Ar⁺ FIB Milling and Measurement of FIB Damage in Silicon

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Minimizing surface damage during FIB specimen preparation is an important factor for high quality images analytical results and S/TEM sample preparation. Using conventional Ga⁺ FIB milling, techniques using reduced accelerating voltages for final polishing to minimize sample damage are commonly employed [1]. Nowadays, new column and source technologies allow for multiple ion species to be employed for sputtering at high and low accelerating voltages. With the introduction of these alternative ion species for milling, it is of interest to characterize the ion-solid interaction with respect to FIB sidewall damage in an effort to understand whether ion species other than Ga⁺ might produce better TEM samples for high resolution STEM (HR-STEM) imaging.

Previous studies on single crystal silicon have characterized gallium and xenon FIB sidewall damage to be ~ 22 nm and ~ 13 nm, respectively, using an accelerating voltage of 30 kV [1, 2]. Smaller sidewall damage layers are observed with lower accelerating voltages. On the same substrate, modeling indicates that milling with heavier ions will produce less sidewall damage than with a lighter ion of the same energy [3]. Therefore, one expects slightly more sidewall damage with argon (atomic number 18) than with gallium (atomic number 31) and xenon (atomic number 54). In this study, the sidewall damage on single crystal silicon after Ar⁺ FIB milling is presented.

Cross-sections of a blanket silicon wafer were prepared using a Thermo Scientific Helios Hydra Plasma FIB DualBeam. Specimens were polished with energies of 30, 5, and 2 kV using incident angles of 88°, 86°, 84° respectively. Each sidewall was protected using electron beam induced deposition (EBID) of 2 keV platinum. Conventional in-situ liftout TEM samples of the milled cross-sections were prepared using the Hydra equipped with an EasyLift nanomanipulator. Sidewall silicon damage was analyzed by HR-STEM on a Thermo Scientific ThemisZ operating at 300 keV.

Figs. 1a, 1b and 1c show HR-STEM images of the amorphous sidewall damage from Ar^+ FIB milling with 30 kV, 5 kV and 2 kV, respectively. As expected, the experimental results follow SRIM calculations and predictions from fundamental ion-solid interactions, which are shown with experimental results in Table 1 [3]. The Ar^+ sidewall amorphous damage decreases dramatically as a function of energy and is nearly 2 times larger than 30 kV Ga⁺ FIB and nearly 3 times larger than 30 kV Xe⁺ PFIB sidewall damage.

References:

- [1] LA Giannuzzi et al, Microscopy and Microanalysis 11 (2005).
- [2] B Van Leer et al, Microscopy and Microanalysis (2013).
- [3] JF Ziegler and JP Biersack, SRIM 2003, www.SRIM.com.



Figure 1. 300 keV HR-STEM images of sidewall amorphization damage in Si from a Ar^+ FIB with a) 30 kV, b) 5 kV, and c) 2 kV accelerating voltages.

Accelerating Voltage of Ions (kV)									
Target Material	30			5			2		
Ion Species	Ga ⁺	Xe ⁺	Ar ⁺	Ga^+	Xe ⁺	Ar^+	Ga ⁺	Xe ⁺	Ar^+
Silicon	~ 22 nm	~ 13 nm	$\sim 37 \text{ nm}$	~ 6 nm	$\sim 4 \text{ nm}$	~ 8.9 nm	$\sim 3 \text{ nm}$	$\sim 2 \text{ nm}$	~ 5.5 nm
SRIM Calculations	27 nm	15 nm	39 nm	5 nm	6 nm	8 nm	5.1 nm	4 nm	5.4 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.