WHITE PAPER

Advanced SEM Imaging with the Trinity Detection System

Petr Wandrol, EJR Vesseur,
Radovan Vasina, Alex Ilitchev

Introduction

Fundamentally, the quality of a scanning electron microscope (SEM) image depends on the quality of the detected electrons. Although traditional SEM design employs an Everhardt-Thornley detector (ETD) for secondary electrons (SE) and a below-the-lens detector for backscattered electrons (BSE), advanced SEMs can potentially be equipped with multiple in-lens detectors. As these can collect both SE and BSE signals, the observed electrons could be sorted according to their energies and/or emission angles. The Thermo Scientific™ Trinity™ Detection System, available in the Thermo Scientific™ Apreo SEM (with NICol column) and Scios™ DualBeam, is described here. It consists of three detectors: two in-lens (T1, T2) and one in-column (T3). This unique system offers unparalleled SE and BSE contrast as well as detailed information on sample composition, morphology, surface features, and more.

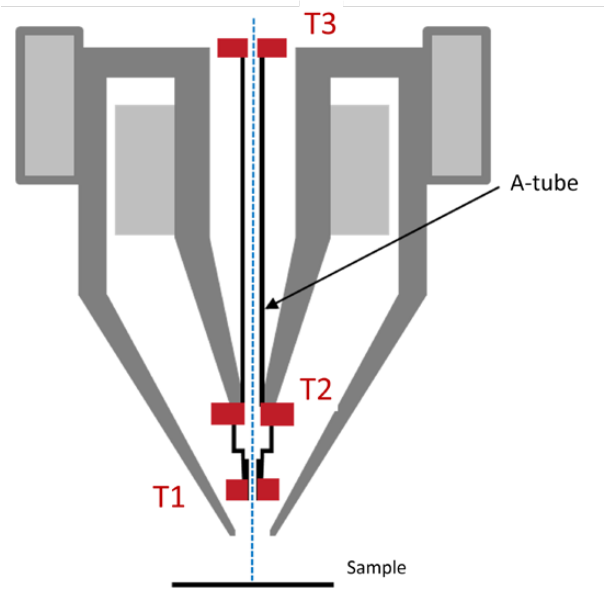

Figure 1. Schematic drawing of the NICol column with the Trinity Detection System. T1 and T2 are in-lens, whereas T3 is in-column.

Trinity Detection System

The Trinity detectors are placed along an A-tube biased at 8 kV; the potential of the A-tube attracts signal electrons to them (**Figure 1**). Positioned very low in the column is T1, which detects the intense signal of the high-energy backscattered electrons. Secondary electrons do not have the energy to reach the T1 detector; the electric field instead focuses them into the hole of T1, and they are subsequently detected by the T2 detector. Secondary electrons with even lower energy (<2 eV) pass T2 and are detected by the T3 detector (**Figure 2**).

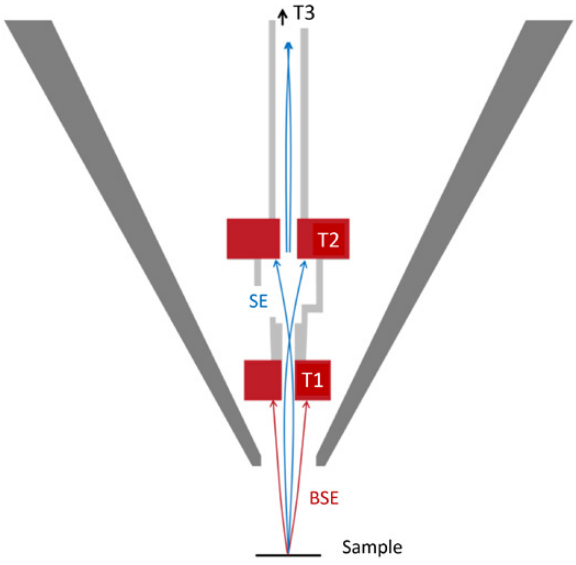

Figure 2. Illustration of the SE and BSE trajectories in the Trinity Detection System at analytical working distance (WD). SE, influenced by the electric field, bend toward the beam axis and pass to T2 and T3. BSE are less sensitive and are detected by T1.

ThermoFisher SCIENTIFIC

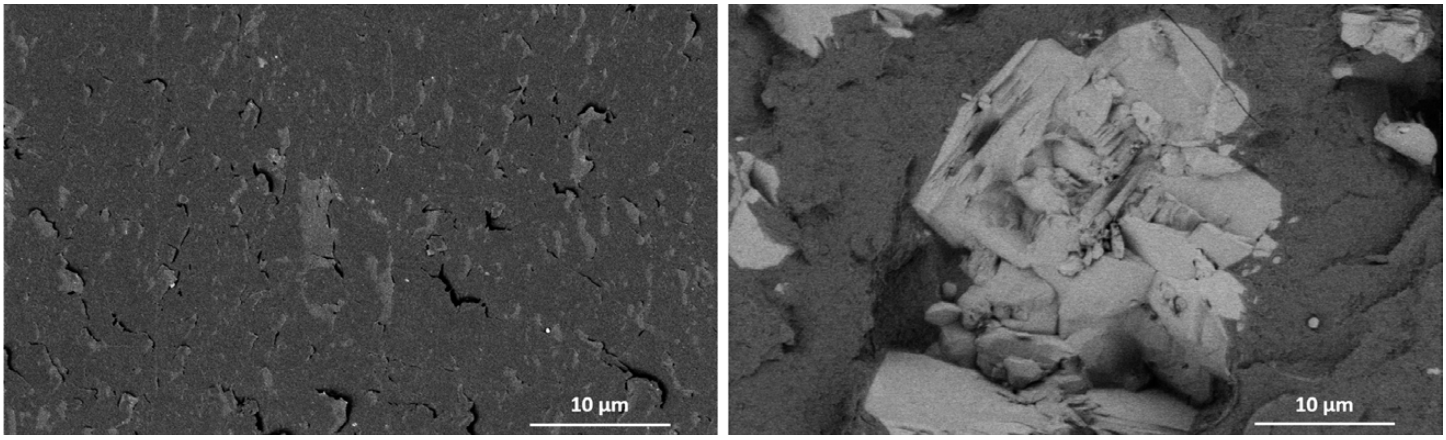

Figure 3. T1 compositional contrast images of a HDPE/COC polymer blend (left) and an epoxy resin filled with aluminum trihydrate microparticles (right). Both images were acquired at 500 V accelerating voltage. *Samples courtesy of T. Vackova and M. Slouf, Institute of Macromolecular Chemistry of the Academy of Sciences of the Czech Republic.*

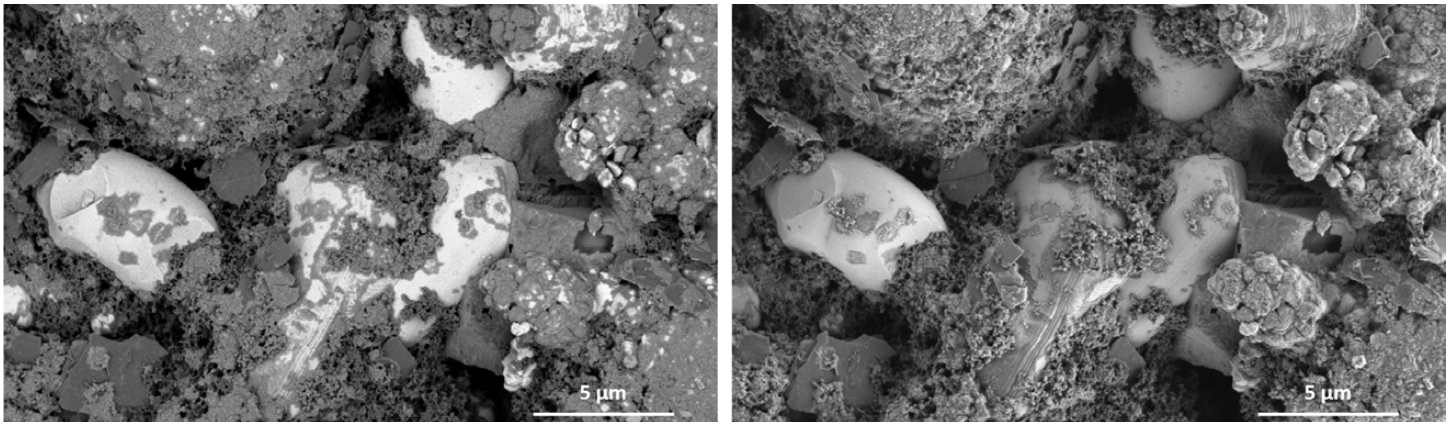

Figure 4. Lithium-ion battery cathode imaged by high-angle BSE (T1 detector, left) and low-angle BSE (ETD detector, right) at a working distance of 10 mm. *Sample courtesy of T. Kazda, Brno University of Technology.*

Backscattered electron detection

The intense BSE signal on T1 is one of the greatest benefits of the NICol column design. In a conventional SEM, BSE are either detected with a below-the-lens detector or by an in-column detector. In the Trinity Detection System, the T1 BSE detector is positioned at the bottom of the final lens. As it is placed only a few millimeters above the pole piece, the opening angle of backscattered electrons that can reach the detector is far greater than it would be in typical systems, where the detector is higher up. This results in a superior signal to noise ratio (SNR), even at an analytical working distance of 10 mm. High SNR is mandatory for low-voltage and low-dose imaging, so this is particularly useful for beam- and charge-sensitive materials such as polymers and polymer composites. Two examples are shown in **Figure 3**: a polymer blend with very similar components (high-density polyethylene (HDPE) and cycloolefin copolymer (COC)) and an epoxy resin filled with aluminum trihydrate microparticles. Clear differentiation can be seen in both samples without any additional etching or contrasting.

Until now, these measurements would be performed with a BSE detector below the pole piece. That space is now free, granting remarkable freedom and safety while tilting and moving the stage, which is critical for DualBeam™ and EBSD applications. Alternatively, that area is now available for an ultra-microtome, large window EDS, cathodoluminescence detector or another accessory.

Angular selective BSE detection

Samples composed of materials with similar atomic numbers require additional analysis to resolve via BSE detection. Collecting a specific part of the angular BSE emission is one way to do so. Backscattered electrons emitted at angles close to the optical axis (high-angle BSE) provide compositional (atomic number) contrast, whereas BSE collected perpendicularly to the optical axis (low-angle BSE) give topographic insight. Using T1 and ETD, these signals can be segmented. When longer working distances are used, the angular range of the T1 detector becomes smaller (only collecting high-angle BSEs), while the ETD detector takes over the low-angle BSEs. (Secondary electrons are attracted by the electric field of the A-tube and do not reach the ETD detector.) The angular dependency of the BSE signal is demonstrated in **Figure 4**, with an acceptance diagram of approximate energies and angles shown in **Figure 5**.

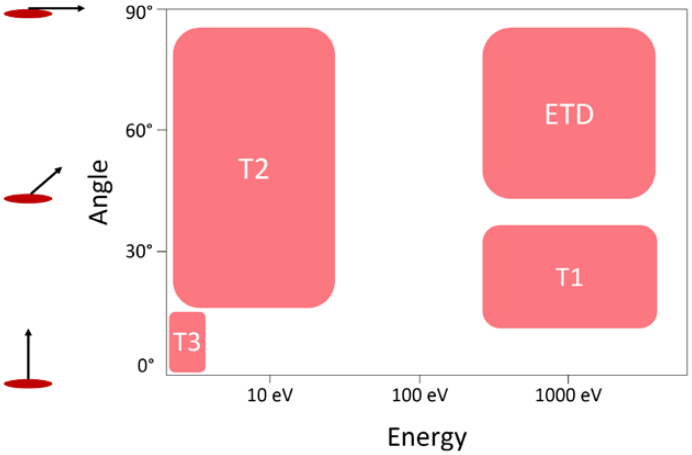

Figure 5. Acceptance diagram of angular and energy distributions collected by each detector at a working distance of 10 mm.

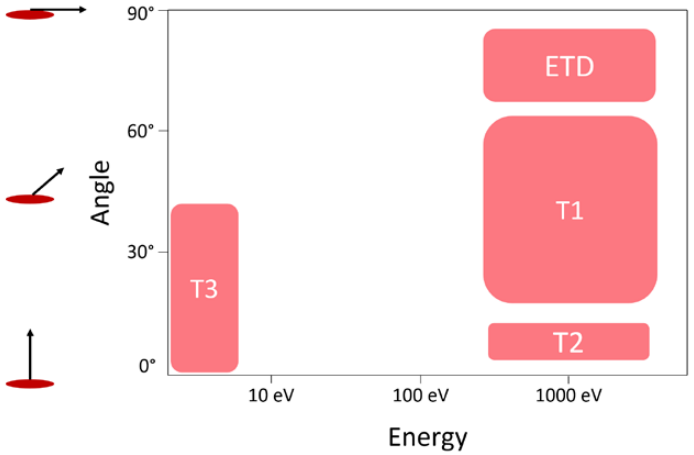

Figure 6. Acceptance diagram of angular and energy distributions collected by each detector at a working distance of 5 mm.

Although the T1 and ETD detectors cover nearly the whole angular range, a subset of highest-angle BSEs is not collected by T1. As these electrons are almost parallel to the beam axis, they provide strong compositional contrast, which is being lost. This can, however, easily be corrected by moving the sample to a 5 mm working distance, where the crossover of secondary electrons shifts into the hole of the T2 detector. The SE, therefore, pass through the hole and only the BSEs reach T2. In this configuration, the Trinity Detection System captures the complete emission of backscattered electrons separated into high, middle, and low angles (**Figure 6**). Imaging with the T1, T2, and ETD detectors can be done simultaneously. **Figure 7** shows the main components of a lithium ion battery cathode. The active material, the carbon filler and the solid electrolyte interface layer are clearly recognizable in the T2 high-angle BSE image (**Figure 7**, left). A mix of topographical and compositional contrast can be seen in the T1 image (**Figure 7**, middle), whereas exclusively topography is present in the ETD low-angle BSE image (**Figure 7**, right).

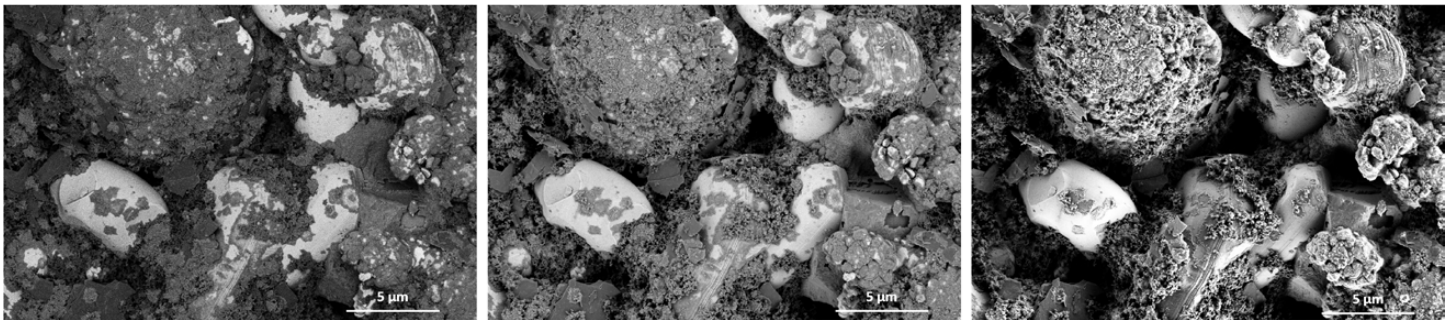

Figure 7. BSE images of a lithium-ion battery cathode acquired simultaneously by the T2 (high angle, left), T1 (medium angle, middle), and ETD (low angle, right) detectors. *Sample courtesy of T. Kazda, Brno University of Technology.*

Energy selective BSE detection

High compositional contrast can also be obtained by selectively detecting the highest-energy BSE, also called low-loss BSE. The dependence of the BSE coefficient (η) on atomic number is strongest for these electrons, so their selective detection would enhance contrast substantially. The Apreo SEM uses a compound final lens to collect low-loss BSE only. This lens combines the magnetic final lens of the pole piece, the immersion magnetic lens, and the electrostatic lens formed by T1. The main function of this compound lens is to focus the primary electron beam to the sample. Independent control of these three lenses is, however, possible and allows for the immersion magnetic lens to control the trajectories of the signal electrons. It directs the high-loss (lower energy) BSEs into the aperture of the annular T1 detector, isolating the low-loss BSEs for detection by T1 (**Figure 8**, left). The acceptance diagram, illustrating the difference between the unfiltered and filter BSE spectrum, is shown in **Figure 8** (right), and an example of this compound lens achieving high compositional contrast is shown in **Figure 9**. Here, clear differentiation of Ga and GaAs is seen on self-catalyzed GaAs nanowires grown by molecular beam epitaxy on Si(111) substrate.

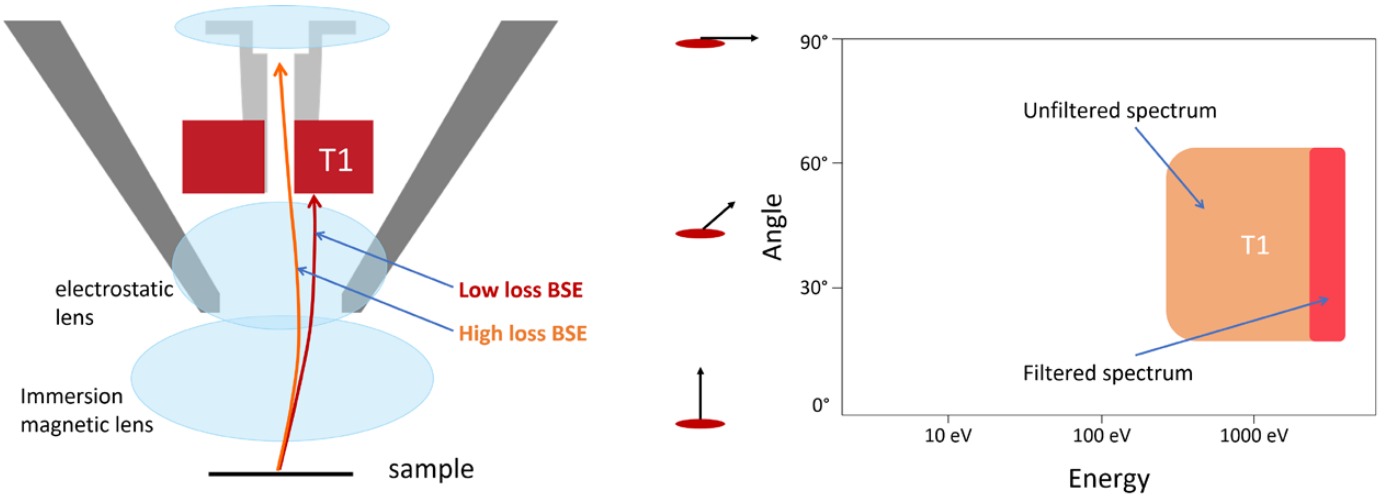

Figure 8. Diagram of BSE energy filtering with the compound final lens (left) with its corresponding acceptance diagram (right).

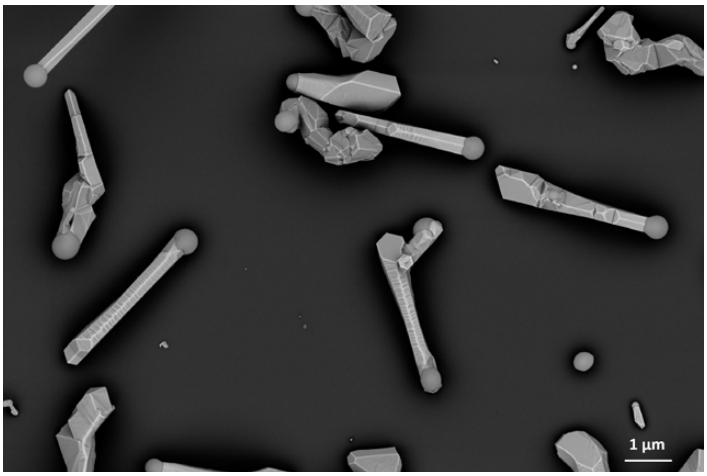

Figure 9. T1 low-loss BSE image of self-catalyzed GaAs nanowires on Si (111) substrate. Sample courtesy of David Fuster, Andrés Raya, Álvaro San Paulo and María Ujue González of the IMM-Instituto de Microelectrónica de Madrid (CNM-CSIC).

Naturally, BSE filtering cuts off a significant part of the T1 signal; however, the T1 detector keeps providing low-noise images at probe currents of 25 pA or lower while retaining atomic number information. The use of such small probe currents is critical for imaging non-conductive samples that often suffer from charging artifacts. Filtering of high-loss BSE that are influenced by sample charging enables charge-free imaging of these samples. Additionally, a side benefit of using the immersion magnetic lens as the BSE filter is improved resolution, as a smaller primary beam diameter results in better resolution.

Secondary electron detection

SE are typically used to characterize surface morphology and behave similarly to BSEs in the electric field. Those emitted parallel to the optical axis, with low energy, pass through the opening in the T2 detector and are collected further up in the column by T3. The slowest SE have energies between 0 and 2 eV and are the most surface-sensitive components of the emitted signal. They provide detailed information regarding small surface features and respond to surface electric fields, thereby delivering charge contrast information. SE with higher angles and energies (>2 eV but <50 eV) are detected by T2. They are less sensitive to sample charging but still provide morphological information. The example in Figure 10 highlights the benefit of simultaneous collection of high- and low-energy secondary electrons. The high-energy SE collected by T2 provide morphological information (**Figure 10**, left), whereas the low-energy SE collected by T3 allow for easy identification of organic residues, or oxidation, due to the local charge (**Figure 10**, right). Insulating samples in particular benefit from T2 SE detection, as these electrons are less sensitive to charging artifacts (**Figure 11**).

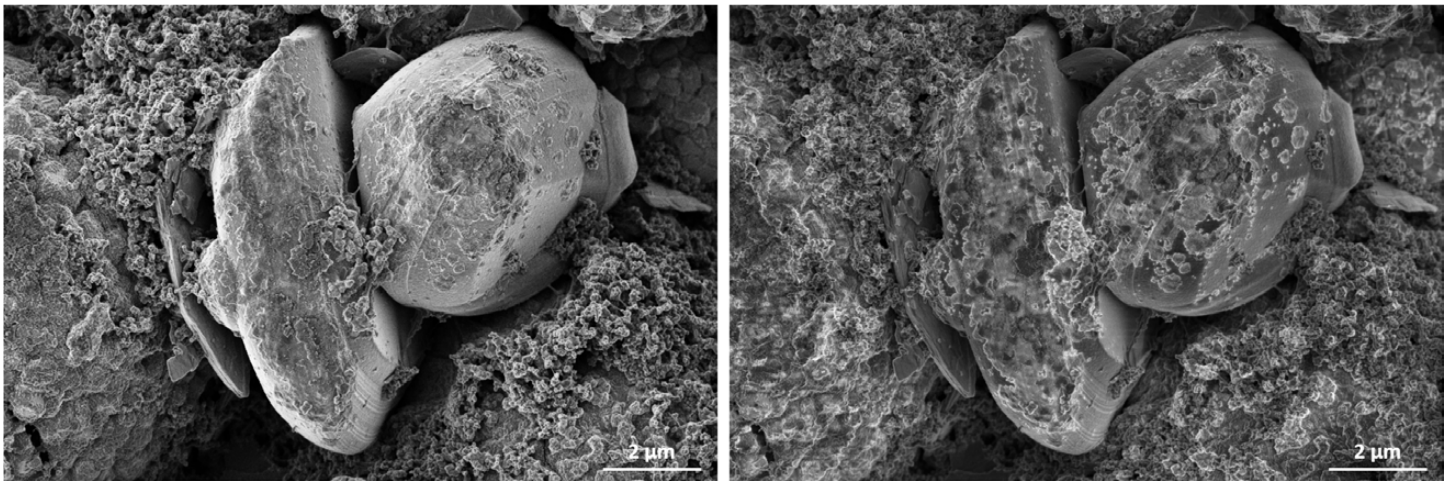

Figure 10. Detail of the active material in a Li ion battery cathode. High-energy SE collected by T2 reveal morphology (left), while low-energy SE on T3 are used to highlight small surface details and charge contrast (right). *Sample courtesy of T. Kazda, Brno University of Technology.*

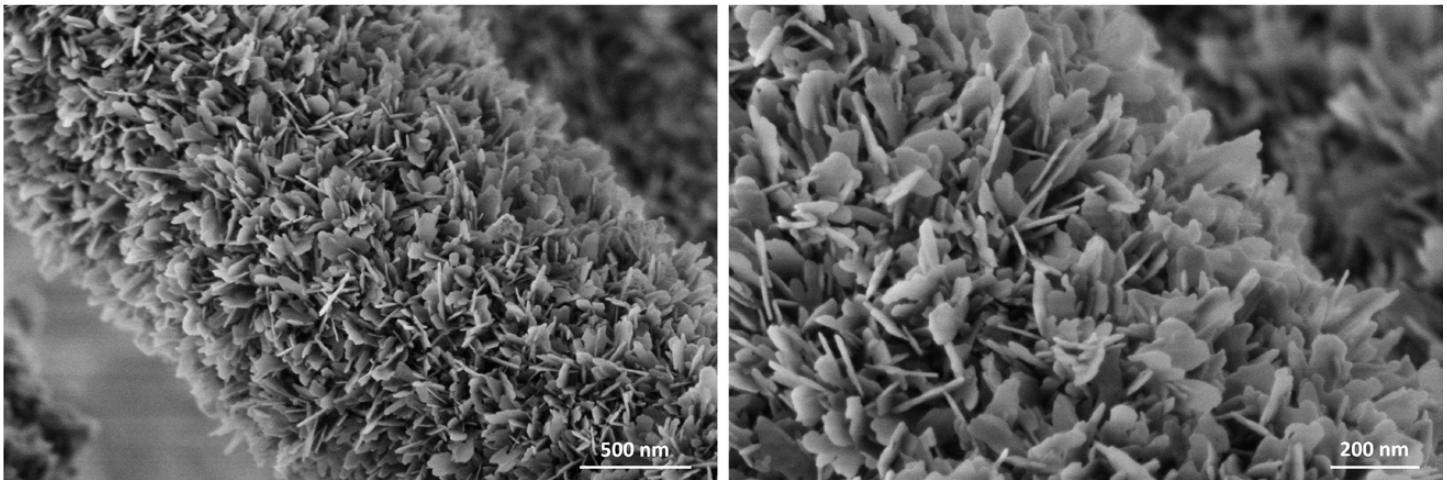

Figure 11. Uncoated hydroxyapatite nanosheets grown on bioactive glass fibers. Secondary electrons collected with the T2 detector are less sensitive to charge artifacts and reveal clear morphological features. *Sample courtesy of Devin Wu, Thermo Fisher Scientific, China.*

thermo scientific

Conclusions

Electron detection is one of the key determinants of quality for state-of-the-art SEMs. The unique design of the Trinity Detection System in Apreo SEMs and Scios DualBeams allows for selective detection based on energy and angle, providing enhanced contrast and information for both BSE and SE. For BSE, the entire angular distribution can be collected in a single scan, and thanks to the high signal to noise ratio of the T1 detector, BSE imaging can take place at low beam energies and currents. This is ideal for beam-sensitive and insulating materials. Finally, low-energy and emission-angle SE provide excellent surface information and charge contrast, whereas high-energy SEs with the Trinity Detection System allow for detailed morphological observations.

Find out more at **thermofisher.com/EM-sales**

ThermoFisher SCIENTIFIC