APPLICATIONNOTE

Model 1040 NanoMill® TEM specimen preparation system

TEM specimen configuration

Preparing specimens for a demonstration of the Model 1040 NanoMill TEM specimen preparation system.

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 eV to 2 keV, excels at targeted milling and specimen surface damage removal. Ion-induced secondary electron imaging is used to locate the FIB-produced specimen and to target the region that will receive low energy milling in either area or point modes.

Only the specimen 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:

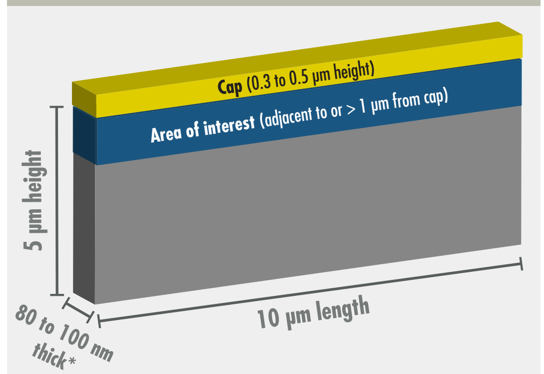
- *Specimen geometry.* Refers to the type of specimen, e.g., FIB-prepared in situ lift-out, ex situ lift-out, APT specimens, etc.
- *Specimen configuration.* Refers to the position in which the specimen is mounted onto a support grid.

Preparing FIB milled specimens

In situ lift-out specimens

This type of specimen is extracted from the bulk specimen inside the FIB tool under vacuum conditions. The in situ lift-out specimen requirements for NanoMilling include:

- Specimen thickness of 80 to 100 nm.
- Specimen dimensions (Figure 1) of 5 x 10 µm (height x length); these dimensions allow optimal ion beam targeting.
- Weld the shorter side of the rectangle-shaped lamella (Figure 2):
    - to the flat surface of the grid's ledge (Figure 3) on the B pillar the Omniprobe® grid, close to the pillar's tip; this will ensure a low-angle tilt can be achieved during milling (other specimen configurations may result in sputtering and subsequent redeposition of the grid material onto the specimen surface), and
    - coplanar with the grid surface so that the specimen may be tilted symmetrically, and
    - on both sides of the lamella at the point of attachment to create a rigid and firm bond.
- Place a protective cap (platinum or carbon may be used) of about 0.5 to 0.8 µm in thickness on the top face of the specimen after FIB tool preparation. This protective cap is sacrificial and is often milled away during the



*If the material is bulk or has a simple interface, thickness is 80 to 100 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 1. Specimen dimensions.

Figure 2. Light optical microscope image of a lamella attached to the B pillar of a support grid.

NanoMilling process. The preferred cap layer material is platinum. Do not use tungsten as a cap layer; its lower sputtering rate results in longer NanoMill system cycle times.

- Polish the specimen at 5 kV or lower; it must be free from curtaining and as parallel-sided as possible. 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.

Ex situ lift-out specimens

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

- Specimen thickness of 80 to 100 nm.
- Specimen dimensions of 5 x 10 µm (height x length) (Figure 1); these dimensions allow optimal ion beam targeting.
- Place a protective cap (platinum or carbon may be used) of about 0.5 to 0.8 µm in thickness on the top face of the lamella after FIB tool preparation. This protective cap is sacrificial and is often milled away during the NanoMilling process. The preferred cap layer material is platinum. Do not use tungsten as a cap layer; its lower sputtering rate results in longer NanoMill system cycle times.
- Polish the specimen at 5 kV or lower; it must be free from curtaining and as parallel-sided as possible. 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.
- Manipulate the specimen onto a TEM support grid; either a carbon-supported grid or an EXpressLO™ grid:
  - **Carbon-supported grid.** Mount the specimen on a grid mesh near the center of the carbon-supported TEM grid (Figure 4); this positioning will ensure visibility of the specimen for easy ion beam targeting during milling.
  - **EXpressLO grid.** Mount the specimen near the top of the slotted grid for easy ion beam targeting during milling (Figure 5); other specimen configurations may result in sputtering and subsequent redeposition of the grid material onto the specimen surface.

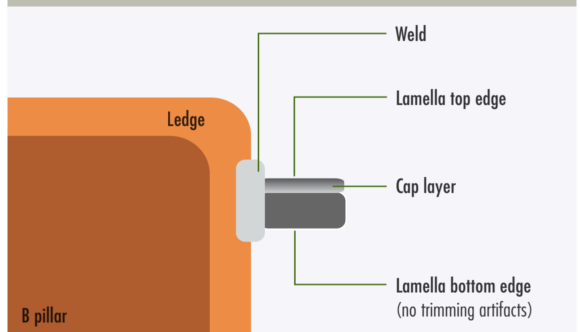


Figure 3. Specimen configuration.

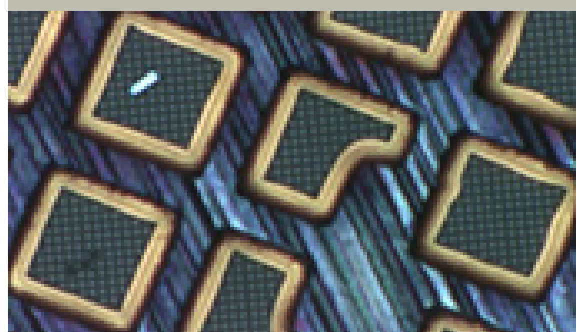


Figure 4. Ex situ specimen mounted on the mesh of a Quantifoil® carbon-supported grid.

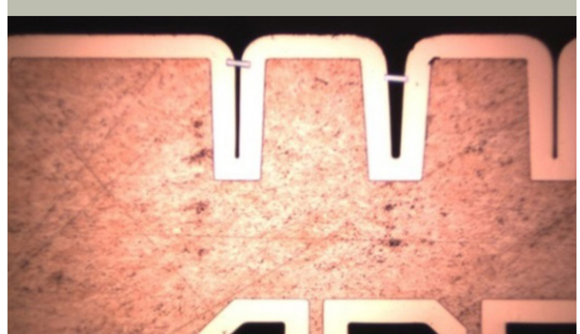


Figure 5. Ex situ specimens mounted near the top of the slots on an EXpressLO™ grid.

Atom probe tomography (APT) specimens

This type of specimen is typically prepared from a bulk wedge specimen mounted on a Si half-grid using a FIB system; the specimen is then subsequently polished to form a fine tip by annular milling. APT specimens are milled at a 0° tilt in the NanoMill system. The APT specimen requirements for NanoMilling include:

- Sufficient protective cap (platinum only) of about 40 to 60 nm in thickness after FIB milling on the top face of the APT apex. This cap is to protect the surface of the specimen during annular milling and is completely removed during NanoMilling. Complete removal of the Pt cap is necessary for successful APT imaging and analyses.
- Bulk wedge lift-out of the specimen is welded:
  - to the surface of a flat pillar on a Si half-grid (Figure 6). The pillar in the Si grid needs to be milled before welding the bulk specimen to ensure attachment of the specimen, and
  - on both sides of the wedge specimen, front and back, at the point of attachment to create a rigid and firm bond.
- Specimen polished in a FIB system 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 for NanoMilling is 80 to 150 nm (Figure 7) after FIB milling.

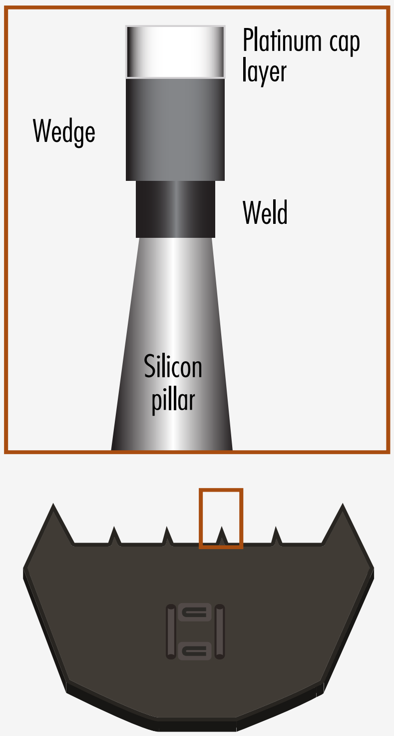


Figure 6. A wedge lift-out specimen attached to the pillar of a Si half-grid (bottom) and an enlarged schematic of the wedge specimen (top).

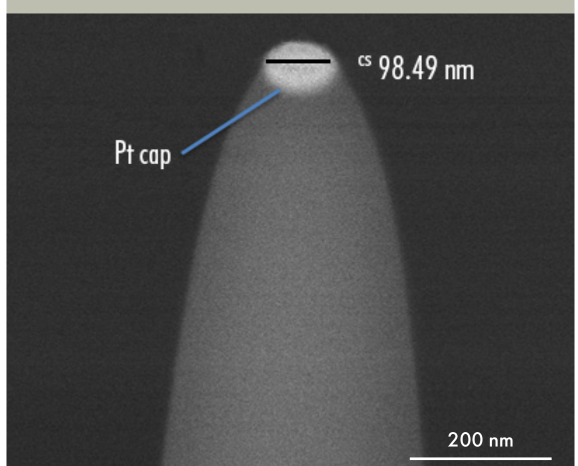


Figure 7. Scanning electron microscope backscattered electrons (SEM-BSE) image of an APT specimen after FIB annular milling. Tip apex diameter is 98.49 nm.

Submitting specimens to Fischione Instruments

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 of the lamella.
- Ship specimens in containers that are standard practice for FIB tool 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 milling (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 or ex situ lift-out and APT tip
Dimensions	Optimal final thickness: • **In situ and ex situ lift-out:** Dimensions dictated by the material type, typically 80 to 100 nm • **APT:** Final tip diameter of 80 to 150 nm
Position	• **In situ lift-out:** Side of B pillar of a copper Omniprobe® grid • **Ex situ lift-out:** On the mesh near the center of a carbon-supported TEM grid • **APT:** Centered on a pillar of a Si half-grid
Attachment	• **In situ and ex situ lift-out:** Platinum weld of top and bottom surfaces to the grid ledge • **APT:** Platinum weld at the front and back of wedge specimen
Cap layer type and thickness	• **In situ and ex situ lift-out:** Platinum or carbon (do not use tungsten) cap 0.3 to 0.5 µm thick after FIB milling, area of interest no more than 1 µm away • **APT:** Platinum cap only, no less than 40 nm thick after FIB milling, area of interest no more than 100 nm away
FIB milling	On all surfaces and edges of the specimen: • **In situ and ex situ lift-out:** 30 kV maximum for bulk cuts and 5 kV or less for final polishing • **APT:** 30 kV maximum for bulk cuts and annular milling for tip sharpening

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