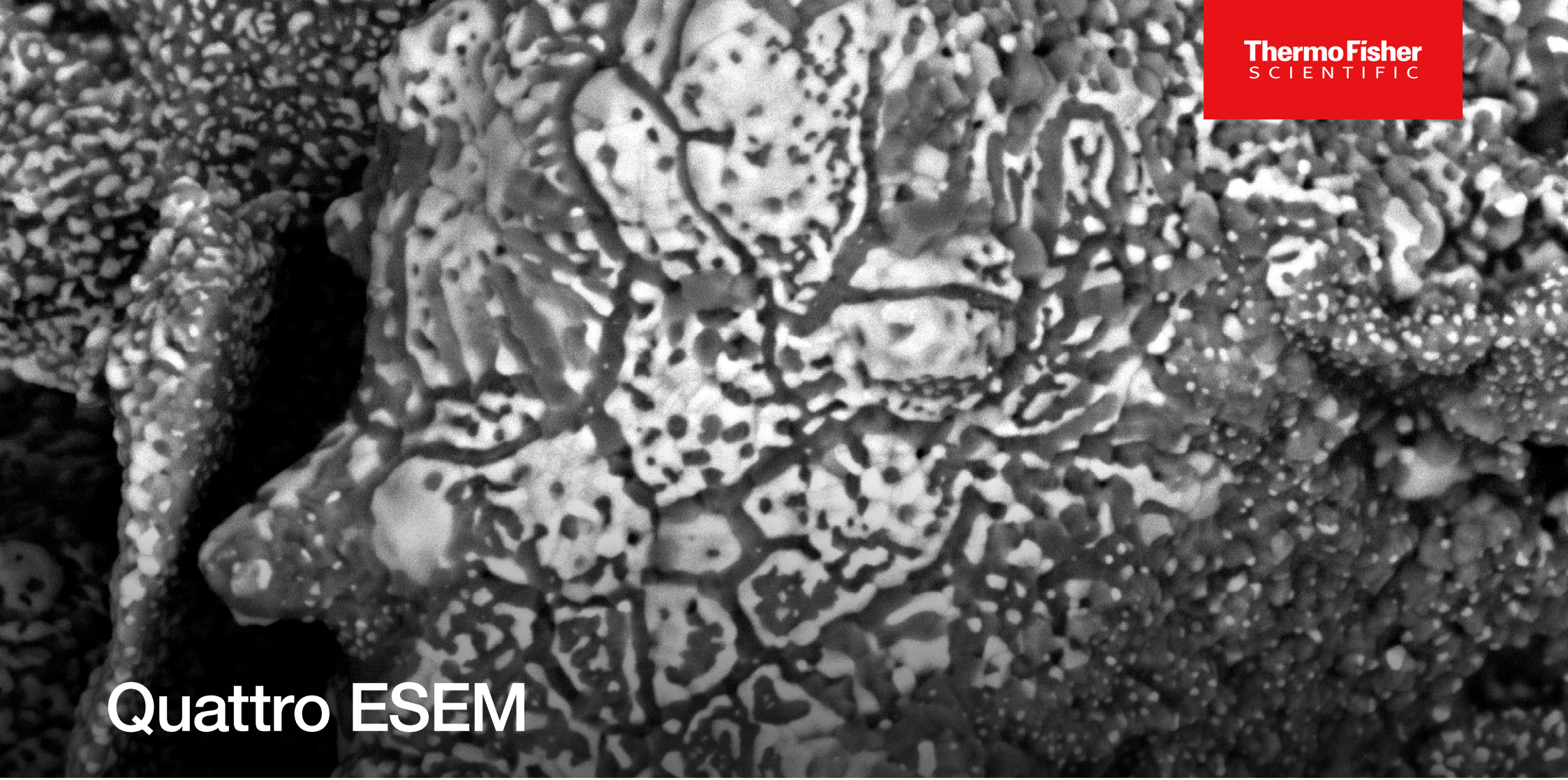


ThermoFisher SCIENTIFIC

Quattro ESEM

Ultra-versatile, high-resolution SEM with unique environmental capability

thermo scientific

Since their introduction, scanning electron microscopes (SEMs) have seen a progressive increase in their use and range of applications. Additionally, field emission (FE-SEM) tools have developed in their resolution performance, analytical capabilities, and the possibility to process a wider mix of materials, ranging from conductive and non-conductive materials to wet and humid specimens in their natural states.

While electron microscopy (EM) has traditionally been a static imaging method, advances in sample handling and rapid imaging have allowed the technique to be used for live, *in situ* observations. The high resolution of FE-SEM tools enables you to investigate nanoscale annealing behavior, phase transformations in metals, structural changes, sintering phenomena in catalysts, segregation/diffusion phenomena, and much more.

As materials research continues to advance, it is becoming increasingly important to not only observe materials in their initial and final states but also throughout their various applications. This might include imaging metal feedstocks as they are heated for additive manufacturing or wetting and drying of functionalized nanoparticles to understand their behavior in real-world conditions. Characterization of these behaviors is crucial, as they impact critical research areas such as clean energy, transportation, catalysis, nano-electronics, and even human health.

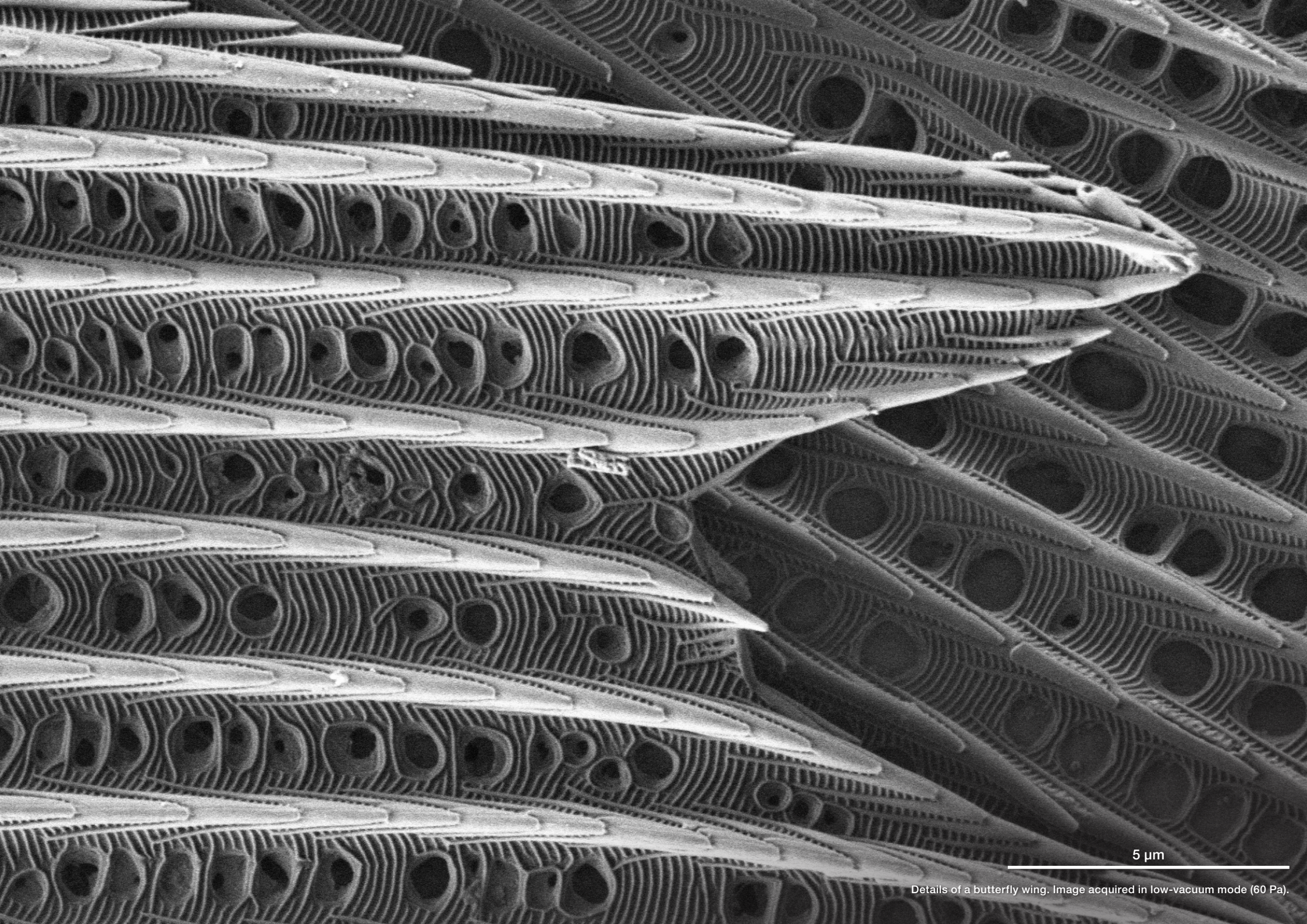
Environmental scanning electron microscopy (ESEM) expands the boundaries of traditional SEM to deliver deeper insights into all types of samples. ESEM™ technology allows for the imaging of samples with minimal preparation and adds variables such as hydration, thermal cycling, and the introduction of gas to characterize *in situ*, dynamic changes. Using water vapor and a temperature control stage, some of the “impossible to image samples” such as highly outgassing and naturally hydrated samples (whose properties will change with drying) are now easily characterized in the Thermo Scientific™ Quattro ESEM.

Introduction

The Quattro ESEM, with its field emission electron source (FEG) ensures excellent resolution and image quality with a broad choice of detectors. It combines unrivaled all-around performance with a unique environmental mode (ESEM) that enables *in situ* studies of materials in real-word conditions with little to no preparation required. No matter what type of sample, in high vacuum or when combined with the unique experimental conditions supported by the Quattro ESEM, reliable analytical results can be obtained, even on samples that are conductive, insulating, wet, or at high temperatures.

Highlights

- High-resolution imaging of any sample with a wide range of detectors in three different vacuum modes
- Excellent analytical capabilities
- Best in class for *in situ* imaging capabilities and dynamic experiments on wet and hot samples
- Advanced automation with Thermo Scientific AutoScript™ Software for *in situ* experiments
- Easy to use with minimal sample preparation time
- Flexible and precise eucentric sample stage



Details of a butterfly wing. Image acquired in low-vacuum mode (60 Pa).

Extreme flexibility in sample loading and navigation

Easy sample mounting with both standard and multi-purpose sample holders

The Quattro ESEM provides extreme versatility thanks to its flexible and precise, 5-axis, motorized, eucentric stage that can load (at 0 degrees tilt) up to 5 kg. Samples are loaded through the door, and the pumping system allows the microscope to be turned on very quickly. It reaches a high vacuum level (pressure below 6×10^{-4} Pa) in less than 3.5 minutes; it allows you to work in ESEM mode in less than 4.5 minutes.

Two different sample holders are available, for increased flexibility. They are mounted directly onto the stage, and each holder's type can mount up to 18 standard stubs (12 mm diameter). In addition, the multi-purpose holder provides a fast way to fit a range of different sample types such as pre-tilted positions, cross-section samples, and STEM samples.

Easy sample navigation

For increased ease of use and navigation, you can take advantage of the Thermo Scientific Nav-Cam Camera, which enables you to track saved positions as well as the current imaging location. It is fully integrated into the SEM user interface and graphically shows holder rotation and beam location.

The Nav-Cam Camera presents an optical image that provides an extremely large field of view of the loaded samples. It allows you to quickly traverse the entire sample holder with point-and-click navigation, letting you reach your area of interest with ease. Because the camera displays a color image, it is easy to differentiate between different samples, letting you take advantage of any multi-sample holder. It provides very easy sample handling and navigation and, in combination with the multi-purpose sample holder, saves you time by loading multiple samples at once.

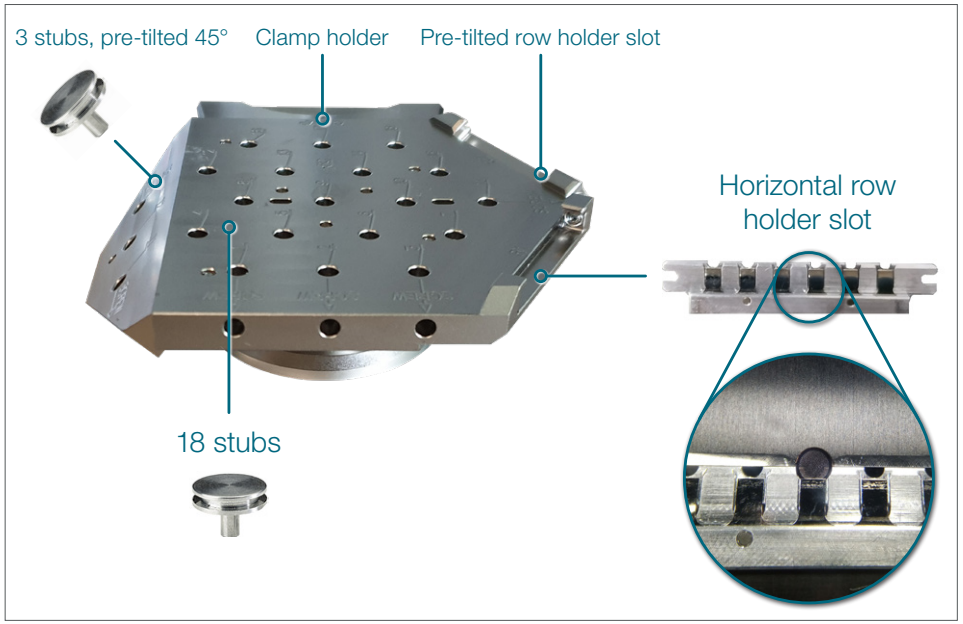


Figure 1. Schematic highlighting key features of the multipurpose sample holder.

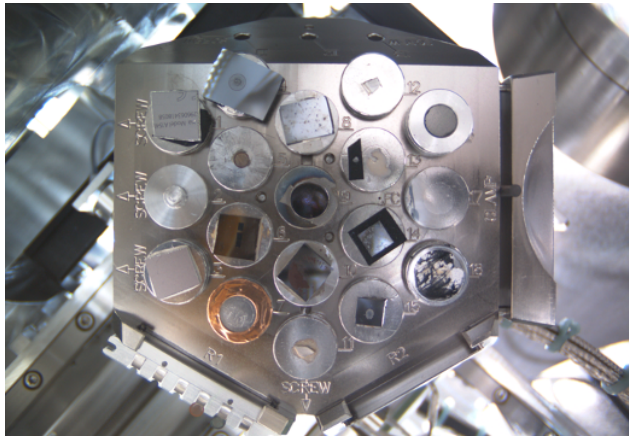


Figure 2. Top view of the multipurpose sample holder.

Extreme versatility for high-resolution imaging with a wide range of detectors

High vacuum

Research facilities and universities have moved from the concept of the expert microscopist as a single user of extremely complicated tools and are now moving towards multi-user laboratories that expect a modern SEM to accommodate a wide variety of samples with excellent image quality and with the least amount of sample preparation.

The Quattro ESEM comes with a field emission gun (FEG) that ensures excellent resolution in a wide range of conditions. Tunable contrast is provided by a vast choice of detectors that, in high vacuum, include the Everhart-Thornley detector (ETD), a retractable under-the lens directional backscatter detector (DBS), and STEM and cathodoluminescence detectors.

The simultaneous acquisition and display of the signal from multiple detectors or detectors' segments is available in a single scan and allows you to improve the time to results. Furthermore, it allows you to reduce the beam exposure of sensitive samples during low-kV imaging.

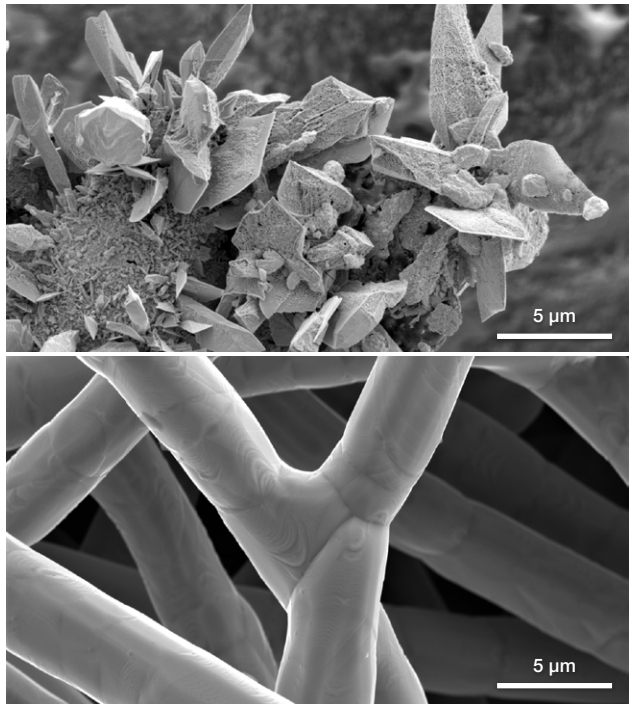


Figure 3. Everhart-Thornley detector (ETD) images. The top image shows beam-sensitive salts imaged at 2 keV and 16 pA. The bottom image shows a metallic mesh of a filter imaged at 15 keV and 0.99 nA.

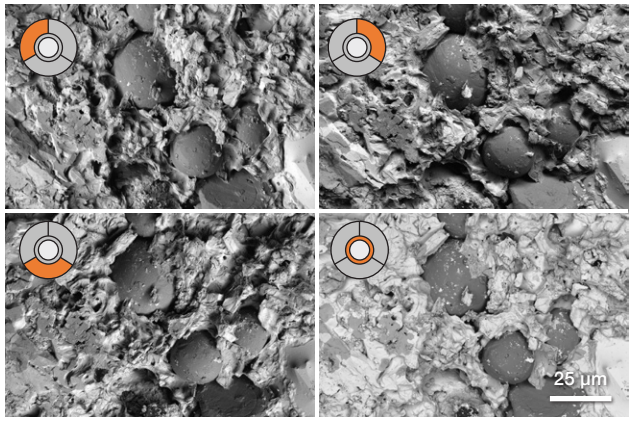


Figure 4. Dental filling material consisting of mercury and a powdered alloy containing silver, tin, and copper. The DBS provides tunable contrast, thanks to the software-based segmentation selection, and is extremely sensitive even at low keV. From top left to bottom right, images acquired using four different segments of the angular backscattered detector (ABS) configuration. The first three images provide enhanced topographical information, while the bottom-right image contains the signal coming from the inner ring, giving mostly compositional information. Acc voltage 2 keV, beam current 0.13 nA.

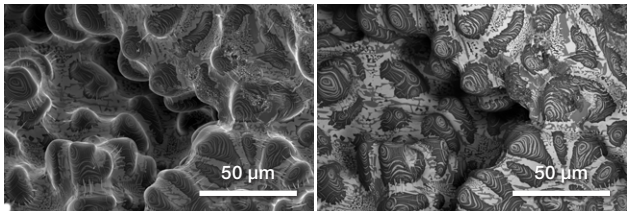


Figure 5. Simultaneous acquisition of SE (left) and BSE (right) imaging on a mix of melted metals. Acc voltage 5 keV, beam current 0.15 nA.

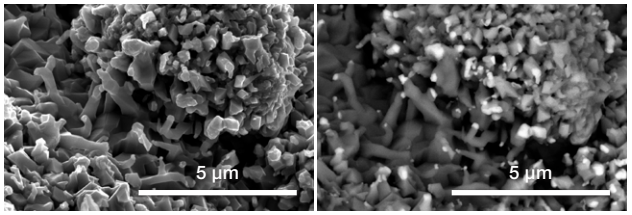


Figure 6. Simultaneous acquisition of SE (left) and BSE (right) imaging on a substrate of NiTiSn. While improving time to result, the simultaneous acquisition offers an easy way to obtain all the needed information at one time. Left image shows roughness and topography of the sample; right image shows the compositional contrast based on the mean atomic number. Acc voltage 10 keV, beam current 0.46 nA.

STEM3 Detector

The Thermo Scientific STEM3 Detector is a retractable detector that offers 11 individually addressable components with flexible segmentation. The level of retrieved information can be enhanced by selecting between STEM3+ and STEM3 configurations that offer bright field, dark field from different scattering angles, and high-angle annular dark-field (HAADF) imaging with a clear improvement in the possibility to customize your selection.

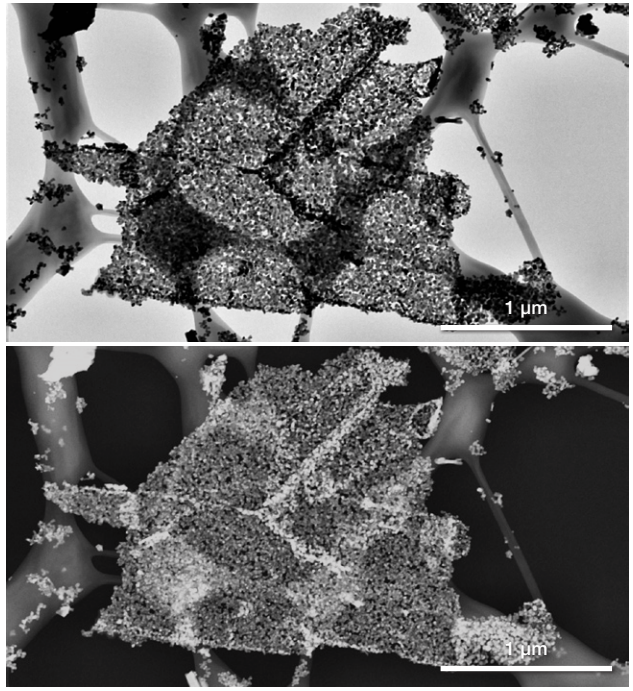


Figure 7. Example of bright-field (BF) and high-angle annular dark-field (HAADF) images of ZnO platelets. Acc voltage 30 keV, beam current 13 pA.

The full integration of the STEM3 Detector in the SEM user interface allows you to load different sample types and switch between SEM and STEM imaging with no need to open the chamber or change instruments.

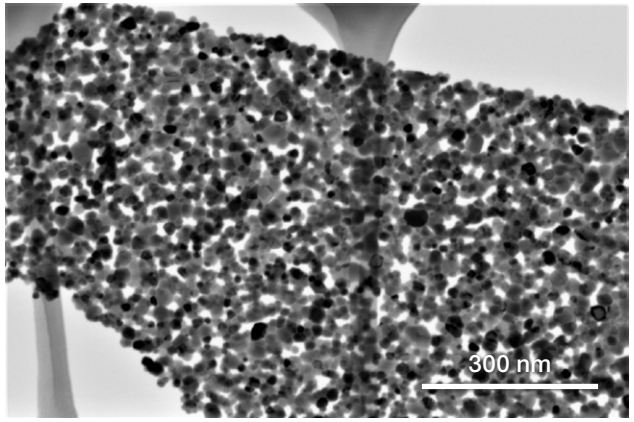


Figure 8. Higher magnification BF image of a ZnO platelet showing its internal structure made of zinc oxide nanoparticles. Acc voltage 30 keV, beam current 11 pA.

Retractable RGB cathodoluminescence detector

The novel, flat detector design of the RGB cathodoluminescence detector (CLD) provides real-color cathodoluminescence data without compromising on ease of use, simultaneous detection, or field of view. With no need for optical alignment, it offers a short time to results with real-time RGB color display. It can work both in high vacuum and low vacuum and is an important option for enhanced compatibility. Its ability to be used simultaneously with SE, BSE, and even EDX is key to complementing conventional SEM information to map trace composition or highlight crystal defects.

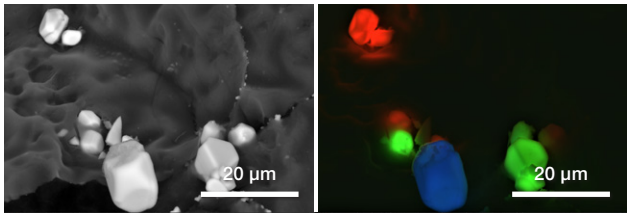


Figure 9. Red, green, and blue emitting particles from a cathode ray tube. Thanks to the information provided by the CLD detector, the different natures of these particles are easily seen, while the different emission properties were not visible by SEM imaging alone. (The compositional information provided by the DBS image on the left does not clarify the different nature of the particles.)

Features

- Wavelength detection range: 350–900 nm
- Large field of view (not limited by the detector)
- Flexible working distance

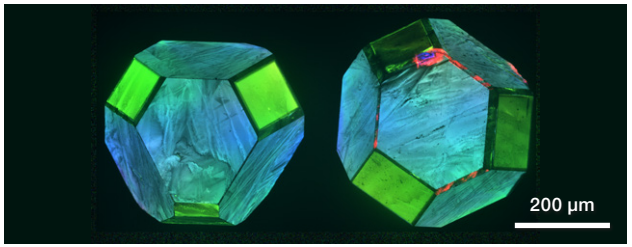


Figure 10. Red, green, and blue emitting areas from two diamonds. Acc voltage 10 keV, beam current 0.7 nA.

Excellent analytical capabilities with ChemiSEM Technology and Pathfinder Software

The Quattro ESEM comes with a chamber with 12 ports, of which the EDS ports are separated 180 degrees, and allows up to three simultaneous EDS detectors. EDS analysis is possible on non-conductive samples and in low-vacuum mode, too. With the Quattro ESEM's through-the-lens pumping, EDS results are extremely accurate.

Thermo Scientific EDS solutions are designed to accommodate all your needs by providing you with elemental information much more quickly and easily than traditional third-party options. Thermo Scientific ChemiSEM™ Technology revolutionize EDS analysis to provide a streamlined user experience. Fully integrated within the user interface, ChemiSEM Technology simultaneously displays SEM and elemental information, with all the tools needed to interpret the data available in one place. ChemiSEM Technology is always-on and provides real-time quantitative compositional information and live reliable quantification (Noran quantification) with a short time to data.

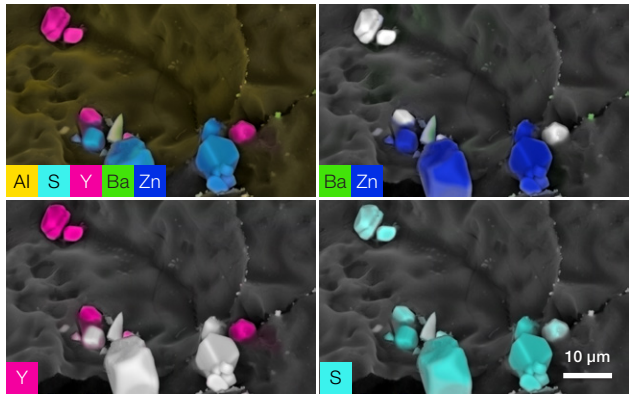


Figure 11. ChemiSEM images of the area previously analyzed with the CLD detector. The top-left image shows the overview distribution of most of the elements present. The remaining images show the separate distribution of barium and zinc (top right), yttrium (bottom left), and sulfur (bottom right). Acc voltage 20 keV, beam current 0.29 nA.

Thanks to its full integration with the SEM user interface, ChemiSEM Technology allows you to combine techniques to obtain different levels of information in a short time to result, with no risk of losing the area of interest. Figure 11 shows the ChemiSEM quantitative maps of the red, green, and blue emitting particles presented in Figure 9. The ChemiSEM elemental maps clarify that the particles' different emissions are due to compositional differences; the red emitting particles are yttrium-rich, but they do not contain any zinc, which is rich in both the green and blue emitting particles.

In Figure 12, in the upper left in grayscale, a DBS image shows two different materials with comparable mean atomic numbers. The compositional contrast provided by the DBS detector is not enough to show the materials' differences. Figure 12's ChemiSEM images of the area of interest immediately disclose the composition of the two different materials. Both left and right alloys contain iron and nickel, shown in light blue and red, respectively. However, what differentiates the two materials is shown in the other ChemiSEM quantitative elemental maps: one of the two materials is rich in both silicon and aluminum, while the other material contains chromium. Note that there is an area in the top part of the image that is not colored. As the left material is slightly lower (in z) than the right alloy, that specific area is masked to the EDS detector; hence, it is not providing any signal to it.

Key benefits

1. Complete information—ChemiSEM Technology offers a comprehensive set of features to obtain the most complete elemental composition possible.
2. Intuitive elemental analysis—ChemiSEM Technology integrates compositional mapping with traditional SEM imaging capabilities, making the characterization of key areas an easy task rather than a long journey.
3. Increased productivity—Its integration and more intuitive color display enable ChemiSEM Technology to make EDS two to four times faster than conventional EDS packages, whether you are looking for a quick screening or a complete characterization of a key feature.
4. Broader userbase—With its exceptional ease-of-use, ChemiSEM Technology is accessible to everyone and provides reliable results to users with no knowledge of the technique.

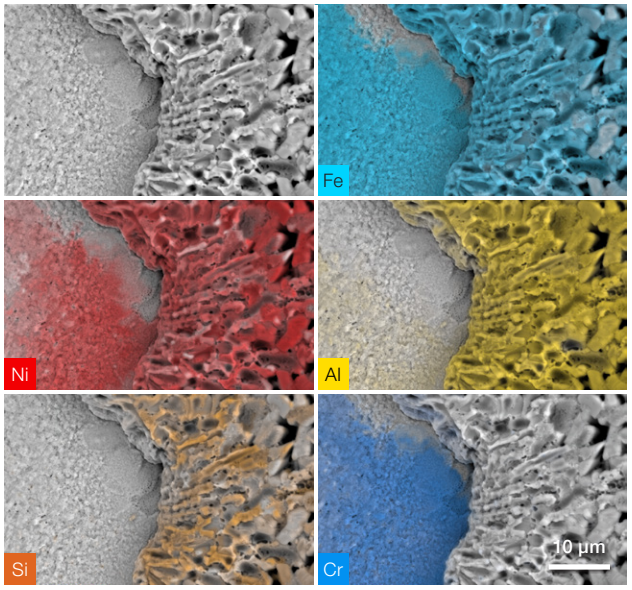


Figure 12. Before-and-after images of two materials with comparable mean atomic numbers. ChemiSEM Technology provides clear differentiation between the two materials. Acc voltage 20 keV, beam current 1.2 nA.

Low-vacuum and environmental SEM (ESEM) modes

The Quattro ESEM comes with three vacuum modes (high-vacuum, low-vacuum, and ESEM) for enhanced flexibility to accommodate the widest range of samples of any available SEM (as of release), especially beam-sensitive samples, outgassing materials, and specimens that would not otherwise be vacuum-compatible.

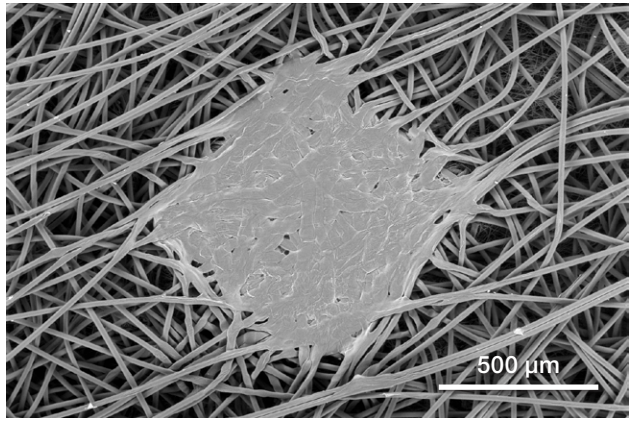


Figure 13. Polymer fibers imaged with a pressure of 80 Pa. Acc voltage 10 keV.

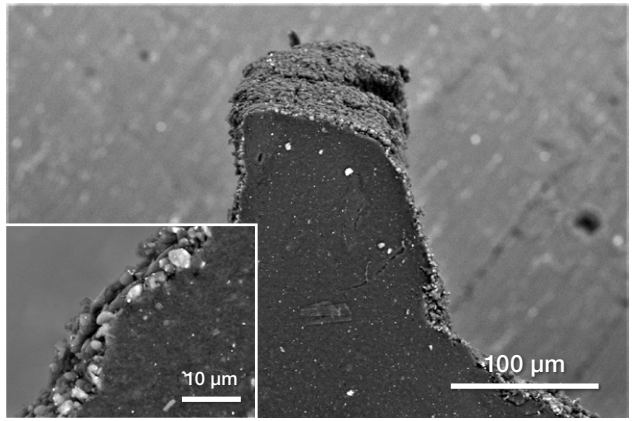


Figure 14. External surface of a tire cross-section imaged with a pressure of 70 Pa. The inset shows a higher magnification of the external part of the tire, which is in contact with the road. Acc voltage 15 keV.

The low-vacuum mode allows you to tune the pressure up to 200 Pa. It provides a huge advantage for imaging non-conductive samples. The ability to tune the pressure in such a wide range is key to enabling charge-free imaging and analyzing hydrated materials.

High-resolution performance is not affected by low-vacuum mode. The Quattro ESEM not only supports high-resolution imaging of any sample in any condition, but, also, for some sample types, the best image quality in low-vacuum mode is obtained with low kV. For very thin and insulated samples, in fact, a high acceleration voltage might give transparent or translucent results that do not give the information that matters on the samples (Figure 16, left image). The ability to use very low-kV imaging in low-vacuum mode without losing resolution performance allows you to highlight surface details as the low-energy electrons probe only the top surface of the material (Figure 16, right image).

Additionally, low-vacuum mode allows you to obtain reliable analytical results on insulating materials. Electron backscatter diffraction (EBSD) analysis, for example, generally requires a relatively high beam current, and that may generate severe charging when dealing with non-conductive materials. Thanks to the Quattro ESEM's low-vacuum mode, you can easily run high-voltage and high beam-current EBSD analysis on any type of material. The example in Figure 17 shows results obtained from a thermal barrier coating (TBC) layer made of ceramic (zirconia). The characterization has been conducted at 15 keV with a beam current of 26 nA with no issue from a charging perspective, as the applied pressure was 100 Pa.

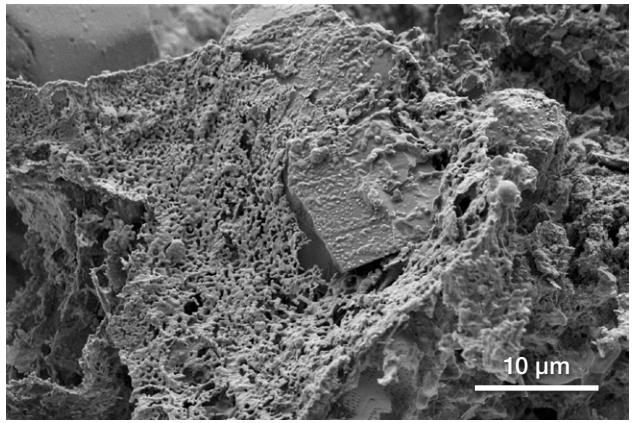


Figure 15. Diatoms and salt crystals in ash imaged with a pressure of 50 Pa. Acc voltage 3 keV.

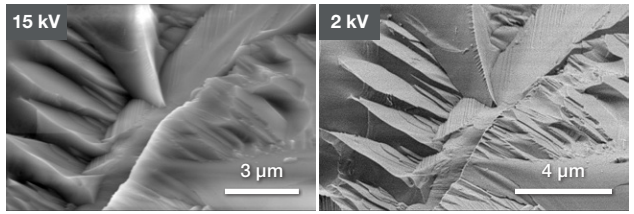


Figure 16. SE images of a rust sample acquired in low-vacuum mode with a pressure of 50 Pa but with different acceleration voltages. The left image was acquired at 15 keV and, in comparison with the right image, which was acquired at 2 keV, shows fewer surface details, much less topographical information, and that the surface appearance is flattened overall.

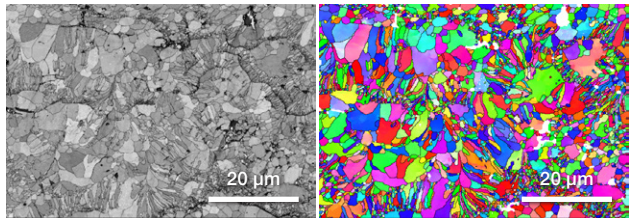


Figure 17. EBSD on zirconia from a thermal barrier coating. Acc voltage 15 keV, beam current 26 nA, pressure 100 Pa.

However, sometimes, the materials of interest are not compatible with high-vacuum SEM imaging: coating is not possible, cleaning the sample may damage or remove interesting surface information, and even low-vacuum mode is not a viable option. For other samples, soft or porous structures just never completely harden, and the continuous outgassing prevents the system from holding the vacuum long enough to allow imaging. The most versatile solution in these cases is available on the Quattro ESEM, thanks to the ESEM option that allows for extremely high chamber pressure that can be tuned up to 4,000 Pa (Figure 18). Thanks to a multi-stage differential pumping system, the column is protected from contamination while a constant flow of imaging gas pushes volatiles out. Several detectors, optimized for such high pressures, allow you to obtain a complete characterization of the sample of interest. ESEM allows you to match the environment inside the SEM to the needs of the sample, rather than adapting the sample to meet the needs of the SEM.

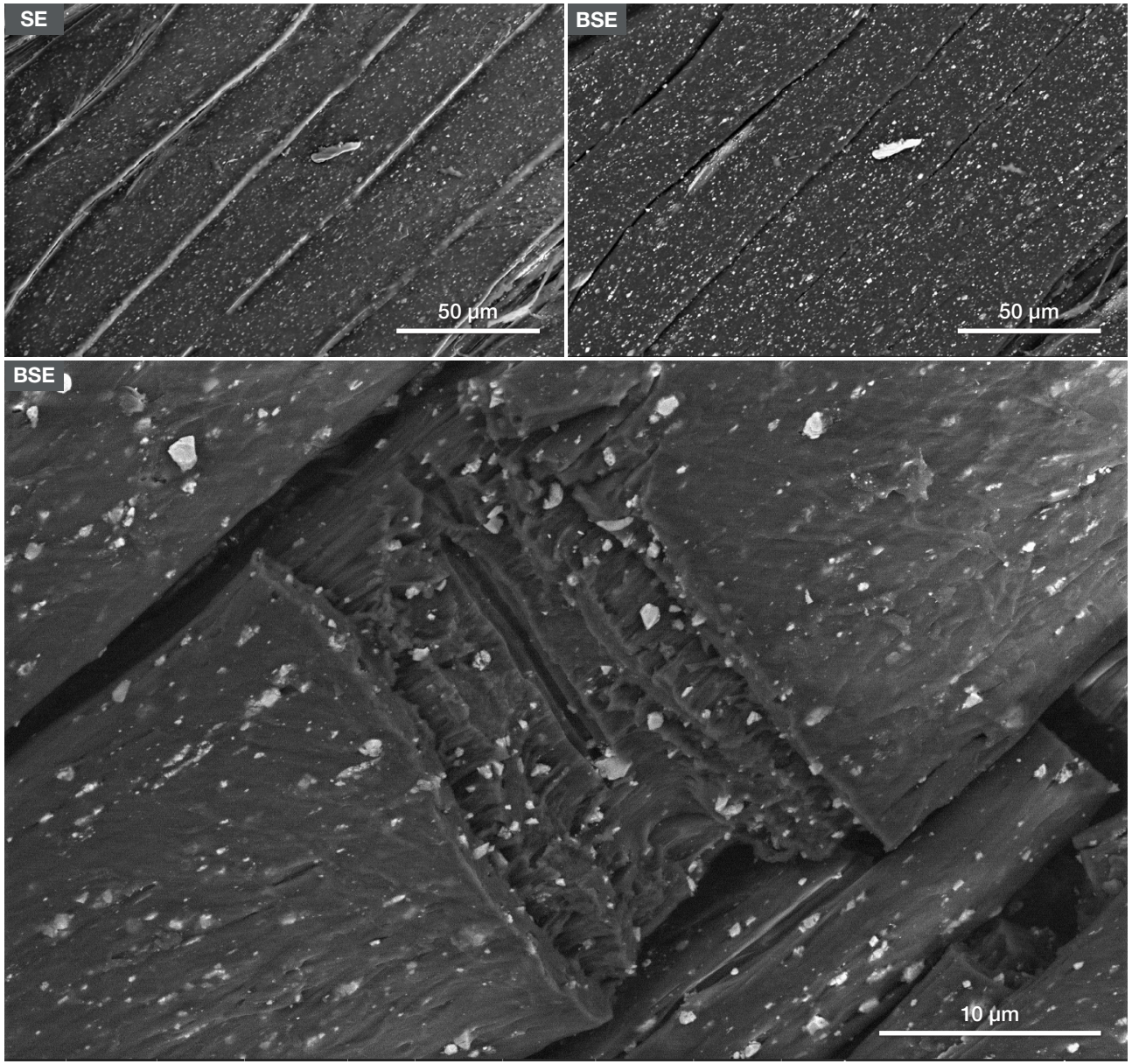
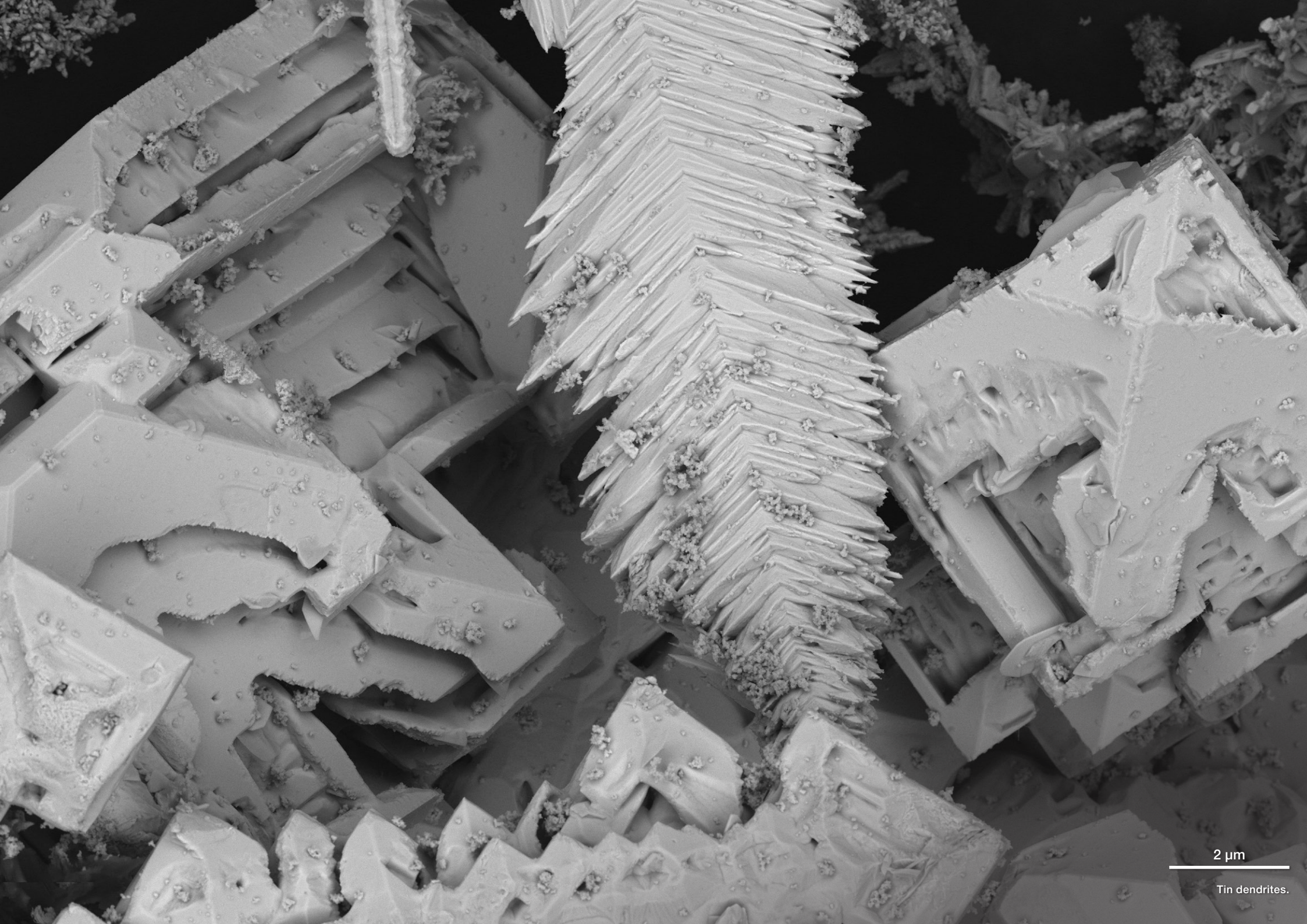


Figure 18. Biomedical braid cord used for sewing tissues. The simultaneous acquisition of SE and BSE images was obtained with a pressure of 300 Pa. The compositional contrast provided by the DBS detector shows the distribution of the ceramic nanoparticles inside the cord. Their distribution and size are highlighted in the higher magnification image. Acc voltage 10 keV.



Tin dendrites.

Best in class for *in situ* capabilities

Wet samples

In the past, SEM has been known generally as a high-vacuum imaging technique. However, industries in many fields such as wood products, chemicals, pharmaceuticals, cotton, and polymers make use of the hygroscopic characteristics of these products to control the humidity in production and to understand how long-term storage of such materials can be affected by changes in water content. In this perspective, ESEM has expanded the boundaries of traditional SEM to deliver deeper insights into a wider variety of sample types by providing a way to observe and record the abovementioned changes at high resolution.

With the Quattro ESEM, materials and life scientists are now able to observe real-time material interactions with water, with the possibility to conduct *in situ* experiments by taking advantage of the introduction of gasses to characterize dynamic changes.

Peltier cooling stage

Thanks to the possibility of pairing ESEM with a Peltier cooling stage, you are now able to take full control of sample hydration by varying pressure and temperature (the allowed temperature range is between -20˚C and +50˚C).

The Peltier cooling stage allows you to study how humidity changes a material and how water interacts at the surface of samples. Additionally, it offers unique methods for testing materials that were commonly obtained with other tools and techniques such as wettability measurements by contact angle, measurements of size, and shape changes during water absorption and desorption.

Dedicated detectors for both SE and BSE detection allow you to obtain excellent resolution whether you need topographic or material contrast information, which can be obtained at all pressures. Furthermore, the SEM user interface integrates different options to capture movies and TIFF image series during the experiment or after data collection.

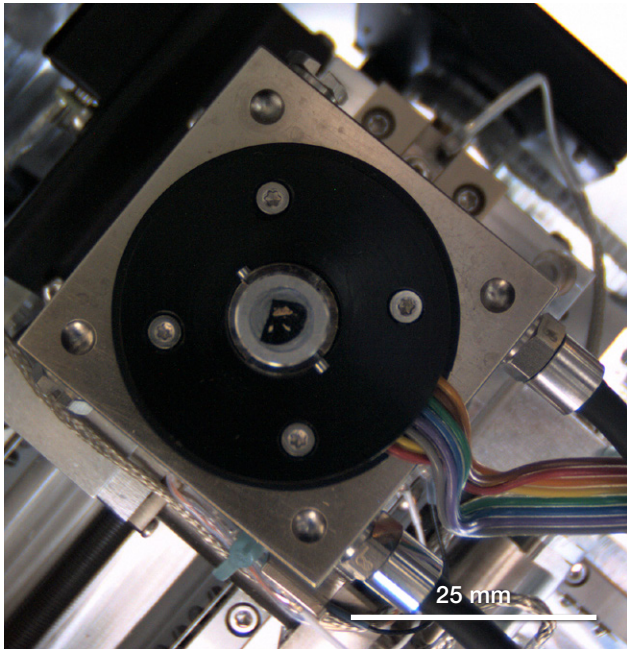


Figure 19. Navigation camera image of the Peltier cooling stage.

In situ characterization of sodium sulfate when exposed to water vapor: a Peltier application example

An example of an *in situ* cooling experiment was conducted on a sandstone sample with crystals of sodium sulphate. The target of the experiment was the exposition of salt crystals to the water vapor in the SEM to study their behavior and observe their dissolution and recrystallization.

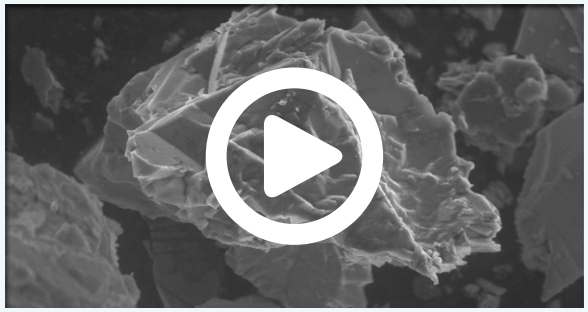
For this experiment, the Peltier cooling stage was used (Figure 19), and the sample was attached to the stub using a small piece of carbon tape.

At the beginning of the experiment, the temperature of the stage was set to 2˚C, and the system was pumped directly to ESEM while applying an optimized purge cycle. The purging allowed the microscope to cycle between a minimum and a maximum pressure (in the range of the desired final pressure) to progressively replace the ambient humidity with the desired pressure of water vapor.

With the stage set at 2˚C and low relative humidity (RH of 70%, around 470–500 Pa), an EDS map was first acquired at 10 keV to identify the sodium sulfate crystals. The Quattro ESEM, in fact, allows you to perform EDS in ESEM mode. Elemental maps are presented in Figure 20, showing a clear concentration of sodium and sulfur in the object in the center of the characterized area.

The crystal shown in Figure 20 was selected and imaged live during the entire experiment.

From a starting pressure of around 480 Pa (RH of 70%), the chamber pressure was gradually increased to raise the RH up to 100%. The temperature was kept stable at 2˚C, and, with the increase of humidity up to an RH of 100% (700 Pa), water could be observed on the surface of the sample (Figure 21, third image). Finally, the chamber pressure was gradually decreased (returning around 400 Pa) to remove water and induce recrystallization of the sample (Figure 21 last image).



***In situ* characterization of sodium sulfate when exposed to water vapor: a Peltier application example. Duration 0.18**

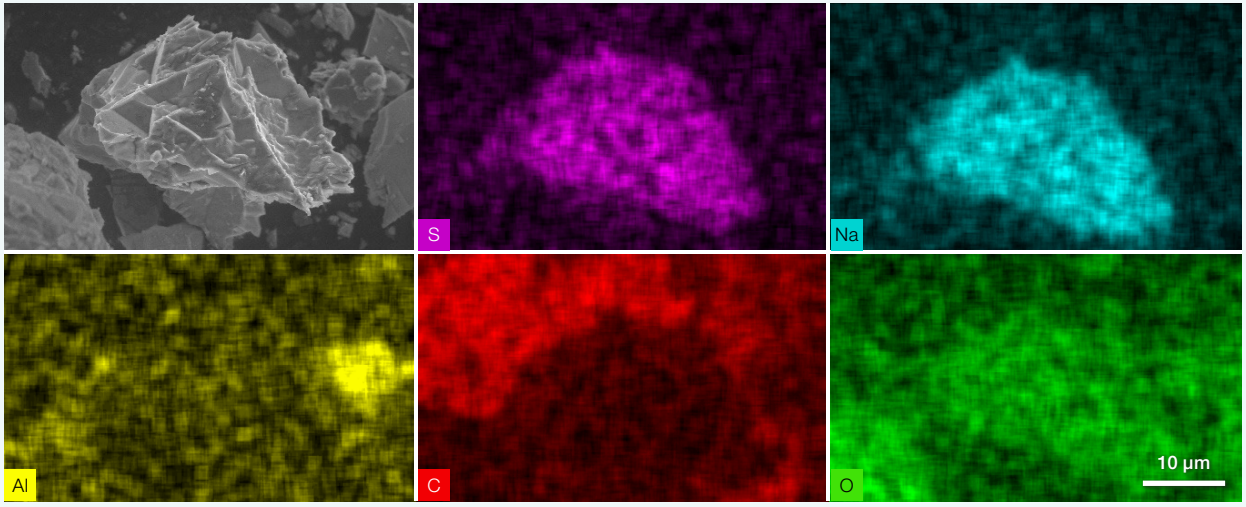


Figure 20. Elemental mapping acquired in ESEM mode, with a pressure of around 500 Pa and a temperature of 2˚C.

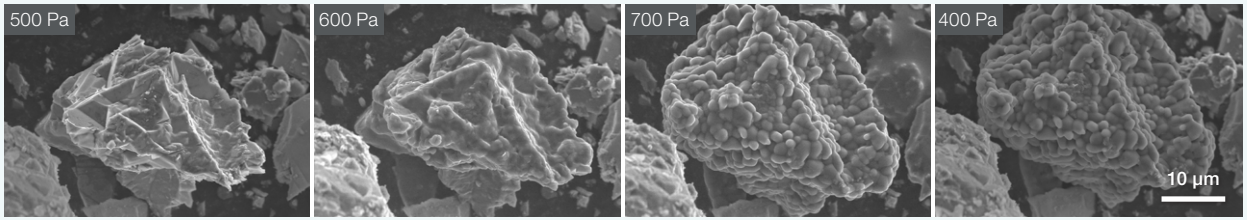


Figure 21. Selection of images showing the progression of the salt's dissolution and recrystallization with the chamber pressure change. The last image at 400 Pa shows the appearance of the salt after the recrystallization. *Sample courtesy of Institute of Theoretical and Applied Mechanics of the Academy of Science (Czech Republic).*

WetSTEM Technology

The Thermo Scientific WetSTEM System allows observation of wet samples in transmission mode through thin membranes of water. Compared to TEM, it has the main advantage of lower acceleration voltages that result in higher contrast. Additionally, EDS analysis is possible and will give high spatial resolution results.

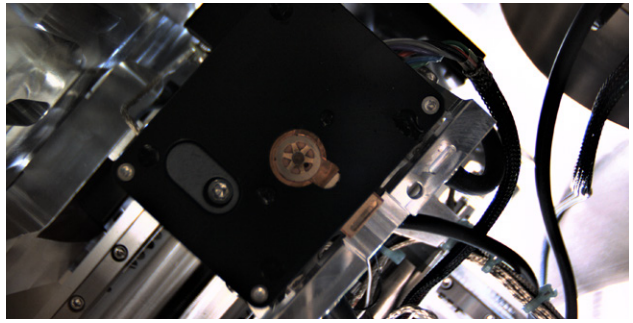


Figure 22. Navigation camera image of the WetSTEM System stage.

The WetSTEM System uses the STEM3+ solid-state detector, which can be mounted at the bottom of the WetSTEM assembly or inserted underneath it with a retractable arm. Fine adjustments in temperature and pressure allow the same dynamic control as with bulk samples in traditional ESEM.

Silver nanoparticle STEM imaging in liquid

The material under study was a sample of silver nanoparticles in water. A 1.5-µl drop of the solution was drop-casted onto a carbon-coated holey copper TEM grid. The characterization was carried out on a Quattro ESEM using the WetSTEM stage.

Water vapor was added to back-fill the chamber, which is essential to accurately control the RH level of the sample. An initial purging cycle was employed to switch from the room pressure (from the venting cycle) to the fully wet and hydrated condition (RH 100%) without drying the sample. The chamber pressure was then decreased with the aim of removing the excess water while keeping the sample fully hydrated. The temperature of the stage was set at 1˚C and maintained at this temperature by the Peltier stage for the entire experiment.

The imaging was conducted with the use of the STEM3+ solid-state detector, which, thanks to its configuration with several segments, allowed the acquisition of bright field (BF), dark field (DF), and high-angle annular dark-field (HAADF) contrast.

This possibility, together with the SEM user interface design that offers simultaneous detection of up to four signals/detector segments, provided a complete and optimal characterization of the experiment.

A first overview of one of the grid's meshes is shown in Figure 23. Liquid water is visible at the edges of the copper mesh, and it appears as either darker or brighter than the sample, depending on the contrast obtained from the different detectors' segments.

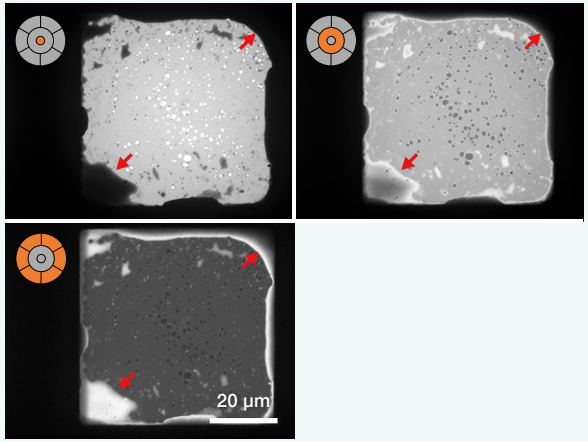


Figure 23. BF (top left), DF (top right), and HAADF (bottom left), images of a large-scale overview of one of the meshes. The images were acquired in wet mode, and liquid water is visible at the edges of the copper mesh (see red arrows).

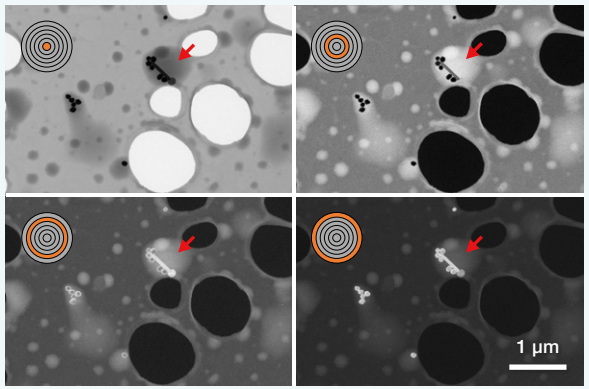


Figure 24. BF (top left), DF2 (top right), DF4 (bottom left), and HAADF (bottom right) imaging of the silver nanoparticles in the water droplets.

Higher magnification images (Figure 24) were acquired to characterize the nanoparticles' shapes. Additionally, with a relative humidity of 100%, the WetSTEM stage allowed for inspection of their arrangement in liquid and how they interacted with each other. (Examples are shown in Figure 25.)

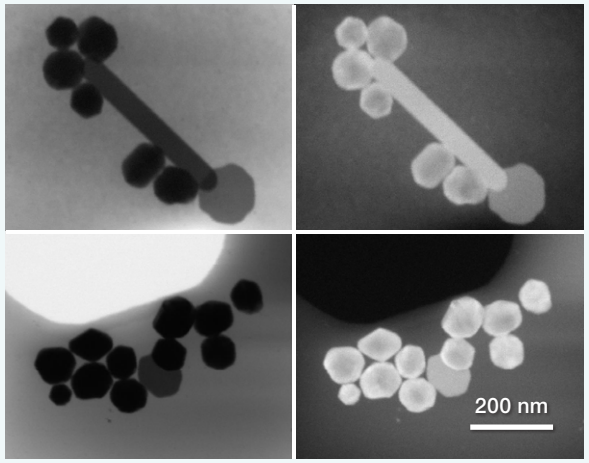


Figure 25. High-magnification, BF (left), and HAADF (right) images of different nanoparticles' shapes.

Top-down imaging application example with the WetSTEM stage

The WetSTEM stage option is designed to allow the characterization of samples in transmission mode. The flexible design of the Quattro ESEM and its accessories allows you to also use the WetSTEM stage in top-down mode with the included stubs. This enables you to image your materials of interest top-down without the need to purchase an additional stage.

The materials of interest for this application example are small pieces obtained from a flower and its pollen. The samples have been mounted onto the Peltier stub with copper tape (Figure 26).

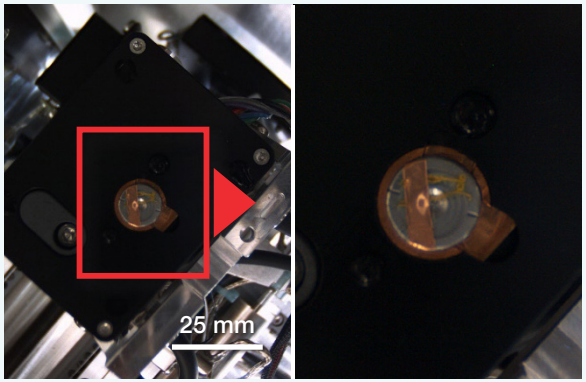


Figure 26. Navigation camera image showing the WetSTEM stage used for a top-down investigation. Two flower sepals are fixed to the Peltier stub using a small piece of copper tape.

For these specific materials, the purging cycle at the beginning of the experiment was optimized to avoid drying the samples while saturating the chamber with water, which would have caused the presence of artifacts on the surface of the sepals.

The imaging was conducted with an acceleration voltage of 8 kV to show enough surface details while keeping the sample fully hydrated. The relative humidity, in fact, was kept at 100% for the entire duration of the imaging, which corresponds to a pressure of around 800 Pa.

Figure 27 shows two different images obtained using the WetSTEM stage for top-down imaging. The pollen was kept hydrated and swollen, as shown in both images, thanks to the high RH. Few water droplets on the aluminum stub are visible on the top left of the left image.

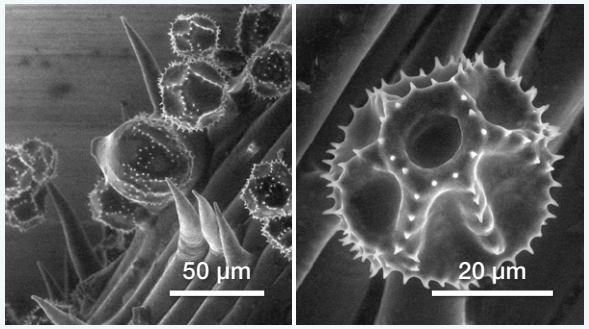
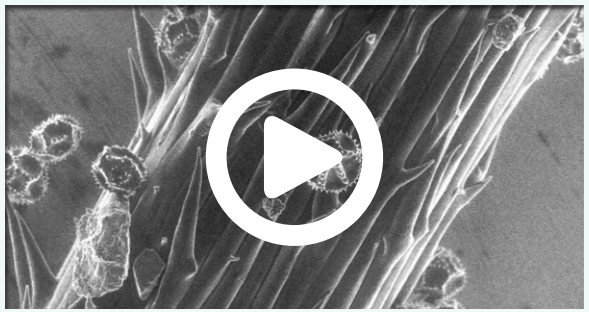


Figure 27. Secondary electron images obtained at 8 keV acceleration voltage in ESEM mode with a temperature of 2˚C and a pressure of 800 Pa.



Top-down imaging application example with the WetSTEM stage. Duration 0.37

Hot samples

Working with ESEM at high temperature has long been a goal for many researchers. In the past, it was generally considered a difficult technique to operate efficiently due to several constraints in sample preparation but also in imaging.

With the Quattro ESEM's unique ESEM technology, microscopists and researchers are now able to study any material's thermal cycling thanks to several different possibilities for heating the samples of interest, both in bulk and powders. Thanks to its extreme versatility, you can perform dynamic experiments at high temperatures both in low vacuum, high vacuum, and on localized areas for better control of the temperature change. Additionally, the Quattro ESEM's architecture with differential pumping is designed to deal with outgassing materials, a common phenomenon during heat-up.

The Quattro ESEM offers two different types of heating stages for low-vacuum and ESEM modes and one heating stage for high-vacuum experiments.

ESEM heating stages

Two ESEM heating stages are available for temperatures up to 1,000°C and 1,400°C. Samples can be in the form of powder or pellet and are placed inside a ceramic crucible surrounded by a heating element. (Graphite crucibles are available for inert atmosphere and temperatures lower than 900°C.) The heating assembly is designed as a micro-furnace. Being inside a crucible, samples are heated from the sides (not just from the bottom), allowing for more uniform and controlled temperature gradients around the sample.

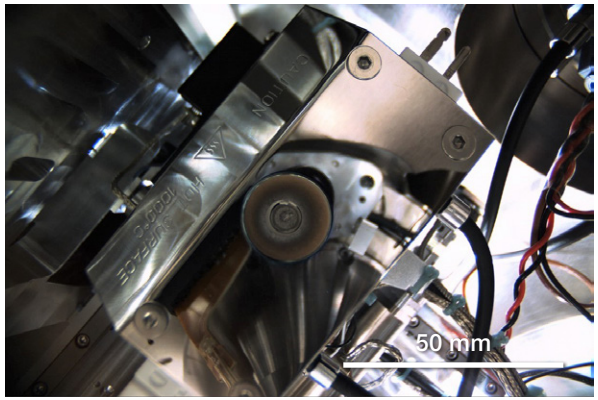


Figure 28. Navigation camera image of the 1,000°C ESEM heating stage.

Several strategies and options, such as sample biasing, detector biasing, and the presence of a specially designed heat shield, are available to improve the image quality, to allow for EDS analysis, and to allow for a more accurate temperature reading. Sample biasing between -50 V and +50 V is available to limit the number of thermal electrons in the total electron signal to be collected. A high-temperature SE detector (HT-GSED) is provided to guarantee the best image quality and to improve the signal yield. Specially designed to work between 70 Pa and 650 Pa, the HT-GSED comes with a fitted pressure-limiting aperture (PLA) and is not sensitive to light or temperature. A movable heat shield is also available to protect the pole piece and, depending on the temperature, to allow for EDS analysis and provide more accurate temperature control.

High-vacuum heating stage

When the material of interest is conductive and high-vacuum compatible, heating in a high vacuum is the best option. The main advantage is that different detectors, such as the Everhart-Thornley (ETD) detector, are available in high-vacuum mode.

This means that better image quality is provided, and, additionally, you can image faster than in low-vacuum mode. Heating in high-vacuum mode also guarantees higher cleanliness while minimizing oxidation. In fact, materials are less likely to oxidize when being heated, or, at least, the oxidation is drastically reduced at high temperatures.

Furthermore, high-vacuum mode offers high enough resolution to allow for good quality with low-kV imaging.

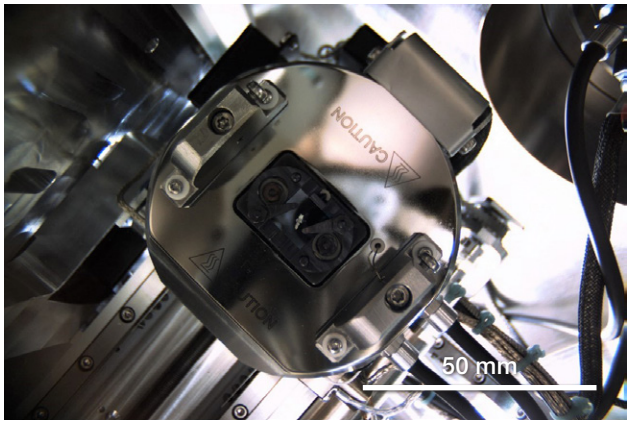


Figure 29. Navigation camera image of the high-vacuum heating stage.

The Quattro ESEM offers a high-vacuum heating stage (HVHS) that allows for sample heating up to 1,100°C. It can mount samples up to 10 mm in size covered by a shield with a 3 mm hole. EDS and EBSD analyses are possible, depending on the temperature employed. (EDS is available up to 500°C and EBSD up to 900°C.)

µHeater Holder

When extreme control of the temperature is needed and the ability to reach 50°C/s is not fast enough, different equipment is needed. The Quattro ESEM, as a very versatile and flexible high-resolution SEM with *in situ* capabilities, provides a solution to that: the Thermo Scientific µHeater Holder, a fully integrated, ultra-fast heating stage for high-resolution imaging.

The µHeater Holder is high-vacuum-compatible and provides rapid and precise heating to 1,200°C in 100 ms (10^4 °C/s).

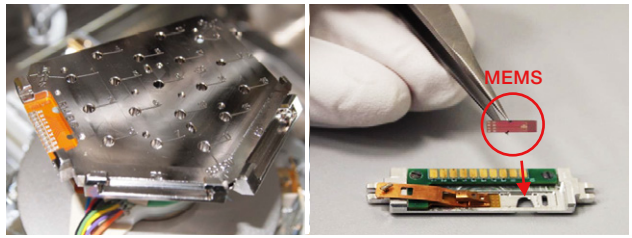


Figure 30. Multi-purpose sample holder with slot for microelectromechanical systems (MEMS)-based µHeater Holder cartridge.

Thanks to the tiny thermal mass of the holder, the MEMS device of the µHeater Holder delivers consistent, reproducible, and uniform temperature distribution over a heated area of 100 µm. The ability to precisely control the temperature in such a small area allows you to run experiments and acquire high-magnification images with very little drift.

Controlled heating of a mix of magnetite and hematite nanoparticles

A mix of magnetite and hematite nanoparticles was diluted in ethanol and sonicated for a few minutes to avoid large aggregates. 3 µl of the solution were drop-casted onto the µHeater Holder's MEMS chip and allowed to air dry. The sample was mounted, and the stage's heating set to 40°C while the SEM was still vented. This helped with the complete drying of the substrate before system pump-down.

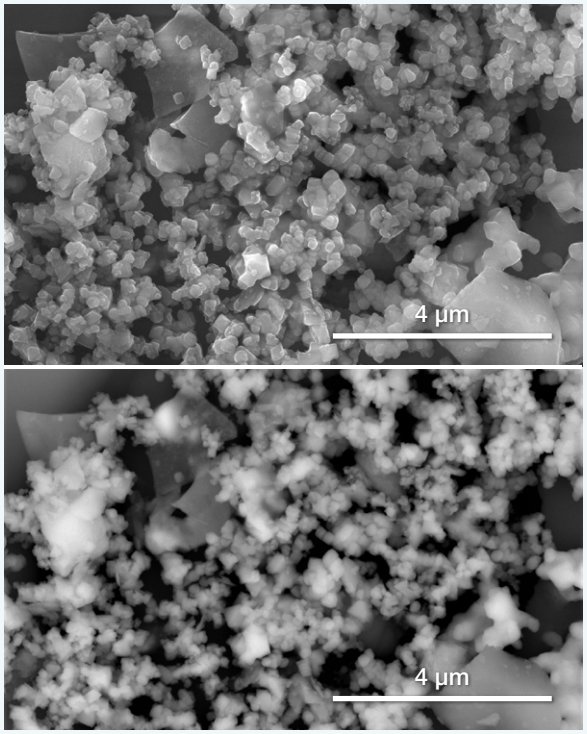


Figure 31. SE (top) and BSE (down) images acquired at the beginning of the experiment with the temperature set at 40°C. Acc voltage 20 keV, beam current 0.13 nA.

Figure 31 shows the SE and BSE images of the area of interest.

The heating was executed in different steps, as shown in the graph of Figure 32 to allow the sample to stabilize and to be able to acquire additional images and EDS maps. Note: the initial steps, up to 800°C, had been executed more quickly and with higher heating rates (°C/s), as the material did not show sensitive changes. The rates had been decreased up to 1°C/s from 800°C up to 1,100°C/s to more accurately monitor the process.

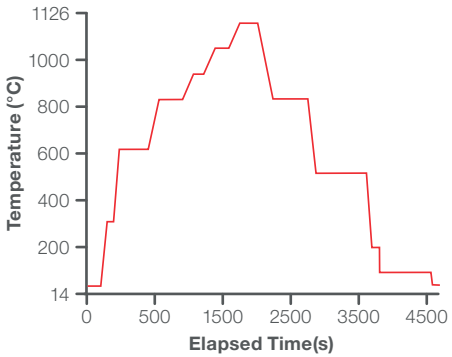
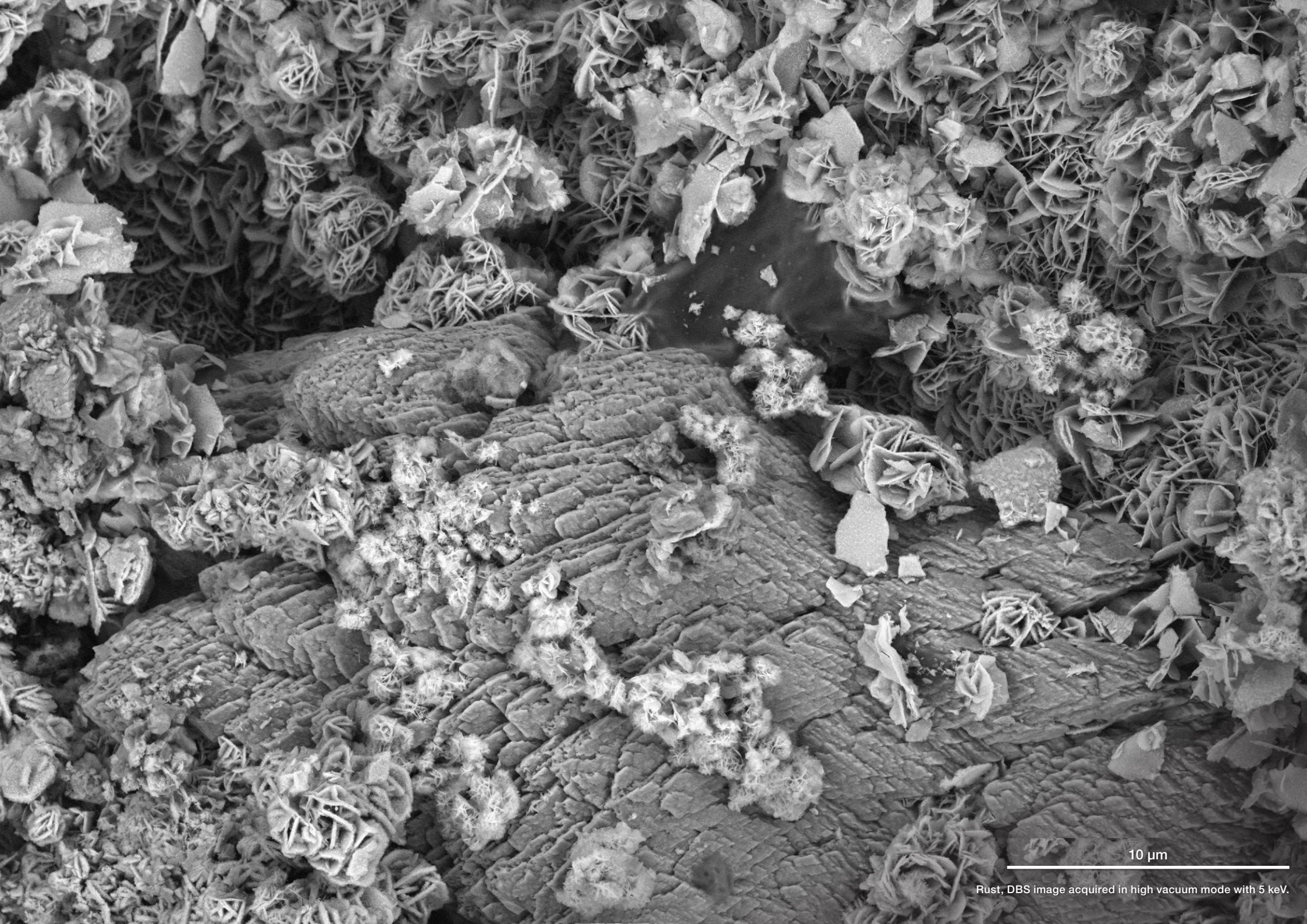


Figure 32. Graph showing the temperature change in time. The first half of the graph shows the heating, in steps, up to 1,100°C, while the second part of the graph is related to the cooling. The number of steps is fewer, as the cooling was more quick than the heating.

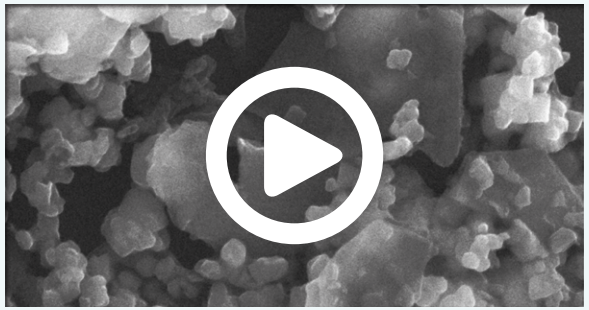
The evolution of the particles' microstructure was followed with both SE and BSE detectors, and each acquired frame was recorded and then converted to a movie.

Some of the frames acquired are shown in Figure 33. Both particle populations melted and changed, both from a microstructural and a compositional point of view. Toward the end of the process, larger particles with squared edges were formed, and they appeared to be laid on top of a uniform layer.



Rust, DBS image acquired in high vacuum mode with 5 keV.

To clarify the compositional changes achieved with the heating experiment, an EDS mapping was performed during the cooling, at 500°C. This specific temperature was chosen to run the EDS, as, at that temperature, the area of interest showed a recrystallization of the melted sample into two different populations of particles.



Controlled heating of a mix of magnetite and hematite nanoparticles. Duration 0.33

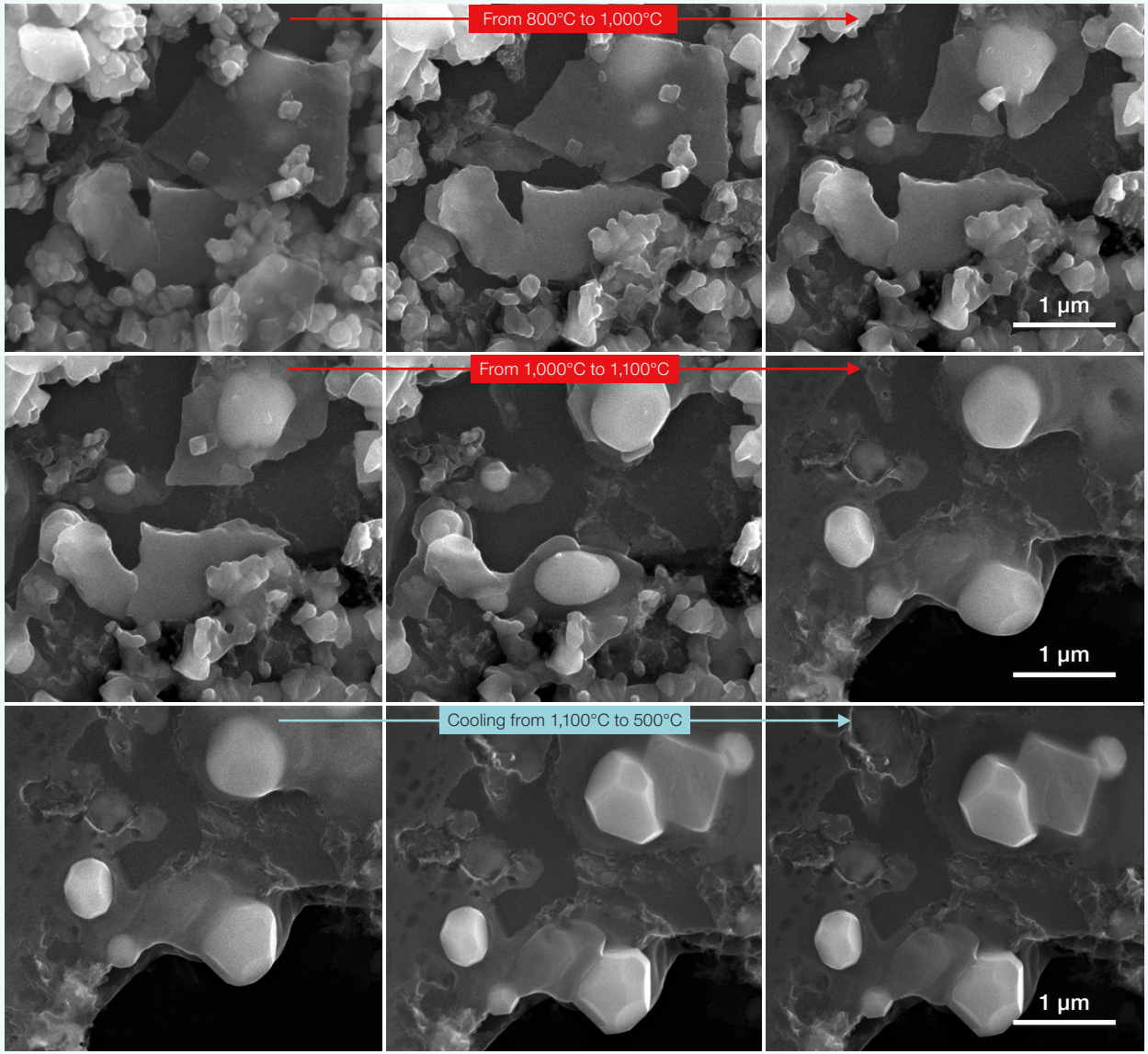


Figure 33. Microstructural changes of the area of interest during the heating and cooling processes. Acc voltage 20 keV, beam current 0.13 nA.

The ability to utilize the EDS detector and run elemental mapping at high temperature allowed for the understanding that the heating experiment led to the formation of two populations of particles, of which the brightest in the BSE image shown in Figure 34 are most likely only iron with a coating of an oxygen-rich layer. The squared particle on the top right of the image, instead, showed a much higher content of oxygen and a lower content of iron.

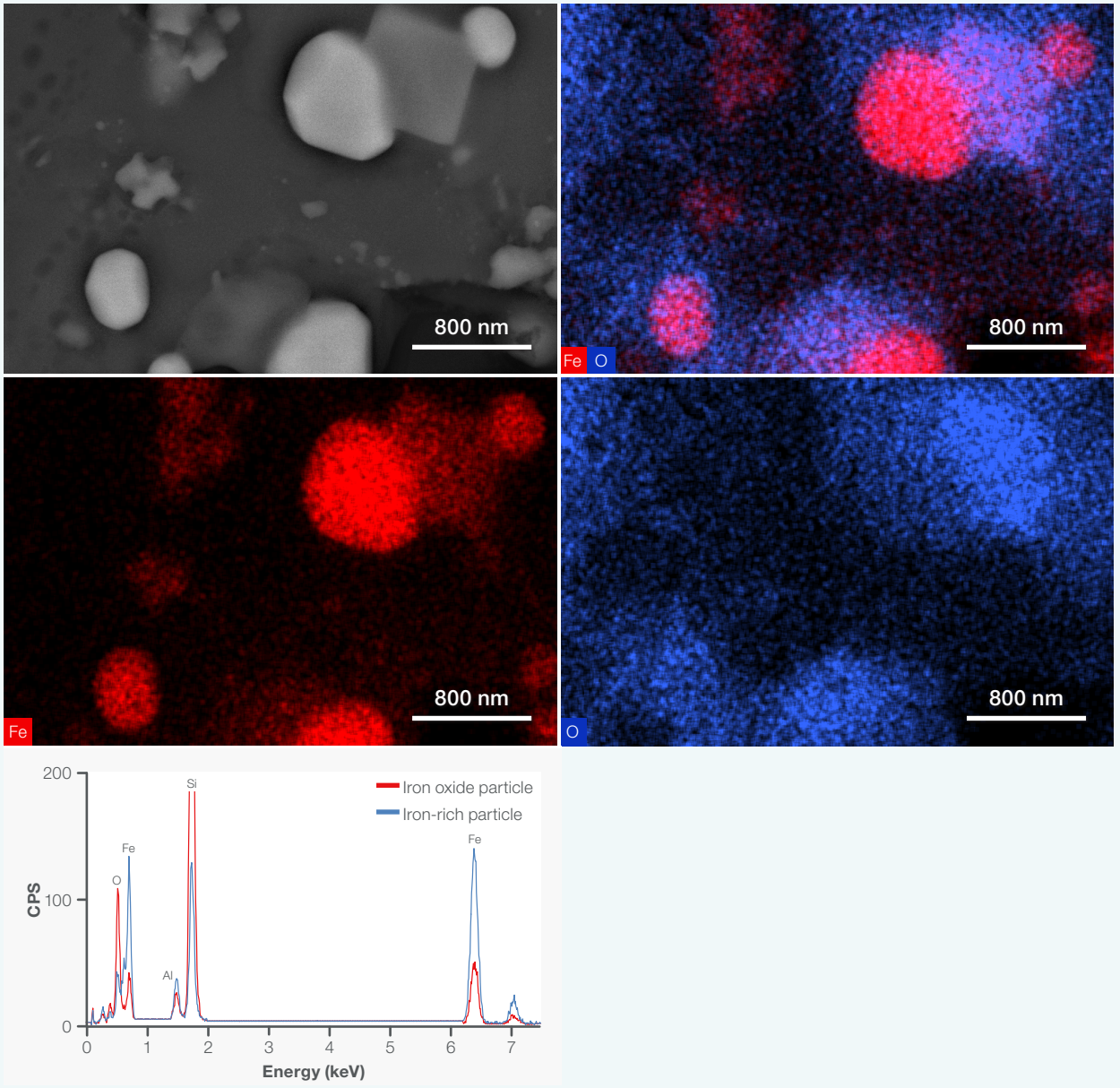


Figure 34. EDS mapping acquired on the heated area. The iron and oxygen maps show an inhomogeneous distribution of the iron in the three particles on the top right area of the image. The two spectra come from two particles showing different composition.

Advanced automation with AutoScript Software

Thermo Scientific AutoScript™ 4 Software is a Python-based application programming interface (API) that offers control of the Quattro ESEM and other Thermo Scientific systems. It opens the microscope to a world of advanced functions that can be used for powerful automation.

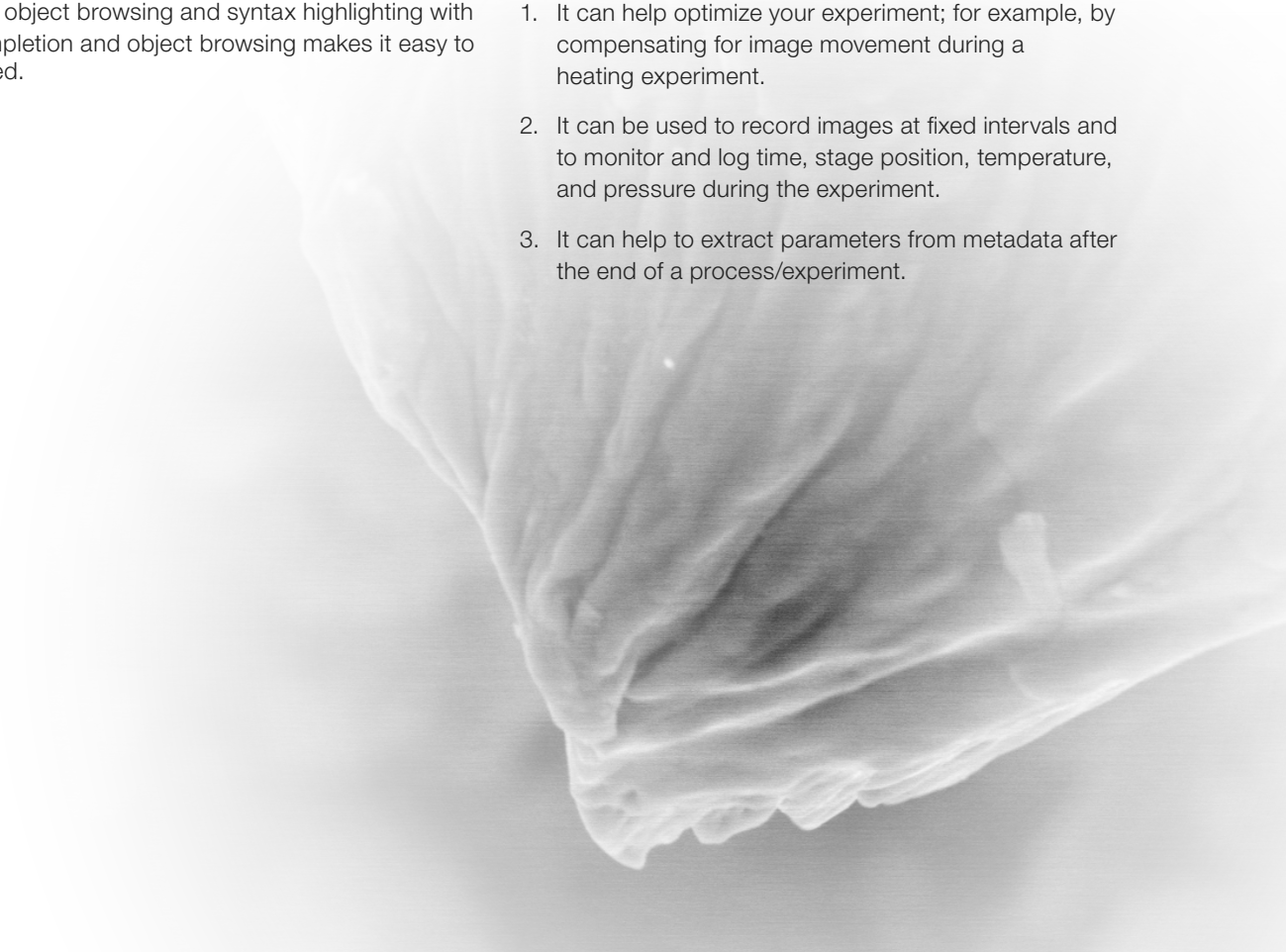
Key benefits

- AutoScript Software provides access to new possibilities for acquisition, analysis, interfacing, imaging, patterning, and data display that were previously inaccessible to manual operators.
- Scripting of repetitive or tedious tasks leads to greatly improved reproducibility and accuracy for higher quality results.
- Unattended, high-throughput imaging and patterning make more effective use of your time and of SEM time.
- Supported by Python 3.x-based scripting environment. Python, the most popular programming language available, and the standard in scientific computing, provides access to a vast collection of pre-installed libraries for data analysis, data visualization, image processing, documentation, and machine learning.
- An integrated development environment (IDE) that supports object browsing and syntax highlighting with auto completion and object browsing makes it easy to get started.

AutoScript Software for *in situ* experiments

AutoScript Software is particularly beneficial for *in situ* experiments, either heating or cooling, especially when it comes to data recording and analysis or even process optimization. Below are some examples of its capabilities, applied to dynamic experiments:

1. It can help optimize your experiment; for example, by compensating for image movement during a heating experiment.
2. It can be used to record images at fixed intervals and to monitor and log time, stage position, temperature, and pressure during the experiment.
3. It can help to extract parameters from metadata after the end of a process/experiment.



ESEM heating stage imaging with AutoScript 4 Software

The following shows an example of the previously mentioned advantages applied to a heating experiment of a silver wire. The 1,000°C ESEM heating stage was used for the experiment; a silver wire was heated from 310°C to 522°C to understand the material behavior and change during melting. A series of secondary electron (SE) images (Figure 36) was automatically captured during heating. (The function is available within the microscope user interface and is extremely useful in cases of dynamic experiments, allowing you to focus on the experiment rather than collecting the images.) The acquisition of an entire set of similar images additionally provides a complete set of metadata embedded in them that will provide information about temperature, pressure, and humidity changes.

In this specific application example, the most important information to be extracted is the beam shift or stage movements. Environment changes generated sample movements, and the area of interest was not always in the same position. The generation of a movie from the acquired set of SE images would have generated a series of frames with a non-centered area of interest. It also would have posed a risk of losing the area of interest. Thanks to a Python script, the feature of interest was tracked down, and live drift compensation was applied through a combination of beam shift and stage moves. The result was a stable image of the silver wire for the entire duration of the movie, even when the wire changed location over the substrate.

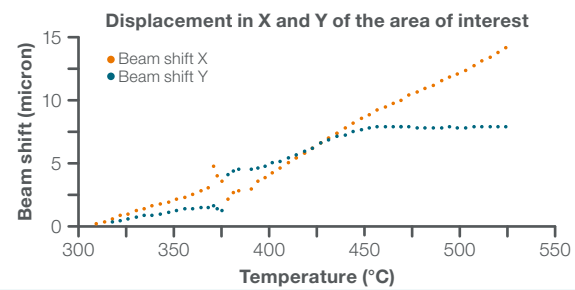


Figure 35. Experiment's temperature profile (°C) versus the elapsed time.

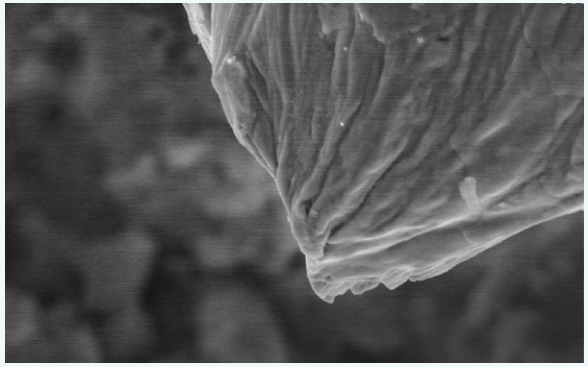


Figure 36. SE image captured during the heating of the silver wire.

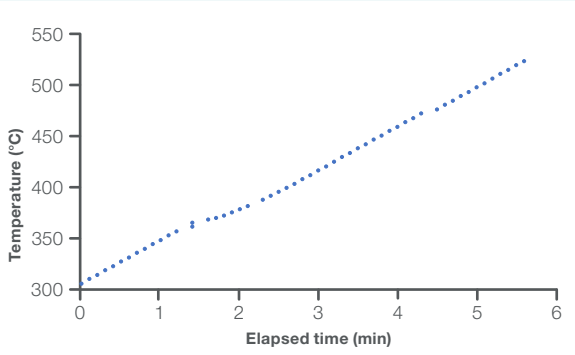
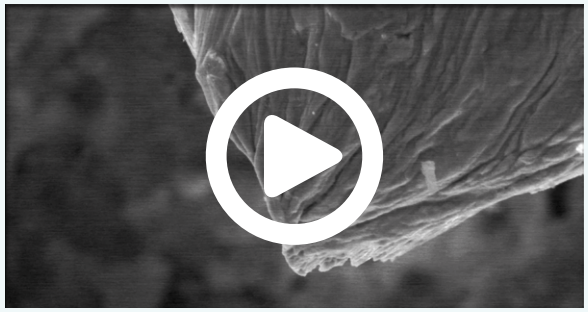


Figure 37. Experiment's temperature profile (°C) versus the elapsed time.



ESEM heating stage imaging with AutoScript 4 Software. Duration 0.08

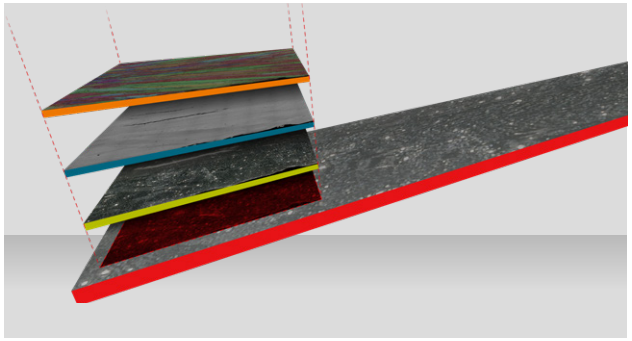
Maps Software

Thermo Scientific Maps™ Software is an intuitive automation and correlative workflow software suite for Thermo Scientific SEM, DualBeam™, and TEM platforms. Maps Software offers distinctive key features such as the ability to automate your acquisitions by running multiple samples in a series to increase system productivity or to automatically acquire up to four simultaneous signals. You can even plan to do this overnight or over a weekend. Furthermore, Maps Software offers a multi-scale, multi-layered visualization environment in which 2D and 3D data and imagery from other modalities (e.g., EDS maps and EBSD) can be imported from any source, easily and accurately correlating layers.

System automation

Maximize the productivity of your microscope by automating imaging routines overnight.

- Included with all SEM/SDB (small DualBeam) platforms
- Automation from single frames to large mosaics
- Auto functions that ensure quality imaging
- Abilty to offload routine imaging to nights and weekends



Correlative microscopy

Explore and interpret all your data efficiently while ensuring that the context of multi-modal collections is preserved.

- Any image format can be Imported and registered
- Multi-modal interpretation and navigation
- Support for 3D data import
- Workflow support for image registration

Visualization, annotation, and sharing

Perform basic visualization, even outside the office; you can also make use of a free offline viewer.

- Correlative functions with full offline version
- Annotation supported online and offline
- Measurement of angles and lines;, abilit to choose ROIs

Maps Software characterization of wood fibers in wet environment after compression test

The use of Maps Software has shown to be key in obtaining a clear overview of most of the sample of interest while running the same experiment with no need to run the experiment multiple times, focusing each time on different areas. The ability to tile and stitch several images can be extremely beneficial, specifically when topographical changes due to the experiment need to be monitored on a large sample surface.

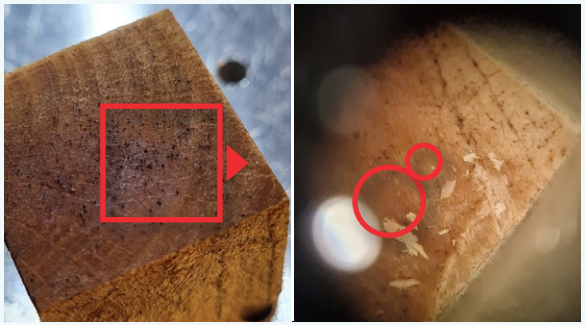


Figure 38. Wood sample. The highlighted fragments have been selected to be characterized.

The material of interest for this characterization is a block of wet wood (Figure 38) that underwent a compression test. Prior to the experiment, the sample was stored in a plastic bag, which, due to the high humidity and condensation, allowed the growth of black and white mold fibers. Two small pieces of wood (the two selected pieces are shown in Figure 38, inside red circles) have been carved out of the block using a scalpel and fixed to the Peltier stub using carbon tape.

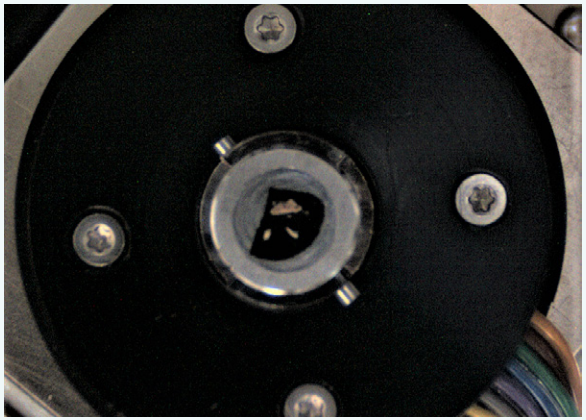


Figure 39. Navigation camera view of the Peltier cooling stage showing the wood fragments at the center of the stage.

A view of the sample's fragments mounted onto the Peltier cooling stage is shown in Figure 39. At the beginning of the experiment, the chamber was pumped down directly to ESEM mode, and the temperature of the stage was kept at 2°C. Two different tile sets (Figure 40) were acquired in ESEM mode. For the entire duration of the Maps Software data acquisition (one tile set was acquired in 15 minutes, while the second was acquired in 11 minutes), the relative humidity (RH) was kept at around 100% (700 Pa) to maintain sample humidity and its starting condition. This allowed the effect of the compression test run before the imaging to be monitored.

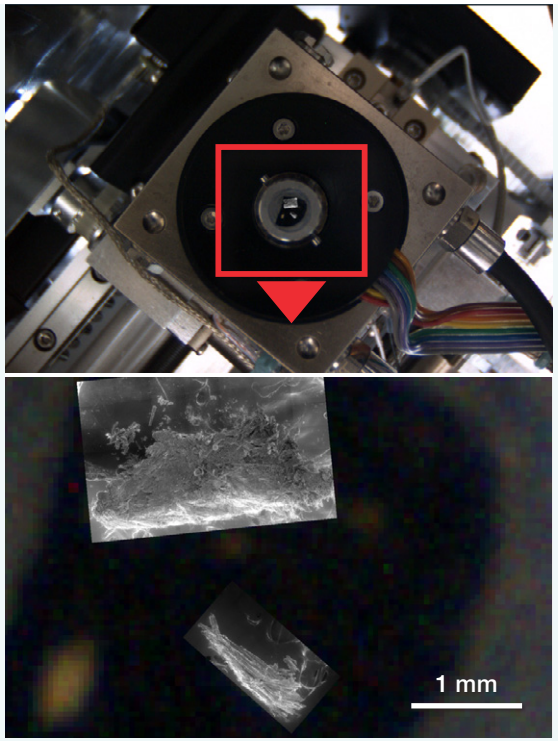


Figure 40. View of Maps Software's user interface showing the presence of two different datasets acquired on each fragment. The image on the bottom shows the overlay of the navigation camera image and the two acquired tile sets.

The Maps Software dataset allowed for inspection of the entire surface of the fragments and was used to navigate the sample to select areas of interest for further higher magnification analyses.

Further higher magnification imaging was conducted while maintaining the system at 2°C with an RH of 100%. Both characterized fragments show the effect of the hydration, as water droplets are visible in Figure 41 and water-coated mycelium is visible from the second fragment (Figure 42).

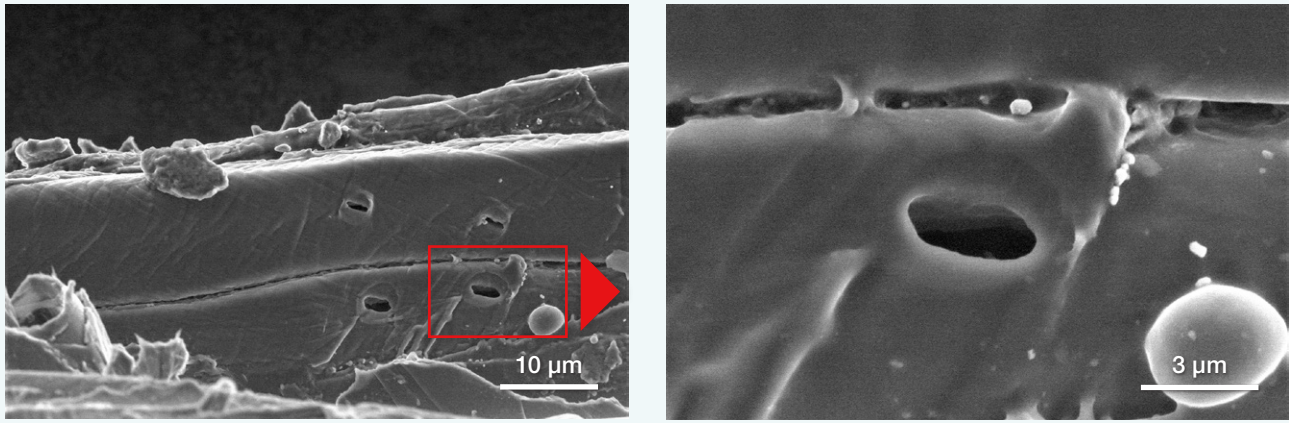


Figure 41. SE images from one of the two fragments, acquired on the fully hydrated sample (pressure 700 Pa, RH around 100%). The right image shows the presence of a water droplet. ***Sample courtesy of Technische Universität Dortmund, Werkstoffe des Bauwesens, Germany***

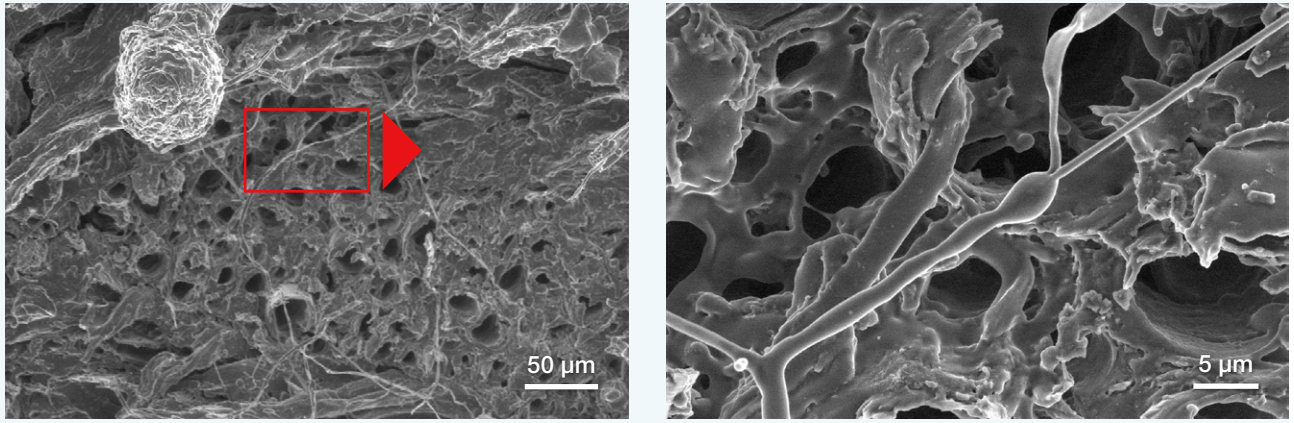
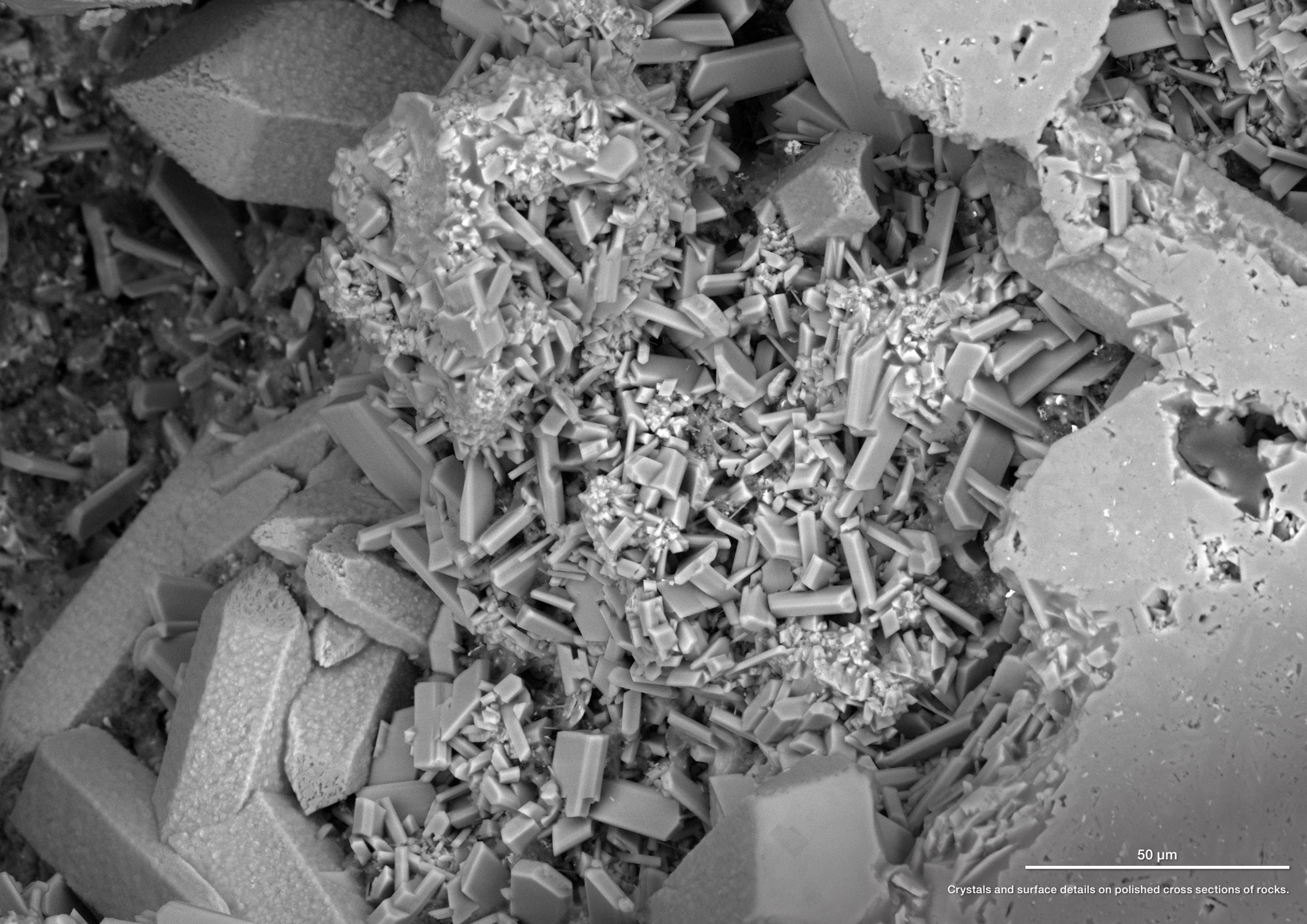


Figure 42. SE images from the second fragment, acquired on the fully hydrated sample (pressure 700 Pa, RH around 100%). The right image highlights a water-coated mycelium.



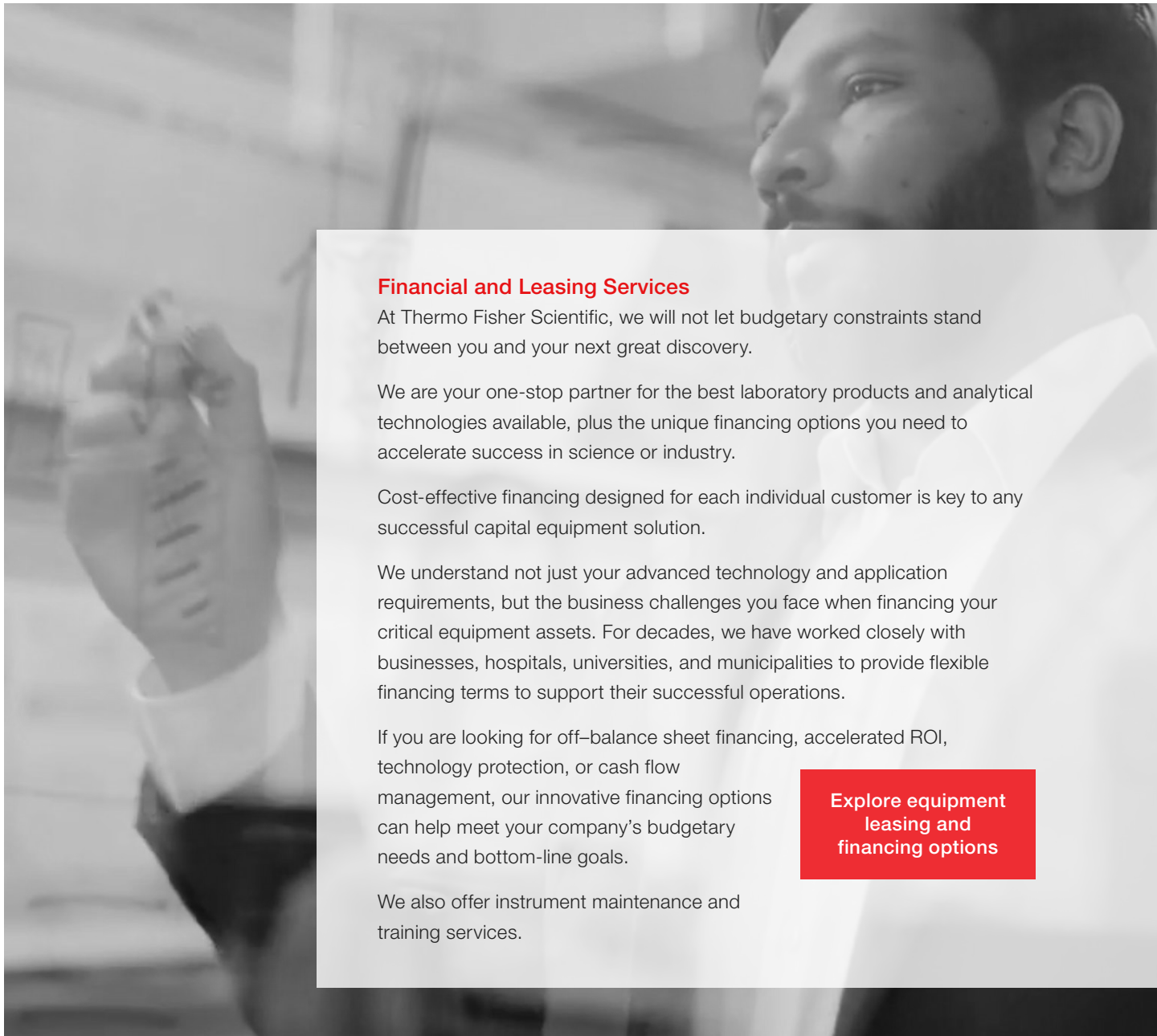
Crystals and surface details on polished cross sections of rocks.

About Thermo Fisher Scientific

We are the world leader in serving science. Our Mission is to enable our customers to make the world healthier, cleaner and safer.

Step ahead. Step beyond. Duration 1.33

Our innovative solutions for electron microscopy, surface analysis, and microanalysis help materials science researchers advance their sample characterization to gain deeper insight into the physical and chemical properties of materials from the macroscale to the nanoscale. Our multiscale, multimodal solutions cover a broad range of applications across dozens of industries and research fields, serving customers in academia, government, and industry. Our TEMs, DualBeam™ FIB-SEMs, comprehensive portfolio of SEMs, XPS, and microanalysis solutions, combined with software suites, take customers from questions to usable data by combining high-resolution imaging with physical, chemical, elemental, mechanical, and electrical analysis across scales and modes.



Financial and Leasing Services

At Thermo Fisher Scientific, we will not let budgetary constraints stand between you and your next great discovery.

We are your one-stop partner for the best laboratory products and analytical technologies available, plus the unique financing options you need to accelerate success in science or industry.

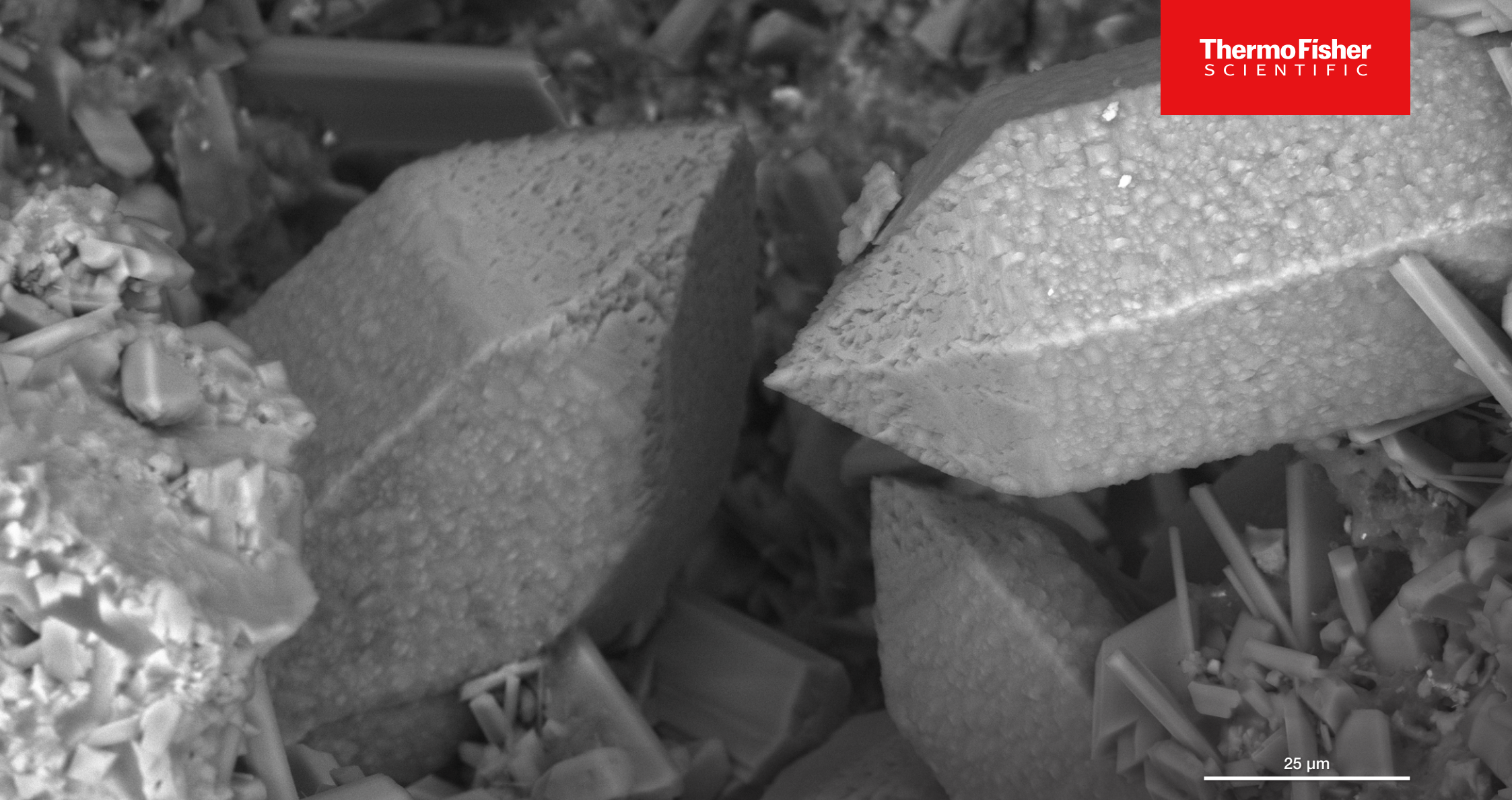
Cost-effective financing designed for each individual customer is key to any successful capital equipment solution.

We understand not just your advanced technology and application requirements, but the business challenges you face when financing your critical equipment assets. For decades, we have worked closely with businesses, hospitals, universities, and municipalities to provide flexible financing terms to support their successful operations.

If you are looking for off–balance sheet financing, accelerated ROI, technology protection, or cash flow management, our innovative financing options can help meet your company's budgetary needs and bottom-line goals.

Explore equipment leasing and financing options

We also offer instrument maintenance and training services.



Crystals and surface details on polished cross sections of rocks.

Learn more at **thermofisher.com/quattro-esem**

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