

Ga⁺ and Xe⁺ FIB Milling and Measurement of FIB Damage in Aluminum

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S/TEM sample preparation of aluminium and aluminium alloys to characterize grain boundary phases by focused ion beam (FIB) continues to be a major interest in metallurgical analysis because of FIB's ability to prepare site specific specimens and eliminating damage from mechanical polishing or electro-polishing [1]. Recent instrumentation using plasma FIB (PFIB) technology and Xe⁺ ions offer 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 μm³/nC [Ga] and 0.41 μm³/nC [Xe]), the ability to use more current for micromachining will allow users to increase throughput significantly and prepare much larger cross-sections for S/TEM sample preparation if PFIB is employed. Therefore, it is of interest to understand the amount of FIB damage introduced into the sidewall of a thin section of aluminum by FIB. 30 kV FIB damage employing a different preparation method has been measured to be ~ 4 nm [2].

Cross-sections of commercial grade 6061 T6 aluminum were prepared using the Helios G4 UX DualBeam™ using Ga⁺ ions and a Helios PFIB DualBeam™ using Xe⁺ ions. Specimens were polished with energies of 30 kV, 5 kV and 2 kV using incident angles of 88.5°, 87° and 85° respectively. After protecting the cross-section surface with 2 keV Pt EBID, conventional in-situ lift-out TEM samples of the milled cross-sections were prepared using a Helios G4 UX DualBeam equipped with an EasyLift™ nanomanipulator. FIB damage was analyzed by HRTEM on a probe corrected Themis Z™ TEM operating at 300 keV.

Figure 1 shows HRTEM images of the FIB sidewall damage in the aluminum using Ga⁺ FIB and Xe⁺ PFIB milling with 30 kV, 5 kV and 2 kV, respectively. Sidewall damage using the Xe⁺ PFIB reduces FIB milled damage at 30 kV by more than 30%. Employing a 2 kV Xe⁺ polish resulted in no measurable FIB damage. Experimental agree with SRIM calculations [3]. Table 1 shows FIB sidewall damage for aluminum and silicon. As expected, decreasing accelerating voltage will decrease FIB damage.

References:

- [1] L.A. Giannuzzi *et al.*, *Mater. Res. Soc. Symp. Proc.* **480** (1997) pp. 19.
- [2] M. Presley in “The Formation of Amorphous and Crystalline Damage in Metallic and Semiconducting Materials under Gallium Ion Irradiation”, Ohio State University Doctoral Dissertation, (2016) p 80
- [3] JF Ziegler and JP Biersack, SRIM 2003, www.SRIM.com

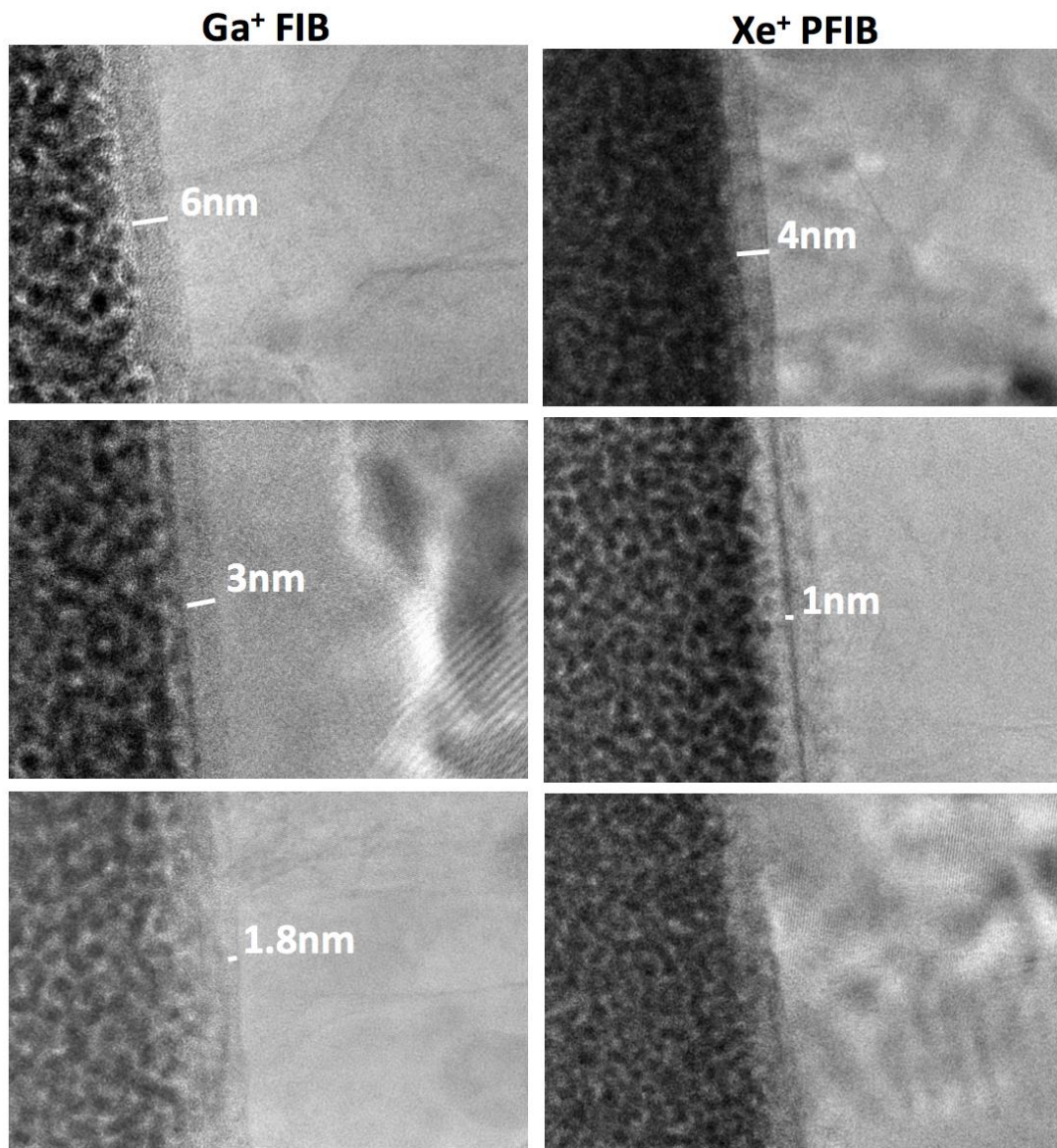


Figure 1. HRTEM images of sidewall FIB damage in Aluminum from a Ga⁺ FIB and Xe⁺ PFIB with 30 kV, 5 kV and 2 kV accelerating voltages.

Accelerating Voltage of Ions (kV)						
Target Material	30		5		2	
Ion Species	Ga ⁺	Xe ⁺	Ga ⁺	Xe ⁺	Ga ⁺	Xe ⁺
Aluminum	~ 6 nm	~ 4 nm	~ 3 nm	~1 nm	~ 1.8 nm	No Measured Damage
SRIM Calculations	6.8 nm	4.2 nm	2.1 nm	1.8 nm	1.4 nm	1.1 nm

Table 1. Summary table of sidewall FIB damage layer thickness (nm) in Aluminum after Ga⁺ and Xe⁺ milling with 30 kV, 5 kV and 2 kV.