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Alternative Post-FIB Polishing Using Low-Energy Argon Ion Milling to Prevent Grid Redeposition

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Focused ion beam (FIB) milling is an indispensable tool for preparing transmission electron microscopy (TEM) specimens because it allows fast, accurate, and site-specific thinning and extraction [1]. Over the years, in addition to a conventional liquid metal ion source (LMIS) using Ga, FIB has expanded to include other species such as Xe, O, N, and Ar as utilized in an inductively coupled plasma FIB. However, for all types of FIB milling ion species, reducing ion energies for the final thinning steps is important to minimize specimen damage [2]. In addition, broad ion beam (BIB) Ar milling systems are often used at low energies as a post-FIB thinning step [3, 4]. Though BIB mills are effective in specimen thinning, redeposition of the grid material onto the TEM specimen is unavoidable due to the large beam size, which can range from hundreds of microns to a few millimeters. This redeposition on the TEM specimen can interfere with TEM imaging and analysis. We present experimental evidence of redeposition by BIB milling at low energies and propose an alternative post-FIB polishing step using concentrated ion beam (CIB) Ar milling. Specimens that were free of grid material redeposition and FIB-induced damage were obtained following CIB Ar milling.

Specimens from bulk Si sample material were prepared in a Ga FIB system [Scios Dualbeam, Thermo Fisher Scientific] by conventional in situ lift-out technique at 30 kV and 5 kV with a final thickness of 200 nm (30 kV step) and 120 nm (5 kV step). Post-FIB thinning was performed at low energies (≤ 1 kV) using a BIB (~1 to 2 mm diameter) and CIB (~1 μm) milling systems; the ion source in the CIB moves in a raster pattern within a user-defined region of interest over the specimen.

High resolution TEM (HRTEM) imaging was performed with a TEM [Tecnai, Thermo Fisher Scientific] at 300 kV to determine the quality of the specimens and the reduction of amorphous damage. HRTEM images of specimens after 30 kV Ga FIB milling, followed by BIB milling (Fig. 1 a-b) show moiré fringes and contrast variations. Analysis of the fast Fourier transforms (FFTs) after BIB milling shows that a diffused halo is present and weak spots near the center spot. Such results indicate the presence of an amorphous damage layer and non-Si material on the specimen. In contrast, HRTEM images (Fig. 1c-d) from specimens after Ga FIB milling followed by CIB Ar milling show uniform contrast, absence of diffused halo, and spots associated with Si on the FFTs. EDS analysis was performed using a low background TEM holder to identify the elemental composition on the specimens. EDS spectra (Figure 2) show that after FIB milling a significant Ga signal is observed. The Ga signal was reduced after both BIB and CIB milling, which indicates the effectivity of post-FIB, low-energy Ar ion milling in removing Ga implantation damage by the FIB. Furthermore, a significant amount of Cu redeposited from the grid is detected after BIB milling, which correlates to the additional spots observed on the FFTs and moiré fringes on the HRTEM images (Fig. 1a-b). Electron energy loss spectroscopy (EELS) acquisition and FFT image analyses are in progress to quantify and verify the redeposition of Cu on the Si specimens after BIB milling.

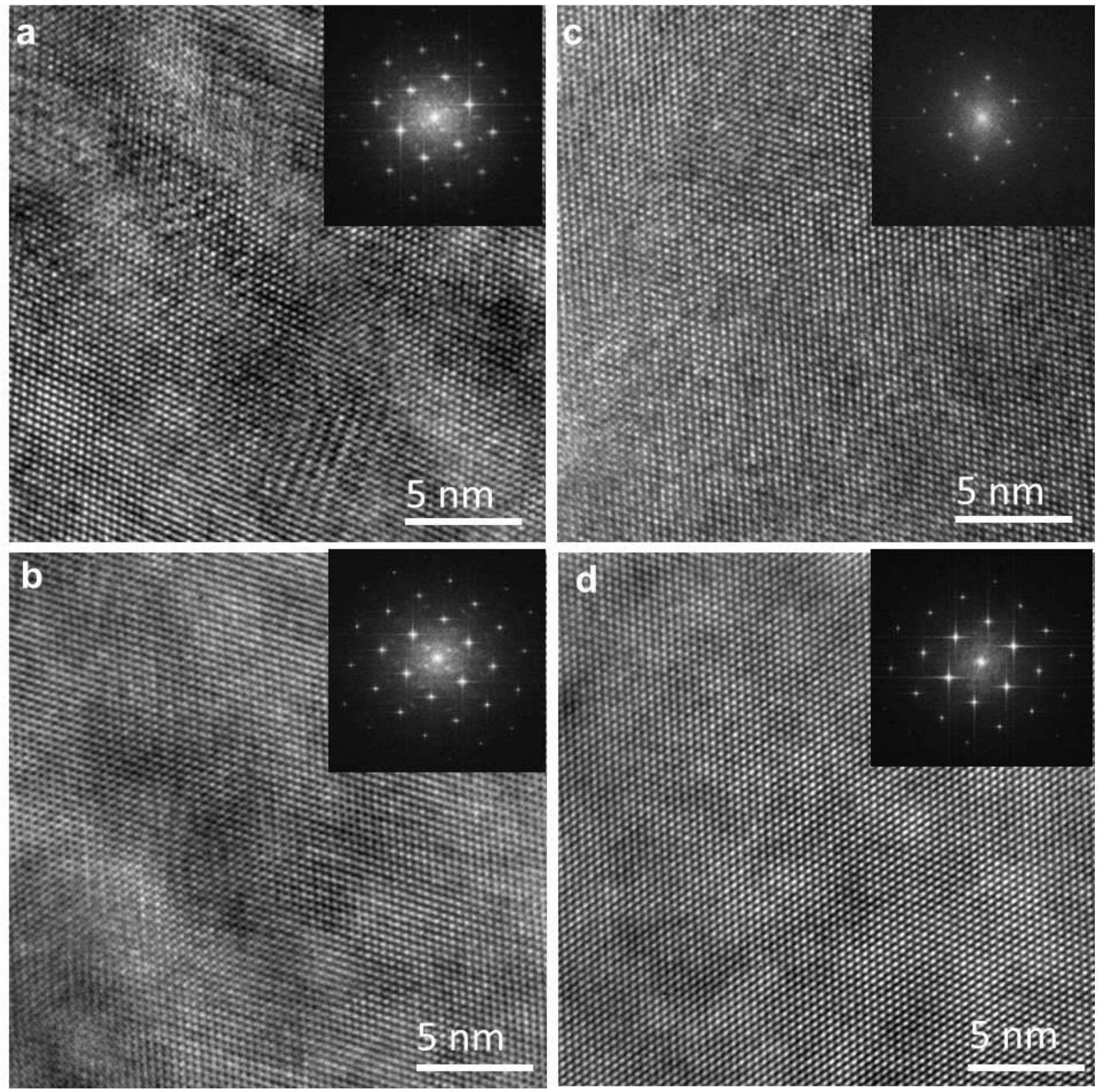

Figure 1. HRTEM images of Si specimens prepared by Ga FIB milling at 30 kV (top row) and 5 kV (bottom row). Insets are fast Fourier transforms (FFTs) generated from the HRTEM images. Images are after 1000 eV BIB milling (a), after 500 eV BIB milling (b), after 900 eV CIB milling (c) and after 500 eV CIB milling (d).

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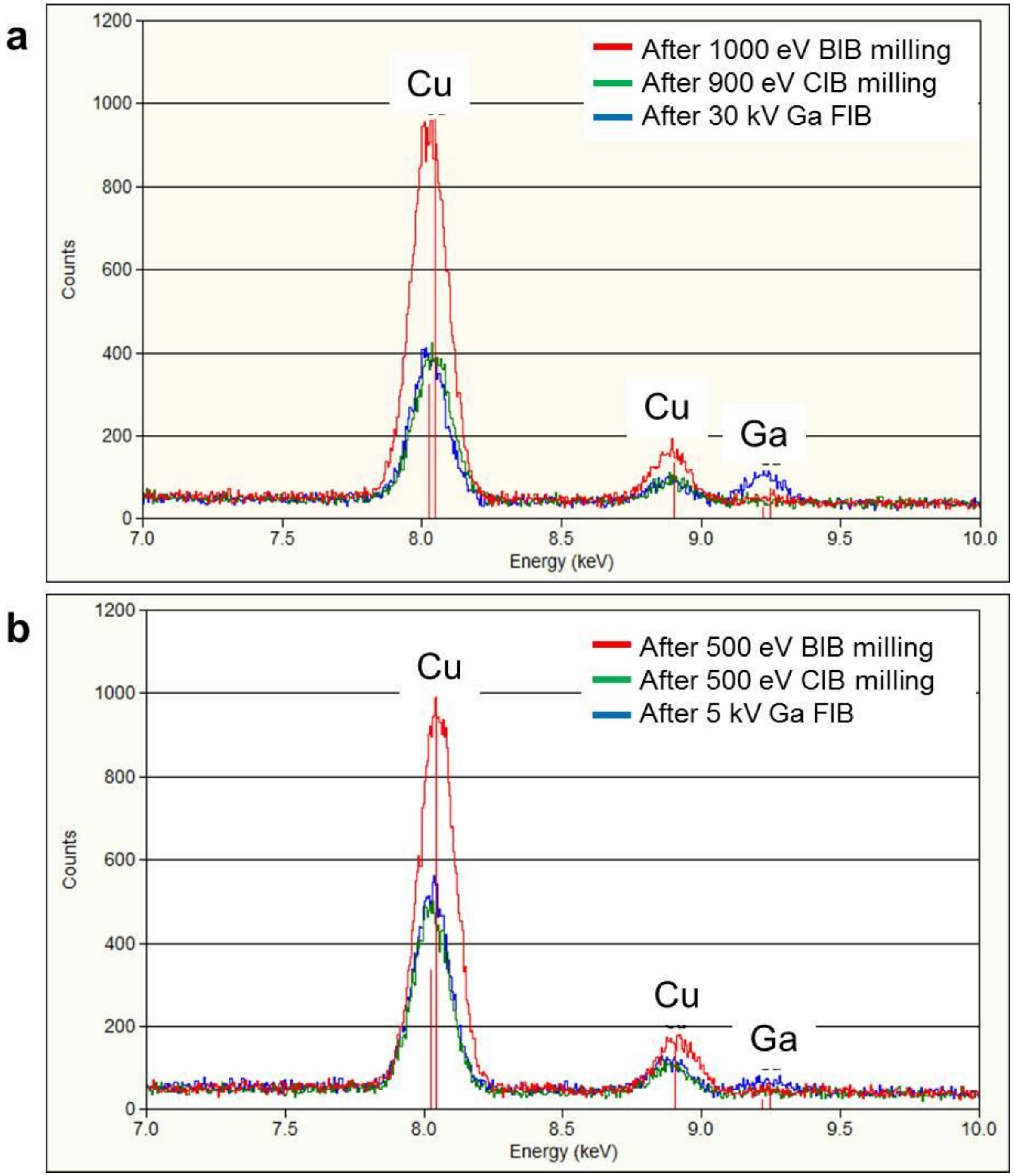

Figure 2. EDS using 300 kV accelerating voltage of Si specimens prepared by Ga FIB milling at 30 kV (a) and 5 kV (b) followed by BIB and CIB milling. Characteristic Cu K and Ga K peaks are shown.

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