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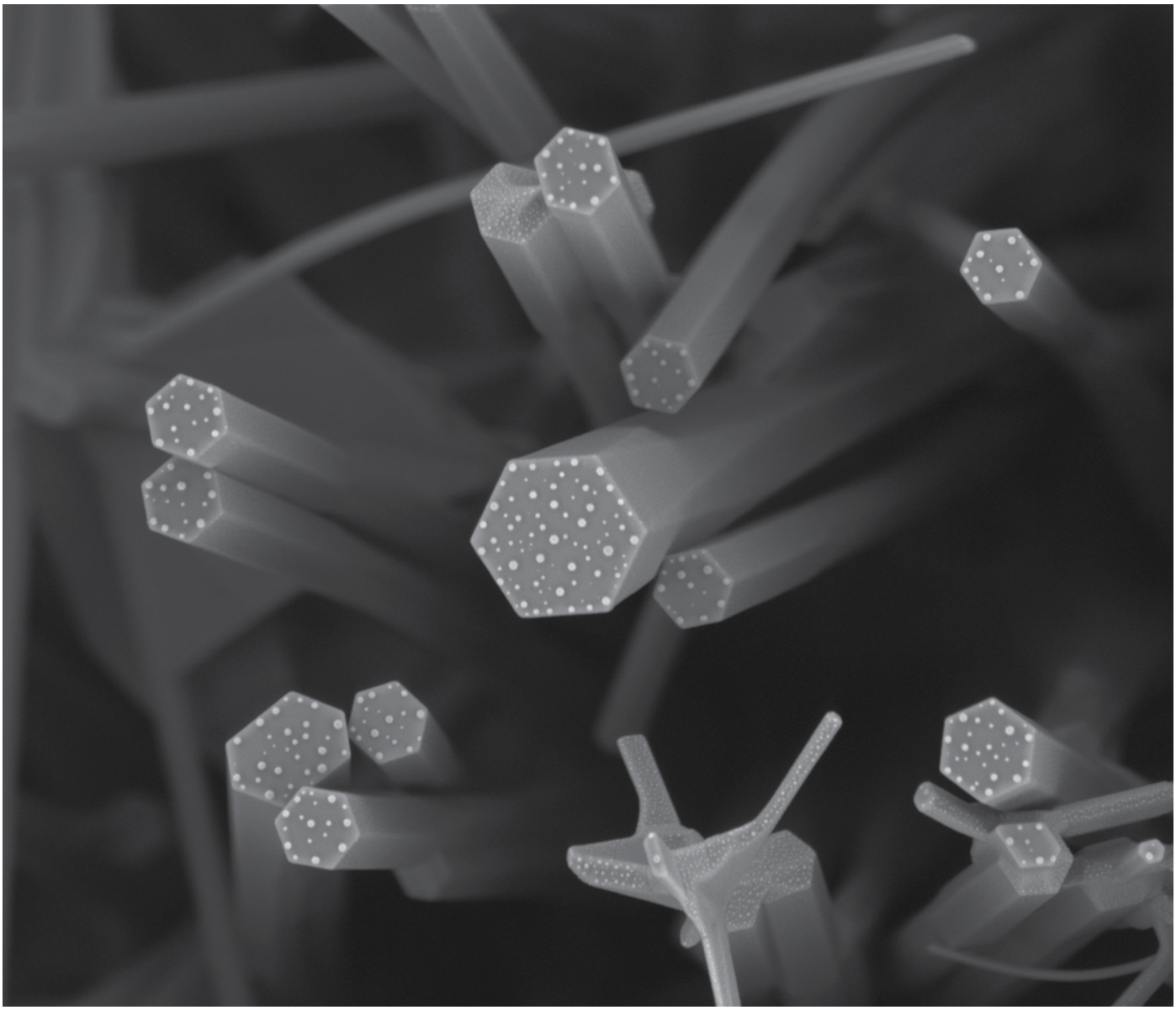
APPLICATION NOTE

Information from Every Angle

Directional BSE detector for next-level imaging



Zinc oxide nanorods with surficial palladium particles imaged at 500 V in high vacuum. Adding palladium increases hydrogen sensitivity for sensor applications.

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Introduction

Due to its ease of use, speed, intuitively interpreted images and analytical results, the Scanning Electron Microscope (SEM) is one of the most commonly used tools to determine the morphology and physical nature of samples. In recent years, the SEM has taken huge steps forward in its achievable resolution, particularly with the introduction of monochromated electron sources. Similarly, SEM detection systems have been evolving rapidly, driven by advancements in nanotechnology and the use of novel materials. Correctly and easily discriminating the different signals available is crucial in understanding the material properties being analyzed and drawing the correct conclusions.

This application note will look at the different signals available and methodologies to capture all of the available information. Repeatedly imaging a sample to gather all of the required information can often lead to sample damage and contamination. The Directional Backscatter Detector, available on all Thermo Scientific™ SEM and DualBeam™ systems, offers a unique way to simultaneously capture information from every angle, while other benefits such as filtering of charge while imaging non-conductive specimen are also realized.

Energy selection

When an electron beam strikes a sample's surface, it generates an interaction volume from which different signals are produced. A generalized diagram in **Figure 1A** shows the primary signals that are detected and used in most SEM applications.

Figure 1B shows the full spectrum of electron energies. For simplification, other signals such as Auger electrons (AEs) will not be considered in this application note. Since secondary electrons (SEs) escape the sample close to the surface with a very low energy (typically <50 keV), they can deliver images with extreme surface sensitivity, generating excellent topographical information.

This can be seen in **Figure 2B**, where the surface of a carbon fiber network is imaged at low energies, providing high-contrast details of the surface with the size and distribution of the fibers. By combining an immersion lens system with a biased suction tube [**Figure 2A**] (i.e., Thermo Scientific™ Helios™ DualBeams and Verios SEM and Nova NanoSEM platforms by Thermo Fisher Scientific), almost 100% of SEs can be directed back into the lens of the SEM, where they are then detected by the in-lens detector. This high-efficiency detection translates into reduced beam damage to sensitive materials since a lower dose is required for image acquisition.

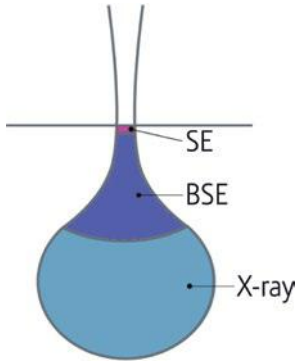

Figure 1A. Generalized diagram showing the interaction volume generated when an electron beam strikes the surface of a specimen.

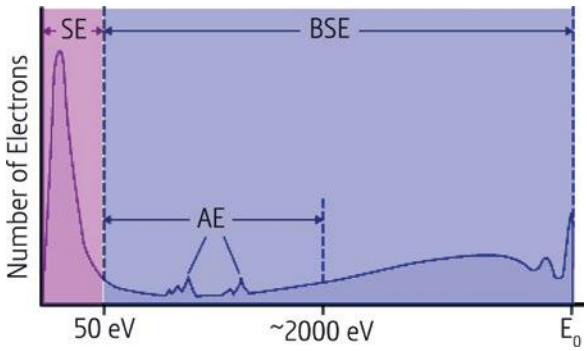

Figure 1B. Electron energy spectrum.

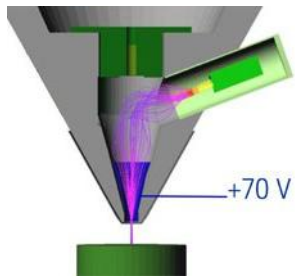

Figure 2A. Schematic of final lens showing in-lens SE detection.

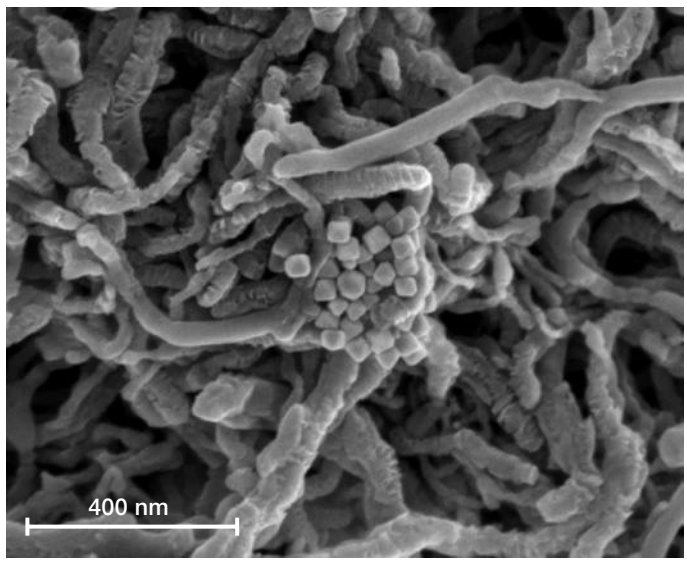

Figure 2B. A resulting SE image acquired using a 2 keV beam.

Backscattered electrons (BSEs), on the other hand, are emitted from deeper in the sample, supplying information on morphology, composition or grain orientation. By simply adjusting the suction tube voltage in the final lens [**Figure 3A**], it is possible to filter the energy of the electrons that enter the detector to distinguish between SE and BSE signals. This can be seen in **Figure 3B**, which shows a 2 keV image in BSE mode in contrast to the same area scanned in **Figure 2B**.

By only considering the energy of the electrons generated, however, some of the signal may go undetected, thereby yielding incomplete sample information. Through simulation, it is possible to plot the angular distribution versus energy, where it is clear in **Figure 4** that there is a large distribution in emission angles present for both SEs and BSEs. Instead of limiting the signal collection to energy alone, it is useful to see not only the electrons' energies but also the angle at which they travel from the sample to gain more information.

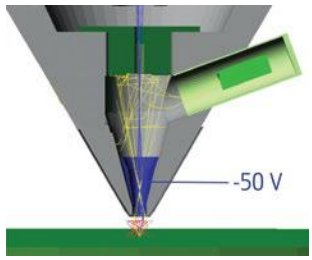

Figure 3A. Schematic of final lens showing in-lens BSE detection.

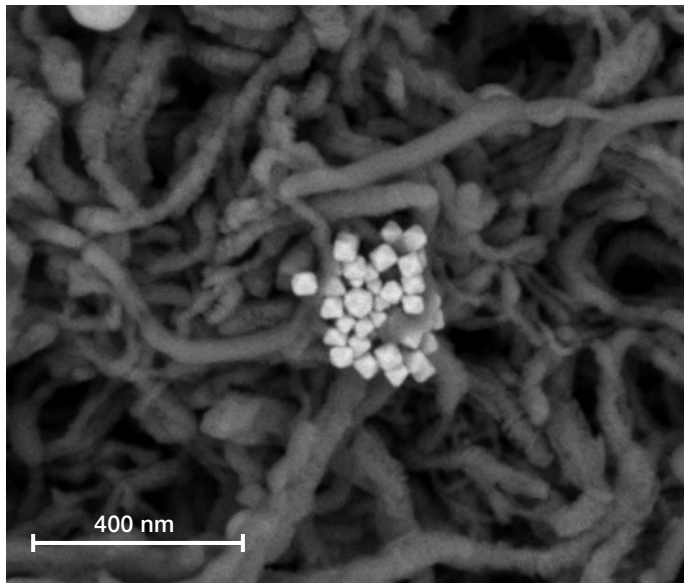

Figure 3B. Resulting BSE image acquired using a 2 keV beam.

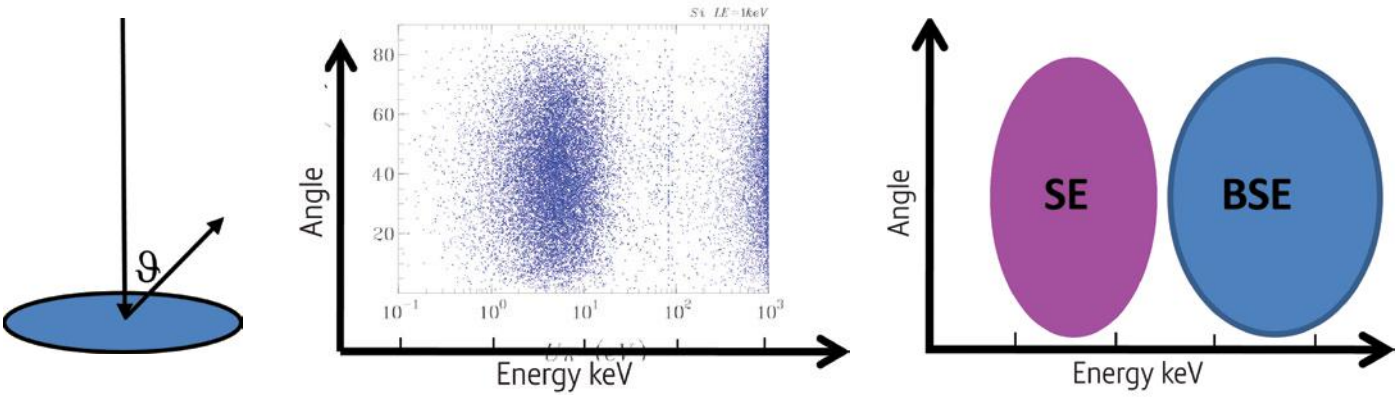

Figure 4. Simulation of electron energy versus emission angle normal to the sample.

Energy and angle information

BSEs leaving the sample at different angles carry different information. Those close to the primary beam axis or at low angles carry more information about the sample's atomic number. Those at high angles closer to the surface carry more information about the sample's topography. **Figures 5B and 5C** show a steel sample imaged at 0.7 keV. **Figure 5B** was acquired using the BSE signal closest to the beam axis, where only material contrast and grain orientation contrast are present. By selecting the BSE signal closer to the sample surface [**Figure 5C**], you can acquire primarily topographical contrast from the specimen.

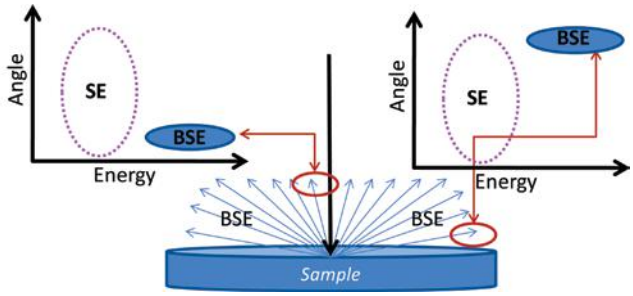

Figure 5A The range of BSEs emitted from the sample. The angle is measured with respect to the primary beam axis.

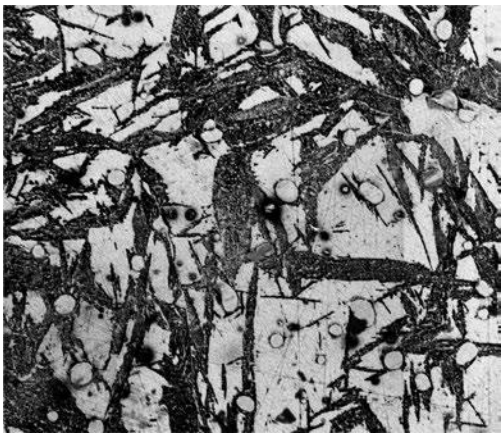

Figure 5B. BSE image of a steel sample acquired at 700 eV. The image is formed by collecting electrons traveling closer to the primary beam axis.

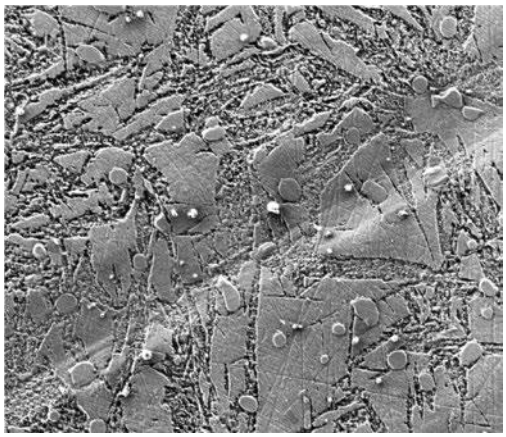

Figure 5C. BSE image of the same location on the steel sample. The image is formed by collecting electrons traveling closer to the sample surface.

By optimizing the angle of collection of the BSE signal, it is possible to drastically affect the contrast and, therefore, the image information. A series of images acquired on a platinum (Pt) sample in **Figure 6 (A-D)** highlights this more clearly, where grain orientation, material contrast and topographic contrast can be seen in the full range of detected angles.

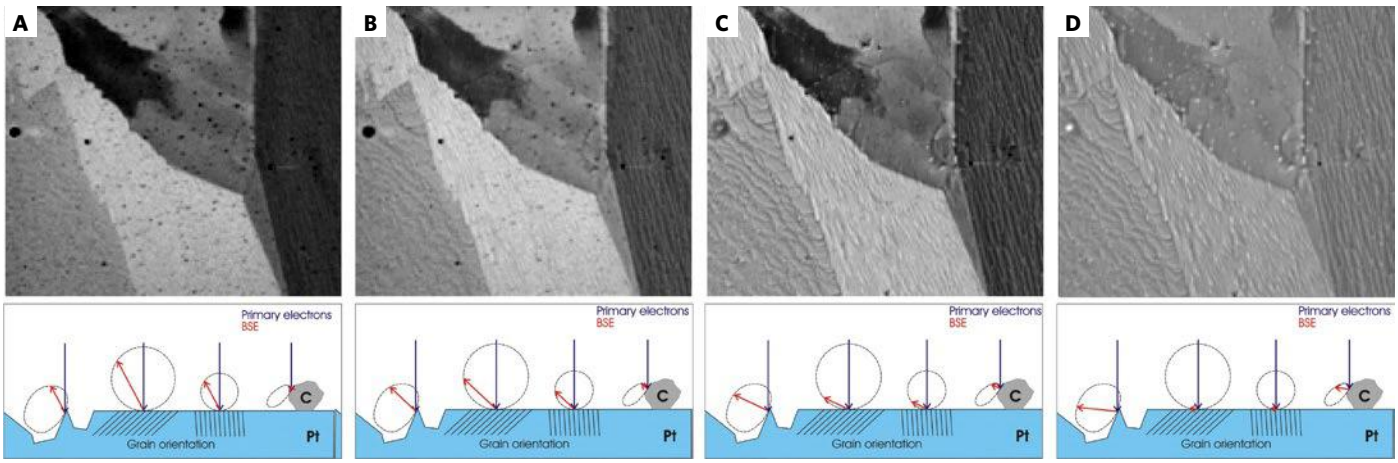

Figure 6. Collecting BSE images based on the angle of emission of back-scattered electrons generates material, topographic and grain orientation contrast.

Capturing all information

Traditionally, this kind of data acquisition has been performed with a four-quadrant BSE detector, where the working distance is altered to control the angle of BSEs detected. This entails repeated scanning of the sample or region of interest, which can lead to a number of unwanted side effects, including:

- **Contamination on the surface:** As the beam interacts with the sample and breaks down hydrocarbons present on the sample surface, details become obscured during further investigation. **Figure 7 (A)** shows where a first scan on the sample (GaAs) has generated a layer of carbon contamination, obscuring the sample surface for further low-voltage imaging.
- **Buildup of charge on non-conductive samples:** As the electron beam injects charge in the specimen, the charge buildup will begin to affect the incident electron beam, leading to deflection. **Figure 7 (B)** shows this effect clearly while imaging a chromosome on glass substrate.
- **Destruction of sample structure due to electron beam sensitivity:** Many materials are sensitive to the electron beam, and their structure can be destroyed during the investigation. For example, the electron beam causes many polymers to cross-link during exposure, which can lead to material shrinkage. This is particularly evident when imaging samples such as photoresists, and it can be critical when metrology is required. **Figure 7 (C)** shows a photoresist sample where the central region has been imaged at high magnification, leading to shrinkage and obvious damage to the photoresist lines.

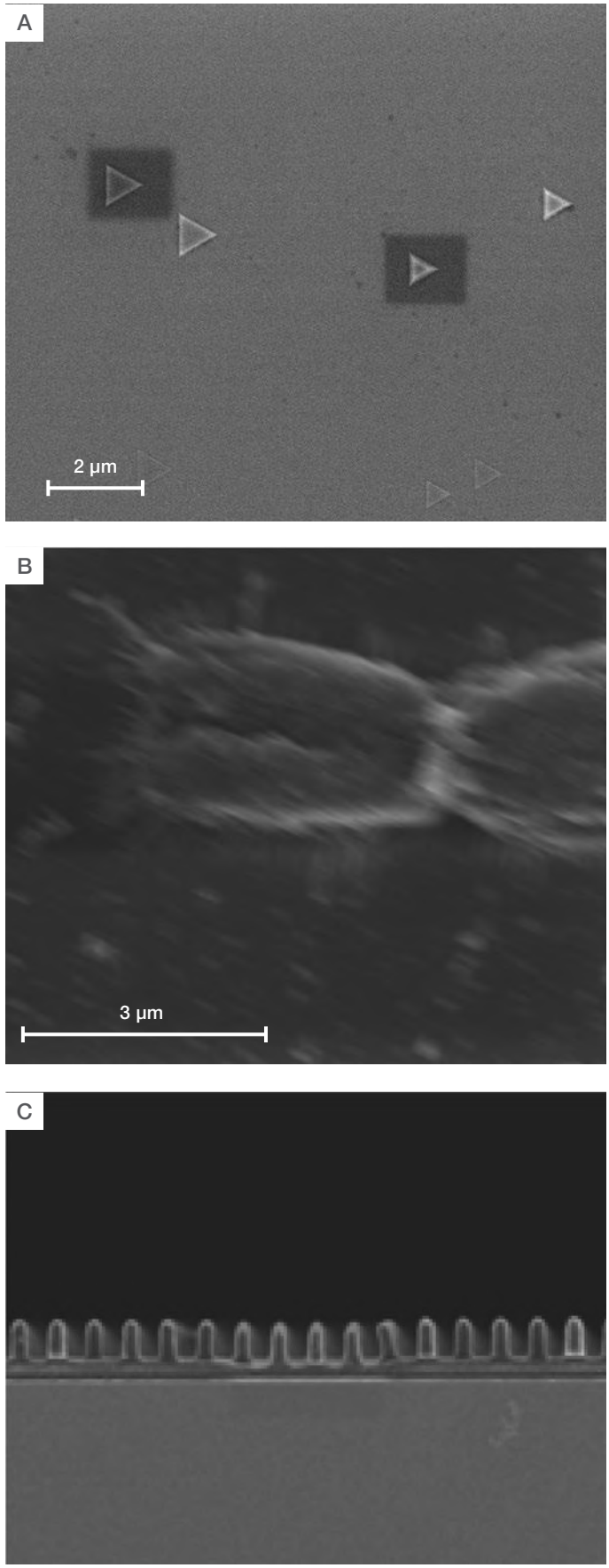

Figure 7. The set of images above depicts common side effects related to repeated sample scanning.

Benefits of a directional BSE detector (DBS)

With the introduction of the DBS family, it is possible to capture critical information without experiencing the aforementioned side effects. Using a concentric ring design (see **Figure 8**), backscatter signal can be easily separated by emittance angle, while four separate rings allow for simultaneous detection of multiple signals.

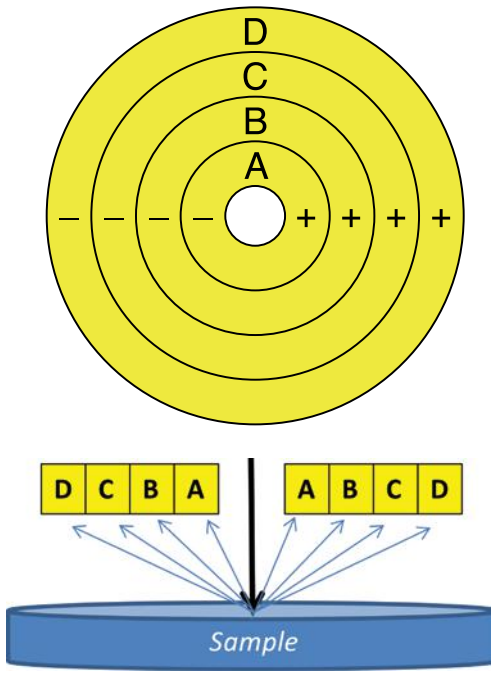

Figure 8. Schematic of angular separation of BSE detection and detector layout.

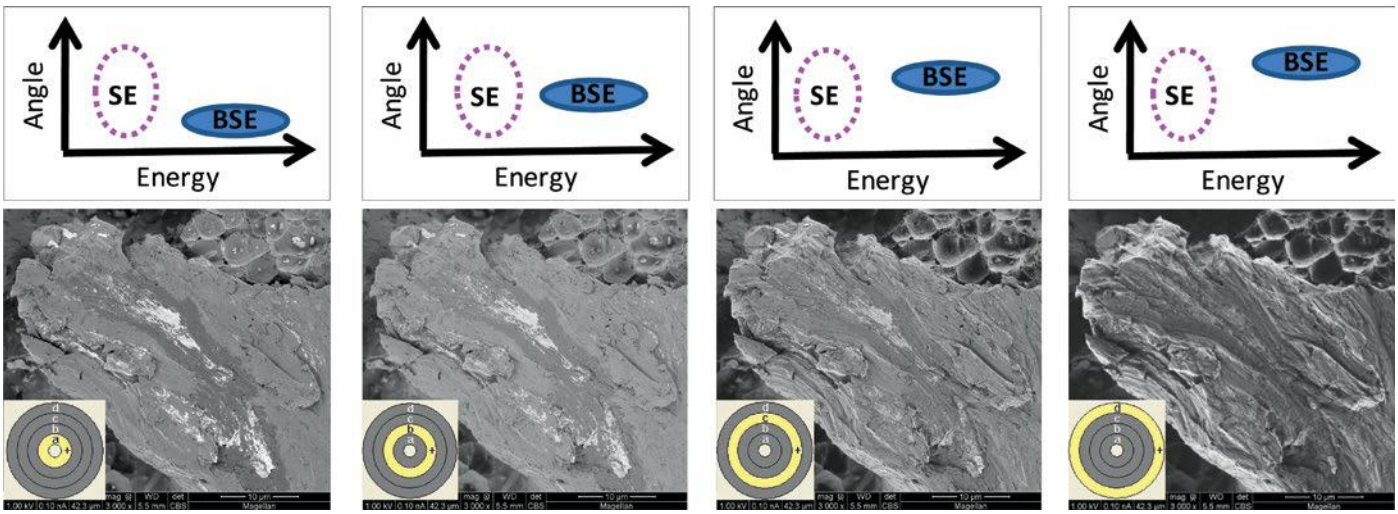

Figure 9. Simultaneously acquired BSE images of a fractured aluminum sample with inclusions acquired at 1 keV.

The example in **Figure 9** shows four separate images of an aluminum fracture, acquired simultaneously by utilizing all four rings of the DBS. Detection data from ring A, which is closest to the primary beam axis, shows a high degree of compositional contrast, highlighting the location of the inclusions in the aluminum. By moving toward ring D, more topographical information about the sample can be obtained.

Most importantly, you can acquire all these images simultaneously at a working distance that is optimized for other analytical techniques [e.g., X-ray energy dispersive spectroscopy (EDX) and electron backscatter diffraction (EBSD) analysis]. The ability to capture available information in a single scan is critical not only to avoid unwanted sample side effects, but also to maintain the SEM's ease of use.

A large number of BSEs leave the sample at high angles, with their trajectories close to the sample surface. This signal typically remains undetected since the angle is too high for collection (see **Figure 10**). This loss of signal can lead to noisier images and loss of information; however, one way to capture this signal is to employ beam deceleration technology. A bias is applied to the stage, which decelerates the primary beam and affects the BSE signal by bending back on axis towards the DBS. By simply varying the stage bias, you can control which BSE angles are detected on each ring. The increased detection efficiency and accelerated signal enable the DBS to detect signals with landing energies as low as 50 eV, as seen in **Figure 10B**.

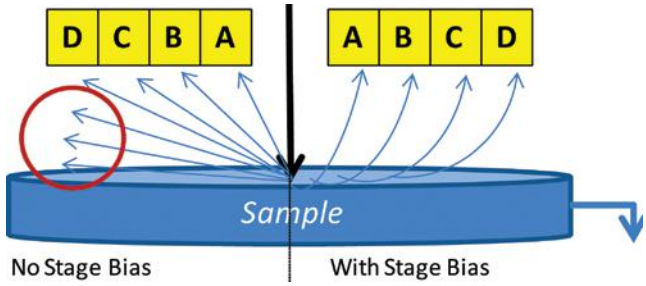

Figure 10A. Schematic showing lost BSE signal when no stage bias is applied (left) and when it is applied (right).

Additional benefits: filtering

The ability to separate signals with an annular detector is beneficial when imaging non-conductive material, where a typical side effect is the buildup of charge on the surface. This charge typically resides in the form of low-energy secondary electrons. By using a magnetic immersion lens, this charge-related signal can be trapped and forced into the final lens of the SEM. This signal is then detected with the in-lens detector, with the charge-related signal easily visualized in **Figure 11 (A)**. Depending on the working distance, some of this signal can still strike the ring closest to the primary beam axis (ring A) when the DBS detector is inserted. With all rings enabled, the reduced charge effect is seen in **Figure 11 (B)**. By simply disabling ring A, it is then possible to filter the charge away and image the non-conductive material, as can be seen in **Figure 11 (C)**, where all charge related information has been removed from the image.

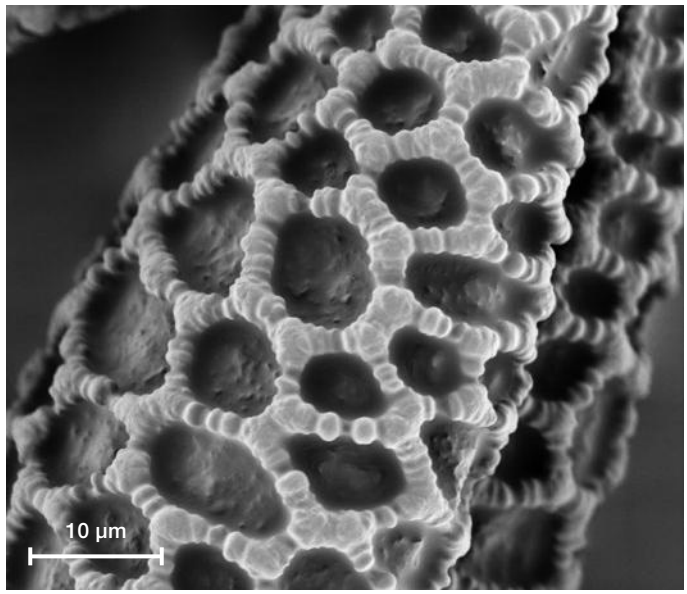

Figure 10B. BSE image of an uncoated pollen sample acquired with a landing energy of just 50 eV.

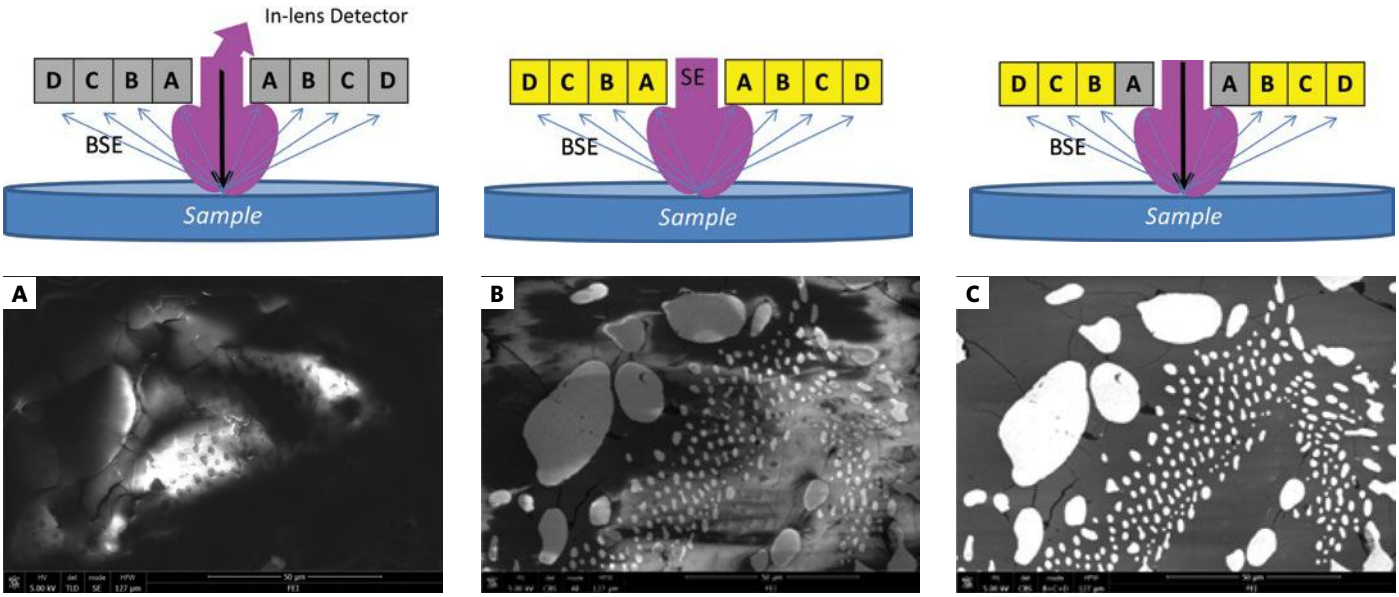

Figure 11. Images of an uncoated ceramic sample acquired at 5 keV. The in-lens detector shows charge-related information (A). Switching to the DBS with all rings selected (B) shows most charge removed, but still some artifacts. Switching off ring A shows a charge-free image (C) without changing other parameters.

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Summary
Providing the ability to capture information from all angles, the DBS offers the following benefits:

- Acquires all information in a single scan
- Eliminates additional sample damage or contamination
- Removes landing energy limitations
- Obtains charge-free imaging of non-conductive samples

Get your information from every angle—the Directional Backscatter Detector is available on all Thermo Scientific SEM and DualBeam systems. Contact your local Thermo Fisher Scientific representative or visit us at ThermoFisher.com/EM to discover how we can help you achieve more with your research.

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