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APPLICATION NOTE

# Impact of Ion Type on FIB Cut Face Quality

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## Do ions matter for cut face quality?

The ability to mill and serial-section material using a focused ion beam (FIB) inherently depends on the interactions between the ion beam and the solid sample. Milling takes place as a result of physical sputtering of the target material. The bombarding ions and their energies, the target material and its crystallographic orientation and the angular direction of impinging ions with respect to the cut face all critically impact the quality of the milled cut face. It is widely recognized that a high-quality cross section is smooth and curtain-free, with a well-defined and sharp top edge along with a damage- and implantation-free superficial surface layer.

For most engineering and semiconductor materials (e.g. metals, cermets and ceramics) high-quality cross sections can be achieved with gallium or xenon FIBs. The best transmission electron microscopy (TEM) lamellas with ultra-low damage and the ultimate quality are prepared with xenon and argon ions. Nitrogen ions can be used to create nitride (e.g. TiN, BN, etc.) coatings with increased surface hardness, abrasion/wear resistance, lower friction and corrosion resistance. However, for soft, non-conductive materials such as polymers, tissue, etc. it is difficult to achieve good cut-face quality with high milling rates. Oxygen plasma FIB (PFIB) can overcome these difficulties.

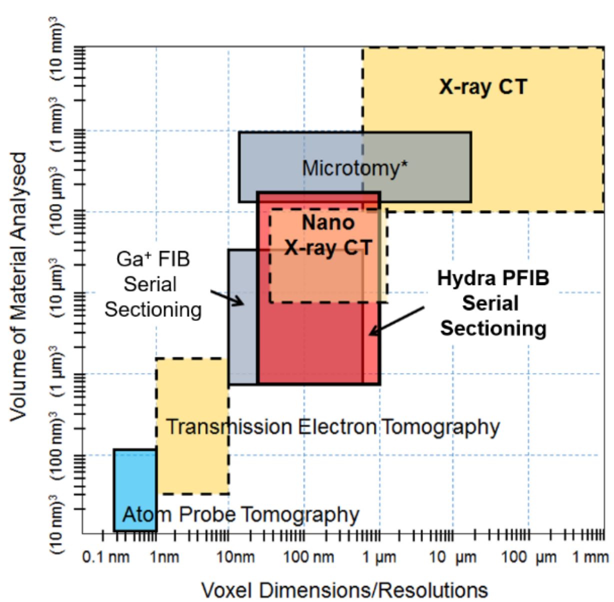


Figure 2. 3D imaging methods in materials science (non-destructive techniques represented by dashed lines; * - for soft materials only).

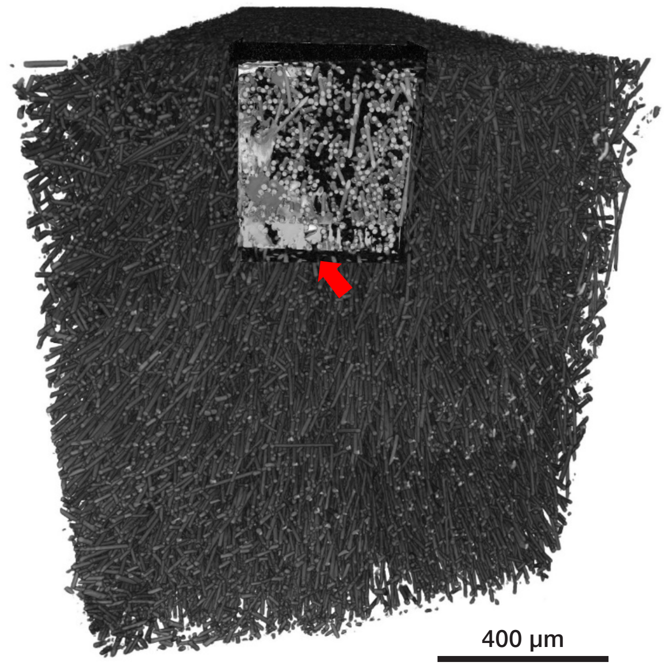


Figure 1. Shows 3D visualization of multi-scale and multi-modal (X-Ray CT followed by Hydra PFIB serial sectioning) investigation of sparsely distributed defects (marked with an arrow) in a glass-fiber-reinfoced polymer composite.

In this application note, the multi-ion species of the Thermo Scientific™ Helios™ Hydra DualBeam, combined with its fast ion switching (<10 minutes), are used to tailor the ion species and beam conditions for the highest quality results with the highest throughput possible. The use of xenon and oxygen ion beams on a glass-fiber-reinforced polymer composite (used in automotive applications) is compared via cut face quality, volume size and resulting 3D reconstruction (Figure 1). The importance of the Helios Hydra DualBeam in a correlative microscopy/tomography workflow will also be highlighted, as the PFIB establishes a vital connection between the macro- and nano-scales (Figure 2).

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In addition to a higher maximum current, which is useful for coarse milling activities, it is shown that the PFIB can operate at currents of 60 nA for the preparation of cross sections, providing good quality images. Note that potentially harmful chemical reactions, such as those which can results from $Ga^+$ FIB milling, are avoided thanks to the multi-ion species of the Helios Hydra DualBeam.

The DualBeam used in this study has a maximum current of 2.5 µA for $Xe^+$ and 3.8 µA for $O^+$ as compared to a top current of ~100 nA typical for many $Ga^+$ FIB systems. Thermo Scientific Auto Slice and View™ 4 (AS&V4) Software is used to manage the slicing and imaging process with integrated rocking polishing.

### Glass-fiber-reinforced polymer composite

The composite material consists of glass fibers (~33% vol.) that are approx. 15 microns in diameter and a nylon 66 resin matrix. This material retains its stiffness, strength and mechanical properties at high and low temperatures, as well as in aggressive chemicals and moisture. This composite is used in injection molding to form the desired shapes of automotive parts such as car oil filter casings (Figure 3a).

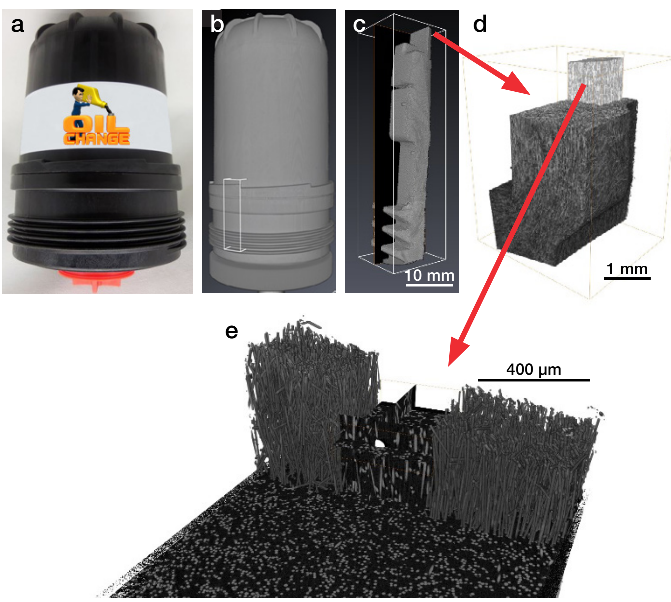


**Figure 3. The steps of locating the region of interest in car oil filter casing. (a) the casing; (b-e) reconstructed microCT volumes showing the location of fibers and the material defect.**

The Thermo Scientific HeliScan™ micro computed tomography (microCT) metrological scanner is used to locate suitable regions of interest (RoI) containing longitudinally arrange fibers and sparsely distributed defects. Based on the reconstructed microCT volume and Thermo Scientific Avizo™ Software, a small piece of material that contains the RoI was cut out (Figure 3b). Next, the correlative microscopy/tomography workflow, based on the universal sample holder and Thermo Scientific Maps™ Software, was used to precisely locate the site for serial sectioning tomography with AS&V4 Software (Figure 4).

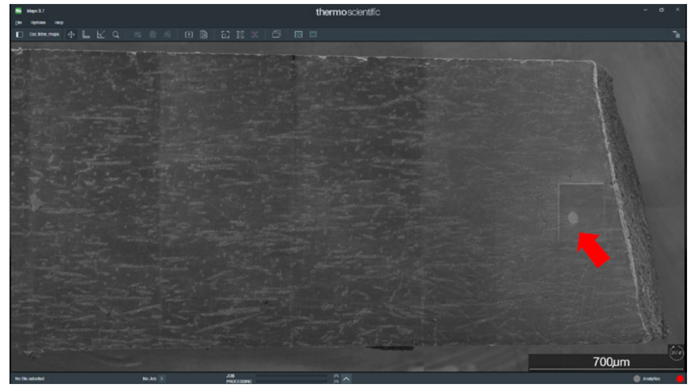


**Figure 4. Maps Software project that contains tiled SEM images of the sample surface, as well as a blended reconstruction of the microCT data volume. The region of interest is indicated with a red arrow.**

Before 3D data collection with AS&V4 Software, a series of experiments in the RoI's adjacent regions are done to optimize the cut face quality and the data acquisition speed using $Xe^+$ and $O^+$ ions. The direct comparison of the cut face quality for both ion species is presented in Figure 5. The top edges of the cut faces are presented in Figure 6. In this example AS&V4 runs are set at 30 kV and 60 nA, rocking polish at 3°, 25 nm/slice and 30 seconds of milling time. Simultaneously secondary electron (SE) imaging with the ETD detector (ETD-SE) and back scattered electron (BSE) imaging with the CBS detector (A, B rings) is performed using a 2 kV acceleration voltage and an electron current of 400 pA with a 1 µs dwell time and 60 s/image. All images have a resolution of 18 nm/pixel.

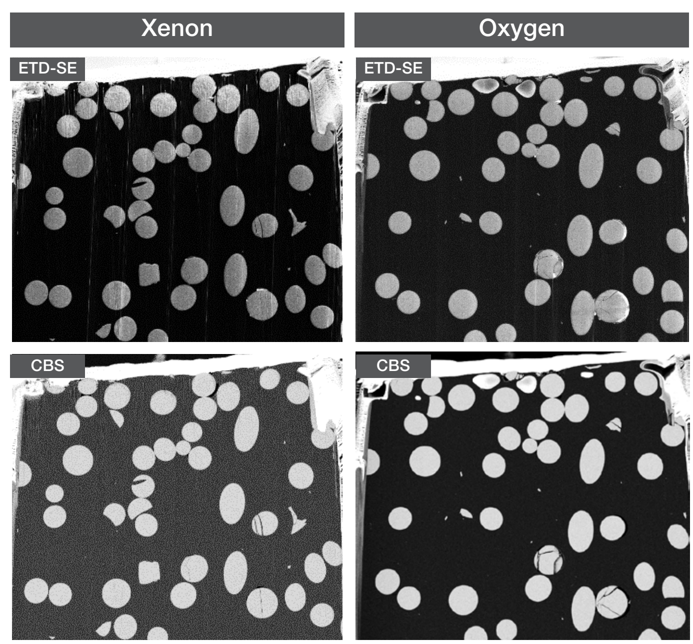


**Figure 5. Direct comparison of the cut face quality of the glass-fiber-reinforced polymer composite generated by xenon and oxygen focused ions (the SEM image width is 150 microns).**

Figures 5 and 6 show that serial sectioning with the $O^+$ PFIB creates far superior cross sections as compared to $Xe^+$. The cut face is smooth and curtain-free, capturing the smallest microstructural details at the nanometer scale and significantly simplifying further analysis, as nearly no post-processing is required. This clearly indicated that, depending on a sample material, the ion type can impact cut-face quality.

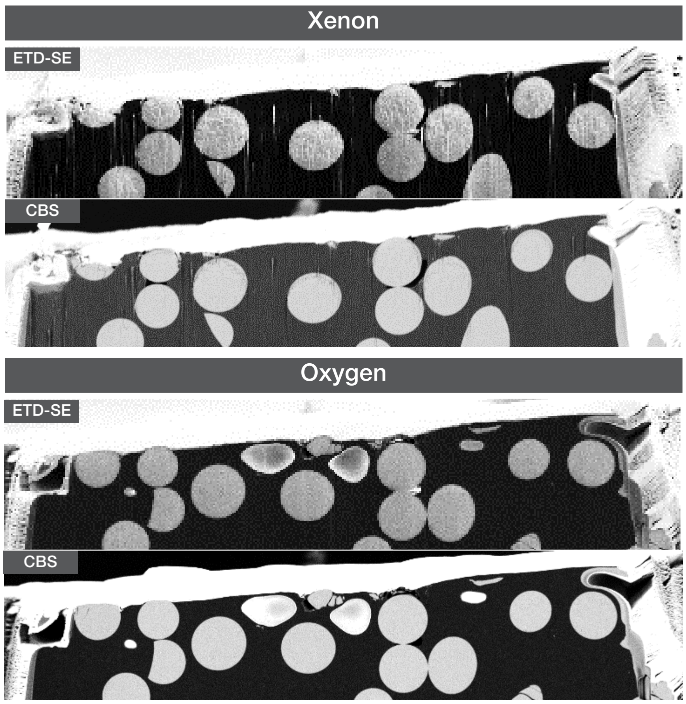


**Figure 6. Direct comparison of the top-edge cut-face quality of the glass-fiber-reinforced polymer composite generated by xenon and oxygen focused ions (the SEM image width is 150 microns).**

The $Xe^+$ PFIB can still be used for this kind of material, but the ion current needs to be reduced by an order of magnitude to efficiently suppress the curtaining phenomenon.

Subsequently 3D analysis with AS&V4 Software and the oxygen PFIB is collected using similar condition as in the test run. The correlated reconstructed material volume of about 400 × 400 × 200 $\mu m^3$ is presented on the front cover.

## Summary

The Thermo Scientific Helios Hydra DualBeam offers researchers the unique opportunity to perform 3D analysis on beam sensitive and difficult to mill microstructures, with sizes ranging from nanometers to hundreds of micrometers. This range of scales also bridges the gap between conventional $Ga^+$ FIB and 3D X-ray tomography. The DualBeam is an ideal instrument for the correlative tomography workflow; it enables the study of RoI over multiple scales by coupling microCT and serial-sectioning tomography, and it enables multiple types of data (structural, crystallographic, chemical, etc.) to be brought together for the same region.

The Helios Hydra DualBeam enables you to:

- Achieve Up to 100× higher throughput milling with various ion species ($Xe^+$, $O^+$, $Ar^+$ and $N^+$) as well as dedicated recipes and chemistries
- Gain the highest-resolution imaging with unique monochromator technology
- Acquire the most reliable and repeatable long-term data acquisition with the unique 5-axis piezo stage
- Prepare highest-quality 3D results with proven and optimized 3D acquisition packages (Auto Slice and View 4 Software)
- Perform the easiest and most powerful 3D data processing with Avizo Software

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## Notes

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