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Ion Milling of Ex Situ Lift-Out FIB Specimens

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Abstract

The semiconductor industry is constantly investigating new methods that can improve both the quality of TEM lamella and the speed at which they can be created. To improve throughput, a combination of FIB-based preparation and ex situ lift-out (EXLO) techniques have been used. Unfortunately, the carbon support on the EXLO grid presents problems if the lamella needs to be thinned once it is on the grid. In this paper, we present low-energy (<1 keV), narrow-beam (<1 µm diameter), Ar^+ ion milling as a method of preparing electron-transparent and gallium-free EXLO FIB specimens.

Introduction

Focused ion beam (FIB) tools are frequently used to prepare transmission electron microscopy (TEM) specimens due to the site specificity and accuracy of specimen thinning and extraction that it provides [1, 2]. In addition, FIB ex situ lift-out (EXLO) TEM preparation is often employed due to its higher throughput and flexibility when compared to in situ FIB preparation [3]. In the case of in situ FIB preparation, it is necessary to perform several in situ welding and cutting steps before beginning to thin the lamella. As the time needed to thin the lamella is reduced through the use of automation hardware and software, the percentage of time spent on the lamella transfer and mounting steps becomes significant. Ultimately, a minimum preparation time is reached, which then limits throughput. If one wants to increase throughput beyond this, TEM specimens can be attached to perforated, carbon supported mesh type grids, which is a common EXLO method used by the semiconductor industry [4].

When performing high-resolution TEM (HRTEM) characterization and failure analysis of materials and devices, it is necessary to create very thin TEM lamellae containing the features of interest. This can be difficult if the spatial location of the features is not precisely known, as can be the case with device failures or processing defects. In addition, there are occasions when it is necessary to perform a comparative analysis of both nearby features and the features of interest. In either case, the lamella may require additional thinning.

TEM lamellae prepared using in situ methods can easily be thinned further for HRTEM analysis or if one is attempting to isolate specific features within the lamella, as in the case when trying to perform root cause failure analysis on devices. The same is not true for TEM lamella prepared using EXLO methods. Further thinning of the TEM specimen mounted on a thin carbon support with a Ga^+ ion beam is limited due to the deleterious milling of the carbon support, which can result in the loss of the specimen. In addition, Ga^+ ion milling causes artifacts such as surface damage and ion-implanted layers, which subsequently limit analytical and high-resolution electron microscopy. In contrast, low energy (< 1 keV) Ar^+ ion milling has been shown to improve specimen quality [5, 6]. Because post-FIB thinning of EXLO TEM lamellae is of growing importance to the semiconductor industry, it is necessary to investigate solutions capable of performing this task.

In this paper, we show that electron-transparent and gallium-free EXLO FIB specimens can be created using a low energy (<1 keV), narrow-beam (<1 µm diameter) Ar^+ ion milling method. In addition, we introduce the feasibility of targeted Ar^+ ion milling in improving specimen quality of carbon-supported EXLO specimens without compromising the integrity of the support.

Discussion

TEM lamellae from a semiconductor wafer were prepared using a CLM dual beam FIB (DB-FIB) [FEI Company]. In this work, we produced thick TEM lamellae with expected thickness > 100 nm at the area of interest. These lamellae were mounted on perforated, carbon-supported, mesh-type grids using standard EXLO methods so that the thinnest part of each lamella was directly over a hole in the grid (Figure 1). By mounting the lamellae in this manner, the grid material did not affect HRTEM imaging and characterization. The Model 1080 PicoMill® TEM specimen preparation system [E.A. Fischione Instruments, Inc.] was then used to mill both sides of the lamella.

Optimization of Milling Conditions

The 9 and 15 nm-thick porous silicon membrane window grids were ion milled in the PicoMill system with the specimen tilted at a 15˚ angle. Milling rates were calculated using the measured

areal dimensions of the perforations and normalized with respect to the raster box size.

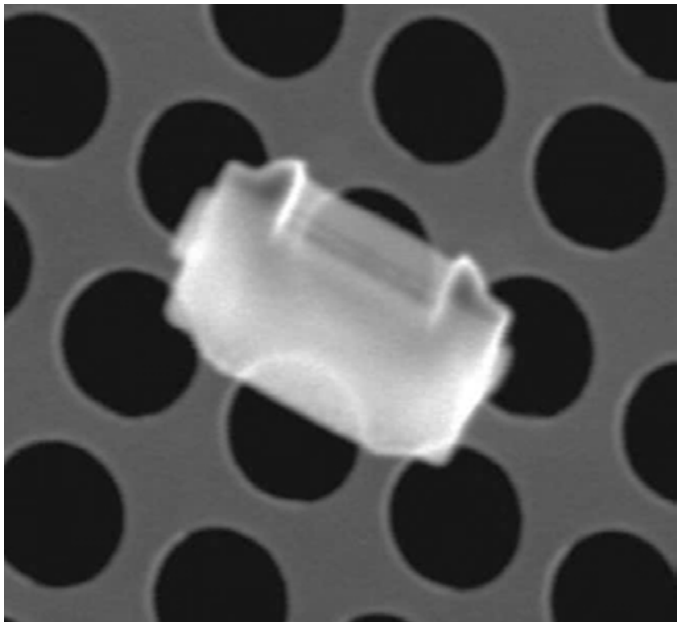

Figure 1: PicoMill SED system image; the thinnest part of the lamella was positioned directly over the hole in the supporting carbon grid.

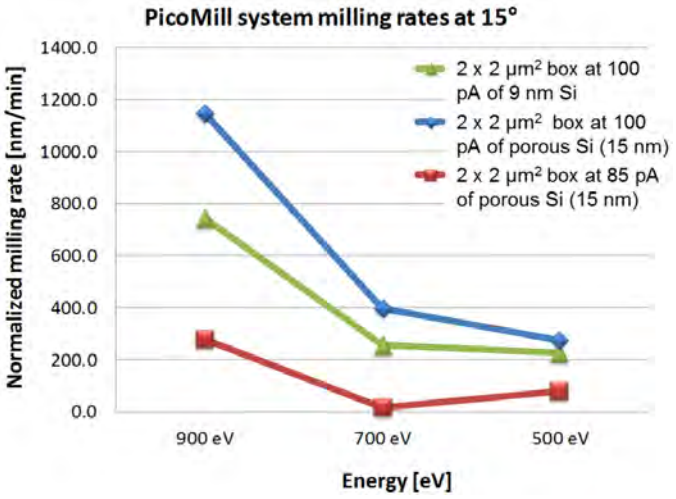

Figure 2: Normalized milling rates vs. energy (eV) for silicon and porous silicon thin films milled at a 15˚ angle with respect to the lamella surface.

Figure 2 shows a plot of the normalized milling rate vs. accelerating voltage for 85 pA and 100 pA beam currents. The milling rate is larger for larger beam currents, which was expected, and the milling rate increases significantly for voltages above 700 eV. This indicates that milling can be controlled by varying either the accelerating voltage or beam current and that by properly selecting these parameters, it should be possible to thin the lamella with minimal deterioration of the carbon support. Because the holes in the grid on which the lamella are placed are 5 µm in diameter and we want to minimize milling of the carbon support, a raster box size of 2 x 2 μm^2 was chosen.

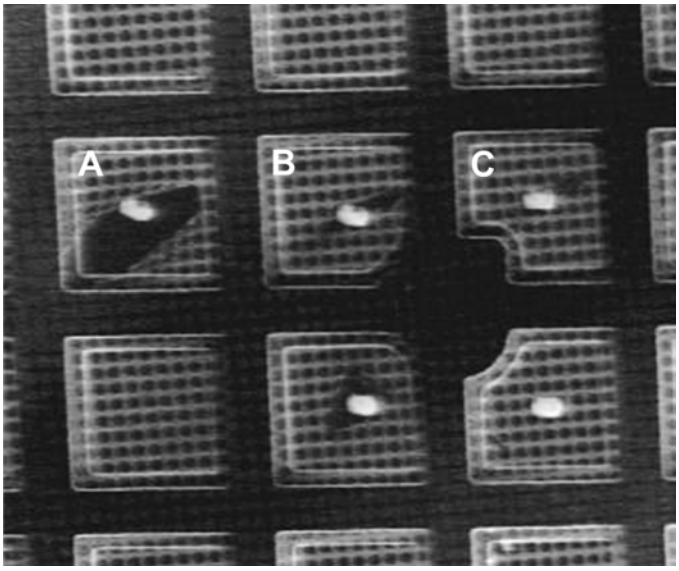

Figure 3: SED image showing the effect of different milling conditions on the lamellae and the supporting carbon grid. All sites were ion milled using a 2 x 2 μm^2 raster box. Site A was milled using 900 eV, 100 pA, for 90 seconds. Site B was ion milled using 900 eV, 85 pA, for 5 minutes. Site C was ion milled using 700 eV, 85 pA, for 5 minutes.

Based on this data, three milling conditions were tested on actual TEM lamellae. A secondary electron detector (SED) PicoMill system image showing the results of these milling tests is shown in Figure 3. The test with 900 eV and 100 pA showed rapid milling of the carbon support within 90 seconds, resulting in large holes in the supporting grid. The test with 900 eV and 85 pA showed less damage to the carbon support even after 5 minutes, but was still not ideal. The test with 700 eV and 85 pA showed only a small amount of degradation of the carbon support after 5 minutes of milling. Thus, accelerating voltages of < 700 eV with a beam current of 85 pA were found to be the optimal conditions for milling with a 2 x 2 μm^2 raster box without compromising the integrity of the carbon support.

In Situ Imaging during Milling

The PicoMill system has both an electron column for SEM imaging and an Ar^+ ion beam column for imaging and milling. In addition, it has both a secondary electron detector (SED) and a bright-field scanning transmission electron microscopy (STEM) detector. In situ imaging of the TEM lamella allows real-time monitoring of milling progress. Figure 4 shows SED and STEM images acquired while milling at 700 eV and 85 pA. During step 1, the raster box was placed at the very top of the lamella, which resulted in a hole in the carbon support above the lamella. During step 2, the raster box was moved towards the bottom of the lamella, which resulted in a hole in the carbon support below the bottom of the lamella. This demonstrates that accurate and precise placement of the raster box during milling is critical to minimize milling of the carbon support.

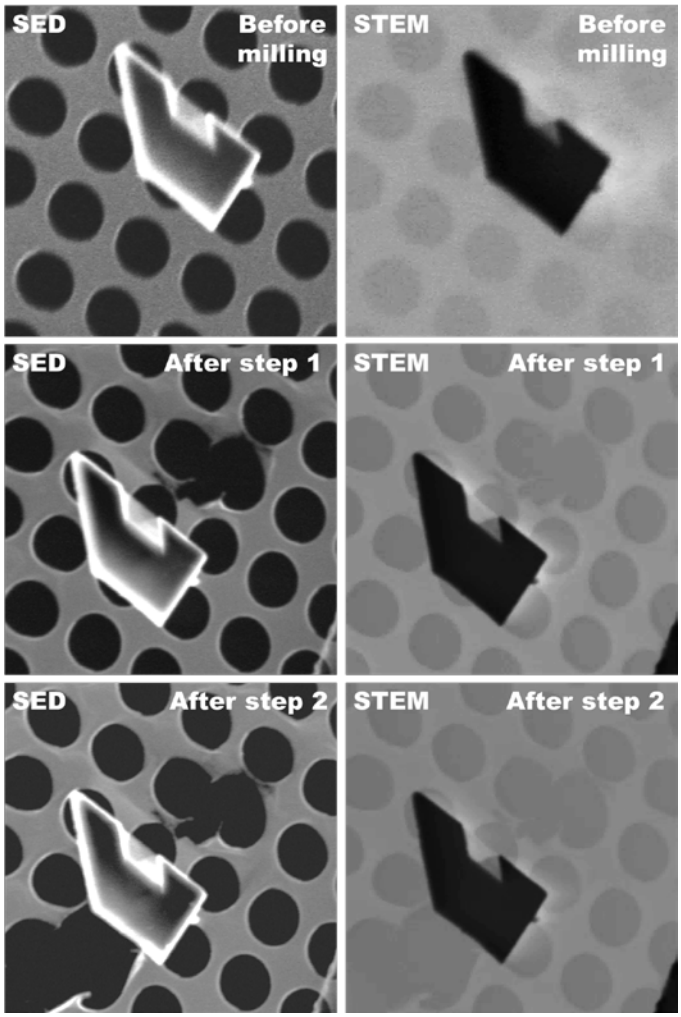

Figure 4: In situ SED and STEM images acquired in the PicoMill system that show the effect of raster box placement on the supporting carbon grid.

Targeting Features and Automated Endpoint Detection

There are times when it is necessary to iteratively mill and image the lamella in the TEM to endpoint on specific features. Qualitative thickness reduction was determined in situ by observing the change in brightness and contrast of the images (image intensities) from both the SED and STEM detector in the PicoMill system. When the image intensities reached a value equal to a predetermined value entered by the user, ion milling automatically stopped. TEM images were then acquired with a Tecnai TF30 TEM (FEI Company) before and after milling. In situ SED and STEM images (Figure 5) acquired in the PicoMill system after ion milling showed minimal damage to the supporting carbon film. Corresponding TEM images (Figure 6) taken before and after ion milling show that the PicoMill system is able to remove very small amounts of material, allowing precise control over the removal of features in the area of interest.

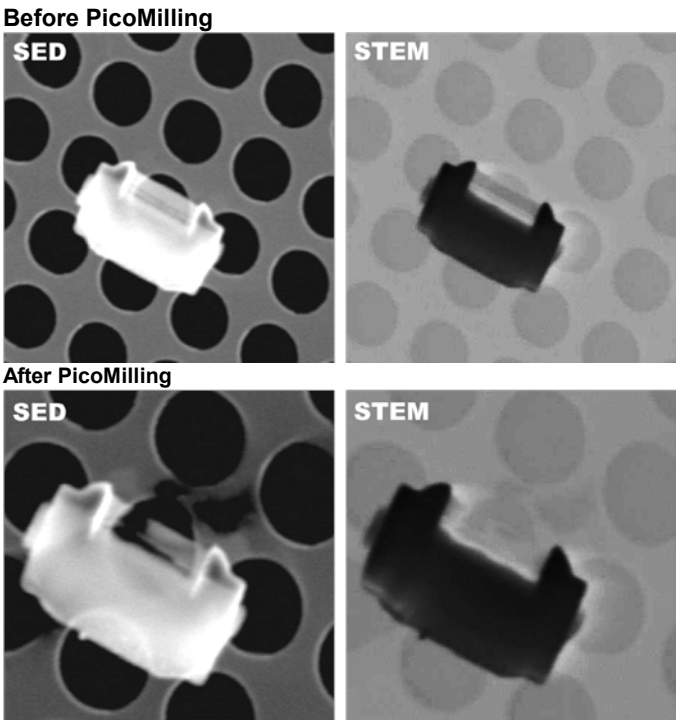

Figure 5: In situ SED and STEM images acquired in the PicoMill system before and after ion milling.

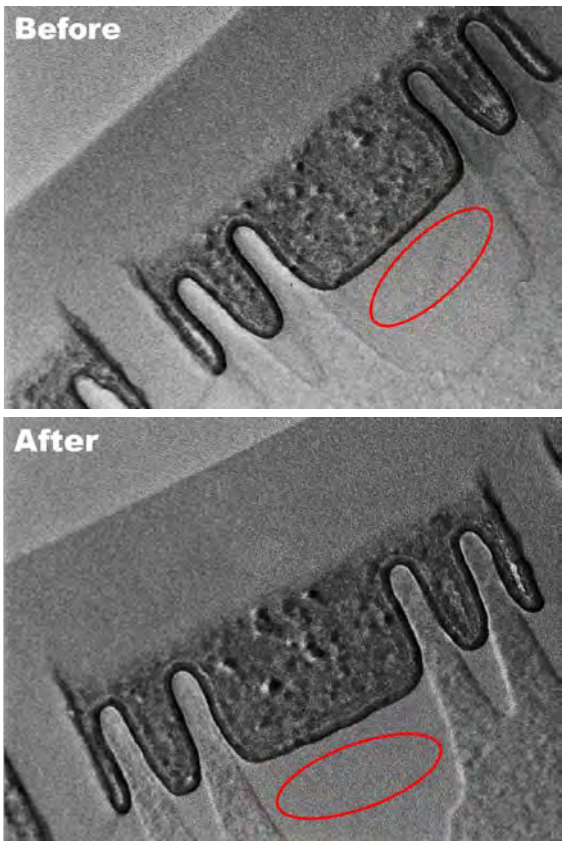

Figure 6: TEM images acquired before and after ion milling. The feature circled in red ("before" image) was removed during ion milling ("after" image).

Gallium Reduction after PicoMilling

Ga^+ ions used in FIB tools are known to implant gallium ions into the specimen surface, which can negatively affect HRTEM imaging and analysis [2]. To remove the gallium implanted layers, it is necessary to mill the lamella surfaces using non-reactive ion species. In this experiment, we also had the additional restriction that we wanted to minimize the milling of the carbon support. This was accomplished using a low energy (< 1 keV), narrow beam (< 1 µm diameter) milling method.

To verify the ability of the PicoMill system to remove gallium, energy dispersive X-ray spectroscopy (EDS) was performed on a TEM lamella before and after milling. A high angle annular dark field (HAADF) STEM image showing the placement of the EDS acquisition box in the area of interest is shown in Figure 7. The EDS data (Figure 8) show no detectable Ga signal after milling, which indicates the removal of Ga from the lamella. In addition, TEM images acquired before and after ion milling (Figure 9) showed little to no change in the features of interest, even after milling. This confirms that it is possible to remove Ga from TEM lamella mounted on carbon-supported grids.

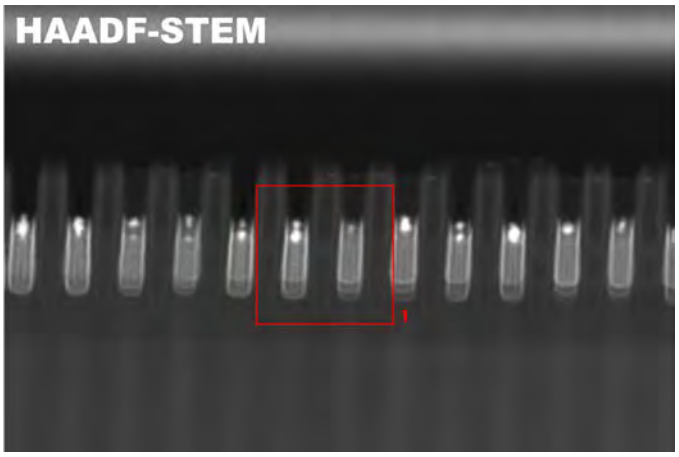

Figure 7: HAADF-STEM image showing the area from which the EDS spectra were acquired.

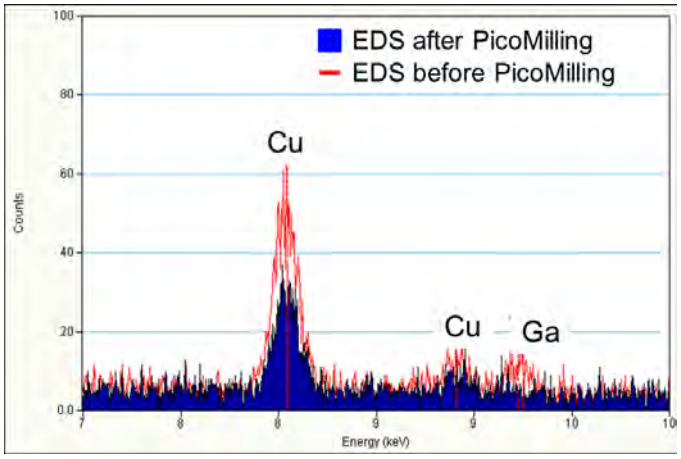

Figure 8: EDS data acquired before and after PicoMilling.

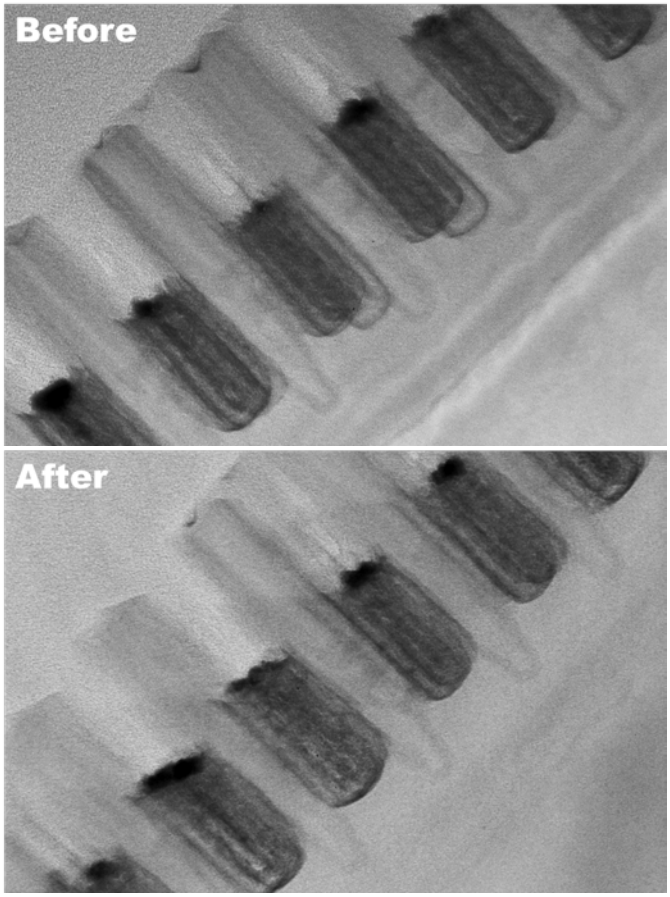

Figure 9: TEM images acquired before and after PicoMilling.

Conclusions

It is possible to thin a TEM lamella mounted on a perforated, carbon-supported, mesh-type TEM grid using the PicoMill system. The low current densities of the Ar^+ ion beam allow thinning of the lamella with minimal damage to the supporting carbon foil. In addition, the sub-micron size and precise positioning of the ion beam allows thinning of both sides of the lamella within the 5 µm opening of the carbon support. This allows thinning of targeted features while minimizing milling of the carbon support. EDS spectra acquired before and after ion milling of the specimen show removal of Ga, which can improve HRTEM imaging. Quantitative analysis of the thickness and Ga reduction is in progress.

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