APPLICATION NOTE

Model 1080 PicoMill® TEM specimen preparation system

Specimen configuration

A guide for preparing TEM specimens for a demonstration of the Model 1080 PicoMill TEM specimen preparation system at Fischione Instruments.

The Model 1080 PicoMill® TEM specimen preparation system is ideal for specimen processing following focused ion beam (FIB) milling. The PicoMill 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 to prevent redeposition. The PicoMill 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 PicoMilling℠ 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, FIB ex situ lift out, FIB atom probe tomography (APT) tip, etc.
- *Specimen configuration.* Refers to the position in which the lamella is mounted onto a support grid.

Preparing FIB in situ lift-out 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 80 to 100 nm.
- has an existing microstructure that is a 2- or 3-dimensional pattern, (i.e., microelectronic devices), the optimal specimen thickness is 50 nm or less.

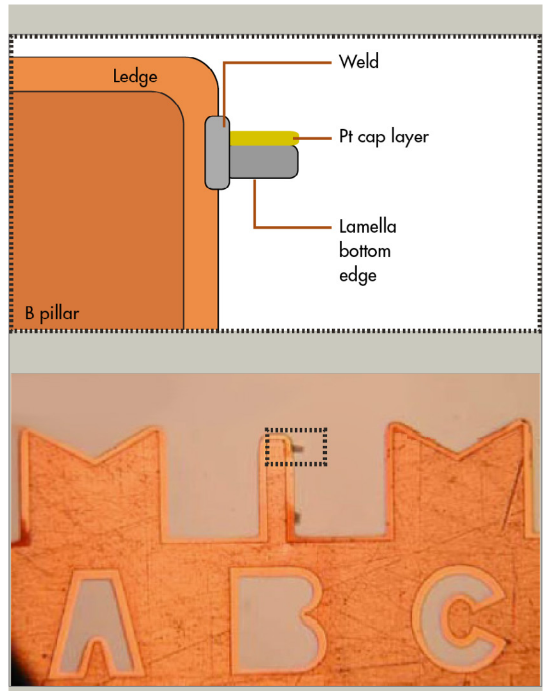
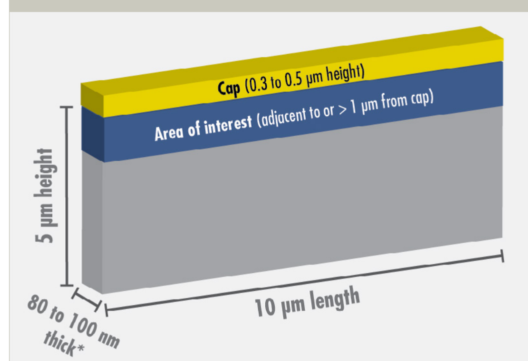


Figure 1. Light optical microscope image of the lamella attached to the support grid (bottom) and an enlarged schematic of the lamella (top).



*If the material is 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 pattern (i.e., microelectronic devices), thickness is 50 nm or less.

Figure 2. Specimen dimensions.

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 PicoMill 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 protective cap of platinum or carbon.

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

- is organic
- is coated
- is multilayer
- contains a low atomic number material

The preferred cap layer material is platinum. Do not use tungsten as a cap layer; its lower sputtering rate results in longer PicoMill system milling 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 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 PicoMill system. This protective layer is sacrificial and is most often sputtered away during the PicoMilling 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 3).

Preparing FIB ex situ lift-out specimens

This type of specimen is extracted from the bulk specimen in the FIB and subsequently manipulated to a TEM supported grid outside the FIB, in ambient conditions. The ex situ lift-out specimen requirements for PicoMilling includes:

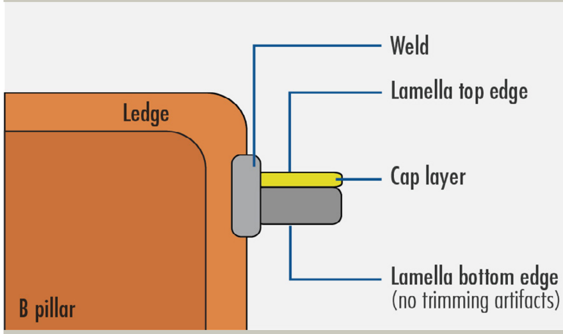


Figure 3. Specimen configuration.

- Optimal specimen thickness of 80 to 100 nm.
- Specimen dimensions of 5 µm in height x 10 µm in length (Figure 2) for optimal ion beam targeting.
- Sufficient protective cap (platinum or carbon) 0.5 to 0.8 µm in thickness on the top face of the lamella.
- FIB specimen polished in the FIB at 5 kV or lower, free from curtaining, and parallel-sided. No trimming artifacts should be present at the bottom edge of the lamella (opposite the cap). Trimming artifacts are usually Ga-rich and may be redeposited onto the specimen surface.
- FIB specimen manipulated on one of the following TEM grids (Figure 4):
  – *A grid mesh.* Specimen should be mounted near the center of the carbon-supported TEM grid.
  – *A slotted grid.* Specimen should be mounted near the top of the slotted grid.

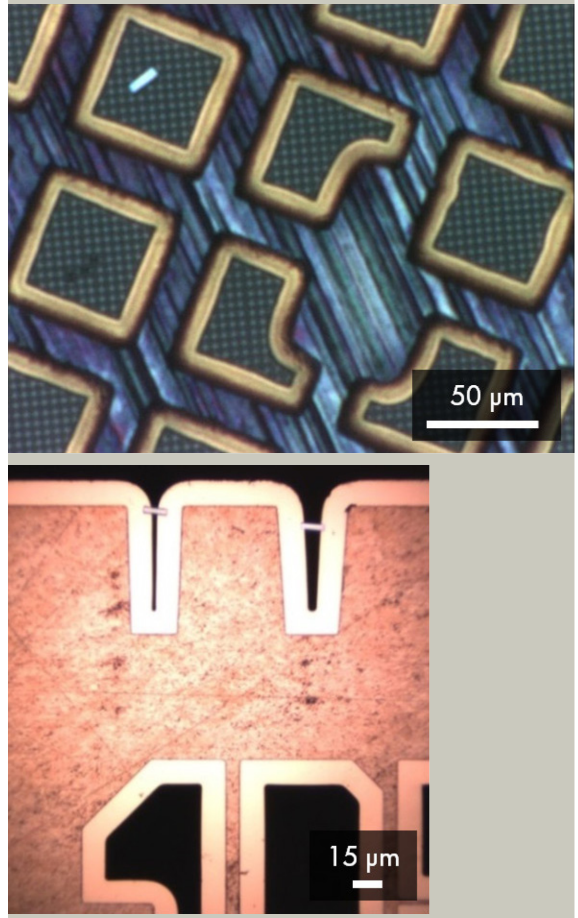


Figure 4. Optical light microscope images of Si lamellae manipulated on a Quantifoil® grid (top) and on a slotted EXpressLO™ grid (bottom).

Preparing FIB APT specimens

This type of specimen is typically prepared using the FIB from a bulk-wedge specimen and subsequently polished to form a fine tip by annular milling. APT specimens on a Si half grid are PicoMilled at 0° tilt only. The APT specimen requirements for PicoMilling includes:

- Sufficient protective cap (platinum only) of 40 to 60 nm in thickness on the top face of the APT apex. The cap protects the surface of the specimen during annular milling and is completely removed during PicoMilling. Removal of the platinum cap by PicoMilling is necessary for a successful APT experiment.
- The bulk wedge lift-out specimen is welded:
  – To the surface of a flat pillar on a Si half-grid (Figure 5). The pillar in the Si grid needs to be milled before welding the bulk specimen to ensure attachment of the specimen.
  – On both sides of the wedge specimen, front and back, at the point of attachment to create a rigid and firm bond.

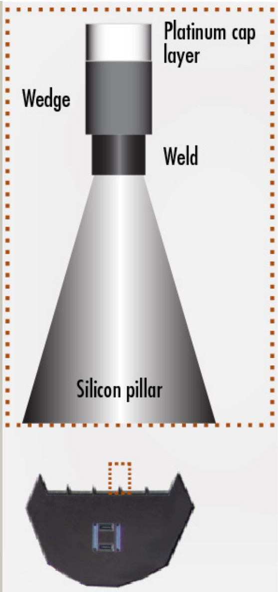


Figure 5. Wedge lift-out specimen attached to the pillar of the Si half grid (bottom) and an enlarged schematic of the attachment (top).

- The specimen is polished in the FIB at 30 kV or 5 kV by annular milling. (Lower kV annular milling is only necessary for specimens sensitive to Ga-implantation.)
- Optimal tip diameter of 80 to 150 nm (Figure 6) after FIB milling

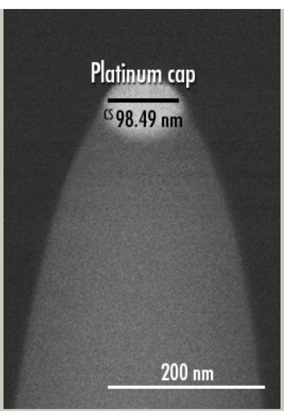


Figure 6. Scanning electron microscope back-scattered electrons (SEM-BSE) image of the APT specimen after FIB annular milling. Tip apex diameter measured as 98.49 nm.

Submitting specimens to Fischione

When submitting a specimen for preparation with the PicoMill 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 of the lamella.
- 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 PicoMill system, follow the suggested practices:

Parameter	Suggested practice by specimen type		
Specimen geometry	**In situ lift-out specimen**	**Ex situ lift-out specimen**	**APT tip specimen**
Specimen dimensions	The optimal final lamella thickness is dictated by the material type; typically 80 to 100 nm.	The optimal final lamella thickness is dictated by the material type; typically 80 to 100 nm.	80 to 150 nm.
Position	On the side of the B pillar of a copper Omniprobe grid.*	On mesh near the center of a TEM support grid.	Center on the pillar of a Si half grid.
Attachment	Use platinum to attach both top and bottom lamella surfaces to the grid ledge.	No mechanical attachment required; van der Waals forces secure the specimen to the grid.	Use platinum to weld the specimen from the front and from the back of the wedge specimen.
Cap layer	The thickness of the cap layer depends upon specimen geometry. After FIB preparation, use platinum or carbon to make a cap 0.3 to 0.5 μm thick with the area of interest no more than 1 μm away from the cap.	The thickness of the cap layer depends upon specimen geometry. After FIB preparation, use platinum or carbon to make a cap 0.3 to 0.5 μm thick with the area of interest no more than 1 μm away from the cap.	After FIB preparation, use platinum (do not use carbon) to make a cap no less than 40 nm thick with the are of interest no more than 100 nm away from the cap.
FIB milling energy	On all surfaces and edges of the specimen: 30 kV maximum for cutting	On all surfaces and edges of the specimen: 30 kV maximum for cutting	30 kV maximum for bulk cuts and annular milling.

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

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