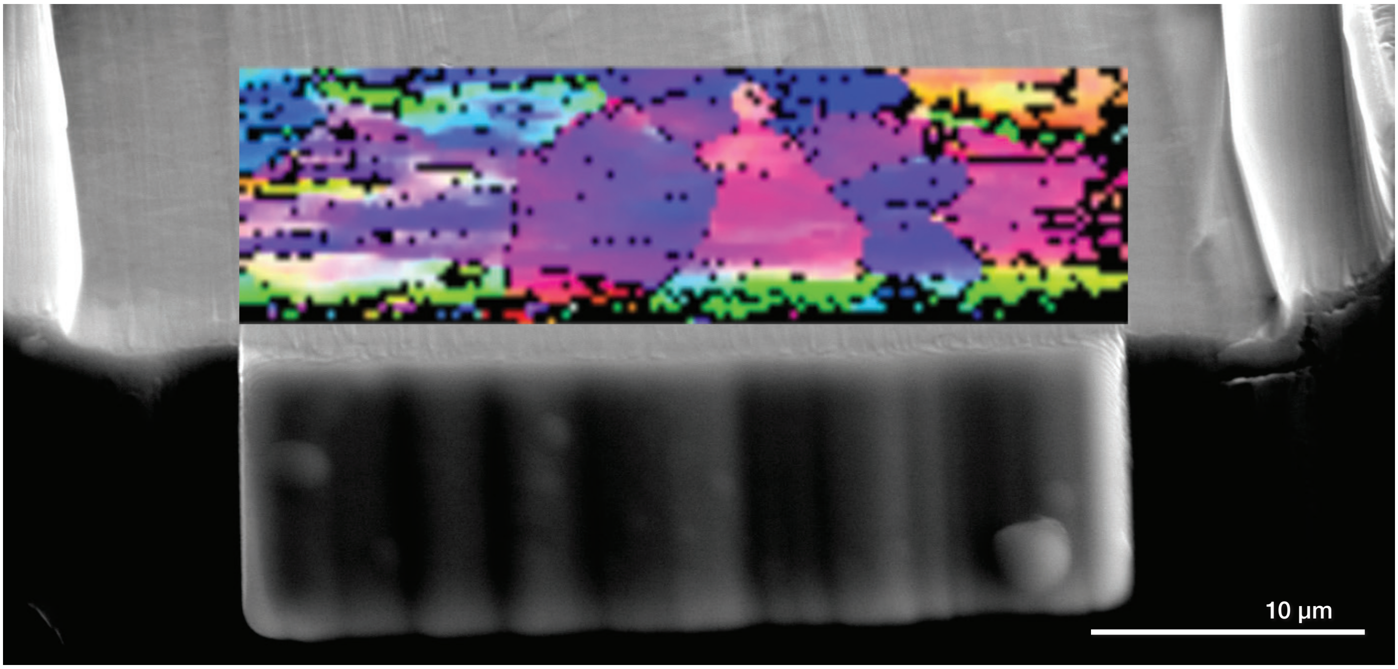
APPLICATION NOTE

Aluminum Sample Preparation

with Focused Ion Beam on Helios DualBeam

It is well known that focused ion beam (FIB) milling of aluminum and aluminum alloys with gallium ions can introduce artifacts, which hinder proper characterization of the material. Gallium has high solubility in aluminum and will generally concentrate along grain boundaries and interfaces. Next-generation plasma FIB (PFIB) instrumentation by Thermo Scientific employs a xenon ion source, allowing for artifact free aluminum micromachining and broadly enhanced sample preparation.



Aluminum cross-section generated on a Thermo Scientific™ Helios™ G4 PFIB DualBeam. Electron backscatter diffraction (EBSD, color) is overlaid onto an SEM image to show surface continuity and quality. Acquisition conditions: 20 keV (22 nA), 1X1 binning, 200 nm step size and acquisition rate of 5.5 Hz.

Gallium ion embrittlement of aluminum

Advanced engineering of aluminum (Al) and aluminum alloys necessitates a micro-scale understanding of the material's structure. Focused Ion Beam (FIB) milling, available on Thermo Scientific Helios™ DualBeam™ instruments, is commonly employed in metallurgical analysis to create TEM-thin samples and cross-sections. However, standard FIB employs a Ga^+ ion source; this is an issue for Al/Al alloy samples as gallium readily diffuses along grain boundaries and interfaces within the sample, causing what is known as liquid metal embrittlement (LME, Figure 1). This integration of gallium not only disrupts the mechanical strength of the aluminum but also acts as a significant imaging artifact. Thermo Scientific Plasma Focused Ion Beam (PFIB) instrumentation offers an alternative Xe^+ ion source which minimizes this damage while also increasing milling rates.

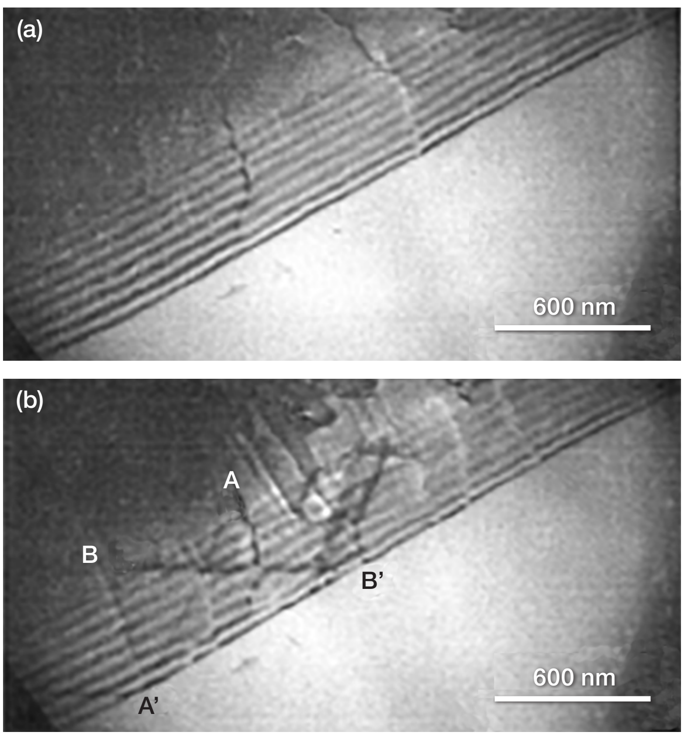


Figure 1. TEM image of aluminum grain boundary before (a) and after (b) introduction of gallium. Newly formed dislocations are shown in (b) as A-A' and B-B'. Adapted from Ref. 1.

Xenon PFIB damage

Whether preparing thin lamella or large cross-sections, the Xe^+ PFIB ion source offers numerous advantages over standard Ga^+ FIBs. The most significant is the total lack of LME milling artifacts in Al/Al alloy samples, improving not just single images but also larger datasets for statistical analysis. As the PFIB is capable of delivering 30-40x more current than standard FIBs there is a marked increase in measured sputter rate as well (about 25%, 0.31 µm3/nC [Ga] vs. 0.41 µm3/nC [Xe]), decreasing overall processing time.

Finally, despite the higher current and sputter rate of the Xe+ PFIB, it has a thinner damage layer than standard FIB. To demonstrate this, cross-sections of commercial aluminum samples (grade 6061 T6) were prepared on Helios G4 DualBeams with either Ga+ FIB or Xe+ PFIB ion sources. A protective platinum layer was applied via electron beam-induced deposition (EBID, at 2 keV). Samples were subsequently polished at 30, 5, and 2 kV using incident angles of 88.5°, 87° and 85° respectively. The resulting lamellae were then imaged using high resolution TEM (HRTEM) on a probe corrected Themis™ Z TEM, operating at 300 keV (Figure 2). At 2 kV the PFIB shows no Xe+ damage whereas the Ga+ FIB has a 1.8 nm damage depth.

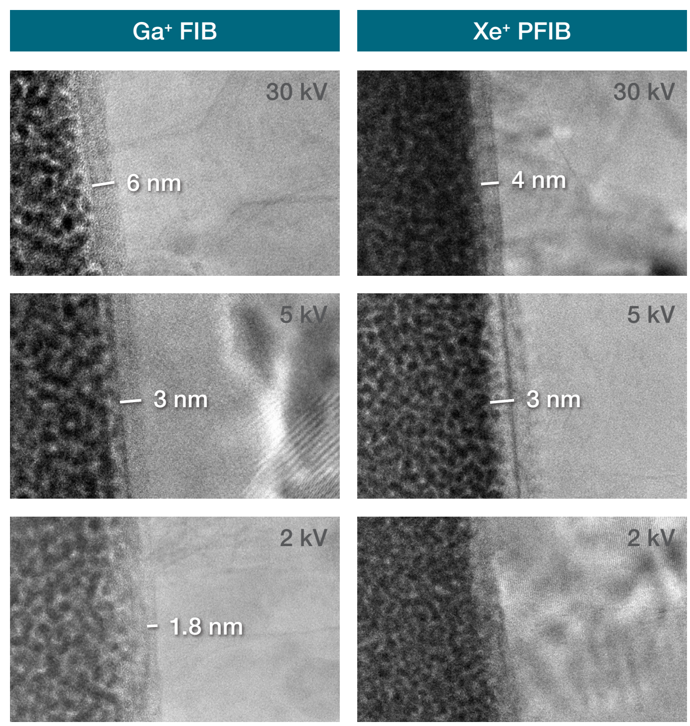


Figure 2. TEM image of ion beam damage depth into an EBID Pt layer on aluminum. At low FIB energies (bottom) the Xe^+ PFIB shows virtually no damage. Adapted from Ref. 2.

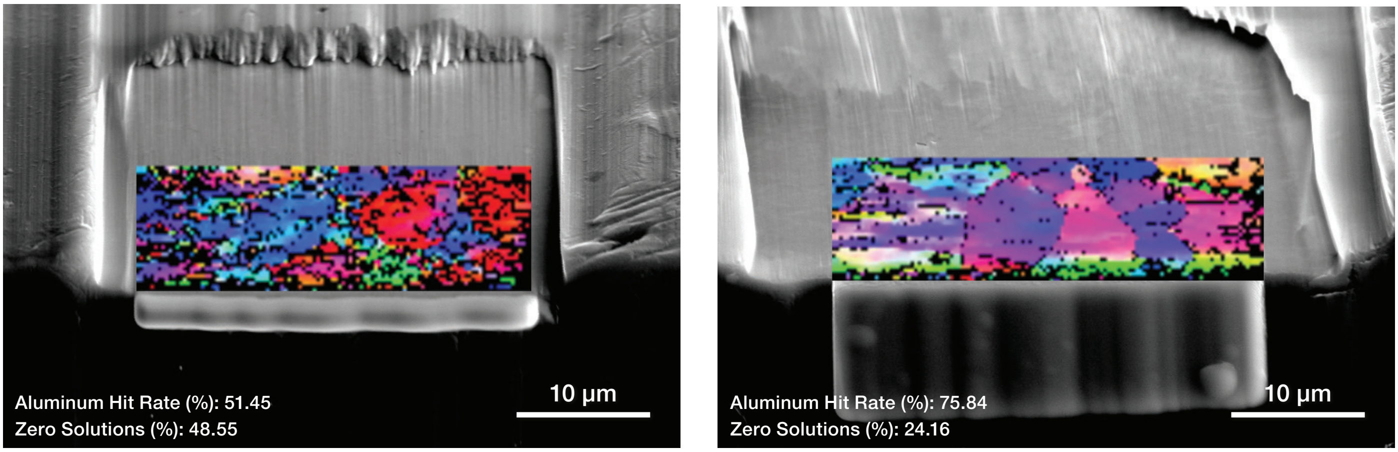


Figure 3. SEM of polished aluminum cross-sections created with Ga^+ FIB (left) and Xe^+ PFIB (right).

Cross-section electron backscattering quality with PFIB

Beyond sidewall damage on TEM samples, cross-section electron backscattering diffraction (EBSD) is also improved with the Xe^+ PFIB. Figure 3 shows comparative SEM images of aluminum cross-sections polished with either Ga^+ FIB or Xe^+ PFIB (at 30 kV). EBSD is overlaid in color. The PFIB sample shows a 25% increase in surface quality compared to the gallium polished surface.

Summary

- Aluminum and aluminum alloys are easily contaminated by the Ga^+ FIB, which can potentially hinder further characterization
- FIB sidewall damage is 25-30% lower with Xe^+ ions (PFIB) compared to Ga^+ ions
- High-current polishing at 30 kV with Xe^+ PFIB provides a significant improvement in EBSD results compared to Ga^+ FIB polishing

References

[1] R.C. Hugo and R.G. Hoagland, "Gallium penetration of aluminum: In-situ TEM observations at the penetration front", *Scr. Mater.*, 41, 1999, p.1341.
[2] F. B. Van Leer et al., "Ga^+ and Xe^+ FIB Milling and Measurement of FIB Damage in Aluminum", *Micros. Microanal.* 23 (S1), 2017, p.296.
[3] K.A. Unocic et al., "Effect of gallium focused ion beam milling on preparation of aluminium thin foils", *Journal of Microscopy*, 240, 2010, p.227.

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Notes

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