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Improvements in Characterization of FIB Prepared Surfaces of Aluminum Using Xe^+ Plasma FIB

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Critical to understanding the microstructure of aluminum and aluminum alloys is the careful surface preparation either by mechanical polishing or by ion beam techniques. Cross-section and S/TEM sample preparation of aluminium and aluminium alloys to characterize grain boundaries by focused ion beam (FIB) continues to be a major interest in metallurgical analysis because of FIB's ability to prepare site specific specimens while eliminating damage from mechanical polishing or electro-polishing [1]. However, gallium is particularly mobile in aluminium and known to diffuse along grain boundaries or interfaces, which can lead to embrittlement [2,3]. Often subsequent polishing in a broad beam polisher is a necessary step to improve the quality of the prepared surface for EBSD analysis. Also, characterizing large grain aluminum 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 and 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 $\mu m^3/nC$ [Ga] and 0.41 $\mu m^3/nC$ [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 as well as improving statistical analysis of grain populations.

Conventional cross-sections by FIB and S/TEM specimens of commercial grade 6061 T6 aluminum were prepared using the Helios G4 UX DualBeam™ using 30 kV Ga^+ ions and a Helios 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 sidewall damage between specimens prepared in Ga^+ FIB and Xe^+ PFIB revealed a decrease in sidewall by as much as 30% depending on the accelerating voltage applied. Figure 1 shows a SEM image the finished TEM lamellae of 6061 T6 aluminum as prepared in the Helios PFIB for FIB sidewall damage. Figure 2 shows a SEM image of a finished thin section of 6061 T6 aluminum across a grain boundary. EBSD results revealed that cross-section surfaces prepared with 30 kV Xe^+ PFIB had better band contrast results and improved indexing rates than cross-sections prepared with 30 kV Ga^+ FIB.

References:

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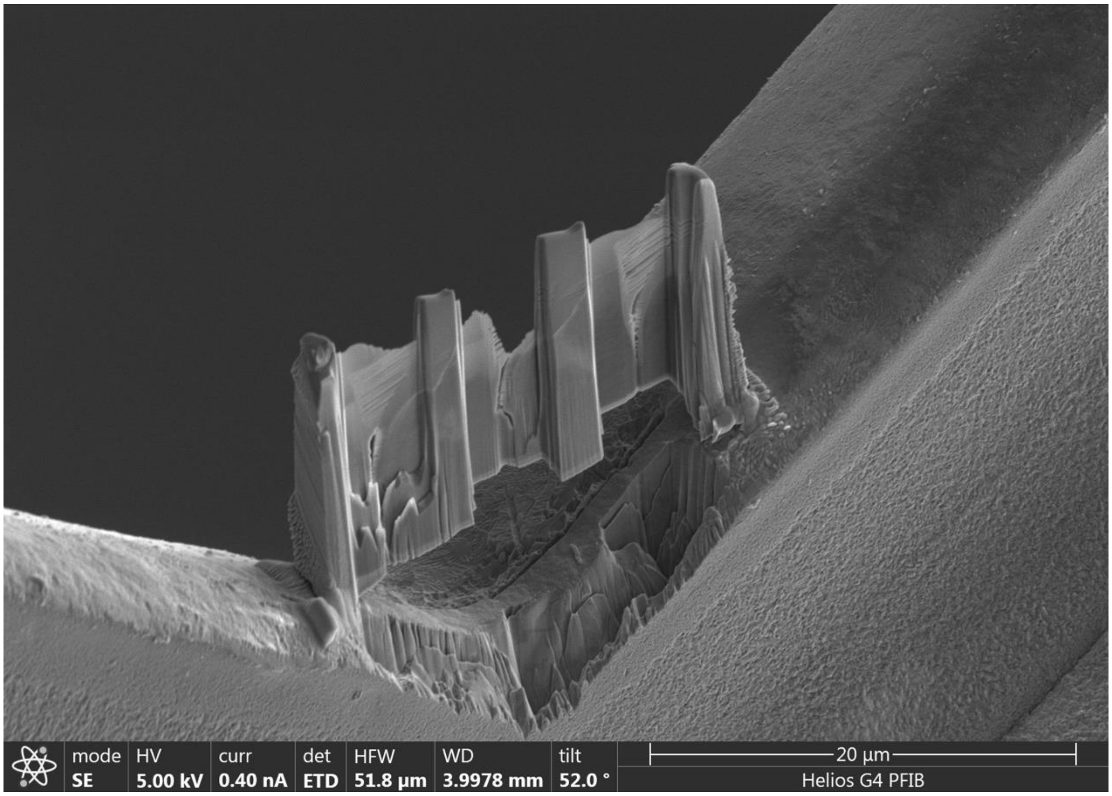

Figure 1. SEM image thin sectioned 6061 T6 aluminum characterizing the effect of accelerating voltage on FIB sidewall damage.

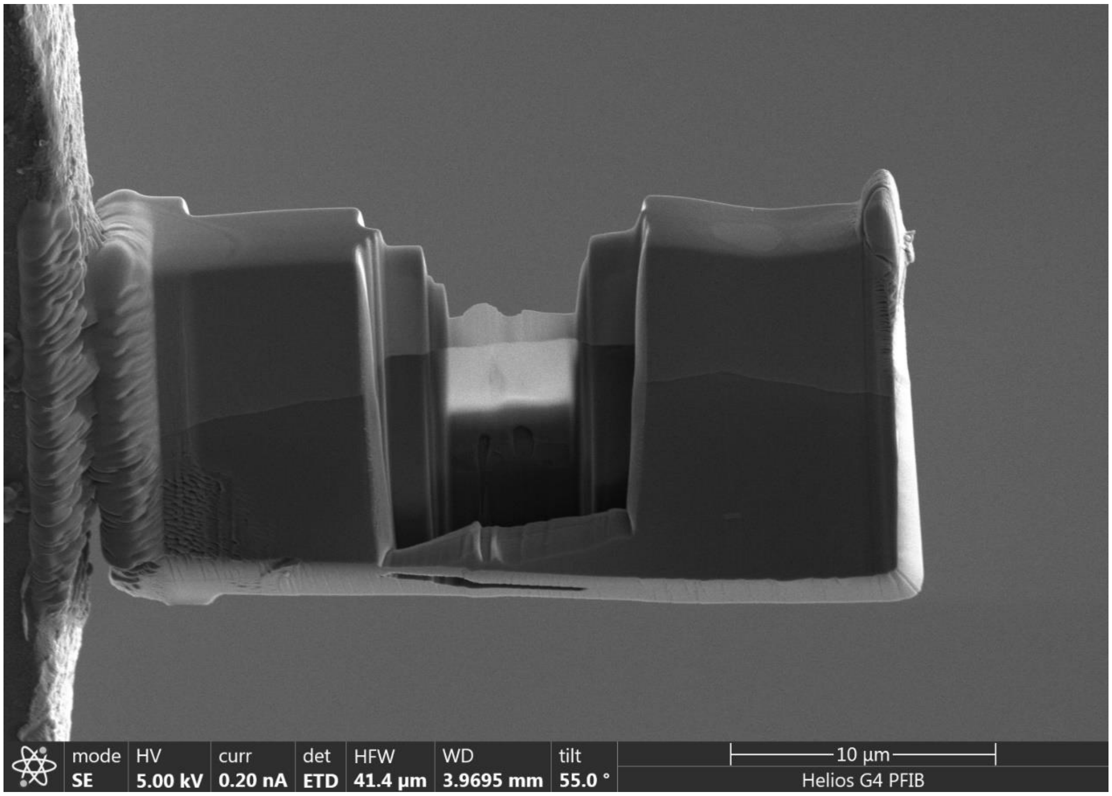

Figure 2. SEM image of thin sectioned 6061 T6 aluminum across a grain boundary.