

MODEL 1040

NanoMill® TEM specimen preparation system

Revolutionary ultra-low-energy, inert gas, concentrated ion beam produces specimens free from amorphous and implanted layers
Ideal for post-FIB processing





TEM REQUIRES HIGH-QUALITY SPECIMENS

For many of today's advanced materials, transmission electron microscopy (TEM) is the best technique for gathering valuable information about microstructure and properties.

Because features in nanotechnology research and semiconductor device specimens continue to decrease in size, it is essential that specimens be very thin and be free of preparation-induced artifacts. These requirements are even more important when using TEMs with aberration-correction and monochromated electron sources where resolution is sub-Ångström.

MODEL 1040

NanoMill® TEM specimen preparation system

The NanoMill system uses an ultra-low energy, concentrated ion beam to produce the highest quality specimens for transmission electron microscopy.

- Ultra-low energy, inert gas ion source
- Concentrated ion beam with scanning capabilities
- Removes damaged layers without redeposition
- Ideal for post-focused ion beam processing
- Enhances the results from conventionally prepared specimens
- Room temperature to cryogenically cooled NanoMillingSM process
- Rapid specimen exchange for high-throughput applications
- Computer-controlled, fully programmable, and easy to use
- Contamination-free, dry vacuum system

Create thin specimens for TEM

Fischione Instruments' Model 1040 NanoMill TEM specimen preparation system is an excellent tool for preparing the ultra-thin, high-quality specimens needed for TEM imaging and analysis.

The variable energy ion source generates ion energies as low as 50 eV. In addition, the beam size is as small as 1 μm , enabling removal of amorphization, implantation, or redeposition from targeted areas.

An ideal application for the NanoMill system is post-focused ion beam (FIB) processing. Although the FIB is highly effective in preparing TEM specimens, its liquid metal (Ga) ion source often results in amorphization and Ga implantation. These damaged layers can be as much as 10 to 30 nm thick. The NanoMill system is ideally suited to removing these layers.

Targeted, ultra-low-energy milling

The NanoMill system's ion source features a filament-based ionization chamber and electrostatic lenses. This source was specifically developed to produce ultra-low ion energies and a small beam diameter. The source uses an inert gas, argon, and has an operating voltage range of 50 eV to 2 kV at variable working distances. The source yields sufficient current density to remove specimen damage within a reasonable time. The

NanoMilling process can be accomplished in as little as 20 minutes.

Because the ion beam can be focused into a 1 μm diameter spot, redeposition of sputtered material onto the area of interest is avoided. The feedback control algorithm for the ion source automatically produces stable and repeatable ion beam conditions over a wide variety of milling parameters.

The beam can be either targeted at a specific point or scanned over the specimen's surface. This is particularly important when targeting a specific area for selective milling or directing the ion beam to a FIB lamella positioned on a support grid.

Ion source parameters are easy to program; simply enter the emission current and accelerating voltage. In addition, it is easy to establish the specimen position. Once you enter the operating parameters, the computer controls the instrument functions.

In the imaging mode, select the scan speed, magnification, focus, brightness, and contrast. With a 1.8 mm field of view, the entire surface of a grid or specimen can be imaged, making it extremely easy to mill the area of interest. This is useful when targeting a FIB lamella.

The variable energy ion source generates ion energies as low as 50 eV and a beam size as small as 1 μm

ION MILLING

Ion milling is used on physical science specimens to reduce the thickness to electron transparency. Inert gas, typically argon, is ionized and then accelerated toward the specimen surface. By momentum transfer, impinging ions sputter material from the specimen at a controlled rate. The NanoMilling process enhances specimen quality by removing amorphous damage and Ga implantation, which optimizes the results achieved by state-of-the-art microscopy techniques.

SED specimen targeting

During operation, it is essential to know the position of the ion beam in relation to the specimen. This is of particular importance for post-FIB processing in which the FIB lamella, mounted onto a support grid, can be as small as 10 μm^2 .

An Everhart-Thornley secondary electron detector (SED) is used to image the ion-induced secondary electrons generated from the targeted area. The SED output is processed by the NanoMill system's imaging electronics to provide a real-time view of the specimen, implicitly aligned with the ion



The Main tab is used to program instrument functions and displays the instrument's operating status in real time. The milling box (red rectangle) is positioned over the area of interest, which is a FIB lamella attached to an Omniprobe™ grid.

beam. You can select the scan speed – either faster imaging or enhanced image quality. Frame averaging is employed to reduce noise.

The SED image is displayed on the Main tab. In point mode, place the cursor on the specimen to focus the ion beam to that point. If you need to thin a larger area, select the area with the milling box (red rectangle) and the ion beam will scan within it. The position and the dimensions of the milling box are displayed in microns.

Computer control

The NanoMill system operates with minimal user intervention. Milling conditions, such as

ion source parameters, milling angle, specimen position, temperature threshold, and processing time, are programmed via a single window. The system software allows you to:

- Control access to the various instrument controls and maintenance functionality through the assignment of user privileges
- Use shortcut keys to speed programming and operation
- Review system operation through the Data and Error Logs
- Configure alerts that will notify you of preventive maintenance tasks

Typical processing sequence

For effective specimen preparation, a series of operational sequences can be established. Typical methodology starts with rapid milling at higher ion energies. As the specimen thins, the ion energy is reduced, which results in a lower milling rate that eliminates artifacts. User-determined ion beam targeting at each step of the operation ensures that the proper area of the specimen is processed.

Automatic gas control

Gas is regulated automatically using precision mass flow control technology. An integral particulate filter ensures that high-purity gas is delivered to the ion source. This reduces specimen contamination and allows the NanoMill system to operate for longer cumulative periods before maintenance is required. The low-flow ion source results in minimal gas consumption.

Contamination-free, fully integrated dry vacuum system

The fully integrated vacuum system includes a turbomolecular drag pump backed by a multistage diaphragm pump. This oil-free system assures a clean environment for specimen processing.

The operating system vacuum is 1×10^{-4} mbar and the base vacuum is 3×10^{-7} mbar. The chamber vacuum level is measured with a combination cold cathode and Pirani gauge. Vacuum status is displayed on the Main tab and the vacuum level is displayed on the Maintenance tab.

Specimen mounting

To prevent specimen shadowing, a unique specimen holder provides unobstructed ion trajectories to the specimen, even at 0° . This is particularly important when the ion beam is targeted at the leading edge of a FIB-prepared specimen.

The specimen is mechanically affixed to the specimen holder, thus eliminating the possibility of contamination from an adhesive. A separate loading station (included) provides a platform for the specimen that eases holder positioning.

Automatic load lock for quick specimen transfer

The NanoMill system features a load lock for rapid specimen exchange. The specimen holder is connected to the end of a conventional transfer rod. After the load lock door is closed and the load lock is evacuated, an automatic gate valve opens and the specimen holder is manually inserted into the specimen stage using the transfer rod.

You can observe the specimen holder through a viewing window during transfer to and from the specimen stage. A chamber light facilitates the transfer process. Once closed, the gate valve prevents light from entering the chamber and affecting the SED signal. After the load lock is vented, the specimen can be rapidly transferred to a TEM holder, thus reducing specimen contamination from ambient conditions.

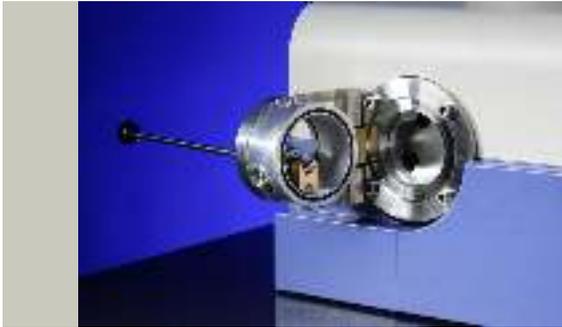
Precise angle adjustment

The impingement angle of the ion beam is programmable from -10° (glancing incidence) to $+30^\circ$. The ion source is fixed in position and the

PLASMA CLEANING

Following the NanoMilling process, Fischione Instruments highly recommends that you plasma clean the specimen and specimen holder.

During fine-probe microanalysis, organic contamination may build up on the specimen. A 10 seconds to 2 minutes cleaning time in the Fischione Instruments' Model 1020 Plasma Cleaner or Model 1070 NanoClean removes the contamination without altering the specimen's structure or composition. Longer cleaning times can remove contamination spots caused by previous TEM viewing of specimens that were not plasma cleaned.



NanoMill system specimen exchange port with load lock door open.

specimen stage tilts to achieve the programmed milling angle that is set through the Main tab. The ion beam can be simultaneously targeted at both specimen surfaces when the angle is set to 0°. The angle can also be set so that the ion beam is directed at either specimen surface.

Milling operations at low angles of incidence (less than 10°) minimizes irradiation damage and specimen heating. Because low-angle milling facilitates the uniform thinning of dissimilar materials, it is highly beneficial when preparing layered or composite materials, as well as conventionally prepared cross-section TEM (XTEM) specimens.

Specimen position control

The stage can be rotated both clockwise and counter-clockwise; the maximum angle is 180°. The stage rotation enables horizontal alignment of the specimen, which enables improved placement of the milling box.

Integrated specimen cooling

Although milling at low angles and ion beam energies reduces specimen heating, temperature-sensitive specimens may require further cooling. Liquid nitrogen cooling of the specimen stage is very effective in eliminating heat-induced artifacts.

The cryogenic cooling system is highly efficient; initial cool down of the system takes only 20 minutes. When the stage is precooled, the specimen reaches cryogenic temperature in less than 5 minutes of insertion.

The cooling system features an integral dewar that is interlocked through the NanoMill system's advanced control. Specimen stage temperatures to -170 °C are common. Dewar hold time is 4 to 6 hours, depending on operating conditions. Stage temperature is displayed on the Main tab.

At the conclusion of the NanoMilling process at cryogenic temperatures, the transfer rod is used to retrieve the specimen holder from the specimen stage and position it in the load lock. The load lock can be vented as soon as the specimen holder temperature reaches 20 °C.

In addition, a thermal safeguard can be programmed to a specific temperature at which the ion source will be automatically deactivated. This is particularly beneficial if the liquid nitrogen in the dewar becomes depleted and a rise in specimen temperature is intolerable.

Process termination

In addition to temperature-based process termination, the NanoMilling process can be automatically stopped at a programmed elapsed time. Following termination by either time or temperature, the specimen holder remains in the chamber under vacuum until you move it into the load lock, vent the load lock, and then transfer the specimen to the TEM. At any time during milling, you can manually stop the process.

Vacuum chamber

The chamber, in combination with the turbomolecular drag pump, provides a hydrocarbon-

free specimen environment for effective processing. Because the processing chamber is under constant vacuum, milling can begin almost immediately after specimen insertion. A spare port on the chamber is ideally suited for connecting a residual gas analyzer to the NanoMill system.

Minimal maintenance

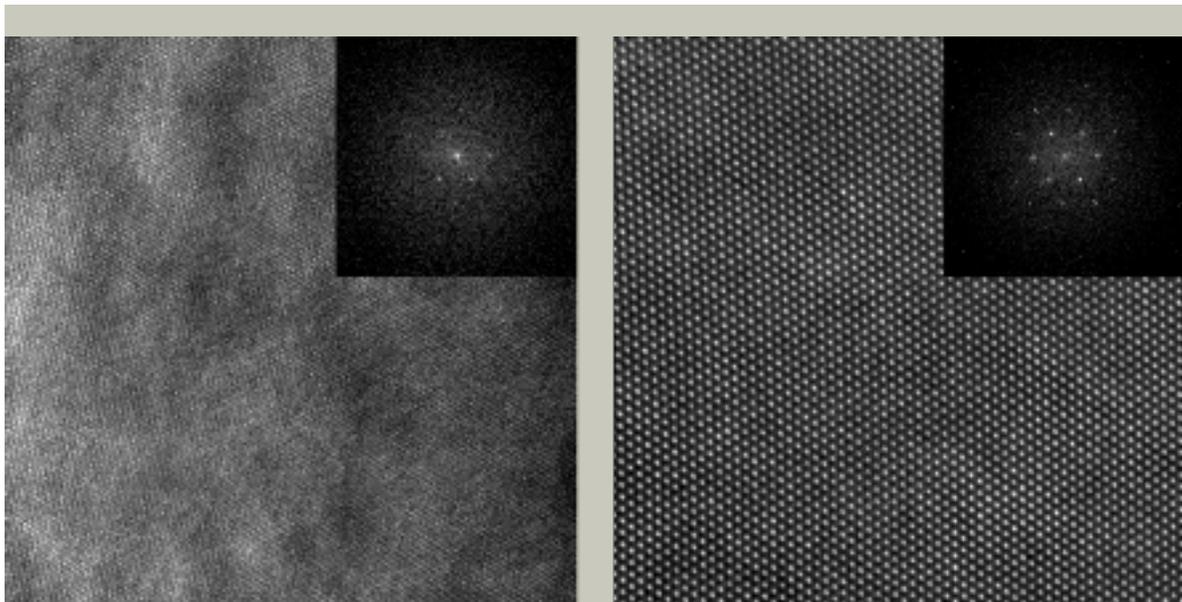
All system components are easily accessible for service. The ion source is designed for both

long component life and extended maintenance intervals. When needed, the filament can be readily replaced.

Password-protected diagnostic software allows complete control over all instrument functions.

Preventive maintenance

Service contracts are available; contact sales@fischione.com.



In situ lift out of Si imaged at 200 kV by a high resolution TEM. Image and fast Fourier transform (FFT) on the left were acquired post-FIB milling using 5 kV Ga. Image and FFT on the right were acquired after low-energy NanoMilling (200 eV beam energy at a 10° specimen tilt). The left FFT (before NanoMilling) shows a diffused halo, which is attributed to amorphous damage. The right FFT (after NanoMilling) shows an absence of the diffused halo, which indicates the NanoMill system removed the amorphous damage. *Images courtesy of P. Midgley, R. Dunin-Borkowski, and D. Cooper, University of Cambridge, Cambridge, UK.*



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