# STEM Imaging of Lattice Fringes and beyond in a UHR In-Lens Field-Emission SEM

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## Lattice Fringe Resolution from an SEM

The phase-contrast imaging of atomic lattices has now become commonplace for both Transmission Electron Microscopes (TEM) and Scanning Transmission Electron Microscopes (STEMs) [1]. Recently, however, bright-field STEM images of multi-wall carbon nanotubes (MWCNTs) recorded from an ultra-high resolution (UHR) in-lens field-emission scanning electron microscope (FE-SEM) operating at 30keV [2] have also demonstrated lattice fringe resolution. One example of such an image containing multiple examples of fringe detail is shown in figure 1. The carbon lattice fringes were analyzed and their origin confirmed by the application of the FFT algorithms in the SMART image analysis program [3]. The resulting power spectrum after thresholding to remove background noise (Figure 2) confirms that phase detail in the image extends down to about 5 Angstroms (0.5nm) and that well defined diffraction spots corresponding to a spacing of 3.4 Angstroms (0.34nm) generated by the (002) basal plane spacing of the graphite lattice are present.

This paper outlines the benefits of STEM technologies in a 30-keV in-lens FE-SEM and further advantages of low-kV STEM in such an SEM. In particular, the in-lens configuration of Hitachi's S-5500 FE-SEM is examined for its world's highest SE resolution at 0.4 nm (Figure 3). The S-5500's motorized high angle annular dark-field (HAADF) imaging mode, which enables true *Z*-contrast imaging for different material composition and thickness, is also discussed in comparison with traditional TEM imaging. Finally, key considerations are proposed for when a STEM-capable UHR in-lens FE-SEM would make the better instrument choice for cutting-edge microscopy.

In the bright field mode of the STEM, the ability to resolve a crystal lattice does not depend on the probe size of the microscope because such contrast is the result of a diffraction effect. Rather, it requires that the total convergence angle of the incident beam be sufficiently large to contain the Bragg angle corresponding to the spacing of interest. To achieve this when operating at 20-30keV requires a beam convergence approaching 0.05 radian (50 milliradians). Such condition is possible only in an SEM equipped with high performance, short focal length, immersion optics and when employing a larger than normal objective aperture. This mode of operation also requires that the instrument be electrically and mechanically stable on the scale of the fringe spacing for the duration of the image recording process. SMART analysis of super-position diffractograms [4] confirmed that the combined drift rate and instability of the S5500 FEG-SEM was less than 1nm per minute under the conditions used for these images.

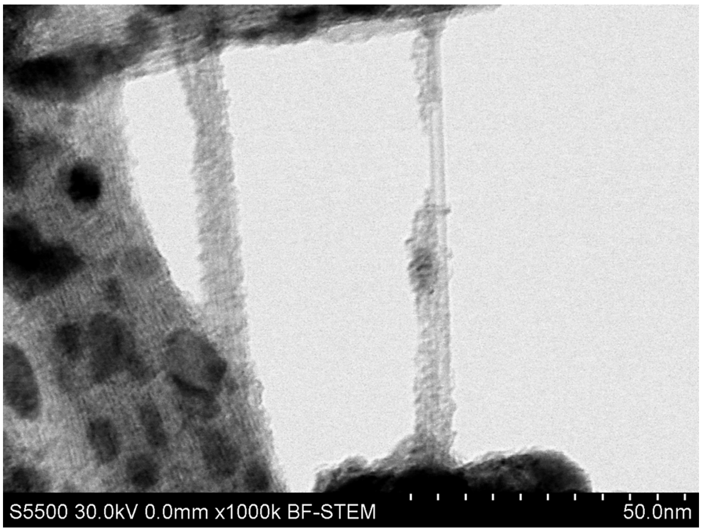


*Figure 1: Bright field (BF) STEM image of CNTs showing carbon lattice fringes.*

The ability to resolve atomic level detail in an image brings the capabilities of in-lens SEMs closer than ever to those of a dedicated STEM (at low kV). This calls for a new consideration of performance boundary of an in-lens FE-SEM and leads to the question of how best to choose between a high performance, low voltage, in-lens FE-SEM and a high energy dedicated STEM.

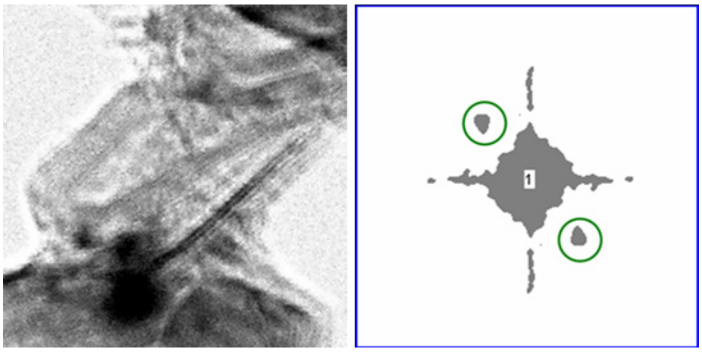


*Figure 2: One analyzed area of a BF-STEM image of MWCNTs (left) showing carbon lattice fringes and (right) FFT diffraction fringe spots corresponding to a spacing of 3.4 Angstroms.*

## Dedicated STEM vs. TEM

A thorough case has been made for the value of additional imaging modes that come with a dedicated STEM compared to the traditional TEM [1]. These two instruments, though different in their mode of operation and their optical configuration, are closely related through the principle of reciprocity. Using this concept we see that in the bright field mode, the STEM is identical with the TEM except that their ray paths are in opposite directions. Comparing these diagrams demonstrates that the STEM detector angle now acts like the condenser aperture angle on a TEM. Making the TEM condenser aperture larger increases the incident beam convergence, raising the beam current but reducing diffraction contrast. Similarly enlarging the bright field STEM detector increases the collection efficiency for bright field signals but reduces the diffraction contrast [5].

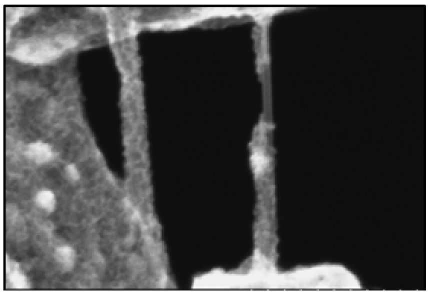
Dedicated STEMs typically operate at 200 keV beam energy and as there are no post-specimen imaging optics to be affected by chromatic aberration concerns, samples up to a few microns in thickness can be imaged in the bright field mode. A TEM operating at this same energy would only be able to image samples up to about 0.5 microns before chromatic aberration became dominant. Typically, TEM samples must be thinned to 100 nm to minimize chromatic aberration of the post-specimen beam and to preserve image quality.

Aside from the benefit for thicker samples, STEM imaging also provides another unique mode that is of significant advantage compared to the traditional TEM—namely, High Angle Angular Dark Field (HAADF) imaging. HAADF signals come from electrons elastically scattered at high angle due to their close approach with sample atomic nuclei, *e.g.* small impact parameter. In HAADF mode, the Bragg diffracted electron signals (*phase contrast*) which often mask structural information are removed, making high resolution *intensity* imaging of single atom columns *routinely* possible [1] provided that the probe size is sufficiently small. The HAADF image contrast varies linearly with the mass-thickness of the sample and is proportional to the atomic number

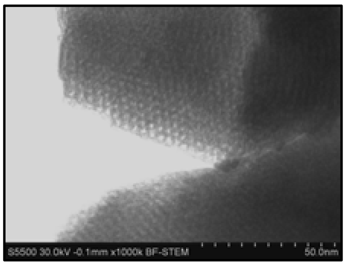
# The Ultimate in Resolution.

## Hitachi S-5500

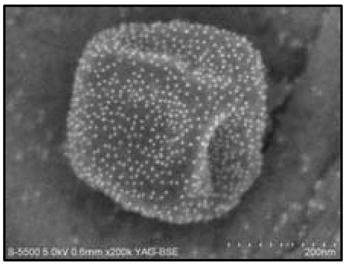
In-lens Field Emission Scanning Electron Microscope



X1000k, 30.0kV Single Walled Nanotube



X1000k, 30.0kV Mesoporous Silica



X200k, 5.0kV Immuno Labeling

The Hitachi S-5500 is a dedicated ultra-high resolution In-lens FE-SEM for leading edge research and development of nanotechnologies.

The patented in-lens technology provides the ultimate performance of 0.5nm guaranteed imaging resolution.

The S-5500 is equipped with a shielding system for reduced EMI and acoustic interferences. These improvements with a completely dry vacuum system ensures Hitachi's high-resolution guarantee for the life of the instrument.

A newly designed BF/DF Duo-STEM detector (patent pending) contains an adjustable dark field detector for tunable collection angles.

Sometimes a single image can change the way we look at life. The new S-5500 with its advances in information collection will lead you to those opportunities.

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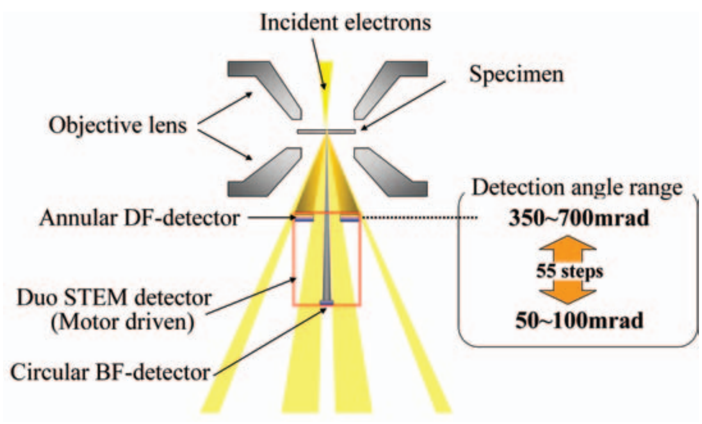


*Figure 3: Schematic of motorized Duo STEM system in the S-5500 UHR in-lens FE-SEM indicating higher maximum scatter angle in HAADF mode and variable dark-field detection angles.*

*Z* of the target squared. Dedicated STEMs also come with secondary electron (SE) imaging and *optional* backscatter electron (BSE) imaging capabilities so surface topography can be observed in conjunction with the bulk information obtained in STEM modes. This added versatility enables STEMs to image samples that are either too thick or cannot be prepared for STEM or TEM.

## What's in a UHR in-lens FE-SEM for applications requiring STEM work?

### *SEM and HAADF STEM:*

Today, all the aforementioned advantages of STEM imaging can also be found in a state-of-the-art in-lens field-emission scanning electron microscope (FE-SEM), albeit in a lower-kV regime. Even at 30 keV, a quality in-lens FE-SEM can demonstrate its invaluable versatility for demanding microscopy work with simultaneous STEM and SE/BSE imaging performance and the benefits that come with low-kV operation.

For example, the best surface details, contrast, and highest SE resolution of 0.4 nm [2] are only attainable with an in-lens FE-SEM because this level of performance requires both the small probe size offered by cold field emitter technology and the high performance provide by in-lens electron optics optimized for SE imaging.

In addition instruments like the S-5500 UHR in-lens FE-SEM [2] offers HAADF imaging at variable detection angles and higher maximum scatter angle than that of a dedicated STEM (Figure 3). For a given beam intensity, higher scatter angles result in better mass-thickness contrast in dark field modes. Motorized adjustments enable optimized detection angle settings to achieve true *Z*-contrast imaging for different material composition and thickness.

Figure 4 shows HAADF images of a 150 nm thick FIB sectioned SRAM specimen at two different optimized detection angle settings. The image to the left, imaged at 400~650 mrad detection angle is optimized for true Z-contrast of high-density materials, such as the W plug and the Ti/Ti$_3$N$_4$ layer. At this detection angle, a MOS transistor containing low-density materials, indicated by the square inset, is imaged with insufficient contrast. However, using the motor- driven Duo-STEM system, the detection angle is easily adjusted to 100~300 mrad, providing the optimized contrast for the MOS transistor gate. Monte Carlo simulation results [10], shown in Figure 5, provide that the differential DF signal is highest for the CoSi$_2$ layer and other low-density materials of the MOS gate at 100-300 mrad, as indicated by the inset box labeled *low*. Accordingly, simulation results also confirm the optimized *Z*-contrast for the high-density SRAM materials at the higher detection angle setting as indicated by the inset box labeled *high*. The former contrast is a mixture of Bragg diffraction and HAADF signals, while the latter contains only HAADF signals, providing true *Z*-contrast.

The high-contrast of low-kV HAADF is well suited for both inorganic and organic samples as well as for crystalline and amorphous materials alike. For example, unstained sections of low-density particles of toner or nanomaterials [10] can be routinely imaged to determine dispersion or inclusion characteristics, respectively. In general, beam-sensitive applications stand to benefit considerably from the high-contrast and TEM-feature details that low-kV STEM provides. For biological applications, tissue sections typically prepared for TEM (up to 200 keV) can be imaged in STEM modes of an in-lens FE-SEM, and for semiconductor device analysis applications (front-end), accurate film thickness measurements from low-kV STEM would overcome any uncertainty from cross-section imaging in SE mode. Comparatively speaking, one gets the value of simultaneous STEM dark-field or bright-field, and BSE, SE UHR imaging for the cost of an SEM at the same SEM operator skill level.

More than that, STEM-type sample size preparation also brings the benefit of very small volume beam interaction. For example, EDS analysis of 10 nm Ni nanoparticles held within carbon nanotubes (CNT) have been performed on the S-5500 [6]. Analyses of these nanoparticles-CNT complexes, which are supported on TEM carbon film grids, are possible due to the fine beam probe of this SEM. At the same time, the incident beam voltage from such a system is high enough to generate easily detectable *K*-line x-ray peaks. At high beam energies, bulk sample analysis by SEM/EDS suffers from a large interaction volume between the incident electron beam and the sample, while lowering the voltage reduces the interaction volume, but this restricts the emission lines that can be used for analysis. This work also demonstrated that it is also possible to detect nanoparticle structures at intermediate voltages, *e.g. 10keV to 30 keV* (Figure 6).

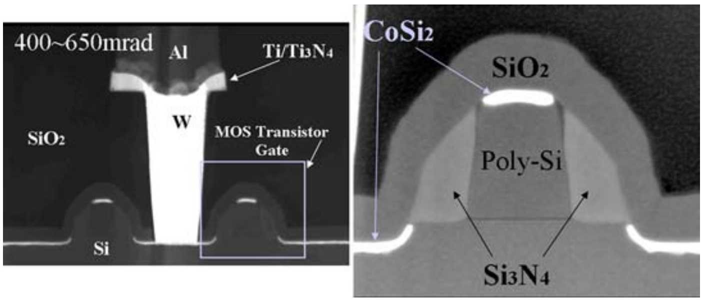


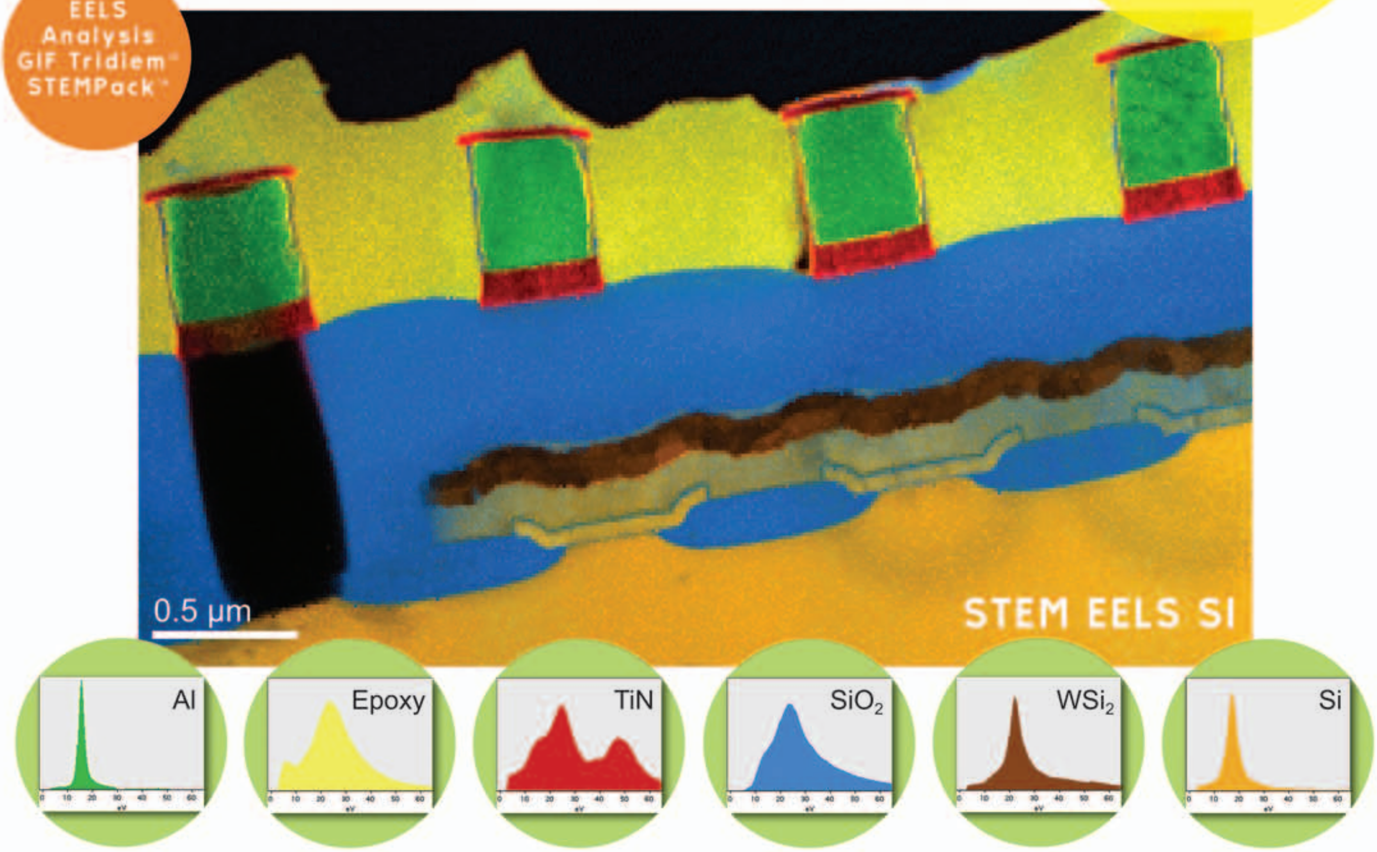
*Figure 4: The HAADF image to the left, imaged at 400~650 mrad detection angle is optimized for true Z-contrast of high-density materials. At this setting, however, a MOS transistor containing low-density materials is imaged with insufficient contrast, as indicated by the inset box (left). At the HAADF scatter angles of 100~300 mrad (right), the MOS transistor is imaged with optimized contrast for its low-density materials.*

## When does your microscopy work demand the best UHR in-lens SEM in the market?

Thus far, we have reviewed key benefits of a dedicated STEM system as compared to a tradition TEM system. We have also demonstrated that in a lower kV regime (and perhaps for slightly lower specimen thickness), the modern UHR in-lens FE-SEM possesses capabilities very close to that of a dedicated STEM while outperforming the dedicated STEM in SE image resolution and arguably HAADF contrast. This discussion naturally begs the question: *How does one decide between a dedicated STEM and a STEM-capable UHR in-lens FE-SEM*?

We propose that the prerequisites for acquiring a STEM-capable UHR in-lens FE-SEM are as follow:

1. *When the world's highest SE image resolution is a must!* SE imaging is expected to approach its spatial resolution limit at a few


0.5 µm
STEM EELS SI
Al
Epoxy
TiN
$SiO_2$
$WSi_2$
Si
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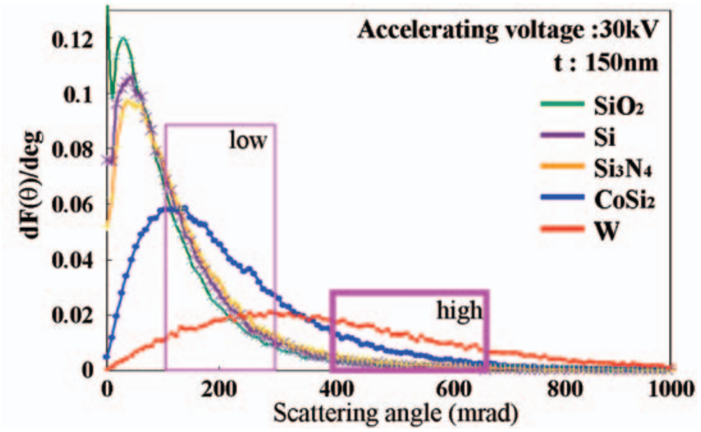

*Figure 5: Monte-Carlo simulation results of differential dark-field signal from 150 nm thick SRAM specimen. Y-axis is differential signal intensity. X-axis is scatter angle.*

Angstroms. At present, state of the art instruments with in-lens design like that of the S-5500 is at about that limit. As discussed above, dedicated STEM often comes with an SE imaging mode as standard, which provide excellent SE performance in conjunction with STEM imaging. However, even at high beam energies, the SE resolution and contrast from a dedicated STEM are not better than the best of the in-lens FE-SEM [**3**]. Therefore, when microscopy work is heavily based on SE-image performance, obtaining the highest SE image resolution will require a UHR in-lens FE-SEM.

2. *When low-kV STEM imaging is required!* Achieving phase-contrast resolution on the order of atomic lattice fringe is commonplace in a dedicated STEM. Obtaining 3.4 Å lattice fringes of carbon nanotubes at 30 keV in an SEM is quite radical and opens up a variety of beneficial and interesting possibilities for STEM-capable in-lens SEMs. For example, for the cost of a high-end SEM, it is now possible in material applications to quickly determine if a specimen is amorphous or measure its lattice fringe. Furthermore, it is conceivable that the immersion objective lens geometry would allow for minor modifications which would then make possible the acquisition of nano-diffraction patterns of STEM samples in such SEMs [**3**].

It is also worth noting that, at 30 keV, the maximum beam energy of most, if not all, FE-SEMs in the market, is well below the knock-on damage threshold. For example, the energy threshold for elastic displacement of carbon in single wall carbon nanotubes (SWNTs) is at 86 kev [7, 8]. This is an important advantage that enables FE-SEMs to image carbon nanotube materials while avoiding the material alteration or damage that would otherwise occur in a STEM or TEM.

This coupled with the growing interest in low kV STEM imaging in the life sciences [9] presents yet another possibility for the future advancement of STEM-capable in-lens FE-SEM. For low mass-density materials often encountered in life science microscopy and nanomaterials research, STEM imaging near 100 keV and below would benefit significantly from the higher contrast. Secondly, the ability to continuously select the operating beam energy common in all SEMs is a perfect match for STEM imaging, where unique beam energy setting is often optimized for different sample material and thickness combinations.

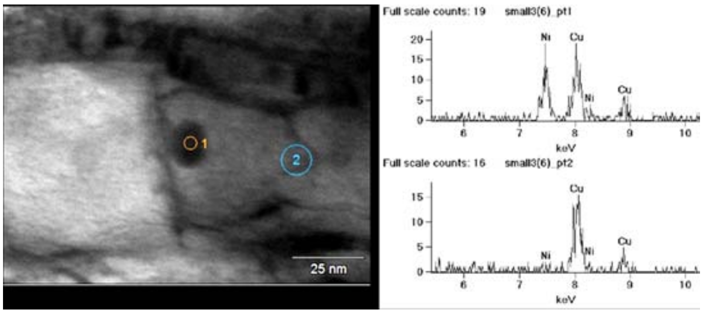

*Figure 6: Small interaction volume EDS analysis of Ni nanoparticles in CNTs at intermediate beam energies: (left) EDS image scan showing 2 analysis points; (right, top) spectrum of Ni particle region vs. that of CNT background area (right, bottom).*

3. *When robust versatility and ease of use of an SEM is needed for a multi-disciplinary facility.* Microscopy work at most facilities today is very interdisciplinary in nature. Today's SEM users are not all strictly dedicated microscopists. Generally, the SEM users can be scientists, researchers, engineers, technicians, or specialists of a field unrelated to microscopy. Yet, in their line of work, the SEM is one of the everyday analytical tools they rely on for data, either frequently or occasionally. This situation has been answered in recent years by most SEM manufacturers as they simplify the SEM operation with computer-controlled and automated functions and streamlined user interface. Although reliability and up-time still vary with manufacturers, the robustness and ease of use of modern SEMs have reduced the prerequisite of entry-level operator skill, allowing them to be easily integrated into a multi-user environment. For the above mentioned FE-SEM, the simplicity of operation remains, for the most part, the same even when the STEM capabilities are introduced. There, such interdisciplinary facilities are able to add higher-level imaging capabilities without the higher costs and operator skills.

4. *When your serious microscopy work is worth a little sample prep!* Even as microscopy on an SEM is simplified and made accessible to a diverse user base, getting at the right image is still an art of the science. With a reasonable degree of experience, the first part of getting at the right image is choosing the right beam, optics, and detector settings, which is aided to a large degree by computerized operation of the SEM. What remains in the practice of this art is getting at the right sample condition. In practice, different specimen of interest often requires different preparation. And when one is serious enough to get to a high performance STEM-capable in-lens FE-SEM for the right image, getting to the right sample condition with a little sample preparation is seriously well worth the work. ■

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