

## Ga<sup>+</sup> and Xe<sup>+</sup> FIB Milling and Measurement of FIB Damage in Aluminum

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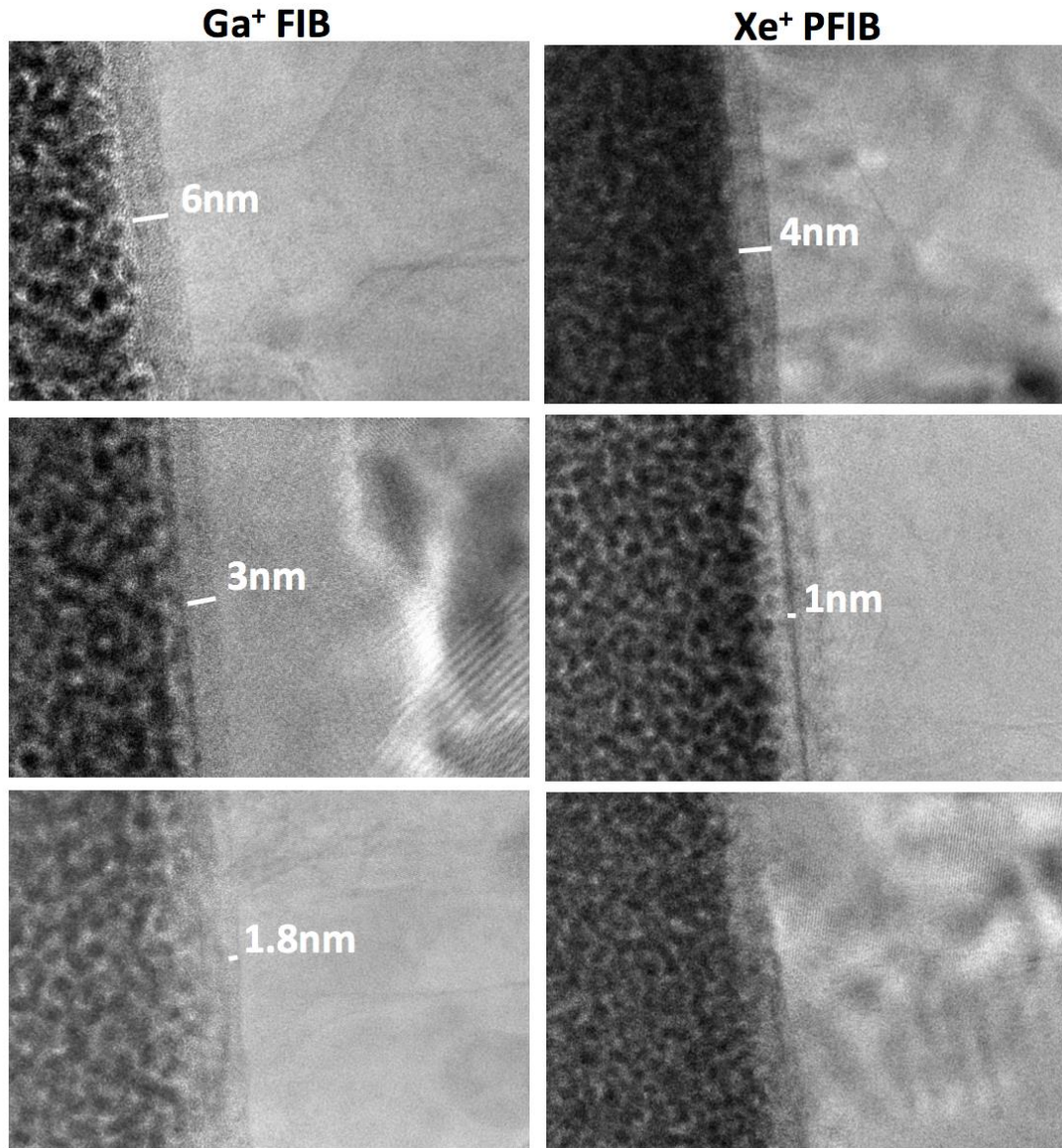
S/TEM sample preparation of aluminium and aluminium alloys to characterize grain boundary phases by focused ion beam (FIB) continues to be a major interest in metallurgical analysis because of FIB's ability to prepare site specific specimens and eliminating damage from mechanical polishing or electro-polishing [1]. Recent instrumentation using plasma FIB (PFIB) technology and Xe<sup>+</sup> ions offer increased milling rates because of its ability to deliver 30 – 40 times more current compared to Ga<sup>+</sup> FIBs. While the measured sputter rate of aluminum using Ga<sup>+</sup> and Xe<sup>+</sup> differs by about 25% (0.31 μm<sup>3</sup>/nC [Ga] and 0.41 μm<sup>3</sup>/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<sup>+</sup> ions and a Helios PFIB DualBeam™ using Xe<sup>+</sup> 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<sup>+</sup> FIB and Xe<sup>+</sup> PFIB milling with 30 kV, 5 kV and 2 kV, respectively. Sidewall damage using the Xe<sup>+</sup> PFIB reduces FIB milled damage at 30 kV by more than 30%. Employing a 2 kV Xe<sup>+</sup> 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](http://www.SRIM.com)



**Figure 1.** HRTEM images of sidewall FIB damage in Aluminum from a  $\text{Ga}^+$  FIB and  $\text{Xe}^+$  PFIB with 30 kV, 5 kV and 2 kV accelerating voltages.

Accelerating Voltage of Ions (kV)						
Target Material	30		5		2	
Ion Species	$\text{Ga}^+$	$\text{Xe}^+$	$\text{Ga}^+$	$\text{Xe}^+$	$\text{Ga}^+$	$\text{Xe}^+$
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  $\text{Ga}^+$  and  $\text{Xe}^+$  milling with 30 kV, 5 kV and 2 kV.