

Ga ion-induced damage on FIB-prepared TEM specimens and its removal using narrow argon ion milling

Bonifacio, C.¹, Nowakowski, P.², Ray, M.², Fischione, P.¹ and Stumpf, M.W.²

¹ E.A. Fischione Instruments, Inc., United States, ² E.A. Fischione Instruments, Inc., United States

Transmission electron microscopy (TEM) specimens are typically prepared using a Ga focused ion beam (FIB) due to its site-specificity for thinning and extraction. [1] However, FIB milling may result in Ga ion-induced damage, which includes surface amorphization, Ga⁺ implanted layers, and defects; the extent of this damage is material-dependent.[2] Characterization of Ga-induced damage and its removal can be limited by the different experimental conditions multiple specimens are subjected to, making correlative and repeatability studies difficult. Here, we present a systematic study using a single TEM specimen to distinctly measure amorphization and Ga implantation and removal of both types of damage by low energy (< 1 keV), narrow beam (< 1 micron), argon ion milling on Si and Al FIB-prepared specimens.

To measure amorphous damage, two cross-section areas were prepared in the FIB at 30 keV at an incident angle of 88.5° similar to reference [3]. The bulk specimen was then removed from the FIB and then one of the cross-section areas was milled by rastering an argon beam within the defined area at 900 eV at an incident angle of 83°. The bulk specimen was returned to the FIB for electron beam-induced deposition (EBID) of Pt followed by conventional in situ TEM lift-out methods.

The thickness of the implanted gallium layer was measured on another set of in situ TEM specimens after FIB milling at 30 keV, which was followed by low energy, argon ion milling. Si and Al specimens were prepared and characterized using TEM and energy dispersive X-ray spectroscopy (EDS).

Figure 1 shows high resolution TEM images of the amorphous damage on Si after 30 keV FIB milling and after 30 keV FIB milling followed by 900 eV argon milling. Based on the TEM images, the initial 22 nm thick amorphous layer after 30 keV FIB milling (Figure 1a) was removed completely (0 nm) after argon ion milling (Figure 1b). EDS results show the amorphous layer after 30 keV FIB milling is Ga-rich (Figure 2a) and the area following argon milling is Ga-free (Figure 2b). Quantification of amorphous damage by 30 keV FIB on Al and its removal by argon milling is underway. X-ray characteristic emission method by EDS will be conducted to determine implanted Ga layer thickness on Si and Al.

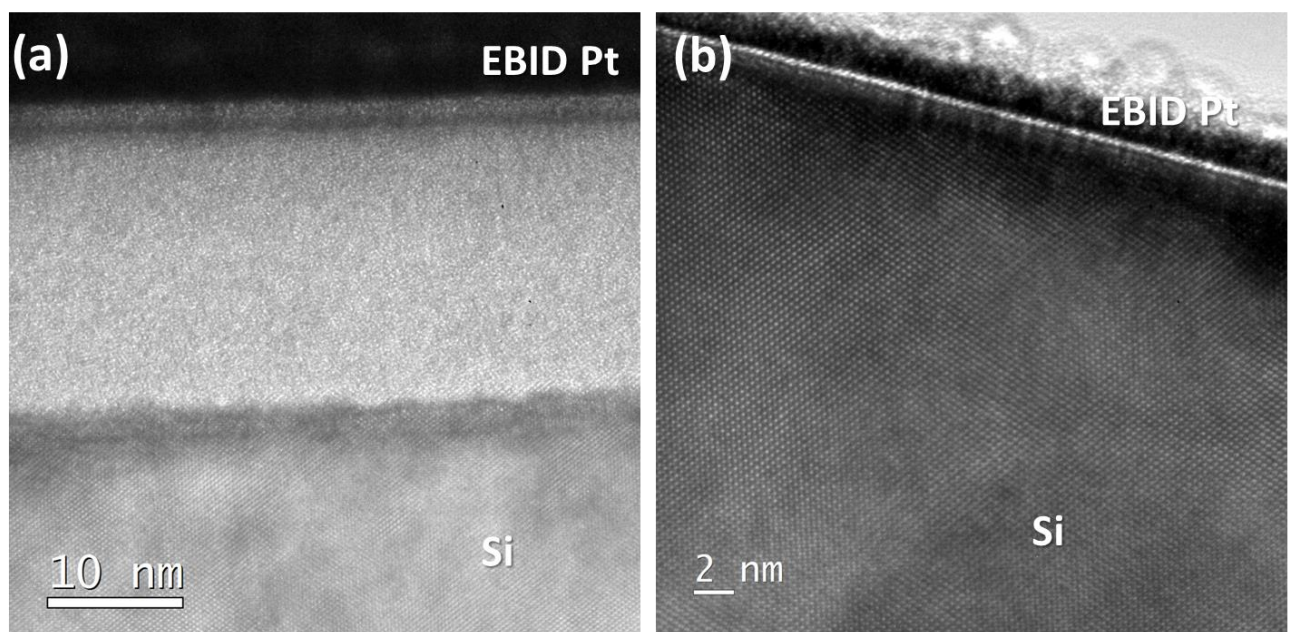


Figure 1. High resolution TEM images of the amorphous damage after 30 keV FIB milling (a) and amorphous layer removal after 30 keV FIB milling followed by 900 eV argon milling (b).

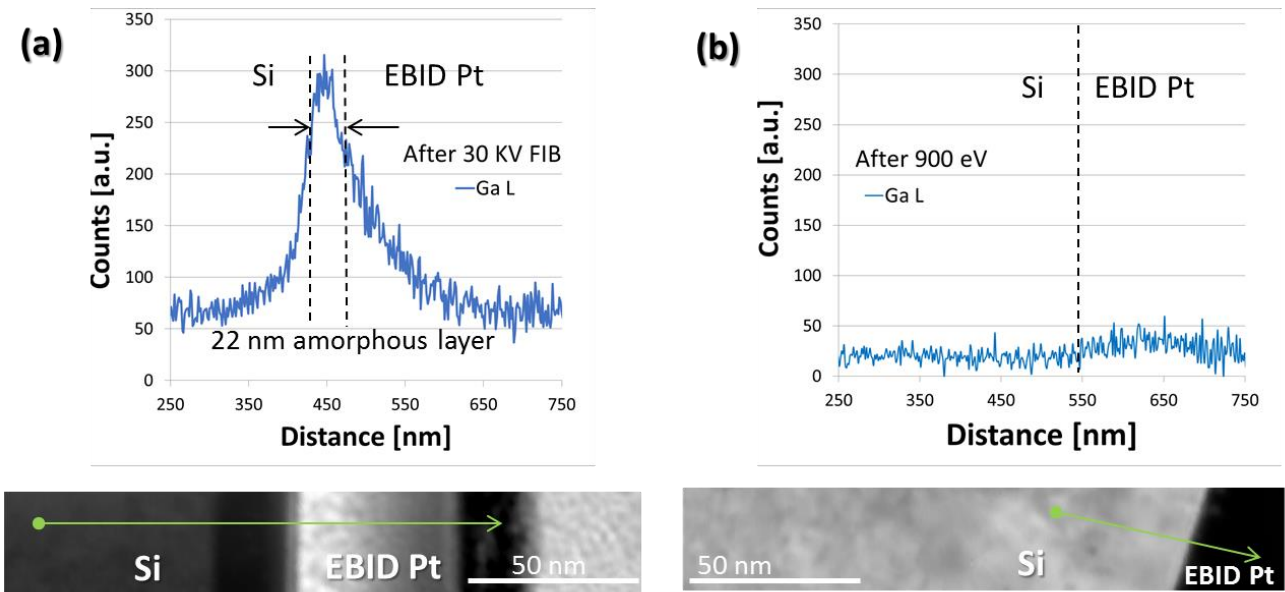


Figure 2. EDS Ga L signal from line scans of the Si specimen to EBID Pt. After 30 keV FIB milling, an amorphous layer containing Ga is detected (a). After 30 keV FIB milling followed by 900 eV argon ion milling, no Ga is detected (b).

References:

- [1] Giannuzzi, L.A. & Stevie, F.A. (1999). A review of focused ion beam milling techniques for TEM specimen preparation. *Micron*, **30**, 197.
- [2] Huh, Y., Hong, K.J., & Sin, K.S. (2013). Amorphization induced by focused ion beam milling in metallic and electronic materials. *Microscopy and Microanalysis*, **19**(S5), 33-37.
- [3] van Leer, B., Genc, A., & Passey, R. (2017). Ga⁺ and Xe⁺ FIB milling and measurement of FIB damage in aluminum. *Microscopy and Microanalysis*, **23**(S1), 296-297.

Acknowledgements:

The authors thank Brandon van Leer of Thermo Fisher Scientific for the FIB sidewall damage specimen preparation procedure.