Quality Improvements of Xe^+ PFIB Prepared Surfaces of Aluminium

Van Leer, B.[1] and Passey, R.[1]

[1] Thermo Fisher Scientific, United States

Advanced engineering of materials containing aluminium requires a critical understanding of the material's microstructure. Cross-section and S/TEM sample preparation of aluminium and aluminium alloys to characterize grain boundaries or prepare surfaces for analytical work such as EBSD by focused ion beam (FIB) continues to be a major interest in metallurgical analysis because of FIB's ability to quickly prepare site specific specimens while eliminating damage from mechanical polishing or electro-polishing [1]. However, it is well known that gallium is particularly mobile in aluminium and its alloys, often diffusing along grain boundaries and interfaces, which can lead to embrittlement [2,3]. One attempt to mitigate this artifact is to subsequently polish the Ga^+ FIB prepared specimens in a board beam polisher to improve the sample's surface quality.

Characterization of large grain aluminium samples by Ga^+ FIB requires longer processing time when investigating multiple grains. Recent instrumentation using plasma FIB (PFIB) technology and Xe^+ ions offers an alternative ion species for FIB milling with increased milling rates because of its ability to deliver 30 - 40 times more current compared to Ga^+ FIBs. While the measured sputter rate of aluminum using Ga^+ and Xe^+ differs by about 25% (0.31 μm^3/nC [Ga] and 0.41 μm^3/nC [Xe]), the ability to prepare gallium free thin sections for S/TEM analysis or large area cross-sections offers a solution to FIB milling artifacts seen with a Ga^+ FIB. This also improves statistical analysis of grain populations.

To compare and contrast the effects of xenon ions to gallium ions, FIB prepared cross-sections and thin S/TEM specimens of commercial grade 6061 T6 aluminum were prepared using the Helios G4 DualBeam™ using 30 kV Ga^+ ions and a Helios G4 PFIB DualBeam using 30 kV Xe^+ ions. Sample quality of the cross-sections were evaluated by comparing EBSD index rates and band contrast. Sample quality of the S/TEM specimens were evaluated by measuring FIB sidewall damage and observation of Ga contamination at grain boundaries in a Themis Z™ TEM operating at 300 keV.

A comparison of FIB prepared cross-sections (30 kV; 15 nA) in 6061 T6 reveals a 25% increase in surface quality when using xenon ions. Figure 1 shows inverse pole figure maps overlaid with SEM images. A comparison of FIB sidewall damage between specimens prepared in Ga^+ FIB and Xe^+ PFIB revealed a decrease in sidewall damage by as much as 30% depending on the accelerating voltage applied. Chart 1 reveals FIB sidewall damage in 6061 T6 for xenon and gallium at 30 kV, 5 kV and 2 kV respectively.

References:

[1] L.A. Giannuzzi et al., Mater. Res. Soc. Symp. Proc. **480 (**1997) p 19.

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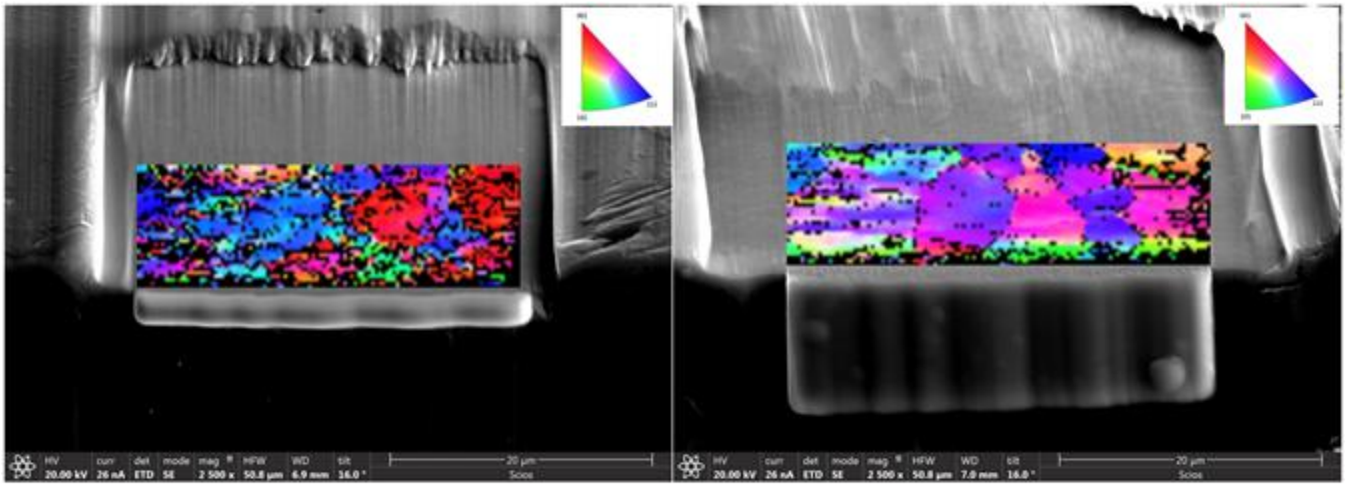

Figure 1. Inverse pole figure results of aluminium 6061 T6 with Ga^{+} prepared surface (left) and Xe^{+} prepared surface (right).

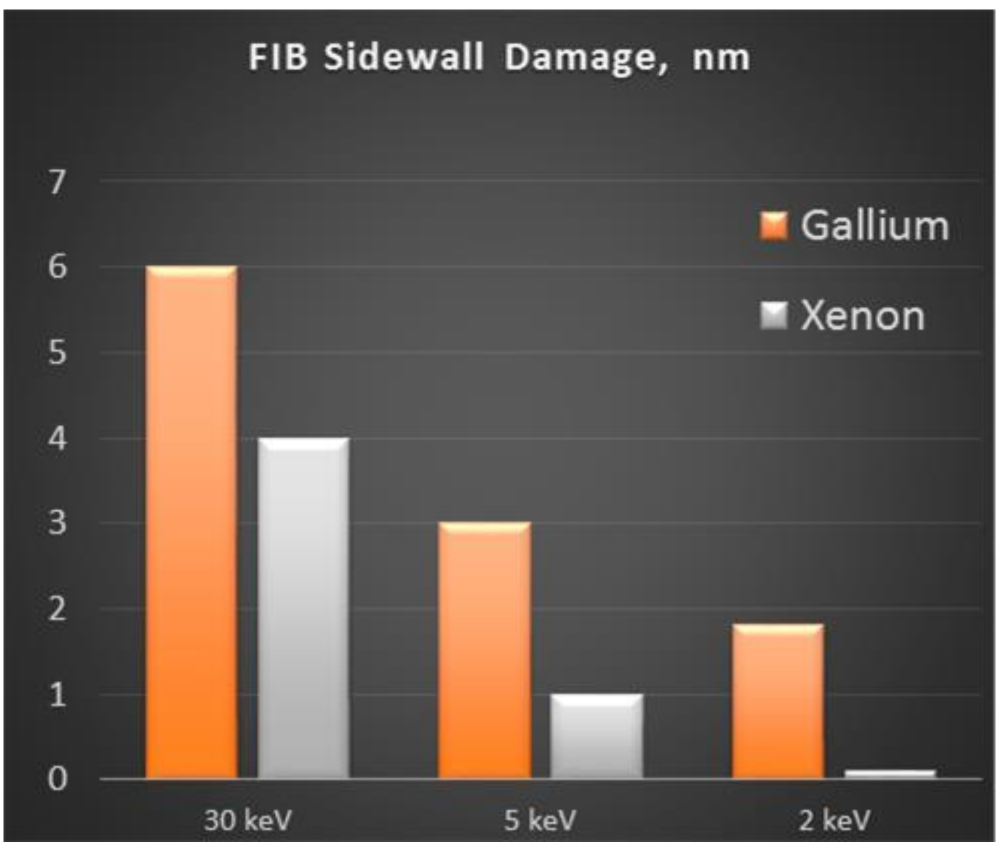

Chart 1. FIB sidewall damage in aluminium 6061 T6 at various accelerating voltages.