COVALENT METROLOGY

eBook

Age of (Scanning)/Transmission Electron Microscopy

Covalent Metrology, S/TEM Analysis Group

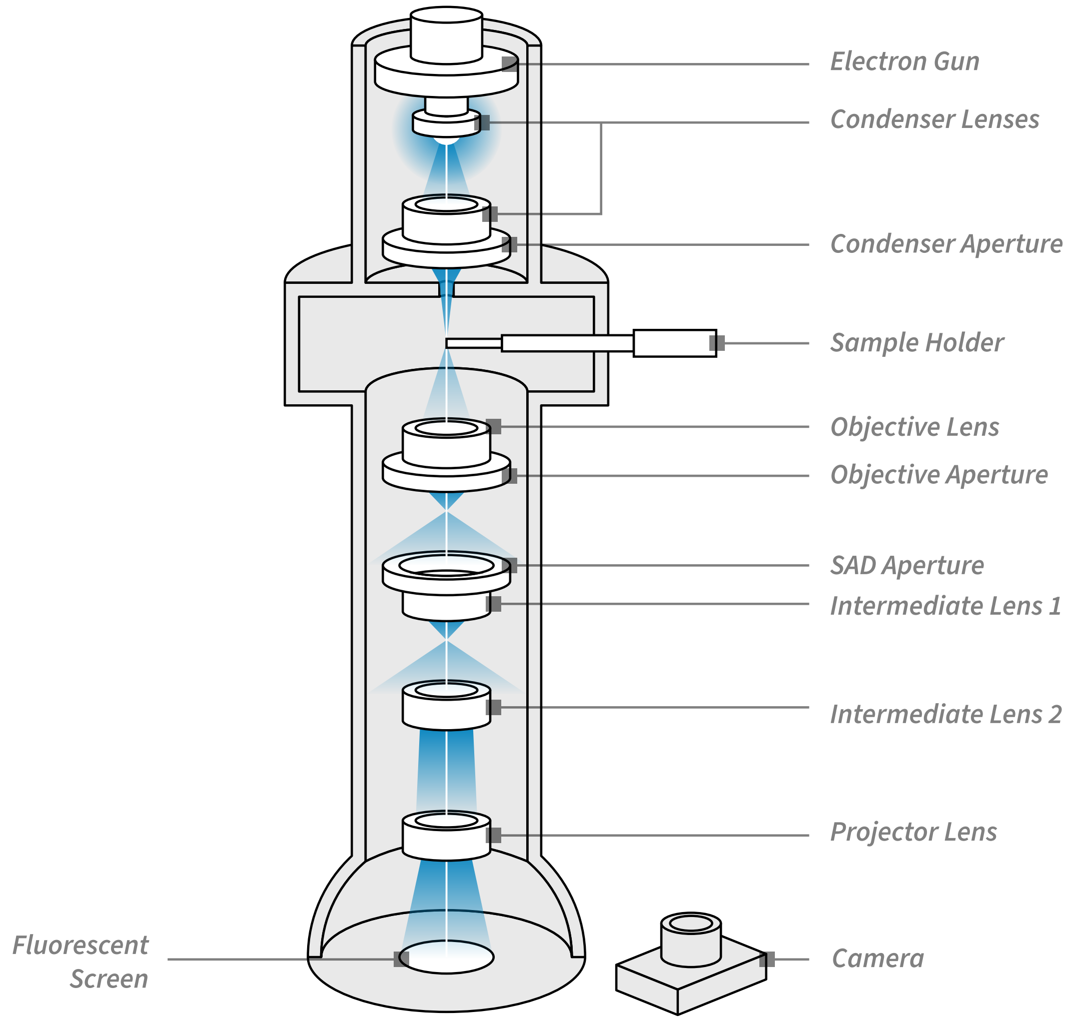
Transmission Electron Microscopy (TEM) is a powerful imaging technique capable of producing images magnified up to two million times.

TEMs can achieve atomic resolution, identifying individual atoms and enabling visual investigation of the building blocks of the sample. Alongside traditional TEM techniques, there are also multiple methods of operation, such as Scanning TEM (STEM) and other signal detection options, which allow researchers to modify the information collected and expand the scope of the data. Due to its high resolving ability and versatility, TEM has become a major analytical method in many fields, including material sciences, nanotechnology, semiconductors, batteries, biology, oncology, virology, medical research, and more.

TEM Instruments at a Glance

TEM is a high-resolution imaging technique in which a beam of electrons passes through a thin sample to produce an image. The electron beam is impacted by the sample's thickness/density, composition, and in some cases, crystallinity. This process starts at the top of the microscope's column; an electron gun produces an electron beam accelerating down the column towards the sample. In the column, condenser lenses and apertures gather the incident electron beam and focus it into a specified size and shape. In conventional TEM, the spot size of the electron beam can cover the entire sample area, whereas, in STEM, the beam is much smaller and is rastered across the entire sample surface. This electron beam then passes through and interacts with the sample fixed onto a sample holder in the main chamber. On the other side of the sample, a series of lenses and apertures focus, magnify, and refine the signals to be picked up by a camera or detection system.

TEMs can produce and interpret various signals to collect data and obtain comprehensive insight into a sample. Similar to other electron microscopy techniques, such as Scanning Electron Microscopy (SEM), the electron beam interacts with the atom and generates backscattered electrons (BSE), which provide compositional information. The electron beam also excites the sample's electrons, causing secondary electrons (SE) and releasing characteristic x-rays. SEs provide topological data, while characteristic x-rays can be detected and measured by Energy Dispersive X-ray Spectroscopy (EDS/EDX) to determine the elemental makeup. In S/TEM, the electron beam can pass through the sample, producing bright-field TEM images, or be elastically scattered from the sample and create diffraction patterns in dark-field images.



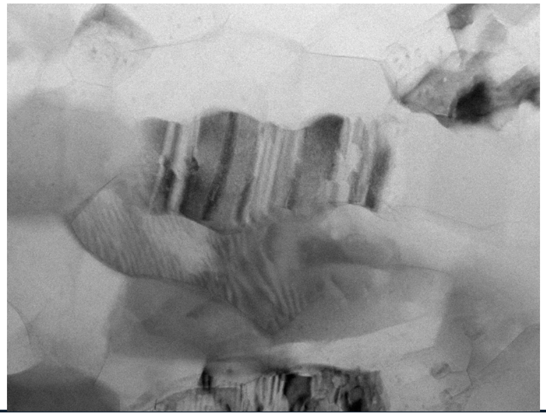
Cross section of a typical TEM instrument from Covalent Academy episode 16

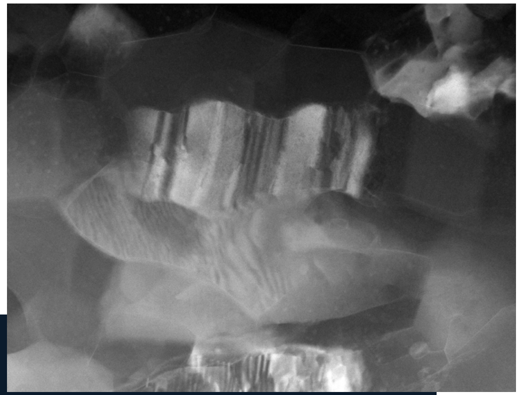
Bright-field TEM

Bright-field imaging is the first of the imaging types for TEM data collection. Bright-field refers to the contrast between the dark regions of the sample structure and the bright areas of empty or less dense space. When the electron beam passes through the sample, some electrons interact with it, scattering off into the chamber at various angles. An aperture is placed behind the sample on the back focal plane of the objective lens, filtering out any widely scattered electrons and only allowing the direct beam through. The direct beam then hits a detector which processes the intensity of electrons throughout the detected area and transforms it into an image of the sample. Sections of the samples where many electrons pass through without being scattered appear bright, showing empty spaces. Whereas sections with few electrons passing through without being scattered appear dark, showing dense, atomically complex, or crystalline regions of the sample. By displaying various intensities, shades of grey can show changes in the structure and regions where the features of interest overlap or interact. This imaging method is the most common imaging in TEM and is suitable for most structure types, providing easily understood results.

Bottom: Bright-field TEM image

Upper Right: Dark-field TEM image of same sample





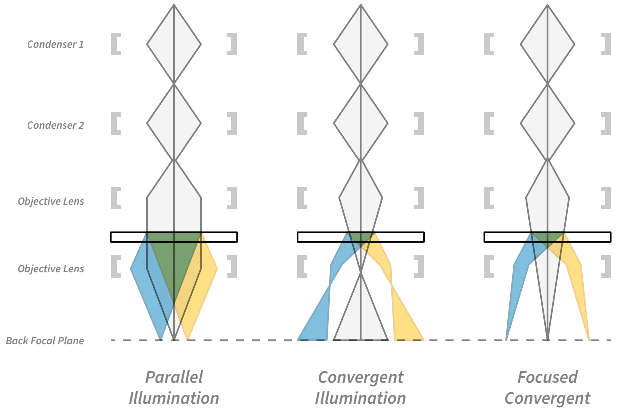
Dark-field TEM

Dark-field imaging is a contrasting method to bright-field imaging. Dark-field imaging shows the sample structures in the lighter regions, contrasting with the darker empty areas. As the electrons interact with the sample, some are deflected while others pass directly through. Dark-field imaging uses an aperture to block the direct beam while allowing one or more of the deflected beams to pass through. These deflected beams then interact with a detector which transforms the signal and produces an image. Dark-field images are read in reverse from bright-field images, with dark regions showing regions where the beam passed through the sample directly and bright regions showing the sample structure. There are also special techniques such as High Angle Annular Dark Field (HAADF), a STEM technique using a washer-like ring detector that can collect scattered electrons without picking up the main beam. HAADF improves the signal efficiency for dark-field images and enables other techniques, such as Electron Energy Loss Spectroscopy (EELS), to be performed simultaneously. When bright-field imaging cannot resolve small crystalline structures or is not clear enough, dark-field imaging can solve these problems. Multiple dark-field modes can be used to investigate small crystalline structures and produce enhanced images.

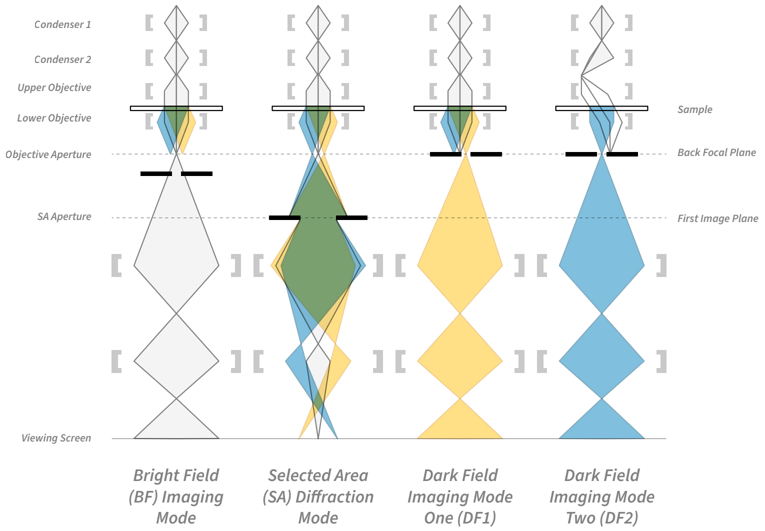
Which Settings to Use?

Understanding how images and contrast are formed can help researchers refine their experiments. The contrast in TEM images emerges from the difference in the electron densities in the image plane, which can arise from various sources. Most contrast sources fall into the amplitude contrast category, which covers contrast obtained by the loss of electrons before the image plane. The electron loss can result from beam interaction with the sample or electrons scattering at too high of an angle for the instrument to detect. A contrast from sample interaction can show mass-thickness differences, where areas with low mass or thickness will scatter less than areas with higher mass or thickness, or diffraction contrast, where crystal formations in Bragg conditions can encourage scattering. These contrast conditions can co-exist but must be interpreted carefully, with expertly selected choices of bright or dark-field technique, sample angles, and other instrument settings. The phase-contrast category includes changes in the electron wave caused by deflections and the sample's electronic or magnetic fields and can be further investigated with specialty techniques.

These imaging methods can be modified by adding and modifying apertures and lenses. Lenses and apertures between the electron gun and the sample create various illumination conditions, changing how the electrons are focused and how the main beam and scattered electrons emerge from the far side of the sample. Illumination conditions can also be combined with various imaging modes which use different apertures to gather different amounts of the main beam and scattered electrons. The combinations of illumination conditions and imaging modes allow researchers to refine the data collection process and the resulting images.



Various possible illumination conditions

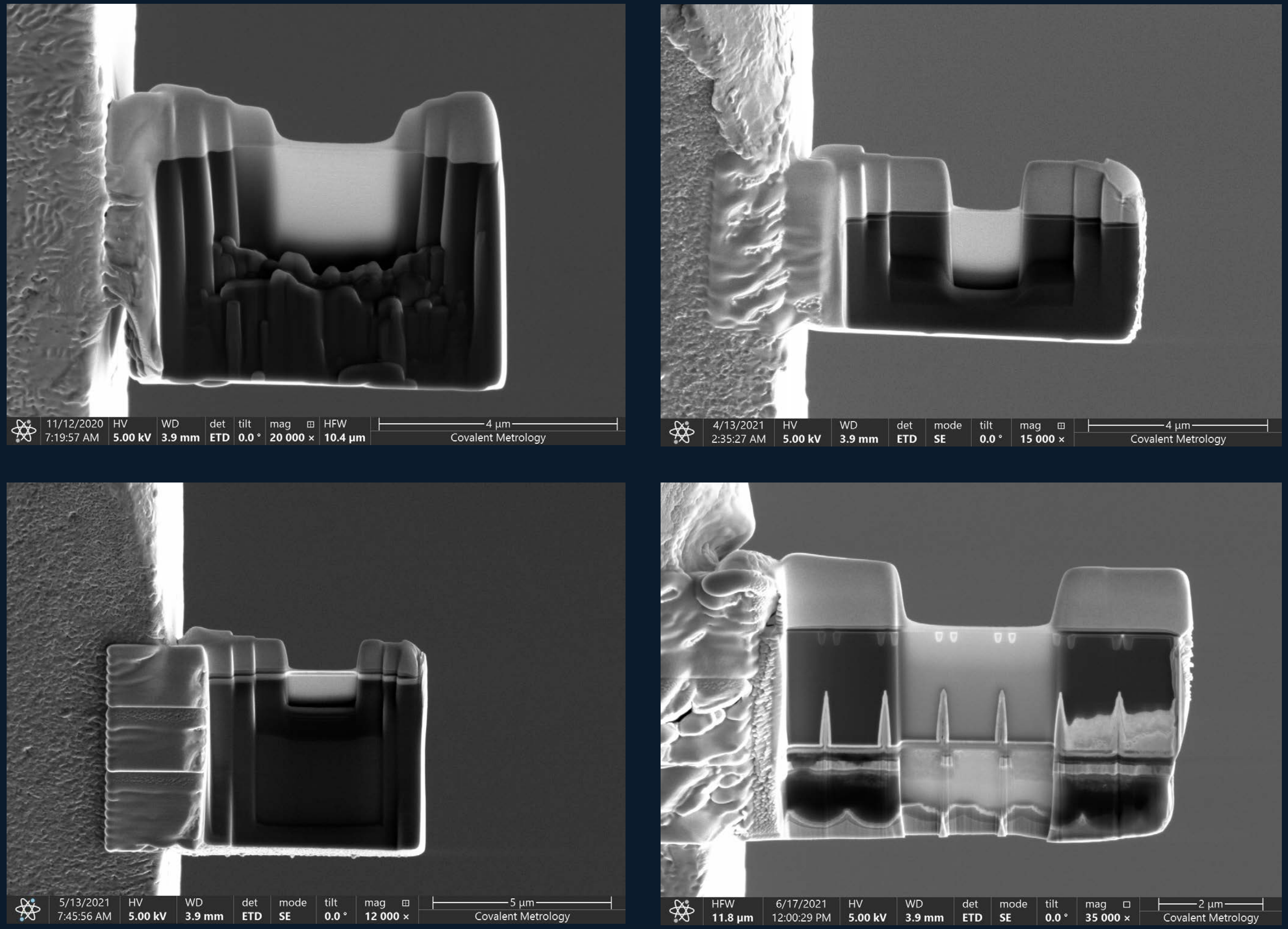


Combined with different imaging modes to produce different images

Thermo Scientific Talos F200X TEM

Covalent uses the Thermo Scientific Talos F200X TEM, designed for high productivity and reduced acquisition time while collecting high-quality data. The instrument combines high-resolution STEM and TEM imaging with state-of-the-art energy-dispersive x-ray spectroscopy (EDS) signal detection and 2D/3D chemical characterization. It is equipped with X-FEG, a high brightness electron source that produces high current beams while maintaining small convergence angles. The X-FEG reduces the signal-to-noise ratio and provides exceptional image resolution for STEM, EDS, and high-resolution TEM applications. The high-powered electron beam also allows more x-rays to be released, increasing sensitivity in x-ray signal techniques. The Super-X G2 detector features four independent symmetrical SDDs (Silicon Drift Detectors), enabling EDS 3D characterization with increased sensitivity. The Gatan OneView camera is optimized for speed versus resolution and includes live drift correction, improving the quality of the images with increased detection efficiency. The instrument also features unique absorption corrections to accurately quantify the sample regardless of the orientation and a sample chamber designed to reduce environmental impacts on the sample measurement.

TEM Line Resolution	≤ 0.10 nm
STEM HAADF Resolution	≤ 0.16 nm
TEM Information Limit	≤ 0.12 nm
Maximum Alpha Tilt	± 90˚ (tomography holder)
Maximum Diffraction Angle	24˚
Electron Source	High-Brightness Field Emission Gun
Gatan OneView CCD	16MP 4K Camera
EDS detectors	4SDD for enhanced sensitivity and detection
EDS mapping time	Pixel dwell times down to µs

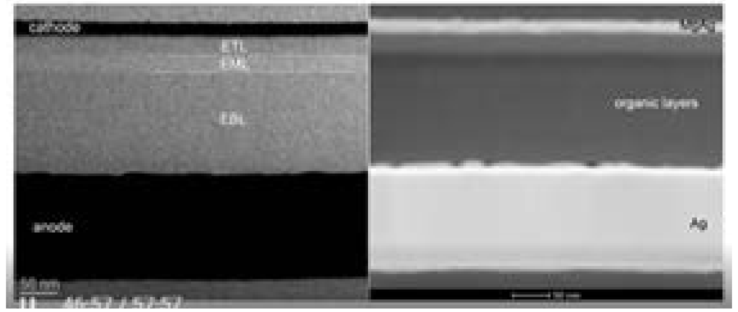


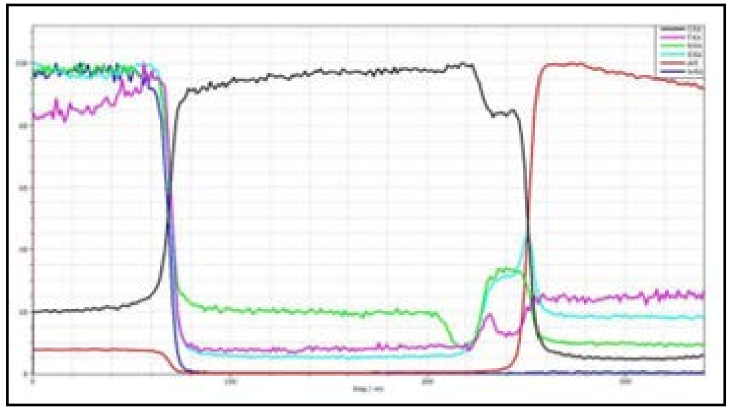
Lamella examples

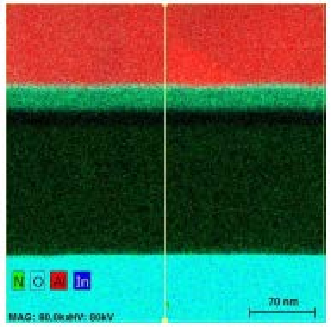
Sample Prep

TEM samples must be less than 100nm thick to allow electrons to pass through. The TEM sample preparation process has significantly improved through recent developments and paved the way for faster and better TEM imaging. Covalent experts use Focused Ion Beam - Scanning Electron Microscopes (FIB-SEM) to create lamella as thin as 20nm. During the sample preparation process, A carbon or platinum coating is applied on the top surface to protect the sample, and materials franking the area of interest are removed by FIB. The sample blade is then attached to the TEM measurement device and thinned down further to ensure electrons can pass through it. This FIB sample preparation process can be automated and fully integrated with TEM imaging. This improved sample preparation method and its seamless integration with TEM has increased TEM usage and made TEM a popular material characterization technique.

Case Study: Display Layers







Top: TEM bright-field and STEM high-angle annular dark-field images showing sections of cell phone display layers.

Middle: Line scan of Energy-dispersive X-ray Spectroscopy data.

Left: False colored images with elemental data.

To understand the process of designing a complete TEM experiment, let's consider the technical challenges of investigating the display layers of a cell phone. Cell phone screens are composed of multiple layers of various organic materials, protective materials, conductive materials, etc. TEM can provide images and elemental data to probe defects and faults in complex systems. Here we used it to understand the layered makeup of the screen.

We used both TEM bright-field and STEM high-angle annular dark-field modes to collect images to show various sections of the layers clearly. The bright-field image on the left clearly shows the cathode and the anode but is less refined when probing the organic layers. The HAADF image on the right clearly shows the density variations in the organic layers, providing information that the bright-field image wasn't showing clearly. By taking images in both modes, researchers can be confident they are imaging the whole system faithfully.

Energy-dispersive X-ray Spectroscopy data were also collected on the layers, providing the elemental composition of the probed layers. The data can be shown in two forms, the first of which is a line scan. The line scan plots the composition as a function of depth into the layer, tracking a specific element through the layers of the display and showing a clear marking of where each layer ends.

The second method of EDS depiction is through false coloring images with elemental data. This method provides a clear visual separation of the elements of concern and allows researchers to visually depict and identify defects or smearing elements out of anticipated areas.

With various imaging modes and additional techniques such as EDS, comprehensive data can be collected on complex systems, allowing researchers to use one instrument to answer challenging research questions.

TEM is a powerful imaging technique capable of achieving atomic resolution and enabling visual investigation of the sample.

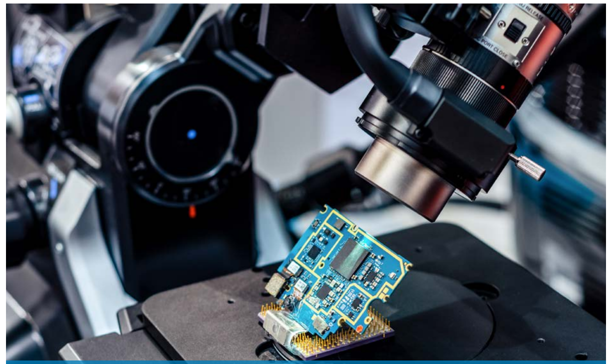
Due to its high resolving ability and versatility, TEM has become a major analytical method in many fields, including material sciences, nanotechnology, semiconductors, batteries, biology, oncology, virology, medical research, etc.

Tips:

1. **Bright-field** is the most common imaging in TEM and provides easily understood results.
2. **Dark-field is strong at identifying small crystalline structures or defects.**
3. **FIB** creates lamellas as thin as 20 nm. The improved sample preparation method allows faster and better TEM imaging.
4. With various imaging modes and additional techniques such as **EDS**, researchers can use TEM to collect comprehensive data on complex systems and answer challenging research questions.

What to Look For in a S/TEM Partner

In the world of S/TEM, it can be a challenge to find the right expertise and hands-on experience, making it difficult to find the right partner. But, when it comes right down to it, some service labs can be slow, expensive or rigid in their approach and often fall flat when it comes to providing adequate data insights. There are a few questions that should be considered if you want a long-term, reliable and strategic partner for your R&D efforts. Do they offer:

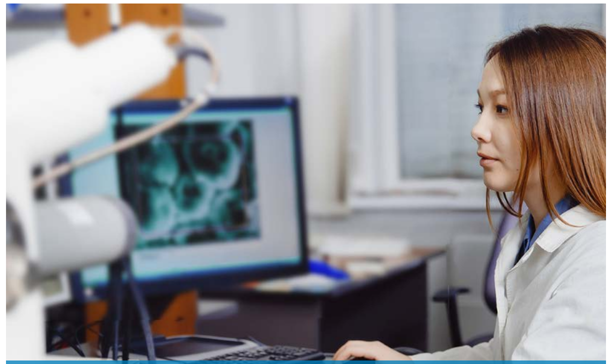


A Comprehensive Solutions Stack

With a diverse offering of techniques, instruments and expertise in advanced modeling, method development and analytical services.

A Flexible Business Model

With the ability to offer custom consulting and onsite support when needed, training and certification on instrumentation, tool-share capabilities.



A Network of Partnerships

With expanded access to specialty labs, instrumentation and community learning.

Speed and Access to Data

With fast turn-around times and a secure portal for uploading, downloading and viewing data.

About Covalent Metrology

Covalent Metrology is ready to serve you with our comprehensive platform of techniques and services. 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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