TEM Sample Preparation using Focused Ion Beams: Fundamental Problems and Solutions

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Transmission electron microscopy (TEM) requires extremely thin specimens, ideally free of preparation artifacts. Conventional focused ion beam (FIB) preparation methods cannot be employed to create high quality specimens much thinner than 25 nm. We have developed a straightforward method for in-situ target preparation of ultra-thin TEM lamellas by FIB milling. With this method we are able to routinely obtain large area co-planar lamellas thinner than 10 nm. The resulting specimens are suitable for high resolution TEM as well as low kV scanning transmission electron microscopy.

TEM sample preparation by FIB has advanced dramatically over the past years and starts to replace conventional techniques when speed and site specificity are required [2]. Despite these improvements the specimen thickness and quality obtainable is not satisfactory for the latest corrected microscopes. Scaling down lamella thickness using conventional FIB techniques is hampered by three detrimental effects: warping, amorphization, and shrinkage. Warping is caused when a lamella is thinned below a certain threshold due to intrinsic or processing induced strain. Special mounting and adaptive milling techniques have been proposed to reduce the effect [3]. Nevertheless mechanically stable large coplanar transparent areas have not been obtained. Amorphization is a side effect of ion milling. Depending on incident angle and energy the lamella is amorphized to a certain depth (e.g. ~30 nm in Si for 30 kV Ga ions at glancing angle). Naturally the amorphization depth has to be much smaller than half of the lamella thickness. This is achieved by polishing the lamella with reduced ion energy. In conventional processing this causes difficulties since the beam shape degrades significantly with reduced energy. This has to be counteracted by steeper milling angles at the penalty of introducing material dependent sputtering effects. Shrinking of the lamella is observed during the final thinning process. With the removal of only a few tens of nanometers in thickness the height of the lamella’s transparent area can be reduced by several micrometers. This effect must be countered by the deposition of very thick protective layers — a very time consuming process.

We demonstrate an elegant method based on conventional lift-out technique [4] that addresses the problems mentioned above. The essential difference is that the final thinning and polishing of the lamella is not performed with the same milling direction for both sides. Instead, the region of interest (ROI) is thinned from one side
to about half the remaining lamella thickness. Then the sample is rotated around its TEM observation direction by \(\sim 90\) degrees and the ROI thinned from the second side. This creates a thin window where the two milling grooves overlap. The process is repeated in several steps while gradually reducing ion energy until the desired thickness is reached. In addition, a final low kV Argon polishing step can be applied to remove residual Ga contamination. The process yields a sturdy lamella with one or several electron transparent windows. Due to the window geometry even a very large region of interest can be milled to extremely small thicknesses without bending. In addition, shrinkage of the lamella does not occur since geometrical sputtering yield effects are efficiently suppressed. This also dramatically lifts milling angle restriction for low polishing with broadened beam diameter. Being able to polish at \(1^\circ \sim 3^\circ\) degree incident angle reduces material selective sputtering effects as well as amorphization depth.

The double-tilt processing is not feasible by using the stage of a FIB/SEM systems alone. Therefore, a dedicated specimen holder was designed to perform the sample tilt with each 180 degrees rotation of the microscope stage. Thus the switching between milling direction takes place reliably and the milled side is always conveniently facing the SEM for visual process control. Planarity of the window can be effortlessly adjusted to well below one degree deviation — enough to theoretically obtain an atomically flat specimen over the relevant field of view.

Motivation

Crystalline Sample Damage Layer

Conventional FIB Target Preparation

1. Auto Sample Preparation
2. In-situ Lift-Out
3. Grid Mounting
4. Thinning + Polishing
Conventional Thinning/Polishing

④ Thinning

Protective Layer

Sample Material

5–10 μm

5–2 μm

Fast Line Scan

10–20 μm

Limits of conventional FIB method…
Reasons for shrinking

Geometric Sputtering Effects I

In order to understand the unexpected shrinkage of the lamella below the threshold thickness, an atomic-level description of the irradiation process is required. Our MD simulation setup, designed for studying ion irradiation effects on the top edge of a short section of a thin Si lamella, is visualized in Fig. 2. In all simulations, the studied system size was 65 nm in the vertical (z) direction and 10 nm in width (x-direction), periodic boundaries being

750 nm

Rounding Overlap
Geometric Sputtering Effects II

FIG. 4. Results for the edges of all studied thicknesses after ion sputtering at 30 keV. For each case, we show the snapshot of the final structure along the x-direction, the positions of sputtered ions, the center of mass \(CM_y\), and the degree of amorphization. Only the upper part of the structure is shown, as this is where all interesting effects appear.

Bending and modest shrinking in the thicker edges (\(\geq 7\) nm) and predominantly shrinking in the thinnest edges. This inference is supported by studying the evolution of the atomic structure of the 10 and 3 nm edges as a function of the 30 keV irradiation dose, as presented in Figs 5(a,b), respectively. As the dose increases, the 10 nm system bends towards the corner where the beam is incident, whereas the 3 nm system shrinks by nearly 10 nm in height as the dose is brought to 1000 ions. The explanation for these two distinct modes of behavior can be found by considering the positions of sputtered atoms in each case and taking into account the resulting surface tension. As noted above and as seen in Fig. 4, sputtering for the \(\geq 7\) nm edges occurs mainly off the front face of the structure. Therefore, the system will relax, i.e., minimize its free energy to a local minimum, by contracting the front face in the vertical dimension, hence pulling the structure into a forward-bent position.

Correspondingly, when atoms are sputtered evenly off both the front and back faces, as for the \(< 7\) nm edges, the bending behavior is taken over by the shrinkage of the system in the vertical direction, as the structure relaxes by contracting along the entire thickness.

Note that the above-described bending is not the same phenomenon as the deleterious warping observed experimentally in the sample during conventional FIB thinning. The empirical warping happens in the opposite direction relative to the beam, throughout the sample.
$X^2\text{--FIB}$
$X^2$–Holder

Sample

$\pm 180^\circ$

$\pm 45^\circ$

Demo
**X²–Holder**

**AURIGA CrossBeam™**

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**SEM @ 1 kV**
- Intense SE detector

**SEM**
- ET–SE detector

**STEM @ 30 kV**
- Darkfield

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**X²–FIB in a Nutshell**

- TEM sample preparation method
  - Site specific
  - Universal
  - Fast
  - High yield

- High quality lamellea
  - Co-planar
  - No bending
  - No shrinkage
  - No thickness limit
  - Excellent mechanical, thermal, and electrical properties

Lechner, et.al. Microscopy and Microanalysis (2012)
Thickness Determination
Thickness Determination
2.0