Low-energy argon ion milling of in situ lift-out FIB specimens with broad and narrow beams

Fischione, P.[1], Campin, M.[1] and Bonifacio, C.[1]

[1] E.A. Fischione Instruments, Inc., United States

Focused ion beam (FIB) tools are commonly used to prepare transmission electron microscopy (TEM) specimens due to the site specificity and accuracy of specimen thinning and extraction that the tools provide.[1,2] However, milling with Ga ions results in a damage layer on each side of the TEM lamella.[3] The damage layer reduces the signal-to-noise ratio and contrast in TEM and scanning transmission electron microscopy (STEM) images, which negatively affects the ability to perform high resolution imaging and analysis. To minimize this, a low-energy Ga ion beam (≤ 2 keV) can be used for final thinning of the TEM lamella.[3-4] Post-FIB milling with a low-energy Ar ion beam can then be done to remove any remaining damage and Ga implantation.[4-6] When used in combination, specimens free of surface damage and Ga implantation can be created.[7]

In this work, TEM lamellae were prepared by a dual-beam FIB using the in situ lift-out technique. Specimens were milled at 30 keV to a targeted thickness of ~100 nm and then cleaned at 5 keV to reduce the thickness of the damage layer. Post-FIB thinning was done at low-energy (≤ 0.5 keV) with either an argon broad ion beam (~1.5 mm diameter) milling tool or an argon narrow ion beam (~1 micron diameter) milling tool, the latter of which rasters the ion beam within a milling box placed over the area of interest. To minimize milling of the protective capping layer, the maximum milling angles of ± 10° were used when milling with the broad ion beam. For consistency, the same angles were used when milling with the narrow ion beam.

High resolution TEM (HRTEM) imaging and energy-dispersive X-ray spectroscopy (EDS) analysis were performed at 200 kV to determine the reduction in amorphous damage and Ga implantation. HRTEM images taken after broad ion beam milling (Figure 1b) show variations in phase contrast across the images and the corresponding fast Fourier transforms (FFTs) show that a diffuse halo is still present. This indicates that some of the damage layer remained after milling. In contrast, HRTEM images taken after narrow ion beam milling (Figure 1c) show uniform phase contrast across the images and the diffuse halo in the corresponding FFTs is gone. This indicates that the damage layer was removed. EDS analysis (Figure 2) shows that broad ion beam milling removed some of the Ga, but that narrow ion beam milling removed all of the Ga. Quantitative analysis of sample thickness and sputtering of grid material onto the TEM lamella at various milling voltages up to 0.5 keV is in progress.

References:
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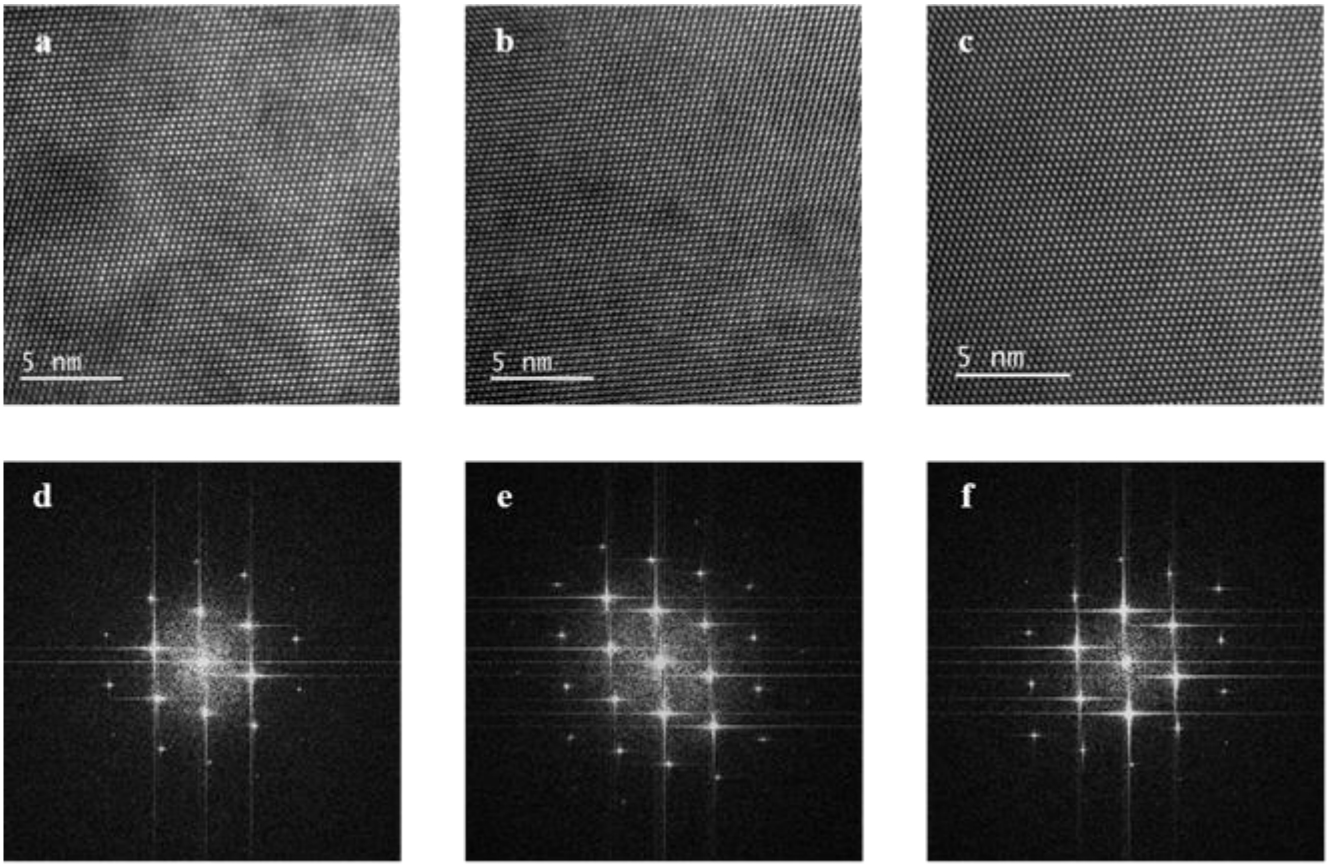

Figure 1. HTEM images acquired after FIB milling (a), after broad ion beam milling (b) and after narrow ion beam milling (c) show that the phase contrast after broad ion beam milling contains non-uniformities, but is very uniform after narrow ion beam milling. Similarly, FFTs corresponding to the HRTEM images (d-f) show that some of the amorphous layer remained after broad ion beam milling, but was completely removed after narrow ion beam milling.

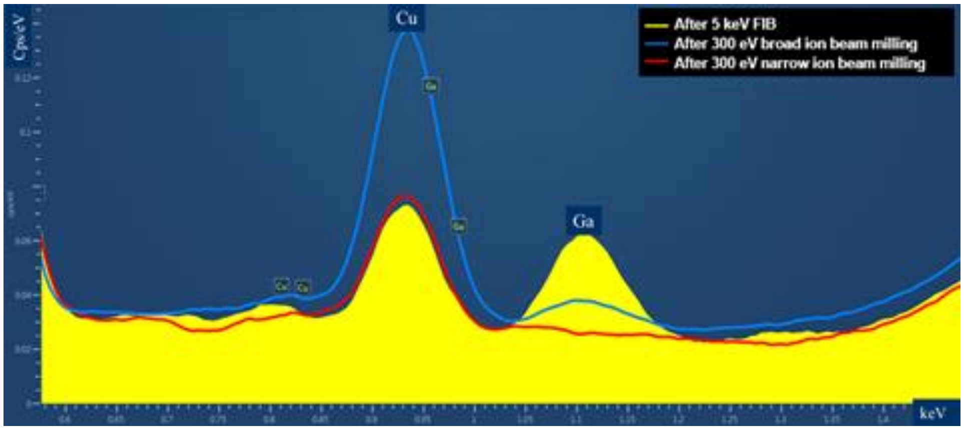

Figure 2. EDS spectra acquired after: FIB milling (yellow); broad ion beam milling (blue); and narrow ion beam milling (red). Complete removal of Ga after narrow ion beam milling is shown.