APPLICATIONOTE

Model 1040 NanoMill® TEM specimen preparation system

Specimen configuration

Preparing focused ion beam (FIB) milled specimens for submission to Fischione Instruments.

The Model 1040 NanoMill® TEM specimen preparation system is ideal for specimen processing following FIB milling. The NanoMill system's concentrated argon ion beam, typically in the energy range of 50 to 2000 eV, excels at targeted milling and specimen surface damage removal. Ion-induced secondary electron imaging is used to locate the FIB-produced lamella and then to target the region that will receive low energy milling in either raster or spot modes.

Only the lamella is targeted during ion milling. Ion bombardment of the supporting grid is eliminated, which helps prevent redeposition. The NanoMill system can also remove damage from electropolished or broad-beam ion milled specimens.

This document explains how to configure specimens to obtain optimal results from the NanoMilling℠ process. Before you start, it is important to understand the following terms:

- *Specimen geometry.* Refers to the type of specimen, i.e., FIB *in situ* lift out, H bar, dimpled, wedge polished, etc.
- *Specimen configuration.* Refers to the position in which the lamella is mounted onto a support grid.

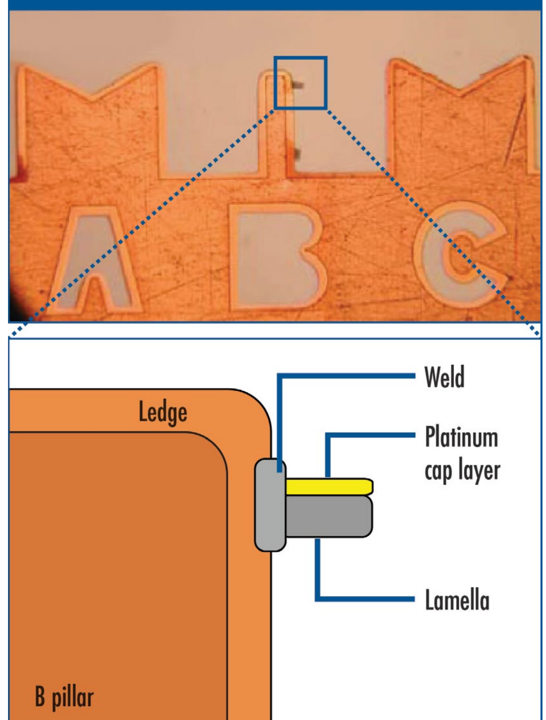
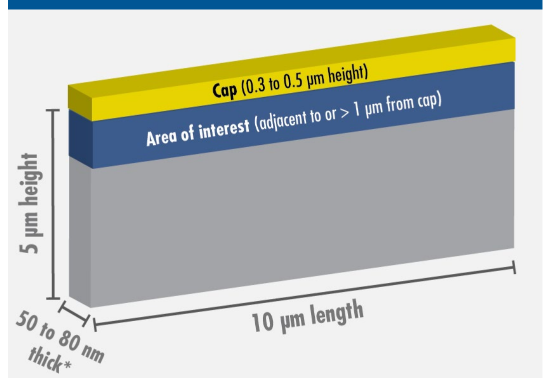


Figure 1. Light optical microscope image of the lamella attached onto the support grid (left) and an enlarged schematic of the lamella (right).

Preparing *in situ* specimens

For FIB-produced specimens, the preferred configuration is an *in situ* lift-out lamella with a thickness that is dictated by the material type. If the material:

- is in a bulk state or has a simple interface, the optimal specimen thickness is 50 to 80 nm.
- has an existing microstructure that is a 2- or 3-dimensional microelectronic pattern, (e.g., integrated circuits and FinFETs), the optimal specimen thickness is 50 nm or less.



*If the material is homogenous, bulk, or has a simple interface, thickness is 50 to 80 nm. If the material has an existing microstructure that is a 2- or 3-dimensional microelectronic pattern, (e.g., integrated circuits and FinFETs), thickness is 50 nm or less.

Figure 2. Specimen dimensions.

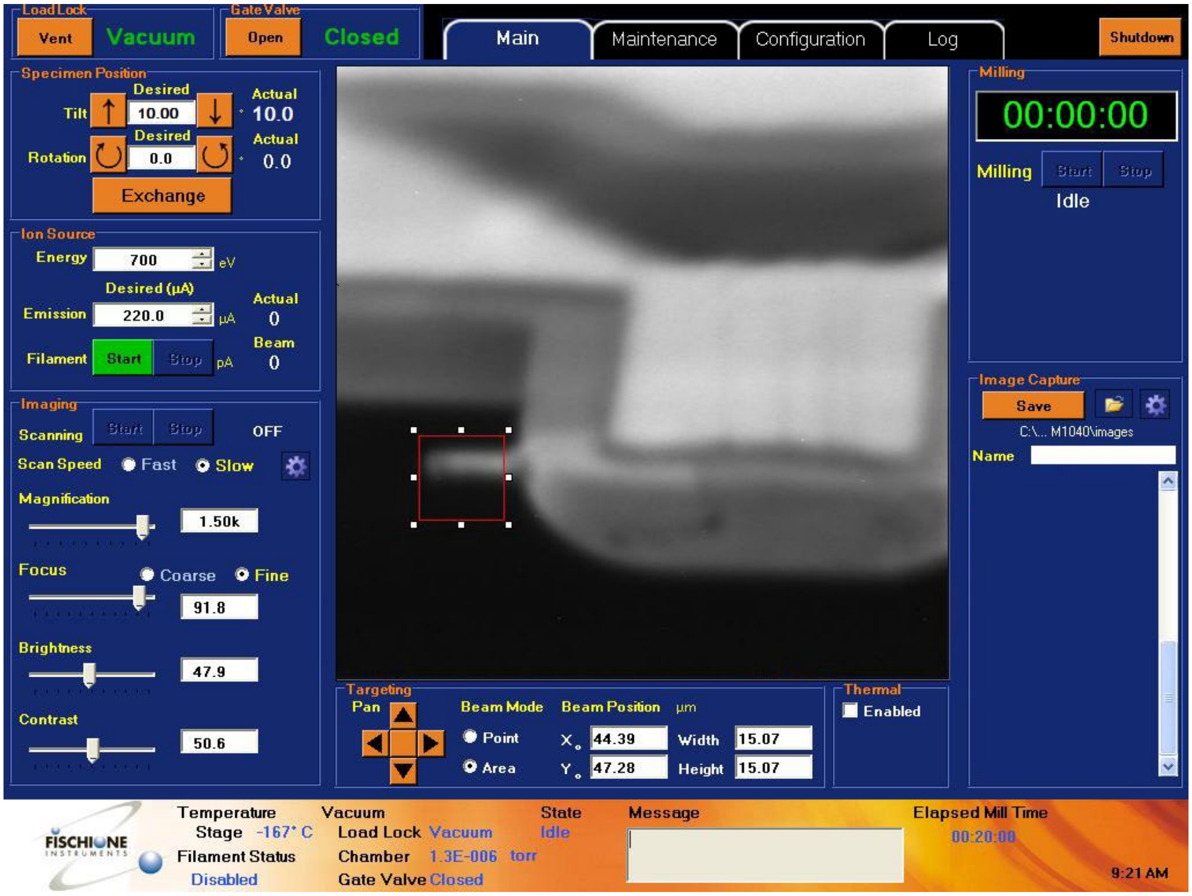


Figure 3. The secondary electron image shows the support grid and the lamella.

Attach the lamella to the side of the central finger (B pillar) of an Omniprobe™ copper* grid (Figure 1).

The three-position ("ABC") grid is preferred because the mounted lamella will be near the eucentric position in both the NanoMill system and the transmission electron microscope (TEM).

The minimum lamella size is 10 µm long x 5 µm wide (Figure 2) as determined by the minimum diameter of the argon ion beam (~1 µm). Larger specimens are preferred for imaging and targeting. It is very important that the shorter side of the lamella be attached:

- To the side and very close to (but not on) the top of the grid's B pillar (Figure 1). This positioning allows for a low-angle tilt during milling. Tilting a grid as it is imaged in a FIB or scanning electron microscope (SEM) can establish the attachment point required for reaching a desired milling angle. The typical range of milling angles is 7 to 10° (the angles are measured with respect to the plane of the grid), both in the negative and positive directions of tilt. Attaching the lamella in this position also protects the lamella during shipping and handling.
- Coplanar with the grid surface so that it may be tilted symmetrically.
- On both top and bottom lamella surfaces at the attached end to create a rigid and firm bond.
- With a metal such as platinum, rather than carbon, because the lamella is subsequently plasma cleaned.

A cap layer is needed if the lamella has any of the following characteristics:

- is organic

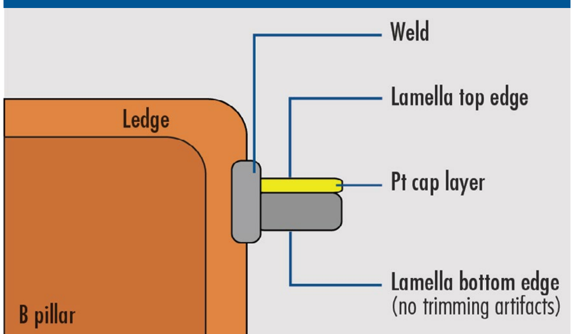


Figure 4. Specimen configuration.

- has a coating
- is multilayer
- is a low atomic number material

The preferred cap layer material is platinum. Carbon is not recommended, because material may be lost during initial plasma cleaning. Do not use tungsten as a cap layer either; its lower sputtering rate results in longer NanoMill system cycle times.

It is important to use the FIB *in situ* lift-out geometry and to attach the lamella to the top left- or right-side of the B pillar because other specimen configurations may result in sputtering and subsequent redeposition of grid material onto the specimen surface.

The described specimen configuration ensures that the argon ion beam bombards only the lamella, even during low-angle milling.

Keep the following guidelines in mind when FIB processing the lamella:

- Do not attach the lamella in the V notch above the A or C pillars in an Omniprobe grid because the short distance between the top edge of the lamella and the grid may result in redeposition of the grid material onto the specimen surface.
- Do not attach more than one lamella onto a grid, even if they are placed on opposites sides of the B pillar. Redeposition of material from one lamella to another may occur.
- Ensure that the protective platinum cap on the top face of the lamella is 0.3 to 0.5 µm in thickness after FIB. Electron beam deposition is recommended because the small grains will not cause shadowing of the ion beam in the NanoMill system. This protective layer is sacrificial and is most often sputtered away during the NanoMilling process. The deposition of gold prior to platinum should be avoided because it may be sputtered preferentially due to its very low sputtering threshold (~ 14 eV).
- Ensure that the lamella is polished in the FIB at 5 kV or lower, it is as free from curtaining, and it is as parallel-sided as possible. No trimming artifacts should be present at the bottom edge of the lamella (opposite the cap). Trimming artifacts reduce the ability to mill from either the top or bottom edge of the lamella (see Figure 4).

Preparing H-bar specimens

The NanoMill system can enhance FIB-milled H-bar specimens. Prevention of sputtering and subsequent redeposition of the specimen substrate onto the lamella depends on the proper construction of the FIB box. A single FIB box at the center of an H bar is preferred (minimum

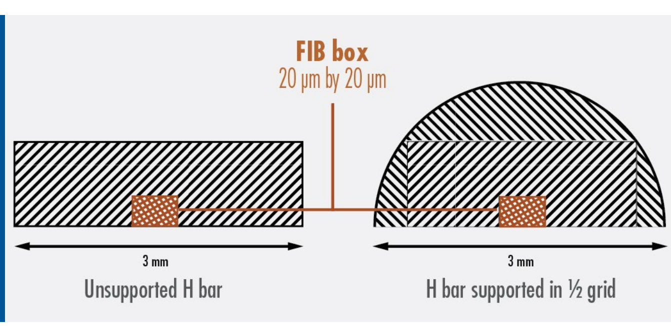


Figure 5. An unsupported (left) and a grid-mounted (right) H bar.

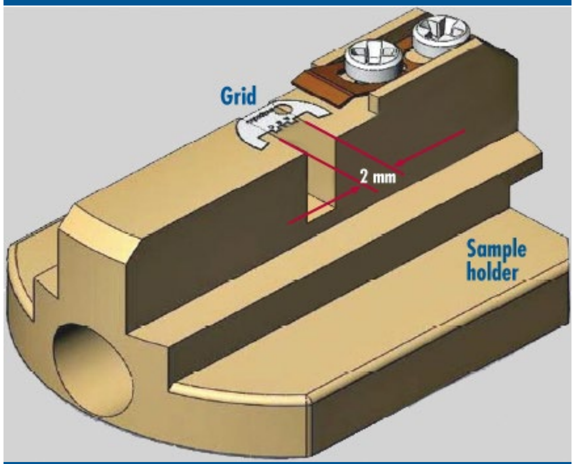


Figure 6. Model 1050 TEM Mill specimen holder with a grid bridging the gap.

dimensions of 20 x 20 µm). Ensure that the lamella receives ion beam polishing, it is free from curtaining, and is as parallel-sided as possible.

The optimal final lamella thickness is dictated by the material type. If the material:

- is in a bulk state or has a simple interface, the optimal specimen thickness is 50 to 80 nm.
- has an existing microstructure that is a 2- or 3-dimensional microelectronic pattern, (e.g., integrated circuits and FinFETs), the optimal specimen thickness is 50 nm or less.

The H bar can be either free standing or attached to a ½ grid with a minimal amount of conductive adhesive. The edge of the H bar containing the FIB box should be positioned near the center line of the 3 mm diameter support grid (Figure 5). The overall dimensions of the specimen geometry must allow specimen loading into a side-entry TEM holder.

Keep the following guidelines in mind when FIB processing the grid-mounted H bar:

- The grid should be electrically conductive.
- The adhesive for the H-bar specimen should be electrically conductive to ensure that the grid and the specimen material have good electrical contact.
- Leave sufficient area surrounding the specimen so that the grid may be clamped securely.

Submitting specimens to Fischione

When submitting a specimen for preparation with the NanoMill system:

- Provide images (TEM/SEM micrographs and/or sketches) to show the areas from which you would like to see FIB-induced damage removed or thinning enhanced.
- Indicate the microscopy that you intend to use to analyze the specimen (e.g., TEM, SEM, high-angle annular dark-field [HAADF], high-resolution electron microscopy [HREM], electron energy loss spectroscopy [EELS], energy-filtered transmission electron microscopy [EFTEM], energy-dispersive X-ray spectroscopy [EDS], etc.).
- Indicate the initial thickness and desired final thickness (following the NanoMilling process) of the lamella.
- The specimen geometry should span the 2 mm gap in the specimen holder (see Figure 6) and fit within the 3 mm diameter of the holder.
- Ship specimens in containers that are standard practice for FIB users and follow standard shipping procedures. Standard membrane boxes used for storage of conventional TEM specimens are suitable; do not insert filter or lens paper into the membrane box.
- Contact sales@fischione.com or applications@fischione.com with questions before FIB (or submission) of samples.

Specimen preparation summary

When configuring FIB-processed specimens for the NanoMill system, follow the suggested practices:

Parameter	Suggested practice
Specimen geometry	*In situ* lift-out or H bar
Lamella dimensions	The optimal final lamella thickness is dictated by the material type. If the material: • is in a bulk state or has a simple interface, the optimal specimen thickness is 50 to 80 nm. • has an existing microstructure that is a 2- or 3-dimensional microelectronic pattern, (e.g., integrated circuits and FinFETs), the optimal specimen thickness is 50 nm or less.
Lamella position	Side of B pillar (finger) of a copper Omniprobe grid
Attachment	Platinum, top and bottom lamella surfaces, to the grid ledge
Cap layer	Platinum, electron beam deposited, 0.3 to 0.5 μm thick after FIB; area of interest no more than 1 μm away from cap
FIB	On all surfaces and edges of lamella: • 30 kV maximum for cutting • 5 kV or less for final polishing

Note

*Use a copper, not molybdenum, Omniprobe grid. Molybdenum grids are too short for these purposes.

E.A. Fischione Instruments, Inc.
9003 Corporate Circle
Export, PA 15632 USA
Tel: +1 724.325.5444
Fax: +1 724.325.5443
info@fischione.com
www.fischione.com

Document Number AN006 Revision 03 01/2014