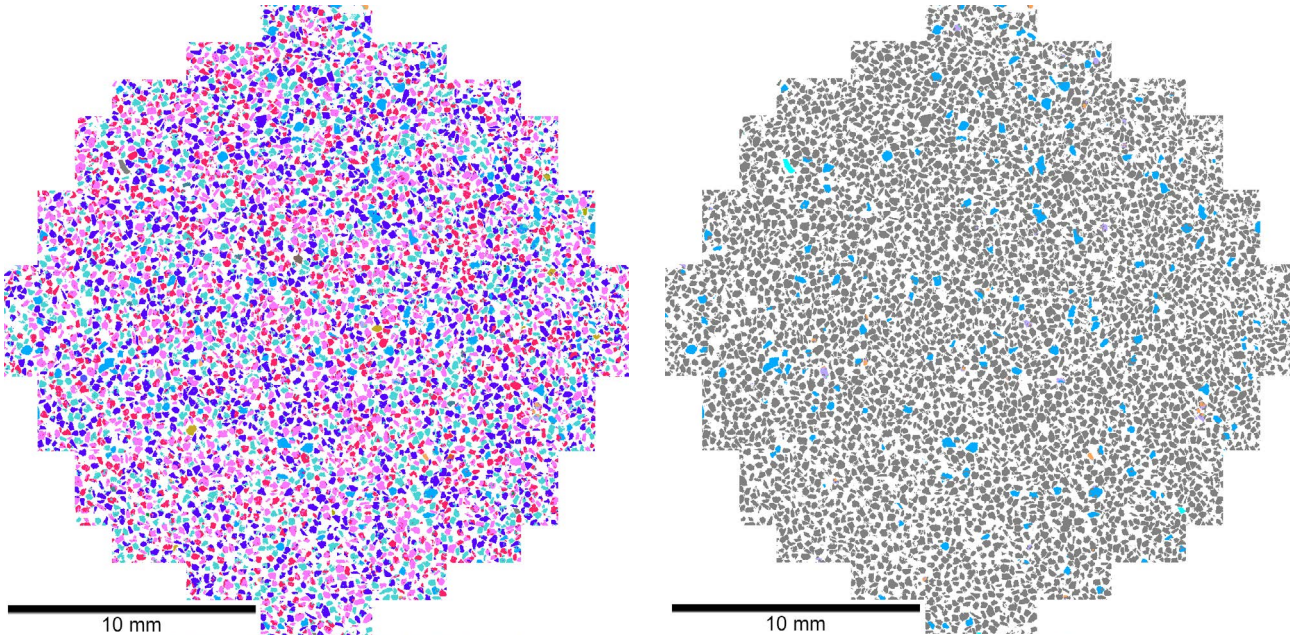
▲ **Figure 2:** Backscattered electron image of the grain mount surface. Brighter grains indicate a higher average atomic number.

To identify the minerals inside the sample, the characteristic x-rays emitted by the sample are analyzed by energy-dispersive spectroscopy (EDS) which generates elemental maps of the sample. Subsequently, automated mineral analysis software – such as the TESCAN TIMA – can use this data to create mineral maps and thus also provide an insight into the texture and composition of the ore. This information is invaluable for metallurgists because it affects the recovery of the elements of interest during mineral processing. A large number of similarly prepared samples is often analyzed automatically to provide statistically relevant data on mineral recovery or losses. However, the images and mineral maps acquired by SEM analysis alone do not provide information on 3D grain shape and empirical models must be used to estimate the true grain size of the particles. Only one single cross-section of the sample can be analyzed at a time which means that minerals in very low occurrences may remain undetected if they are not present in that cross section. Another potential issue arises if mistakes were made in the sample preparation, as heavier grains can sink to the bottom if an epoxy-based grain mount is prepared improperly. As the bottom of the sample becomes the surface that is being prepared for analysis, this density gradient can lead to overestimation of the occurrence of denser phases such as native gold, galena and other heavy minerals.

To acquire valuable 3D information, complementary analytical techniques such as micro-computed tomography (micro-CT) can be used to assess mineralogical samples. Using micro-CT, a larger volume can be analyzed, for example the entire grain mount or even unprepared rock core samples, as presented in this application note. Micro-CT volumes show a 3D distribution of x-ray attenuation and,

just like in backscattered electron images, the contrast is mainly based on the atomic number of the elements in the sample. Thus, it is possible to provide 3D shape, volume and distribution results, with a grey-level based segmentation of the minerals in the sample. In cases where sufficient grey level contrast is present, micro-CT analysis provides all the required information for 3D mineralogical analysis of samples. But, when minerals have similar average atomic numbers because they are composed of elements very close together in the periodic table, conventional micro-CT is unable to differentiate them and provide meaningful data on the occurrence of these minerals.

In this application note, we show how TESCAN Spectral CT can be used to provide contrast for phases of similar density (overlapping gray level), and how spectral CT can be used to identify heavy elements present in important ore minerals.



▲ **Figure 3:** 2D mineral maps produced with TESCAN TIMA. **Left:** all minerals. **Right:** Gangue phases are represented using grey while ore minerals are highlighted in blue.

Materials and methods

To demonstrate the flexibility of our approach, we analyzed two different samples: 1) a grain mount sample with a diameter of 3 cm, and 2) an unprocessed, 5 cm diameter half drill core.

The grain mount sample was polished and analyzed in the TESCAN TIMA (Figure 3).TIMA analysis revealed three main ore materials apart from the silicate gangue: cassiterite (SnO_2), a tin ore, scheelite ($CaWO_4$), a tungsten ore, and tantalite ($[Fe,Mn][Nb,Ta]_2O_6$), a tantalum ore. Next, conventional and spectral CT scanning were applied to the same section using the TESCAN UniTOM XL SPECTRAL. TESCAN Spectral CT makes it possible to perform K-edge imaging for higher atomic number elements at any point within the sample volume. The K-edge is marked by a sudden increase in X-ray absorption, due to the binding energy of the innermost electron shell. This provides specific elemental information in addition to the structural information obtained by conventional micro-CT.

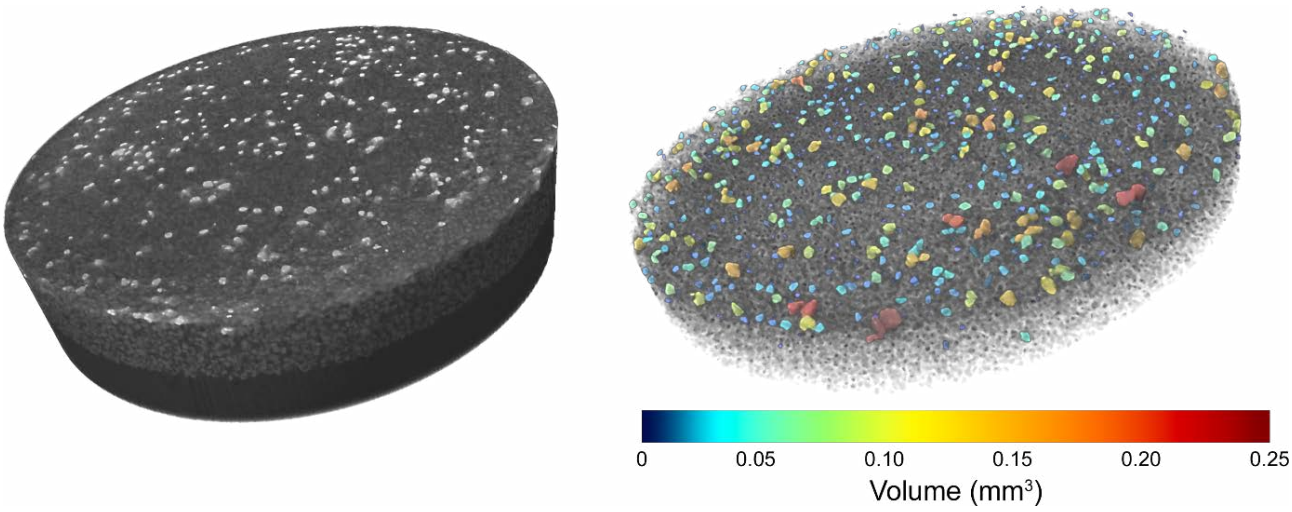
The full core sample is a 5 cm diameter, 20 cm height, gold bearing sample. Both the distribution and the volume of the gold particles inside are unknown and therefore are the subject of our non-destructive investigation. Also this sample was scanned using the UniTOM XL SPECTRAL, using both conventional CT and spectral CT.

Conventional CT scans were reconstructed using Panthera™. For 3D visualization and analysis a combination of Panthera™ and ORS Dragonfly was used. Spectral CT images were acquired using the TESCAN PolyDET II detector, and analysis was performed using the TESCAN Spectral Suite™.

Results and discussion

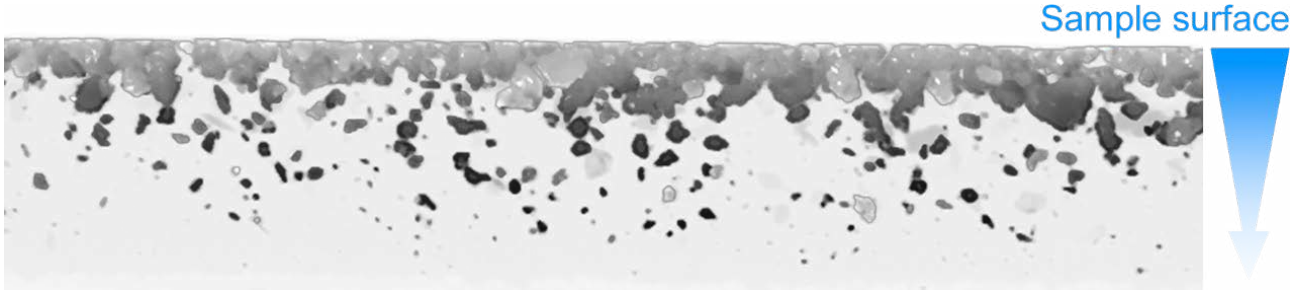
Grain mount sample

The conventional CT scan of the grain mount sample was performed using an accelerating voltage of 160 kV, 15 W power and a voxel size of 13 µm. Using this scan, the full volume of the grain mount can be analyzed. The important minerals (scheelite, tantalite and cassiterite) clearly stand out from the bulk silica minerals, and can be segmented easily based on grey value. We can easily calculate that these minerals make up 0.9 vol.% of all mineral grains in the sample. The segmented grains could then be analyzed for important parameters such as volume, sphericity, orientation, etc. Figure 4 shows the segmented heavy minerals color-coded according to their size.



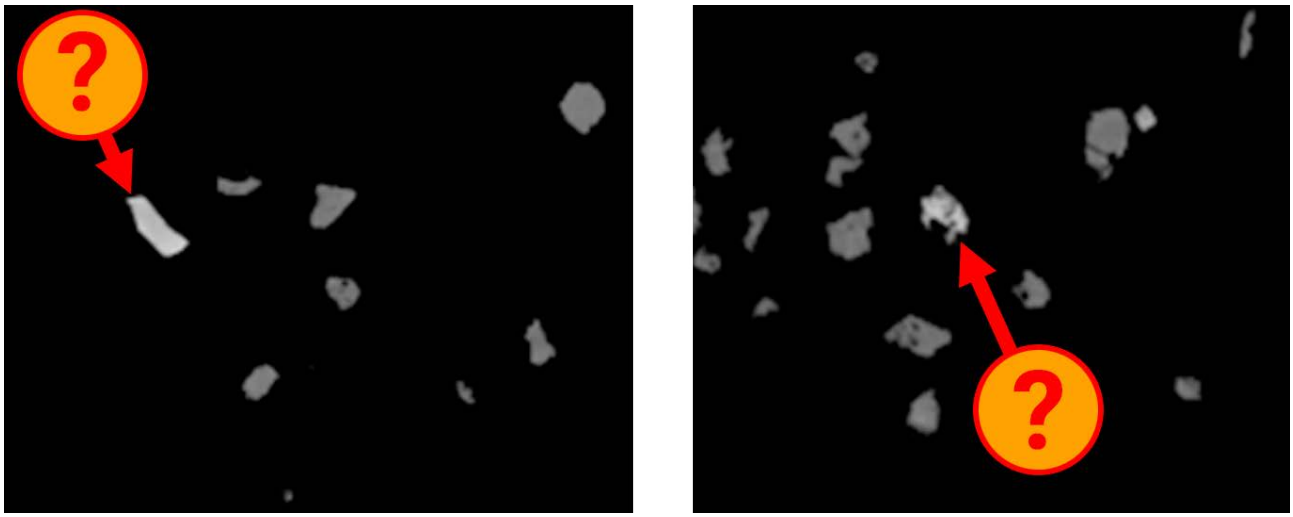
▲ **Figure 4: Left:** volume render of the conventional CT scan of the grain mount. **Right:** dense particles color-coded to size.

The resulting dataset also shows how non-destructive 3D analysis can complement traditional 2D analysis methods. When looking at a lateral view of the sample and looking at the distribution of dense grains in that direction by means of a maximum intensity projection, it is clear that there is an enrichment of dense particles towards the surface of the sample. In fact, the bottom half of the sample contains silica gangue, and only a small amount of very fine grains of the ore minerals. This clearly indicates that the electron microscopy data show an overestimation of the three important minerals in the sample, caused by gravitational settling of the heavy particles during sample preparation. Figure 5 shows how this improper sample mounting can lead to incorrect conclusions: where the 3D analysis of high grey value particles resulted in a volume fraction of below one percent, surface analysis suggested over 12 percent of the mineral grains to be cassiterite, scheelite or tantalite. Figure 5 also shows that this overestimation is both in number of particles, but also in particle size, as larger particles sink faster to the bottom of the epoxy during settling.



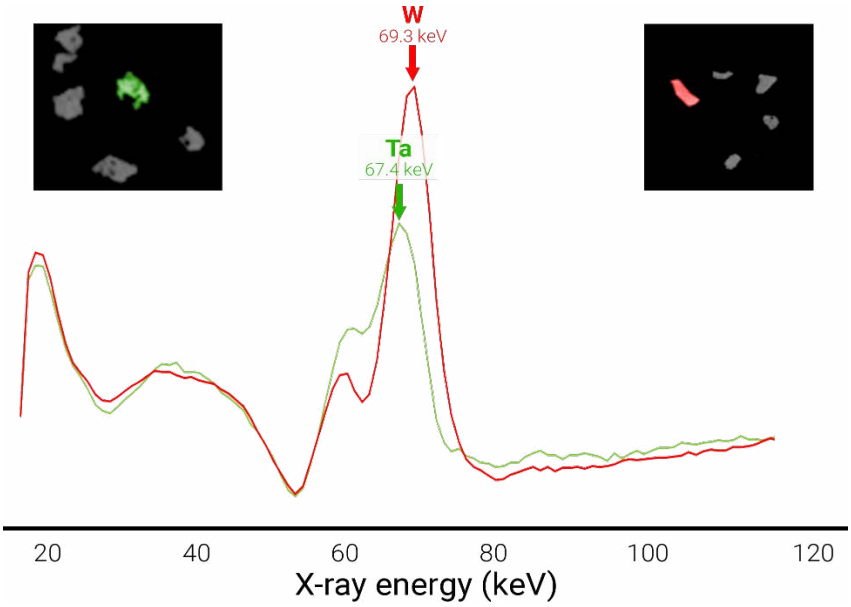
▲ **Figure 5:** Maximum intensity projection in the lateral view of the sample, clearly showing an enriched layer of ore mineral grains at the sample's surface, and a gradual to no occurrence of dense grains toward the bottom.

Of course, these minerals need to be differentiated from each other for a full characterization of the ore. As can be seen in Figure 6 which shows a micro-CT slice through a few mineral grains, the tin ore is clearly different from the other two important minerals, as the average atomic number of cassiterite (40.1) is significantly lower than that of scheelite (51.8) and tantalite (51.9). This leads to an initial differentiation in the minerals of 30 vol.% of cassiterite and 70 vol.% of scheelite/tantalite. However, based on the attenuation of both scheelite and tantalite, it is not possible to determine to which mineral the brightest grey values in the dataset correspond. Furthermore, TIMA analysis showed that many of the Ta and W ore grains are in the smallest grain fraction, making greyvalue based segmentation unreliable due to the partial volume effect.



▲ **Figure 6:** Zoomed view shows a micro-CT slice of the grain mount at two locations. The majority of grains in these images correspond to the tin ore cassiterite. It is not possible to tell the mineral to which the two brighter grains belong.

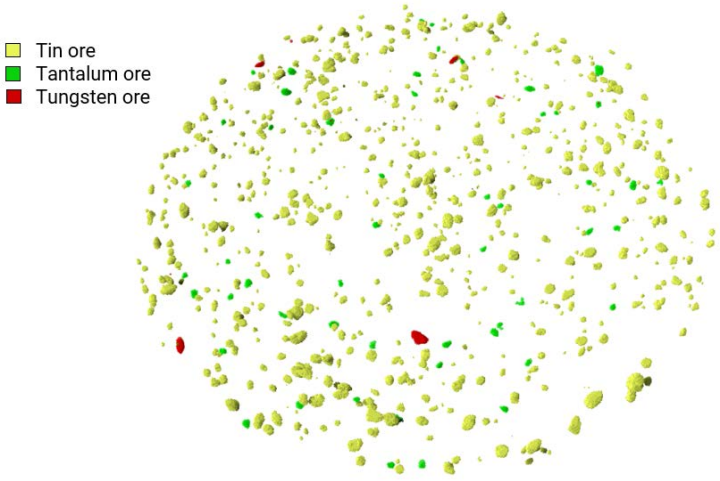
To positively identify the mineral inside this dataset, further analysis using spectral CT was performed. Using spectral CT imaging, it is possible to analyze the spectrum of both highlighted grains in Figure 6 to determine the identity of the main mineral at that location using K-edge imaging. Because Ta and W are adjacent elements on the periodic table, the absorption curves of Ta and W show k-edges at 67.4 keV and 69.3 keV respectively. Figure 7 shows the spectra of the bright minerals highlighted in Figure 6, and reveals that one grain can be identified as scheelite as it contains W, and the other grain as the Ta-containing tantalite.



▲ **Figure 7:** Identification of the mineral grains from Figure 6 as W- or Ta-bearing mineral grains.

Since spectral CT is, just like conventional CT, a complete 3D method, it is possible to segment the full 3D spectral volume. As a result, all scheelite and tantalite grains inside the dataset are differentiated (figure 8). Using this approach, a reliable calculation of the volume fractions of all the valuable minerals in the sample can be performed. By eliminating the effect of partial volume, the tin fraction can be corrected to 83 vol.% of the dense fraction (compared to 70 % using conventional CT). Tantalum (tantalite) is present in 13.9 vol.% of the dense fraction and 2.6 vol.% contains tungsten (scheelite).

By considering that the dense fraction is just 0.9 vol.% of all grains, we can conclude that the sample contains 0.75 vol.% cassiterite, 0.12 vol.% tantalite and only 0.02 vol.% scheelite.



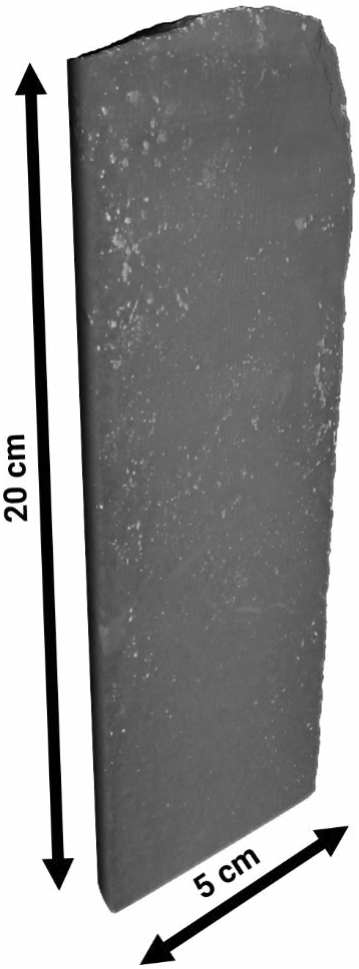
▲ **Figure 8:** Segmentation of all grains in tin ore (yellow), tantalum ore (green) and tungsten ore (red).

Full core sample

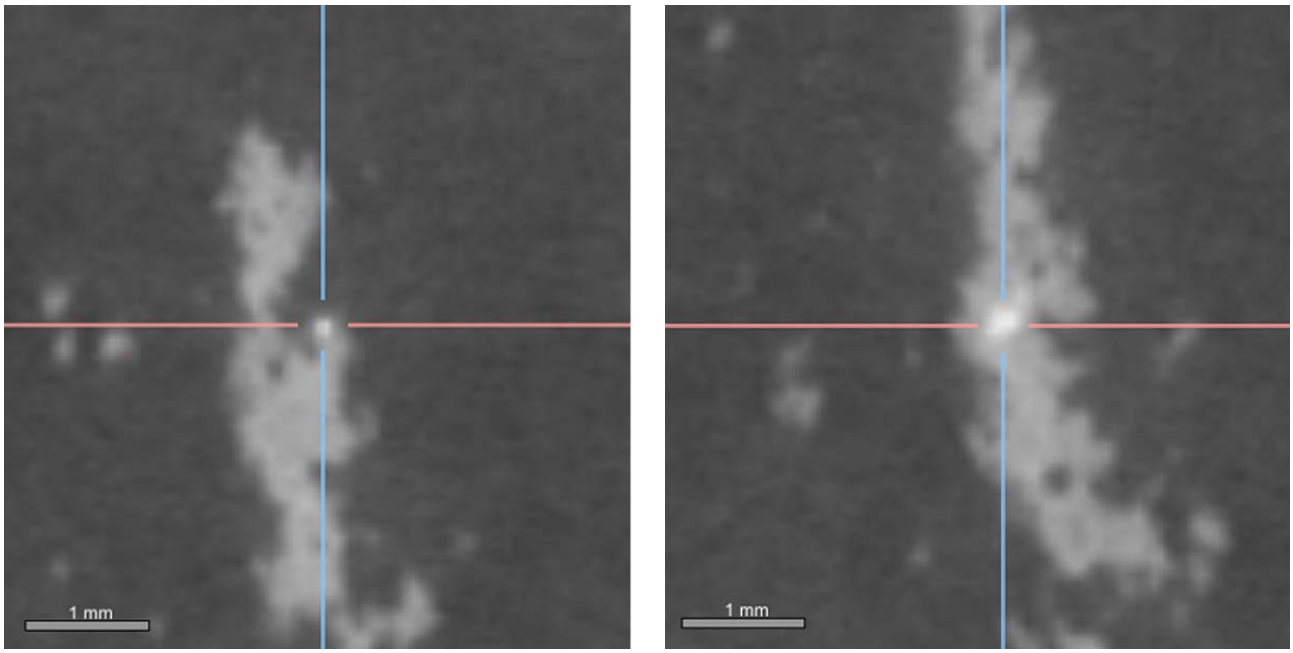
Although the previous example shows the benefit of 3D spectral CT on traditionally prepared samples for electron microscopy, the method can also be used for screening of unprocessed samples. In the next example, a 5 cm diameter core – sliced in half over its longest axis – was analyzed using the TESCAN UniTOM XL SPECTRAL to locate gold grains inside the sample. The sample was sourced directly from a mine and sliced in half to expose a flat surface for additional research such as x-ray diffraction and micro-x-ray fluorescence. These initial tests proved the sample did contain gold, but it was sparsely distributed, and in relatively small volume fractions of 100 µm and smaller. An initial CT scan of 40 minutes at 30 µm voxel size (Figure 9) was used to screen the sample for dense particles that could potentially be gold inside the sample.

Because gold is the highest attenuating material inside the sample, theoretically it should be possible to detect the gold by segmenting the sample based on the brightest grey values in the dataset. However, due to the small dimensions of the gold grains, the partial volume clearly affects the grey value of potential gold particles, and segmentation at this resolution is not a reliable identification method. Due to this partial volume effect, smaller gold grains show the same grey value as larger grains with lower attenuation coefficients, as illustrated in Figure 9.

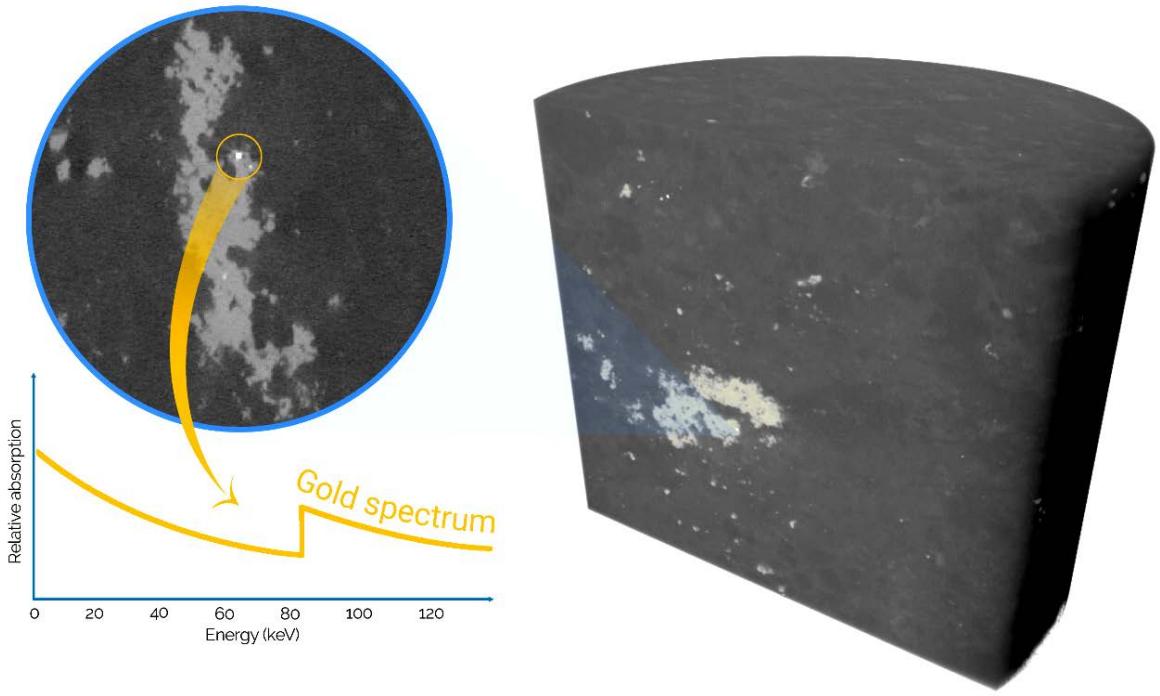
By analyzing all potential gold particles using spectral CT, it is possible to identify particles that contain gold, with absolute certainty. When analyzing the spectrum of the small particle in Figure 9, a clear absorption edge at 80.7 keV is observed, indicating the presence of gold in that location. The larger particle did not show this absorption edge, confirming our presumption that this particle was not a gold grain.



▶ **Figure 9: Volume render of the gold-containing rock core.**



▲ **Figure 10:** A small (80 µm size) potential gold grain (left) has the same grey value as a larger (300 µm size) grain that's most likely not gold (right).



▲ **Figure 11:** Spectral analysis of targeted grains inside a large core indicates the presence of gold.

Conclusions and outlook

We have shown that combining conventional micro-CT and spectral CT provides a powerful method for compositional analysis of geological specimens. Spectral CT is able to quantify the amount of valuable ore minerals inside a sample—without the need for sample preparation. Additionally, spectral CT provides a valuable addition to traditional 2D analysis techniques, by providing 3D information on grain shape, size and distribution. When the concentration of important minerals is very low, micro-CT is able to scan large 3D volumes in short times as a sample screening method for these materials. New developments in the field of spectral CT, make micro-CT even more powerful as an analysis technique, as it is now possible to differentiate between minerals with very similar x-ray attenuation properties, and identify them at any point inside a sample, both internally and externally.

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