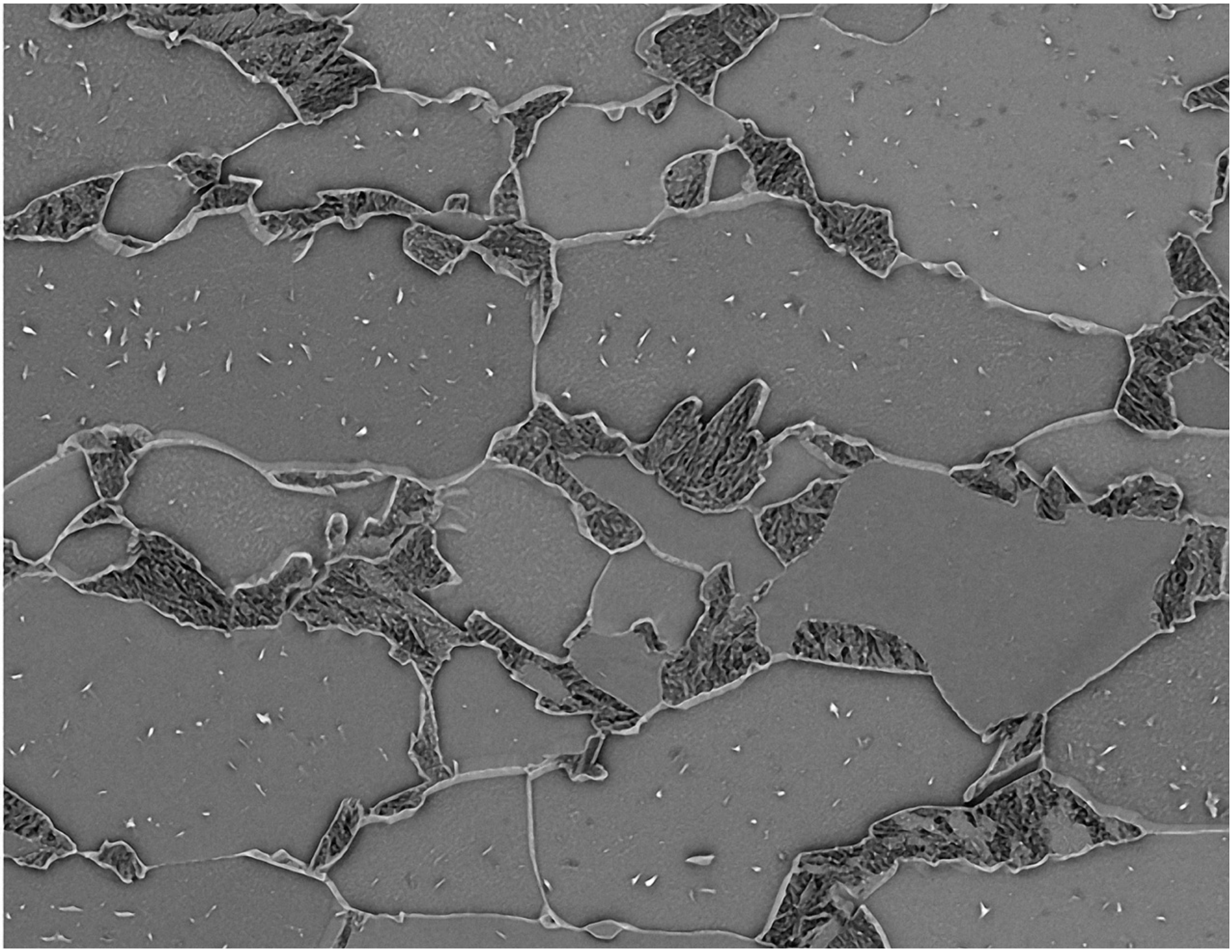
APPLICATION NOTE

Imaging Magnetic Materials

Using the Helios G4 DualBeam, Apreo SEM and Verios G4 SEM



Dual-phase steel imaged at low energy with the Nova NanoSEM

Immersion lens systems

In recent years, the race for the highest resolution in scanning electron microscopy (SEM) has been driven by nanotechnology developments, new materials and smaller devices. Ultimate resolution can be approached by minimizing the focal length of the optical system, and one way to do so is to use an immersion lens. A magnetic immersion lens system delivers increased detection efficiency, with the highest resolution, at all accelerating voltages—without sensitivity to sample tilting. Recent developments have shown that sub-nanometer resolution can be achieved when combining the monochromated electron source of the Helios NanoLab DualBeam, formerly produced by FEI, and Thermo Scientific™ Verios G4 SEM with a magnetic immersion lens system, even with such low landing energies as 1 keV[1]. This improvement enables similar resolution from 1 to 30 keV, meaning that operators only need to consider the information depth required, rather than limitations in resolution. Figure 1 shows the benefits of using a monochromated source when performing high-resolution imaging on a gold-on-carbon reference sample.

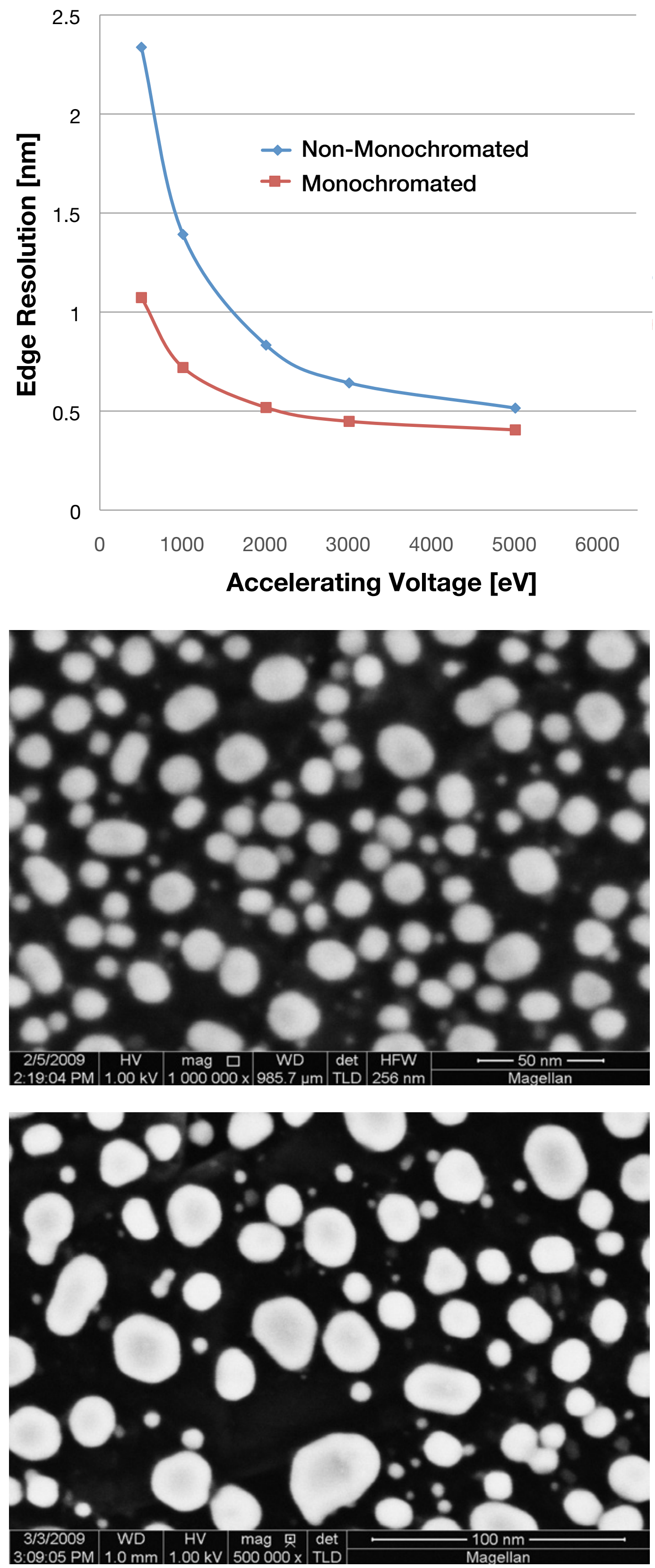


Figure 1. (Top) Plot of resolution versus accelerating voltage at the optimum working distance for a non-monochromated beam (red) and a monochromated beam (blue). (Middle) and (bottom) show resulting images acquired at 1 keV with monochromator disabled and enabled, respectively. Images acquired with the Magellan 400L SEM.

[1] Extreme High-Resolution SEM: A Paradigm Shift. Young, R., Templeton, T., Roussel, L., Gestmann, I., Van Veen, G., Dingle, T., Henstra, S. (2008) Microscopy Today, 16 (4), pp. 24-28.

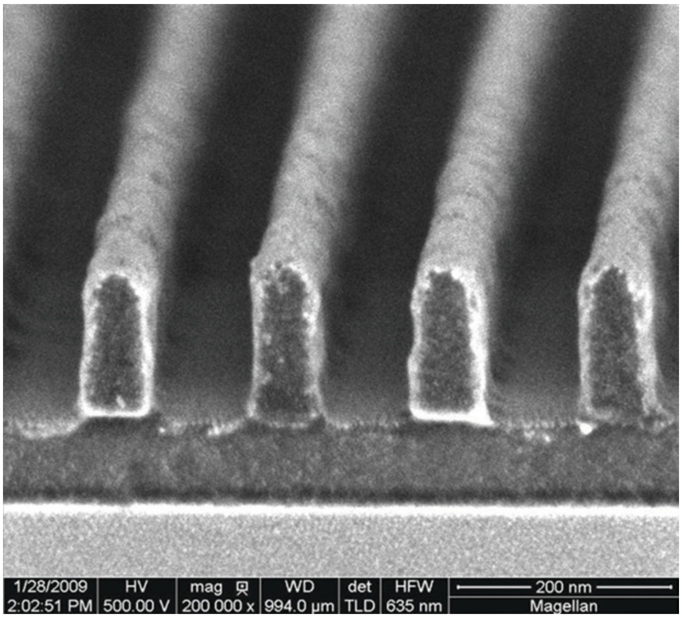


Figure 2. Low-dose imaging of ArF resist for 193 nm lithography. Image acquired at 500 eV with a tilt applied on the Magellan 400L SEM.

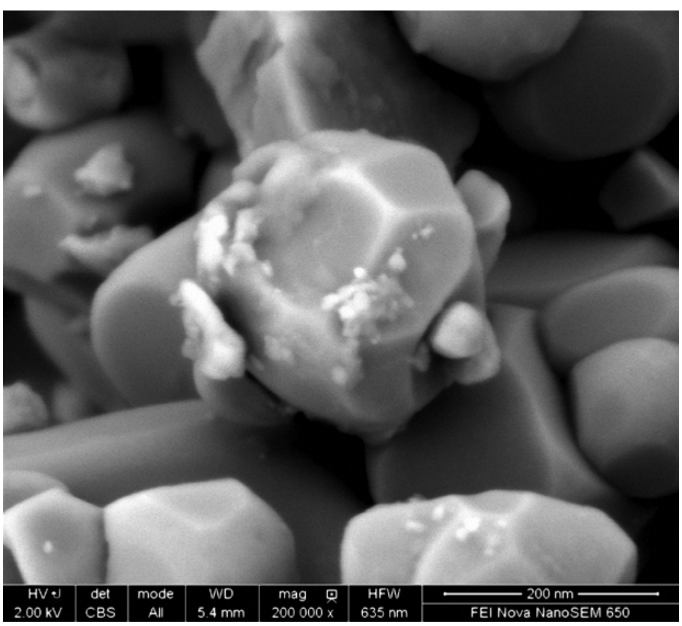


Figure 3. Magnetic particles imaged in magnetic immersion. Image is acquired at 2 keV with the Nova NanoSEM.

Immersion lens benefits

An additional benefit of using a magnetic immersion mode is high-efficiency secondary electron imaging. Almost all secondary electrons escaping the sample are forced back into the SEM's final lens where they can be detected. This permits the use of much lower electron doses while imaging, thereby avoiding contamination of the sample surface and damaging or shrinking beam-sensitive materials. Highly efficient detection is available even while tilting the sample, without any distortion or loss of signal. Figure 2, which shows a beam-sensitive resist sample imaged using a 500 eV beam on a tilted specimen, demonstrates high resolution with no visible shrinkage of the photoresist.

Magnetic material

When working in magnetic immersion mode on magnetic or magnetizeable samples, one can achieve excellent results as long as the following elements are considered. Since bulk magnetic samples can be attracted toward the final lens when this mode is enabled, sample preparation is key for getting the most out of this technique. One way to reduce this attraction is to decrease the overall size of the specimen, with different techniques available depending on the sample.

When working with magnetic powders, it's helpful to place the powder directly onto an aluminum stub, and then grind a second stub on top. This causes the powder to become embedded in the grooves of the bottom Al stub. Using a "dust-off" will remove most of the excess particles. Passing a magnet over the surface of the stub will then remove any additional loose particles before the stub is placed into the SEM. The example shown in Figure 3 demonstrates the high resolution achievable on magnetic particles in magnetic immersion mode using the Nova NanoSEM, formerly produced by FEI.

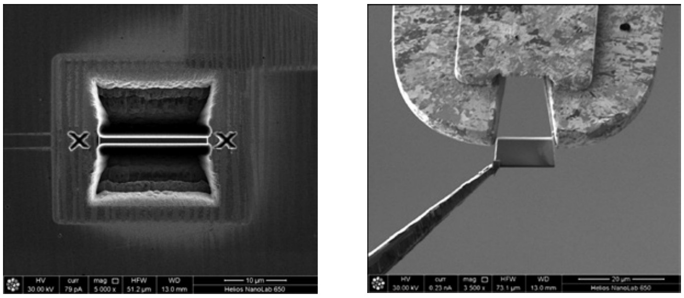


Figure 4. Views (Left) and (Right) show the steps involved in preparing *in situ* S/TEM sample lift-out from bulk sample to sub-50 nm-thick lamella. Preparation performed on the Helios NanoLab DualBeam.

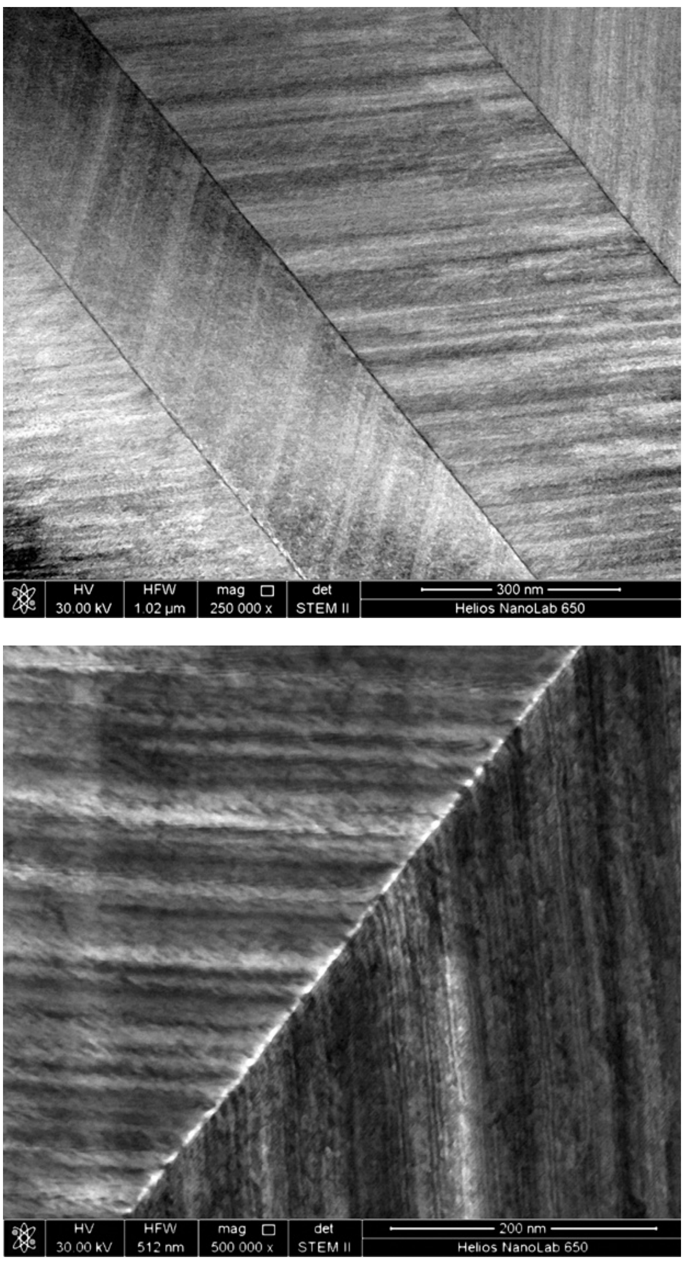
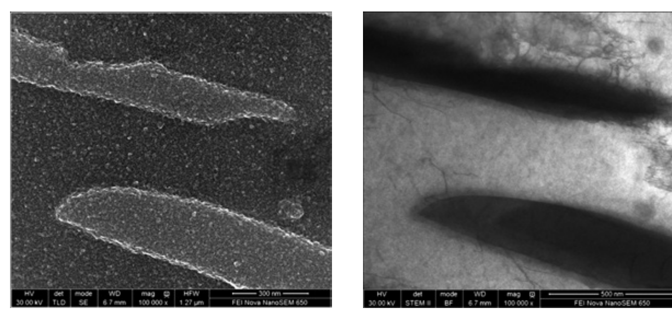


Figure 5. Ultra-high resolution High Angle Annular Dark Field [HAADF (top)] and Bright Field [BF (bottom)] STEM images of a magnetic NiMnGa martensitic alloy. Acquired at 30 keV using the 8-segment STEM detector with the Helios NanoLab DualBeam.

When large bulk samples need the highest resolution investigation, an *in situ* STEM (Scanning Transmission Electron Microscopy) sample preparation can be performed using the Helios NanoLab DualBeam to reduce sample size. This will also provide best lateral resolution and contrast when STEM imaging is performed. In the example shown in Figure 4, a 1 µm-thick lamella is easily prepared from a magnetic NiMnGa martensitic alloy bulk sample using the AutoTEM™ automation software. Using an *in situ* manipulator, the lamella then can be transferred to a support STEM grid, where a final, low-energy ion beam cleaning step is performed to thin the sample to electron transparency.

After sample preparation is complete, high-resolution imaging can be performed on the lamella since the sample's bulk magnetism was removed. With a final thickness <50 nm, it's possible to achieve an ultra-high resolution image using the 8-segment STEM detector as demonstrated in Figure 5.

Combining STEM imaging with in-lens detection can also be a powerful technique for capturing all generated signals simultaneously and correlate the resulting information. The magnetic immersion on the Helios NanoLab DualBeam, Nova NanoSEM and Verios G4 SEM allows simultaneous use of in-lens detectors and STEM at maximum accelerating voltages, which is essential on steels or other high atomic number materials. The example in Figure 6 shows the simultaneous collection of in-lens SE, BF STEM and HAADF STEM images. This concurrent detection enables the user to correlate surface effects on the lamella versus diffraction and materials contrast. Increasing the magnification further enables visualization of dislocation loops within the sample.



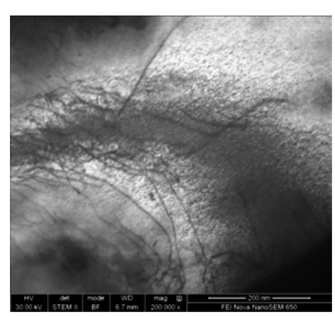


Figure 6. Simultaneous in-lens SE (top left) and BF STEM (top right) images of a TRIP steel lamella. A higher magnification BF STEM image (bottom left) shows dislocation loops within the sample. Images acquired with the Nova NanoSEM.

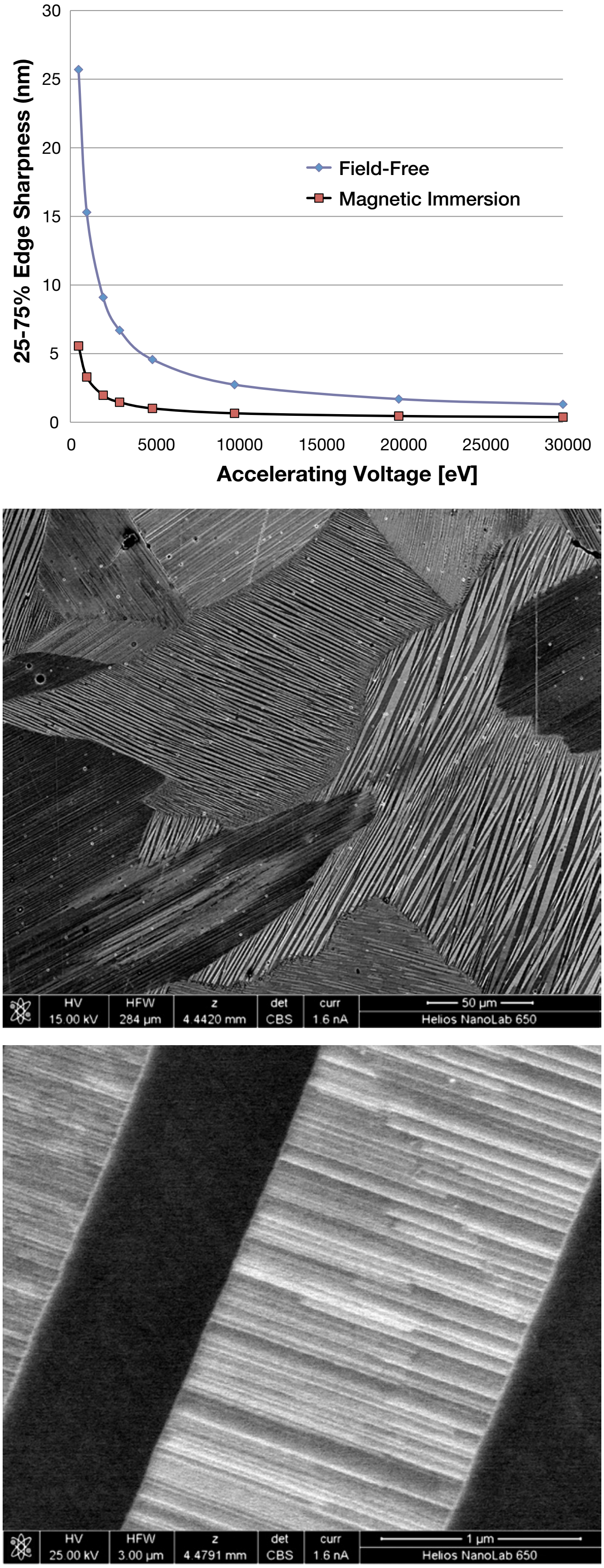


Figure 7. (Top) Plot showing resolution versus accelerating voltage for magnetic immersion and field-free imaging modes. (Middle) and (bottom): NiMnGa martensite alloy imaged at high keV with the CBS detector in field-free imaging mode. Images acquired with the Helios NanoLab DualBeam.

Field-free imaging mode

An additional strategy for imaging magnetic material is the use of a field-free imaging mode. Figure 7 shows a comparison of the relative resolution acquired at eucentric height for magnetic immersion and field-free imaging modes. At higher voltages, it's clear that differences in resolution are relatively small. An example of the excellent resolution attainable in field-free mode is visible in Figure 7 (top and middle) while imaging a magnetic NiMnGa sample using the Helios NanoLab DualBeam. Stacking faults and twinning can be observed clearly when imaged at 25 keV with the concentric backscatter detector (CBS).

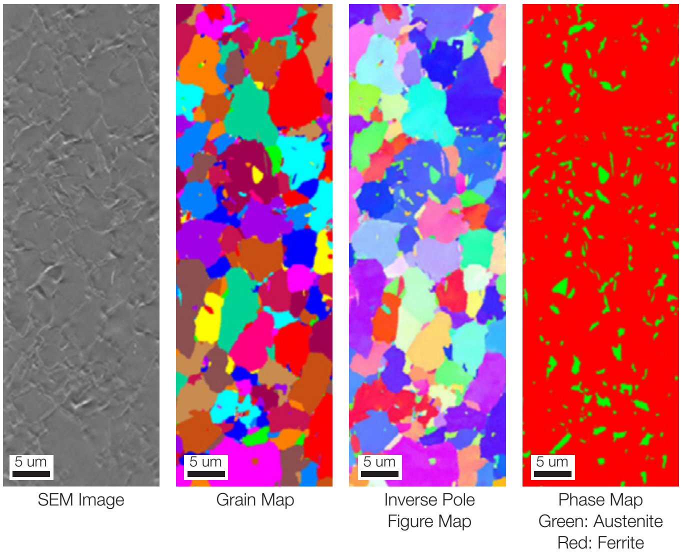


Figure 8. EBSD mapping of TRIP steel acquired at 25 keV.

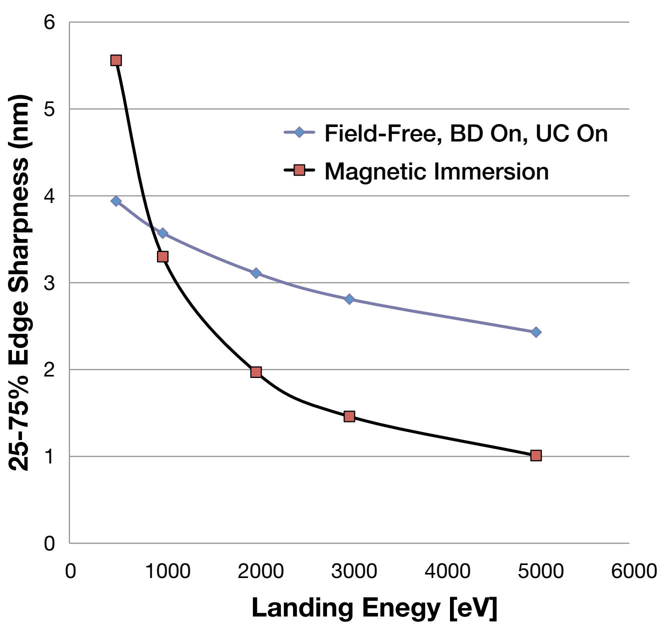


Figure 9. Plot of resolution versus landing voltage for immersion mode and field-free imaging mode at eucentric height.

The field-free imaging mode is also helpful in other high-energy applications, such as electron back scatter diffraction (EBSD) or energy dispersive X-ray spectrometry mapping. Benefits include the long depth of focus and ultra-wide field of view, which assist when high tilt angles (70˚) and large grain sizes place large demands on the SEM. To gain sufficient signal for mapping, beam currents on the order of several nanoAmps are required. Both the Sirion Electron Column, formerly produced by FEI, and the Thermo Scientific™ Elstar™ Electron Column are designed to deliver the highest beam current density possible, even at high beam currents. The example shown in Figure 8 demonstrates the high-resolution EBSD mapping achievable from a TRIP steel sample.

Beam deceleration

For increased surface sensitivity when imaging magnetic materials, a monochromator and beam deceleration technology can be used independently. Both are excellent tools in gaining resolution and signal at low-landing energies. Decelerating the beam outside of the SEM's final lens reduces lens aberrations that typically affect the low energy beam's resolution. By applying a stage bias of 4 keV, the resolution of a 5 keV beam can be achieved with the surface sensitivity of a 1 keV beam. The increase in resolution from beam deceleration and a monochromated beam can be seen in Figure 9. The achievable resolution is similar to that of immersion mode at 1 kEV, and even surpasses it at lower voltages.

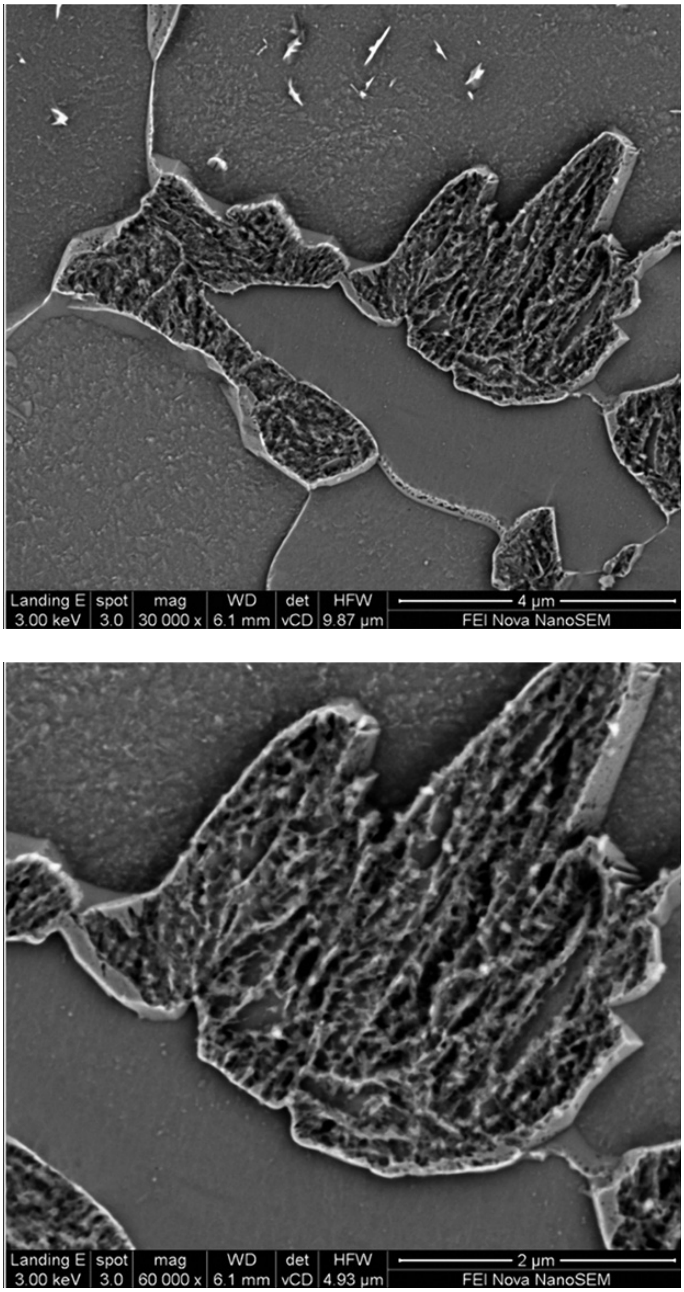


FIgure 10. Dual-phase steel observed at 3 keV in field-free imaging mode with beam deceleration applied. Images acquired with the Nova NanoSEM.

Beam deceleration also delivers highly efficient detection, as all signal generated is redirected and re-accelerated back toward the detectors, delivering high-contrast, noise-free imaging. High-resolution images in Figure 10 show a dual-phase steel imaged with the Nova NanoSEM in field-free imaging mode with beam deceleration applied.

Summary

- A magnetic immersion lens is superior in delivering the best resolution on the majority of samples, with almost 100% secondary electron detection efficiency.
- When magnetic materials need to be imaged, use appropriate sample preparation, or a field-free imaging mode, to avoid any negative side effects.
- Unique technology solutions, including a monochromated electron source or beam deceleration, can be used to improve resolution at low landing energies.

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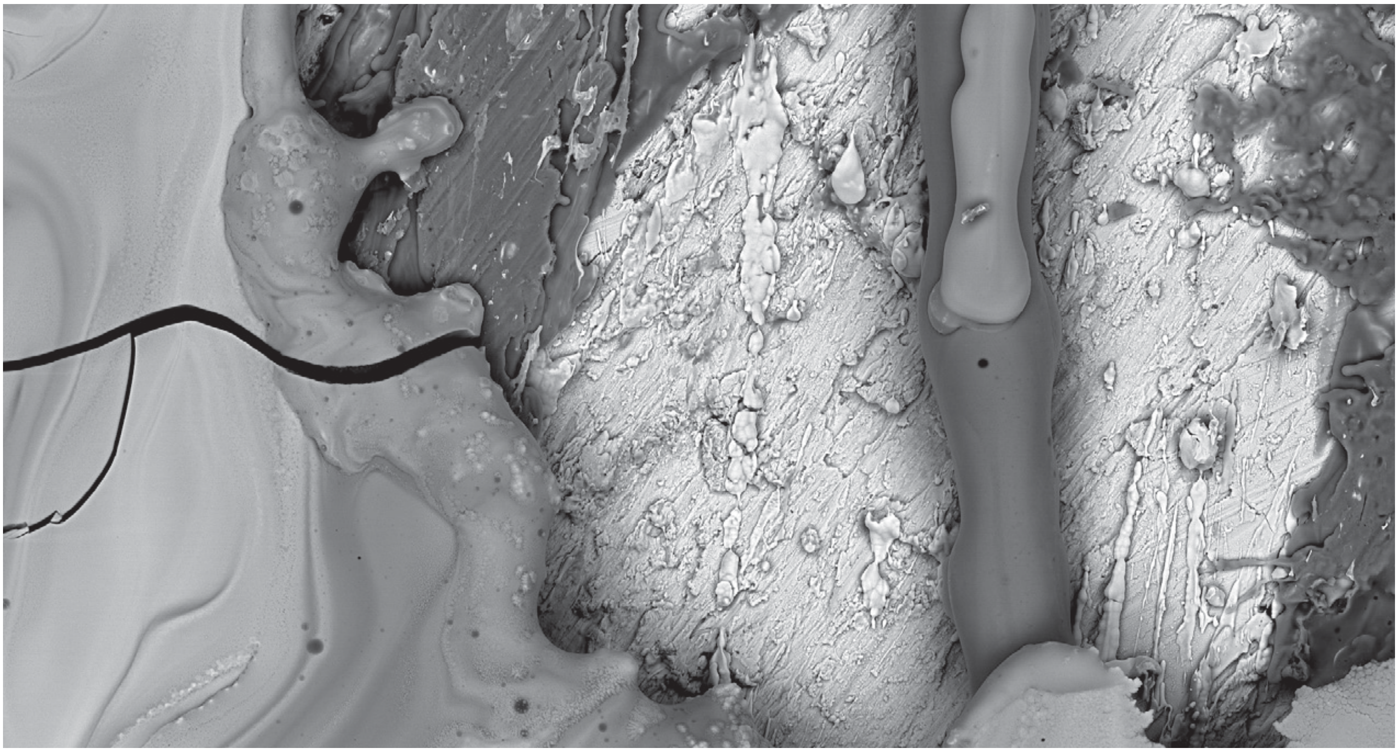
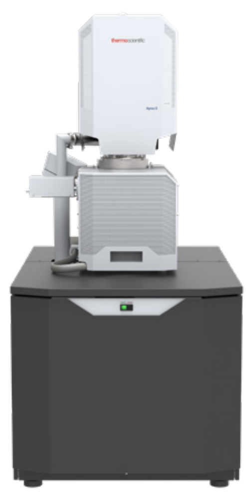


Tungsten carbide coating on a steel substrate imaged at low energy using the DBS detector on the Nova NanoSEM

Helios G4 DualBeam



Apreo DualBeam

Verios G4 SEM

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