COVALENT METROLOGY

eBook

Scanning Electron Microscope (SEM) Optimization & Analysis

Key considerations and steps to optimize your SEM images

Covalent Metrology, SEM and FIB Analysis Group

A Scanning Electron Microscope (SEM) is a powerful magnification tool that produces high-resolution, three-dimensional images and provides topographical, morphological, and compositional data.

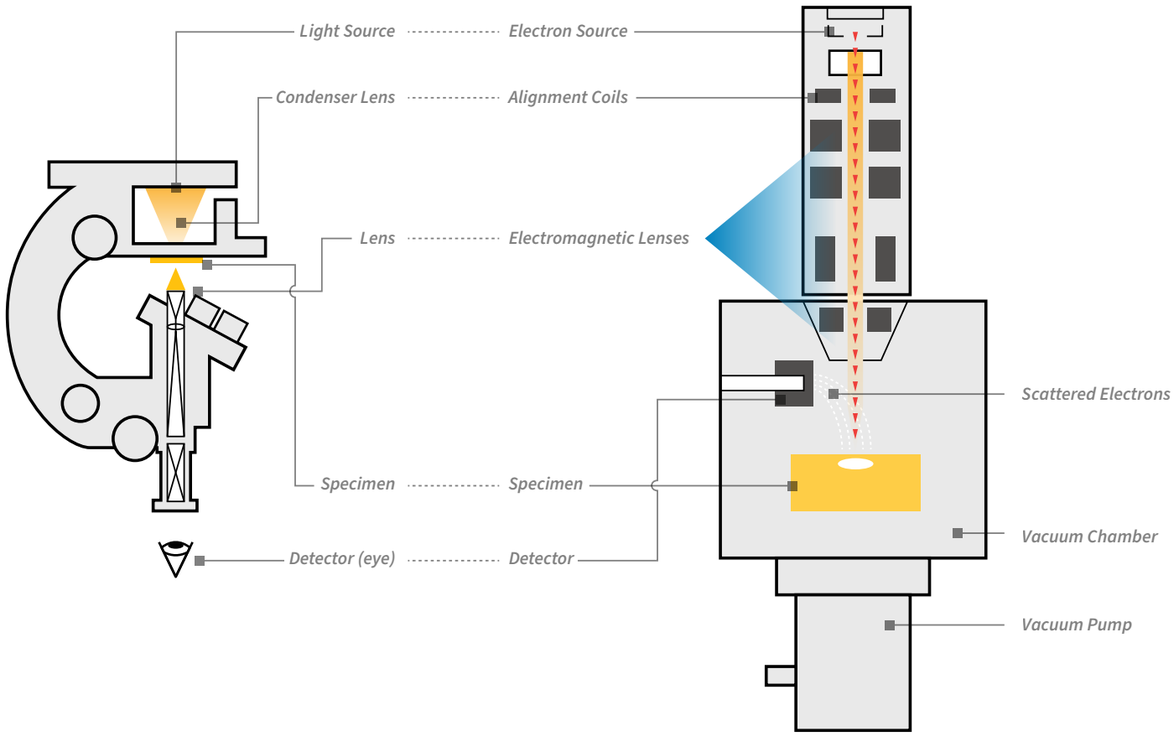
SEMs are invaluable in various industries and applications, such as material science, failure analysis, microelectronics, semiconductors, medical devices, general manufacturing, etc.

SEMs come in a range of sizes, ranging from small benchtop instruments that are more affordable while providing basic capabilities of an SEM to more advanced and larger SEMs that contain expanded features and detectors for refinable and precise measurements.

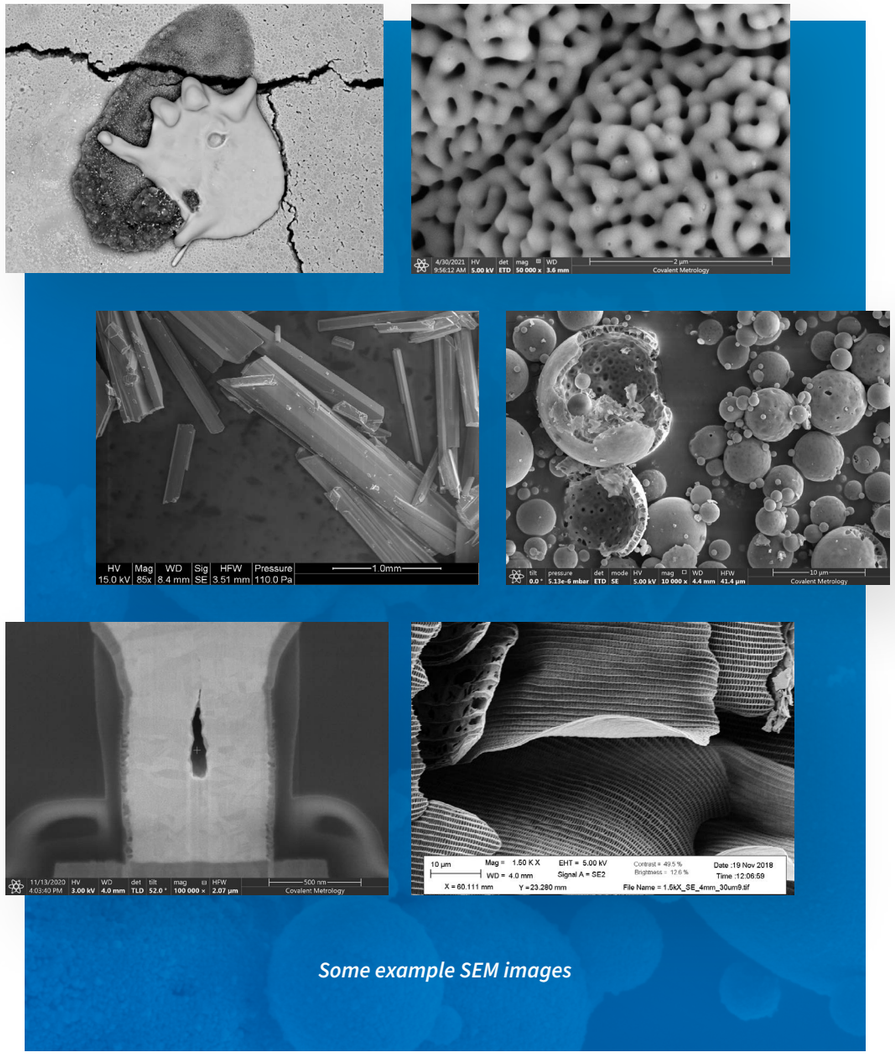
This ebook will cover key considerations when approaching an SEM experiment. A typical SEM experiment will require several decisions, including selecting the instrument and the detector(s) and making changes to refine the measurement. This ebook will help you gain great insights into SEM and feel confident in your future SEM experimental design.

Instrument setup

Regardless of size or customizations, the basic setup of SEM instruments comprises an enclosed column under a vacuum to allow electrons to travel without being deflected by particles in the atmosphere. At the top of the column is a beam source that produces a steady stream of electrons that travel further down the column. As the electrons travel down the column, they pass through a series of coils and lenses, which focus the bulk electron beam into a refined point beam. This refined beam then enters the chamber and contacts the sample mounted to a moveable stage, and signals scatter from the sample in various directions. When the beam interacts with the sample, multiple types of emissions are produced, each signal carrying different information. There are primary beam electrons that can travel up to 100's of nanometers into the sample before interacting with atoms and being returned back up the column as high-energy backscattered electrons (BSE). Other primary electrons interact with the electrons of the sample's atoms, exciting lower energy secondary electrons (SE), which can escape from the top few nanometers of the sample before being detected. As the signals return either towards the beam origin or scatter around the chamber, they are picked up by detectors located within the instrument. Although most SEMs have detectors to pick up both BSEs and SEs, larger equipment has multiple options for BSE or SE detectors and capabilities to collect and analyze other secondary signals.



Comparison between traditional microscope and Scanning Electron Microscope (SEM) designs from Covalent Academy episode 18



Some example SEM images

Data Collection and Processing

During measurement, the beam rasters across the sample's surface, generating signals at resting on each 'pixel' of the sample. The collected signals are then collected by the detector(s), processed, and transformed into a grayscale image that the user can view and interpret.

SEM Applications in Research and Technology

Due to the versatility of large-scale SEMs and the possibility to perform both topographic and compositional measurements, SEMs are used in a wide array of fields with increasingly diverse applications across different disciplines and sectors.

SEM technology can help with process and quality control in industrial settings by allowing automated multi-scale measurements to ensure high-quality processes and results. Many researchers who work on fundamental material research or novel material research use SEM to glean key insights into the material properties at the micro and nano-scale. A more specific field where SEM is extensively used is semiconductors development. SEMs are used to help guide research directions for semiconductors by analyzing yield and failure points for newly developed semiconductors.

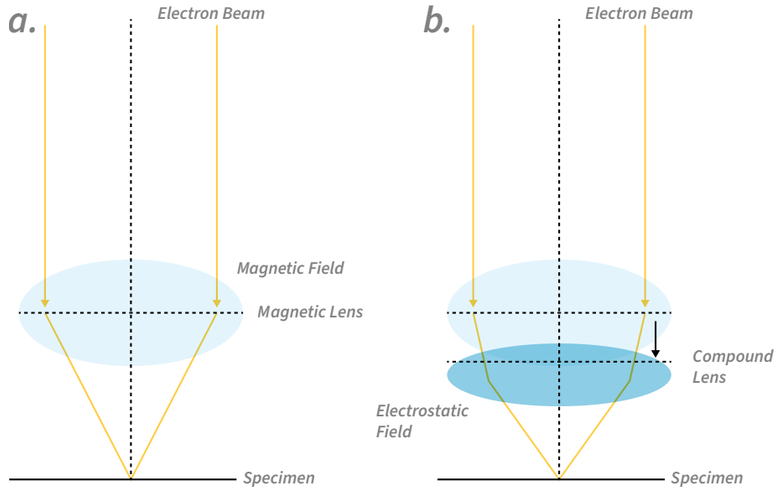
More generally, SEMs can be used when investigations of the surface topology or composition are needed, including research on batteries, semiconductors, polymers, biological samples, metals, crystals, adhesives, fossils, corrosion, fibers, composites, and much more.

Approaching an SEM instrument

Although the fundamentals of SEM operation are practically the same from instrument to instrument, some settings will significantly impact the quality of the measurements and should be appropriately adjusted to achieve the best possible results and images. When choosing among multiple SEMs, it is important to consider accelerating voltage, beam current, detectors, and samples/stage position. These factors will be the most significant variables affecting the measurements and must be duly refined to optimize the final results.

To illustrate some of these differences, we can use the example of two SEM instruments available at Covalent Metrology: the Thermo Scientific Helios 5 DualBeam, and the Thermo Scientific Scios DualBeam. While these two instruments are fairly similar and many samples can be run interchangeably between them, many differences can drastically affect the measurement and determine the instrument to be used. The biggest difference between the two instruments lies in the objective lens. As touched on above, the electron beam passes through a series of lenses ending in an objective lens which focuses the refined beam to a small point. This point beam is then swept across the sample to collect data. In the Helios 5, the objective lens is magnetic, while in the Scios, the objective lens is electrostatic. Both objective lenses narrow the beam and allow precise data collection, but there are tradeoffs in the lens type.

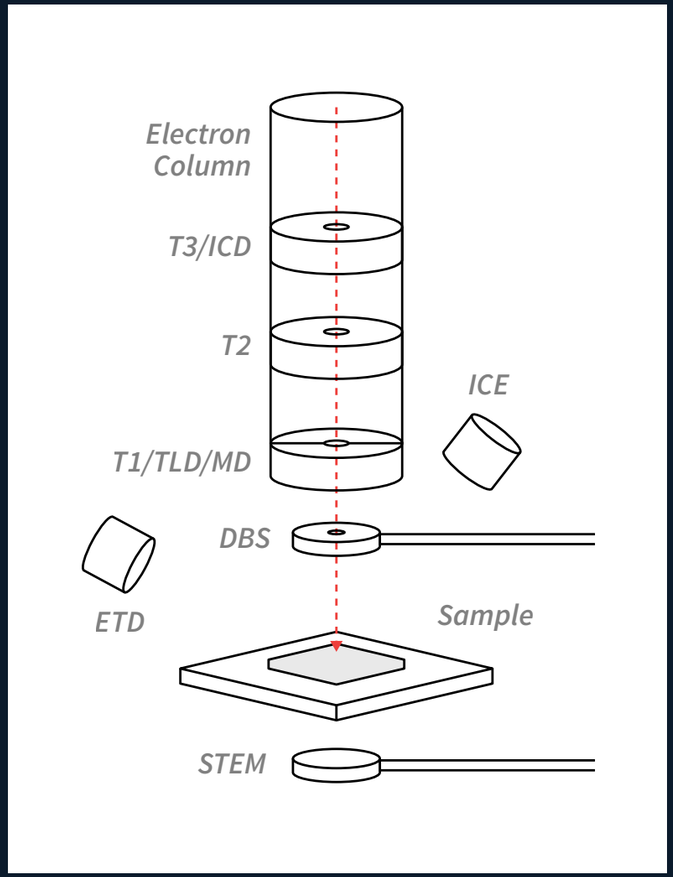
The magnetic objective lens allows for very high resolution, but creates difficulty in handling magnetic samples, sometimes lifting magnetic samples from the stage. In contrast, the electrostatic objective lens in the Scios provides slightly lower resolution at lower energies (although it can still reach similar resolutions at high voltages) but can handle magnetic samples with ease while allowing more consistent focusing. The two instruments also work in slightly different current and accelerating voltage ranges, both of which can be refined to optimize samples in slightly different ways. These instruments are also outfitted with a few different detectors. There are some generic SEM detectors, such as the Everhart-Thornley Detector, and some Thermo Scientific specific detectors, such as ICE, which appear on both instruments, but there are instrument-specific detectors as well, such as the Trinity System on the Scios.



Difference of the electron probe on the specimen for the magnetic objective lens and for the electrostatic/ electromagnetic field superposed objective lens.

a. Lens action only for the magnetic lens. The electron beam is focused onto the specimen only by the action of the magnetic lens.

b. Lens action for the electrostatic/electromagnetic field superposed lens. An electrostatic field is generated at the bottom of the magnetic lens and thus, the electron beam is further focused. As a result, the focal length is shortened and a small probe is produced while the same working distance (WD) is maintained.



Overlaid detector position schematics of both scios and helios from Covalent Academy episode 18

Detectors Overview

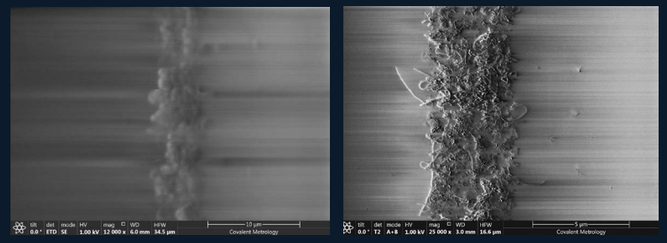
Different detectors collect signals differently; therefore, the type of detector(s) that the instrument is equipped with will determine the measurements and information provided by the instrument. Choosing a detector can be daunting, but a good understanding of the desired information can considerably narrow down the number of available options. For instance, backscattered electron (BSE) detectors are better suited to compositional investigations, while secondary electron (SE) detectors are better suited to topological and microstructural information. Suppose the research goal is unknown or a general sample survey is wanted. In that case, using multiple detectors or detector systems, such as the Trinity Detection system, can allow for a wide swath of information to be collected at once.

Electric Charge Optimization

When running SEM samples, charge adjustment is a common way to refine and optimize samples. Applying electric bias to specific areas of the sample, stage, detectors, or column can change how signals are received. Excess charges in unwanted areas can affect the resulting image quality.

Charge Refinement

SEs are often low energy and require detectors located in the bulk of the chamber to collect the signal. Applying an electric field to the stage, wafer, or sample can improve the efficiency of the SE signal and push SEs to work their way back up through the column. This allows the user to reduce the working distance between the objective lens and the sample, drastically increasing the resolution and contrast.



Left: A sample without charge refinement

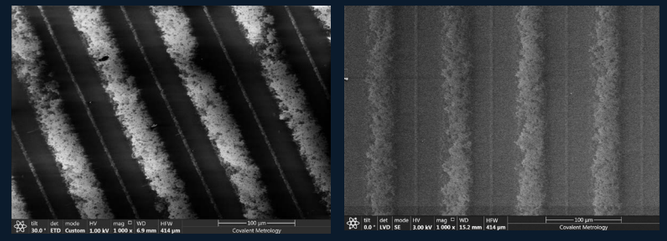
Right: A sample with a retarding field and reduced WD

Charge Mitigation

While there are benefits to adding extra charges to the stage or the sample to improve contrast and signal, charge or lack of charge in a sample can create troubles. When the electron beam scans a non-conductive sample, such as plastic, quartz, or biological materials, the sample can often pick up charges. This charge accumulation can create scanning issues or image artifacts. Typically, ideal samples are electrically conductive and electrically grounded to prevent these accumulations, but with a few adjustments, non-conductive samples can still be measured without issues. Non-conducive samples can be coated with an ultrathin conductive layer or measured using Environmental SEM or low vacuum mode.

Environmental SEM

Environmental SEM mode helps with conductivity issues through charge mitigation using water vapor. Water vapor is introduced into the chamber as the sample is measured at low pressure. If the sample begins to charge, the water helps to neutralize the charge by donating or accepting electrons from the sample. A biased positive low voltage detector then picks up these ionized water molecules. This technique allows non-conductive samples to be imaged without charging the sample and creating image artifacts. The refined image usually has lower contrast and magnification, but the image is more stable, and the sample does not change during the scan.



Left: A sample without charge mitigation

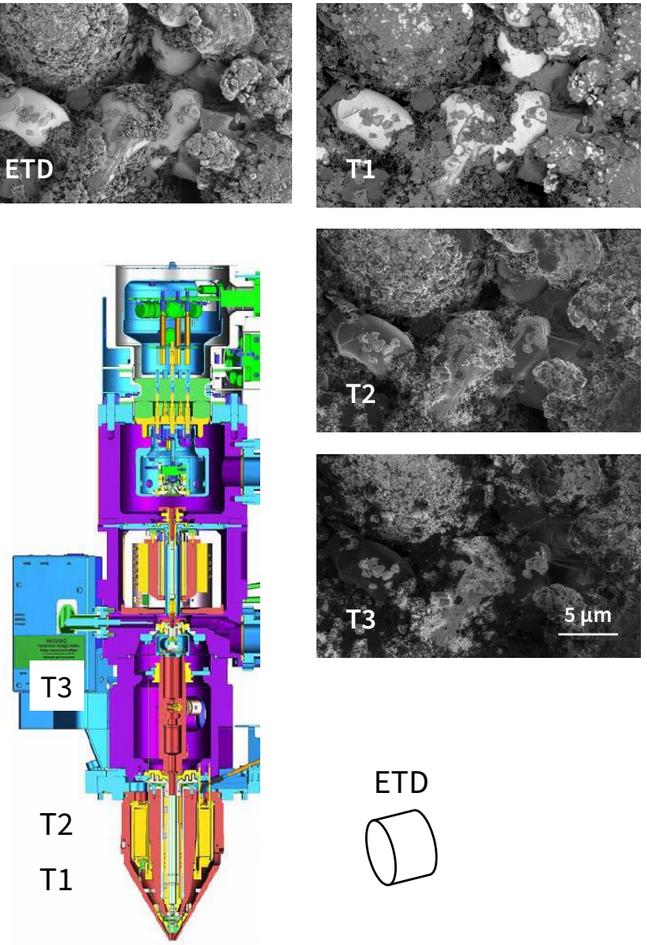
Right: An environmental SEM sample with charge mitigation

Multi-angle Single-shot: Scios 2's Trinity Detectors

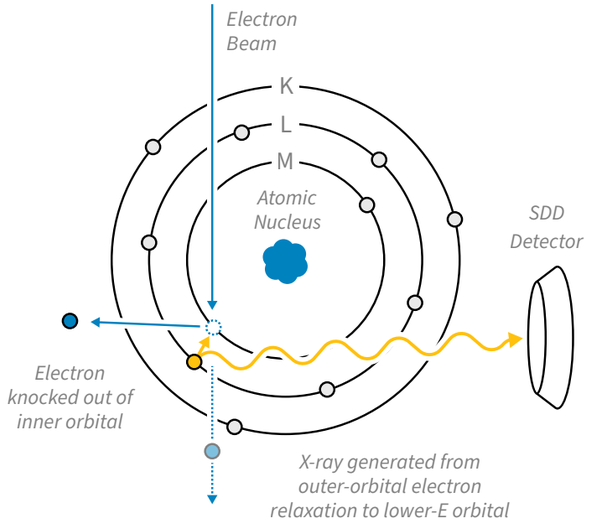
When approaching a new system, there is often uncertainty in the specific zones or focus for running samples. Choosing a particular detector or instrument setting can be challenging when working with restricted samples. But with a few of the multi-detector systems in these SEM instruments, a general survey can be conducted quickly and efficiently. A detector system addressed above is the Trinity Detector system within the Scios DualBeam, which includes three separate detectors that can collect both secondary and backscattered electrons and operate simultaneously. This simultaneous multi-detector system is ideal for general investigations, such as in this Li+ Battery case study.

Understanding the topography and the surface make-up of the Lithium-Ion cathode can help researchers understand the batteries they are making and testing. There is a wide range of data that is helpful to gather and understand. When approaching this measurement, it was important to collect both BSE and SE and take scans with different instrument settings. Using the Scios DualBeam Trinity systems, simultaneous measurements can be taken with the ETD, T1, T2, and T3 detectors, allowing researchers to collect BSE signals with one detector and SE signals with the other three all at the same time.

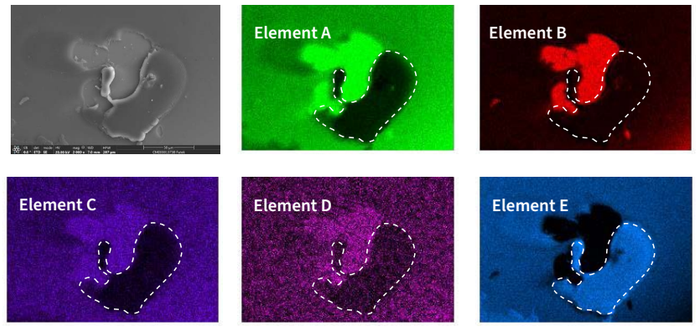
In looking at the images, we see that each detector took the measurements that differ significantly from one another. The Everhart-Thornley in chamber detector (ETD) picks up SE signals and provides a clear look at the topography and surface structures, creating a solid base of understanding. The T1 detector is a backscattering detector, and the BSE image shows high sensitivity to differences in atomic number. The higher the atomic number, the brighter the material appears in the image. The T2 and T3 detectors are both tuned to be SE detectors, with T2 supplying a high-resolution image for smaller-scale investigation and T3 tuned to low-energy SEs, revealing surface features that are not visible in the other images.



Information from every angle captured in a single scan



Principle of EDS



Original SEM Image & Compositional Mapping

EDS Analysis of Defect

Many researchers use SEMs to investigate defects or contamination in a sample, which is a task well suited to this method. If the defect is physical, for example, a scuff, scrape, or scab, then topological insights can help identify the extent of the defect, but SEM can also identify the elemental makeup of a defect. This case study will examine a sample containing an atomically and topologically defined scab. This analysis can be done using BSE detectors or energy dispersive spectroscopy (EDS). When using BSE detectors, the resulting image gives an idea of where the heavier atoms lie but doesn't identify their elements. EDS can pick up specific spectra and create images mapping each identified element separately.

Topographical images were collected using traditional SEM methods, and then EDS was used to collect elemental mapping. While the electron beam focuses on the sample and interacts with atoms, it can knock out electrons of the inner orbitals, which causes outer orbital electrons to drop down to fill the gaps. This orbital change generates an x-ray with a characteristic wavelength picked up by a silicon drift detector, which records the wavelength and the signal intensity to create images similar to traditional SEM measurements.

This method can then be used to understand the specific elements making up the sample and the compositional mapping of the sample's surface. Compositional mapping allows for the separation of the signals of each element and can produce composite images with an explicit focus on where each element occurs.

This compositional mapping shows that the scab is formed almost entirely from 'element C,' which can be found in the surrounding surface but not in the depressed area. EDS allows for a precise mapping of where each element is in the investigated region and allows for a deeper understanding of how each element makes up the sample.

SEM is an essential technique across many fields. Knowing your project needs and optimizing SEM analysis towards your goals helps solve product development problems effectively.

Tips:

1. **Instruments:** Understand strengths and weakness of each SEM instrument.
2. **Detectors**: Select the right detector for the desired information type. Use multiple detectors to gain a general understanding.
3. **Electric Charge Optimization:** Apply an electric field to improve the SE signal resolution and contrast. Make additional adjustments to measure non-conductive samples.

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LIVE VIEW

SEM analysis with Covalent Live View

Covalent Live View makes data collection more immersive.

Wherever you are in the world – you can now participate in your sample's live analysis session with a Covalent Expert. You'll see what our experts see through Covalent Live View. Experience cutting-edge instrumentation first-hand and discuss your analytical project in real-time as it unfolds.

Live View with Covalent Experts to:

- Deepen your understanding of the analysis with full context
- Ensure the critical information targets are gathered with imaging or EDS
- Collaborate with the measurement expert to optimize your metrology project in real time
- Invite your colleagues to combine measurements, analysis, and pathfinding simultaneously!

The Live View service is available with SEM analysis free of charge!

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Covalent Metrology is ready to serve you with our comprehensive platform of techniques and services, even during COVID 19. Reach out to our friendly team of experts to receive our answers to your research questions and to start a conversation about how we can help your team achieve your materials characterization goals.

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