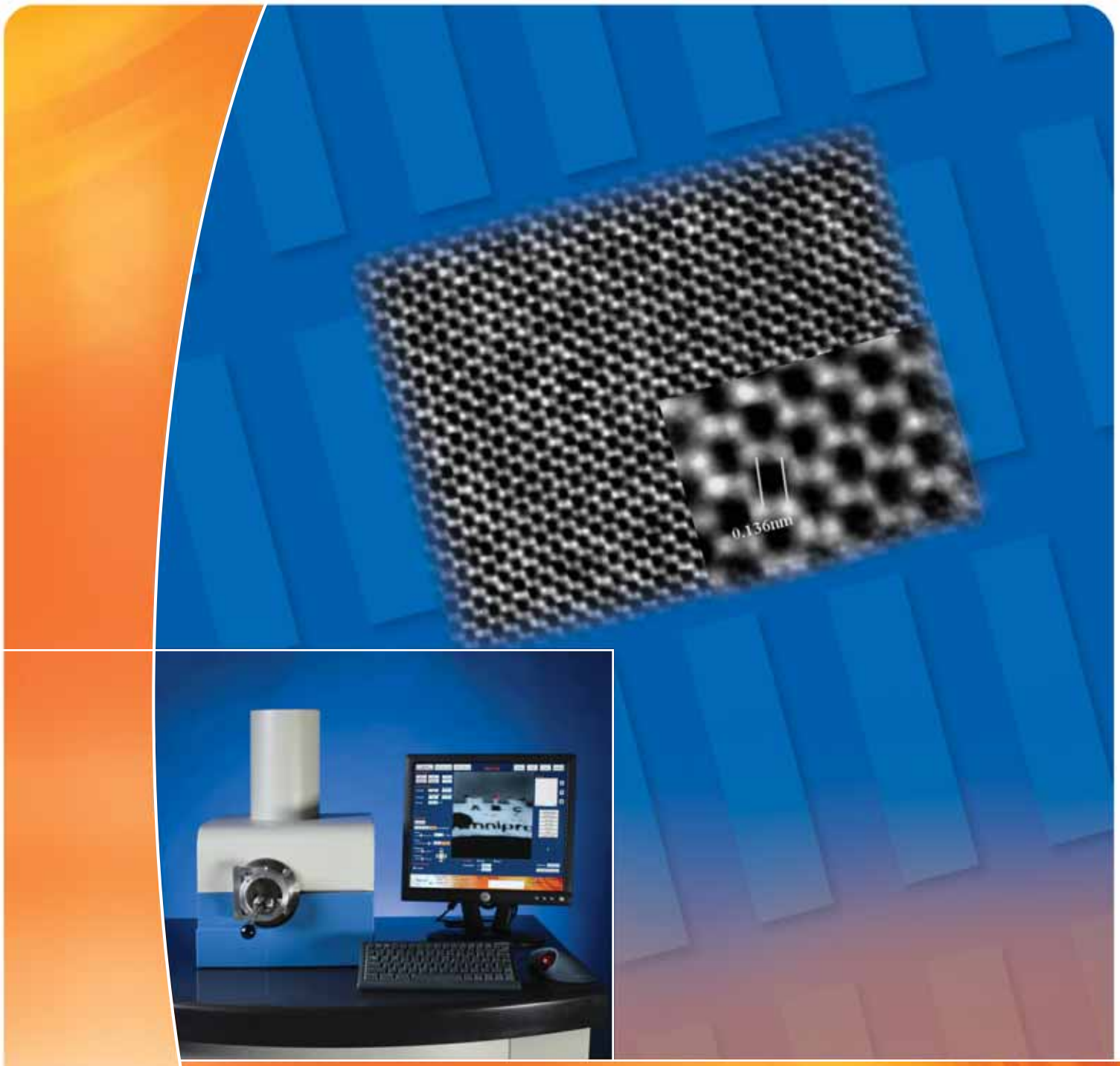


Model 1040 NanoMill®

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

FISCHIONE INSTRUMENTS

EXCELLENCE...MAGNIFIED

Model 1040 NanoMill®

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

Model 1040 uses an ultra-low-energy, concentrated ion beam to produce the highest quality specimens for transmission electron microscopy (TEM).

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.

Since features in nanotechnology research and semiconductor device specimens continue to decrease in size, it is essential that specimens be both very thin and 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-Angstrom.

Create thin specimens for TEM

Fischione's Model 1040 NanoMill system is an excellent tool for preparing the ultra-thin, high-quality specimens needed for advanced transmission electron microscopy (TEM) imaging and analysis.

The variable energy ion source generates ion energies as low as 50eV. In addition, the beam size is as small as 2 microns, enabling removal of amorphization, implantation, or redeposition from targeted areas.

An ideal application for the Model 1040 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 30nm thick. The Model 1040 is ideally suited to removing these layers.

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[sm] process enhances specimen quality, which optimizes the results achieved by state-of-the-art microscopy techniques.

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, typically argon, and has an operating voltage range of 50eV to 2kV at variable working distances. The source yields sufficient current density to remove specimen damage within a reasonable time. The NanoMilling[sm] process can be accomplished in as little as 20 minutes.

Since the ion beam can be focused into a 2-micron diameter spot, redeposition of sputtered material onto the area of interest is avoided. Beam current and spot size are adjusted by using different sized TEM-type apertures. 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.

All ion source parameters are readily programmable. The user simply inputs the desired emission current and accelerating voltage. Additionally, specimen position is easily established. Once the operating parameters have been programmed, the computer controls all instrument functions.

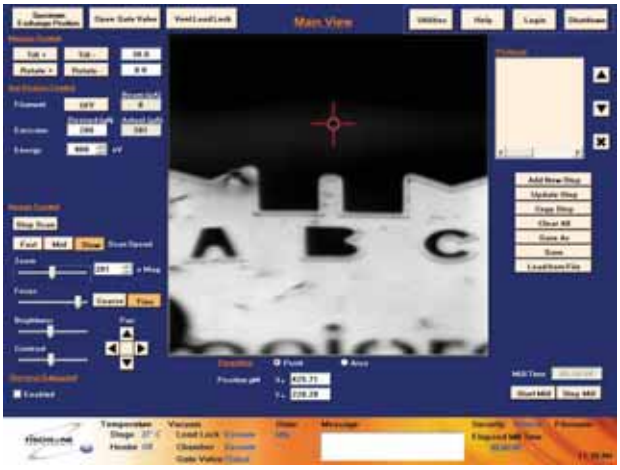
In the imaging mode, the user selects scan speed, zoom, focus, brightness, and contrast. With a 3mm field of view, the entire surface of a grid or specimen can be imaged, making it extremely easy to mill the area of interest. This is particularly useful when targeting a FIB lamella.

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 microns square.

Targeting directs the beam to a specific area of interest. An Everhart-Thornley secondary electron detector (SED) is used to image the ion-induced secondary electrons generated from the targeted area of the specimen. The SED output is processed by the Model 1040's imaging electronics to provide a real-time view of the specimen, implicitly aligned with the ion beam. Variable scan speeds are selectable, allowing either faster imaging or enhanced image quality. Frame averaging is employed to reduce noise.

The SED image is displayed on the Main View window. In spot mode, placing the cursor at the desired position on the specimen focuses the ion beam to that point. If a larger area is to be thinned, that area can be pre-selected by drawing a box with the cursor and the ion beam will be scanned within the selected area. Both the position and the dimensions of the scan box are displayed (in microns) on the Main View window.



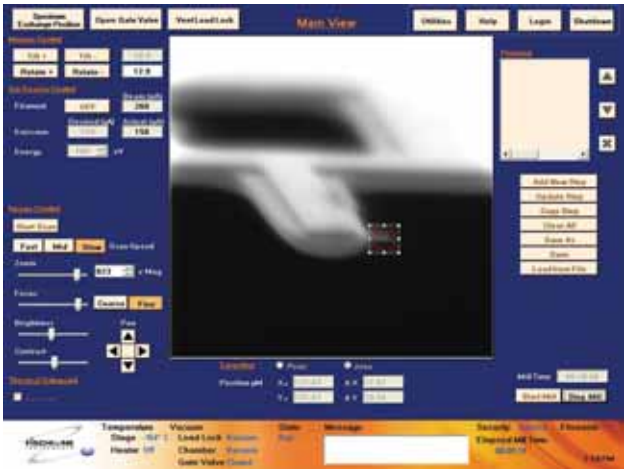
SED image of TEM grid

Computer control

The fully programmable NanoMill system operates with minimal user intervention. NanoMilling℠ conditions such as ion source parameters, milling angle, specimen position, temperature threshold, and processing time are programmed via a single window, easy-to-use interface.

- A sequence of individual milling steps can be stored and recalled. Recipes are created for given materials resulting in highly reproducible results.
- The software allows selective access to the various instrument controls corresponding to levels of expertise and needs for instrument operation. Administrative rights can be provided to qualified maintenance staff.
- Shortcut keys facilitate programming and instrument operation.
- Utilities menus include Data Log, Error Log, Maintenance, and Configuration.

- The Maintenance menu is configurable to provide alerts when various types of preventative maintenance are required.
- The Model 1040 can be networked. Its software is protected by an internal hardware firewall.



The Main View window both facilitates the programming of all instrument functions and displays a real-time indication of the instrument's operating status.

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, resulting 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 Model 1040 to operate for longer cumulative periods before maintenance is required. The ion source uses low flow resulting in minimal gas consumption.

Contamination-free, fully integrated dry vacuum system

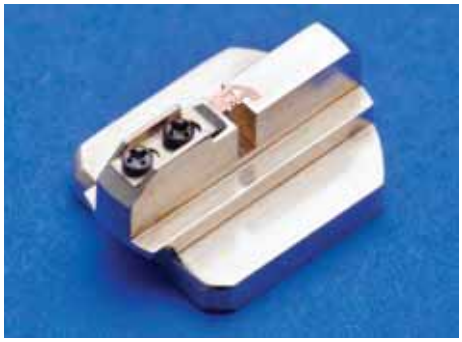
The fully integrated vacuum system includes a turbomolecular drag pump backed by a multistage diaphragm pump. This oil-free system assures a clean environment for specimen processing.

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

Specimen mounting

To prevent specimen shadowing, a unique specimen holder provides unobstructed ion trajectories to the specimen, even at zero degrees. 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; therefore, there is no possibility of specimen contamination from an adhesive. A separate loading station (included) provides a platform for the specimen that facilitates its positioning into the specimen holder.

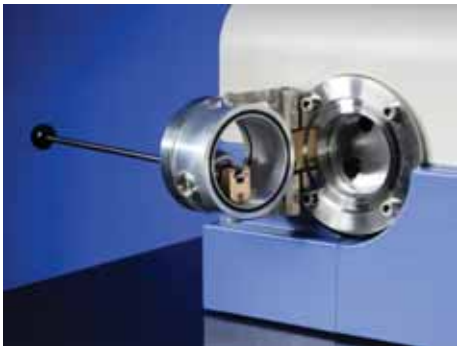


Specimen holder

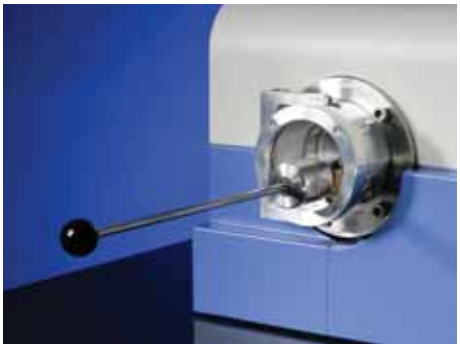
Automatic load lock for quick specimen transfer

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

The specimen holder can be continuously observed 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 the TEM, thus reducing specimen contamination from ambient conditions.



Specimen exchange port with load lock door open



Specimen exchange port with load lock door closed

Plasma Cleaning

Following the NanoMilling[sm] process, plasma cleaning is highly recommended for the specimen and the TEM specimen holder.

During fine probe microanalysis, organic contamination may build up on the specimen. A cleaning time of ten seconds to two minutes in the Fischione Model 1020 Plasma Cleaner 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.

Precise angle adjustment

The impingement angle of the ion beam is programmable from 0° (glancing incidence) to ±10°. The ion source is fixed in position and the specimen stage tilts to achieve the programmed milling angle that is established through the Main View window. 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.

Utilizing the NanoMilling[sm] process at low angles of incidence (less than 10°) minimizes irradiation damage and specimen heating. Since 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 NanoMill system is ideally suited for preparing specimens from heterogeneous or layered materials. Its specimen holder and stage make it easy to position the specimen with respect to the ion beam for optimal milling.

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 cooldown of the complete system takes only 20 minutes. Once the stage has been pre-cooled, the specimen reaches cryogenic temperature within 5 minutes after it is inserted.

The liquid nitrogen cooling system features an integral dewar that is interlocked through the Model 1040's advanced control. Specimen stage temperatures to –175°C are common. Dewar hold time is between 4 and 6 hours, depending on operating conditions. Stage temperature is continuously displayed on the Main View window.

At the conclusion of the NanoMilling[sm] 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. In order to avoid specimen contamination, an integral heater automatically raises the specimen holder temperature to 20°C before venting.

continued

Integrated specimen cooling *(continued)*

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[sm] process can be automatically stopped at a programmed elapsed time.

Following termination by either time or temperature, the specimen holder remains in the specimen stage under vacuum until the user extracts it and moves it into the load lock for subsequent venting and transfer to the TEM.

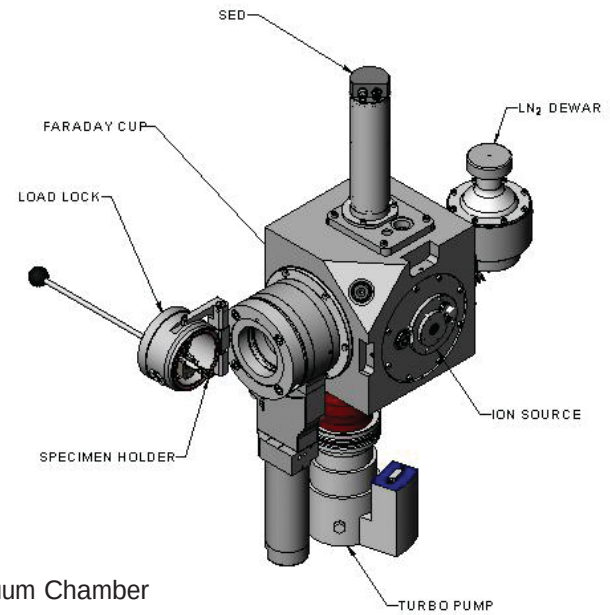
If so desired, at any time during milling, the user can manually stop the process.

Vacuum chamber

The chamber, in combination with the turbomolecular drag pump, is highly beneficial in that it provides a hydrocarbon-free specimen environment for effective processing. Additionally, it allows the ion source to be positioned in an orientation that creates ideal ion beam properties at the specimen. Because the processing chamber is under constant vacuum, milling can begin almost immediately after the specimen is inserted.

To further enhance vacuum integrity, the Fischione Model 190 Cryo-Can can be connected to the load lock. Once it is filled with liquid nitrogen, contaminants condense onto the cold surface of its inner vessel. Removing the cold vessel eliminates the trapped contamination from the Model 1040 chamber.

A spare port on the chamber is ideally suited for connecting a residual gas analyzer (RGA) to the Model 1040.



Vacuum Chamber

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 and easily aligned in relation to the Wehnelt assembly using a stereomicroscope.

For calibration and alignment of the ion source, a Faraday cup is positioned within the chamber opposite the source. Ion beam current, as detected by the Faraday cup, is monitored by the Model 1040's internal picoampmeter and is continuously displayed on the Main View window.

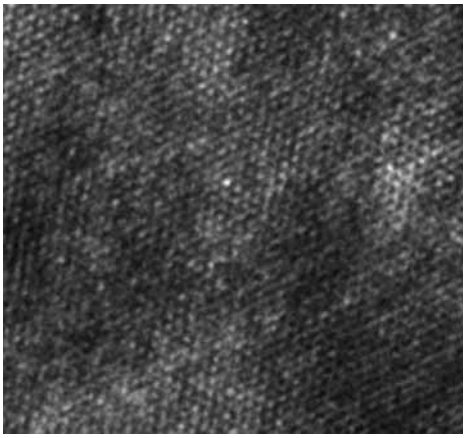
The user can program the system to provide notification when preventative maintenance is needed.

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

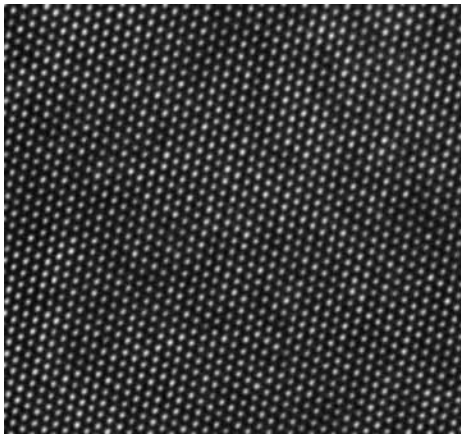
Data and error logs compile historical information about the operating conditions for completed specimen preparation processes.

Results

Transmission electron microscope (TEM) images of Si.



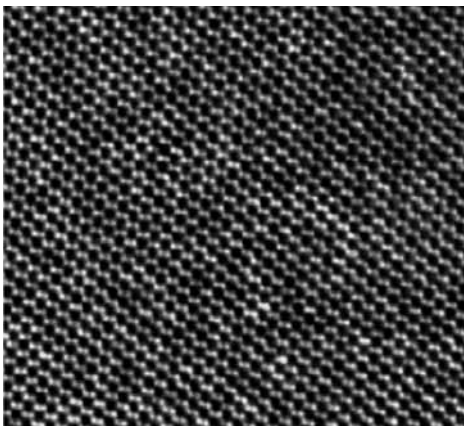
High resolution TEM image of Si in [110] orientation, showing the effect of Ga implantation and surface amorphization on phase contrast imaging.



High resolution TEM image of Si in [110] orientation, demonstrating the effect on phase contrast imaging after Ga implantation and amorphization are removed by the NanoMilling℠ process.

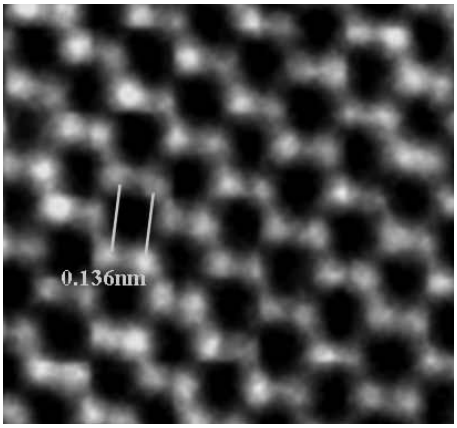
continued

Results *(continued)*



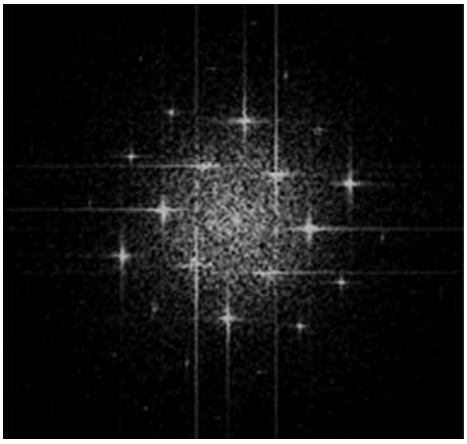
Aberration-corrected high-resolution TEM image showing Si atomic structure (dumbbells) clearly resolved after removal of the Ga implanted and amorphized surface layers.

Image courtesy Professor Angus Kirkland and Dr. Crispin Hetherington, Oxford University (United Kingdom)



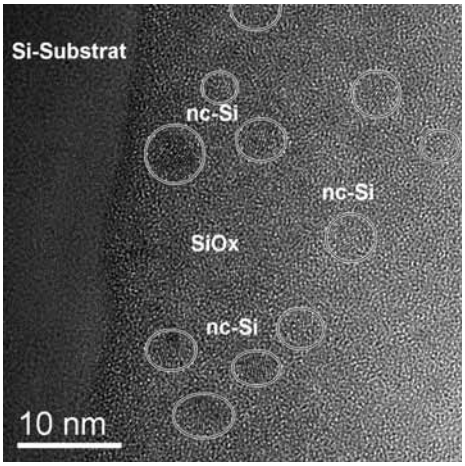
Enlarged region of the Si dumbbell image, left.

Image courtesy Professor Angus Kirkland and Dr. Crispin Hetherington, Oxford University (United Kingdom)



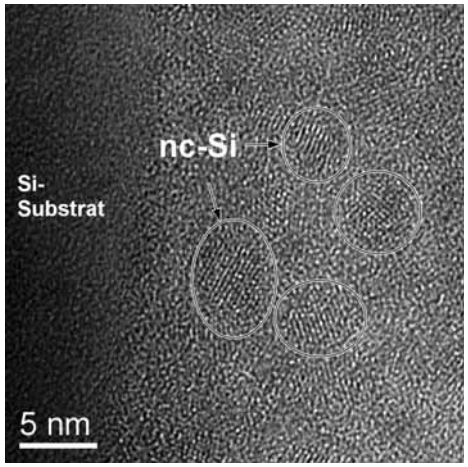
FFT of the Si lattice image presented in figure above, showing the higher order specimen periodicities conveyed after removal of the Ga implanted and amorphized surface layers.

Image courtesy Professor Angus Kirkland and Dr. Crispin Hetherington, Oxford University (United Kingdom)



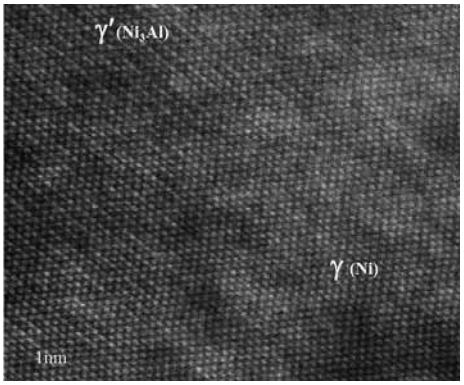
Si Quantum Dots embedded in an amorphous SiO_x Matrix. Because of their size and beam sensitivity, these quantum dots were not visible in the as-prepared FIB lamella.

Sample courtesy of J. Mayer, A. Dimyati, RWTH Aachen University and Ernst Ruska-Centre, Research Centre Juelich (Germany)



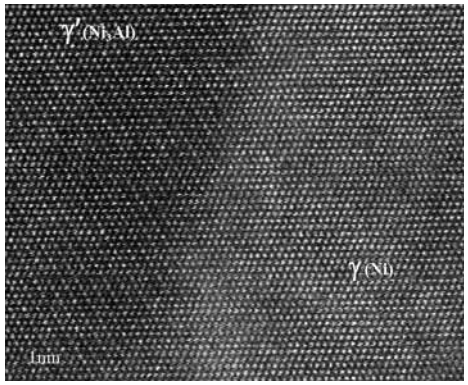
Si Quantum Dots embedded in an amorphous SiO_x Matrix. Because of their size and beam sensitivity, these quantum dots were not visible in the as-prepared FIB lamella.

Sample courtesy of J. Mayer, A. Dimyati, RWTH Aachen University and Ernst Ruska-Centre, Research Centre Juelich (Germany)



Uncorrected Z-contrast STEM image of Ni-based superalloy Renè 88DT prepared by conventional ion milling.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)

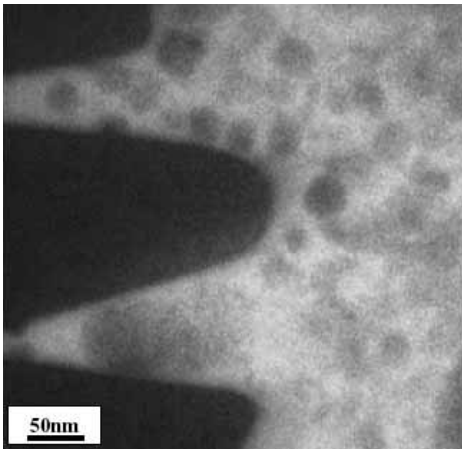


Probe aberration-corrected Z-contrast STEM image after a 500eV Ar NanoMilling[sm] process shows the removal of the surface damage from the highly ordered Ni – based superalloy. The coherent boundaries which possess a small degree of lattice mismatch are revealed.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)

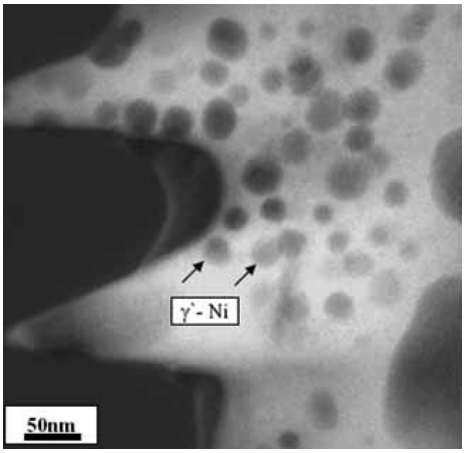
continued

Results
(continued)



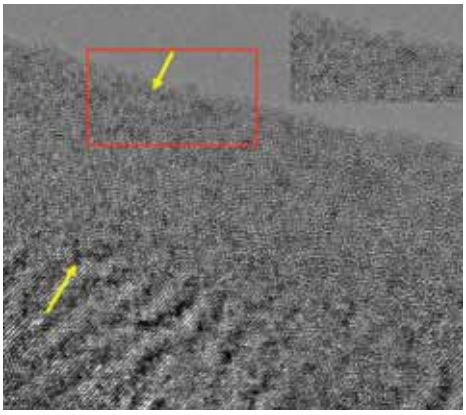
EFTEM image of Ni-based superalloy Renè 88DT prepared by 30keV FIB.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)



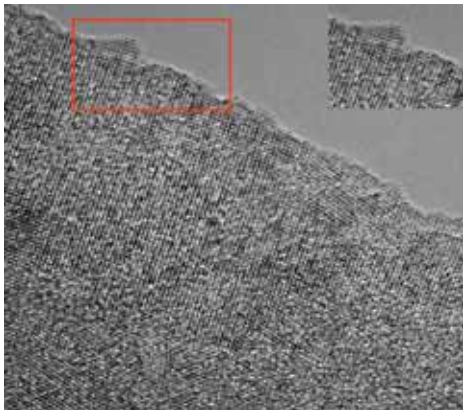
EFTEM image after a 2000eV NanoMilling℠ process enabled reliable phase identification by EFTEM.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)



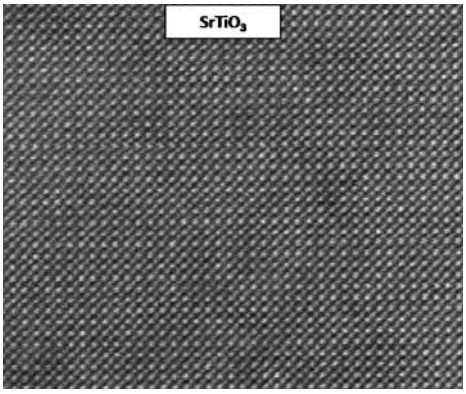
High resolution TEM image of a Ti alloy. The specimen, initially prepared by conventional ion milling, exhibited a 10nm to 12nm amorphous edge.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)



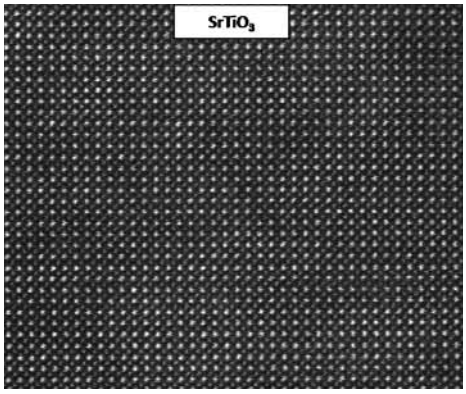
High resolution TEM image after a 500eV Ar NanoMilling[sm] process, yields lattice fringes to the edge of the perforation.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)



Probe aberration-corrected HAADF STEM image of <100> $SrTiO_3$ prepared with 5 keV FIB.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)

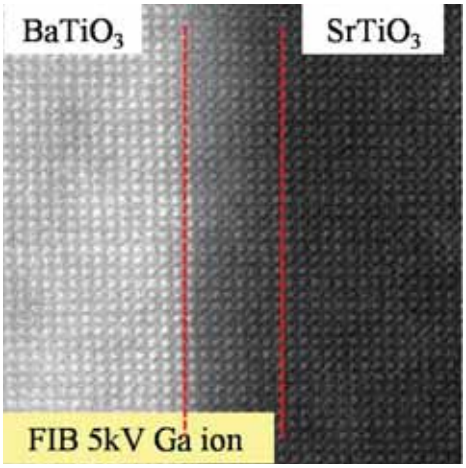


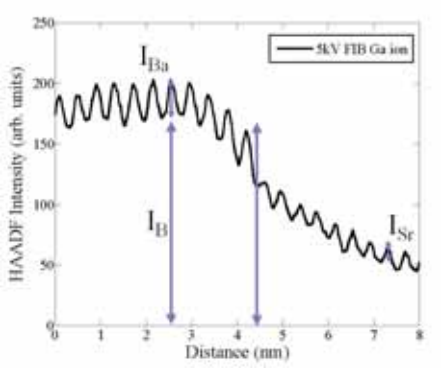
Probe aberration-corrected Z-contrast STEM image of $SrTiO_3$ after a 500eV Ar NanoMilling[sm] process shows that the Sr and Ti interpenetrating lattices can be observed.

Image courtesy of Professor Hamish Fraser, The Ohio State University (U.S.A.)

continued

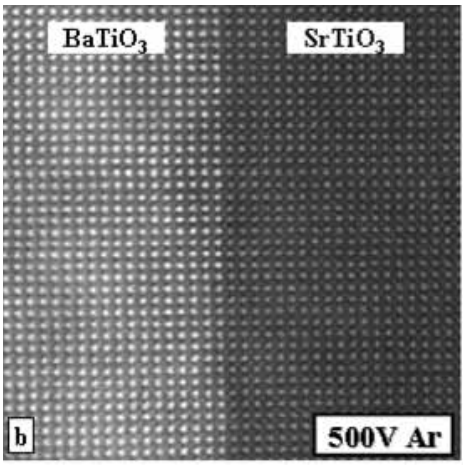
Results *(continued)*

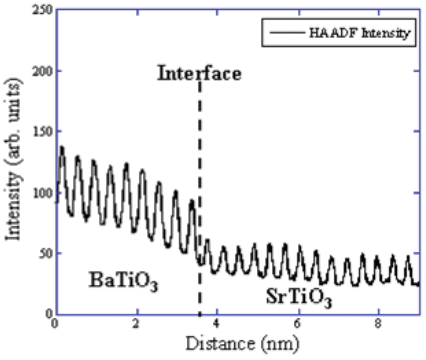




HAADF STEM image of $BaTiO_3$ / $SrTiO_3$ prepared by FIB operated at 5keV. Damage masks the interface separating the $BaTiO_3$ and $SrTiO_3$.

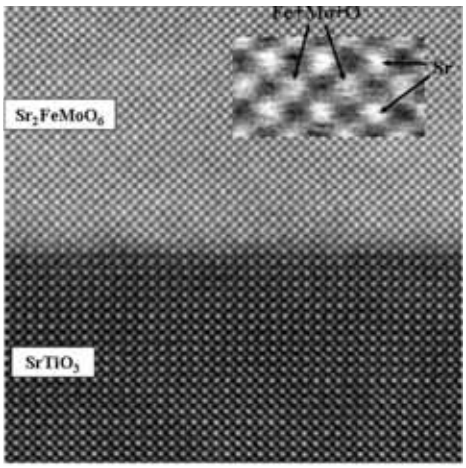
Image courtesy of Professor Hamish Fraser and Dr. Arda Genc, The Ohio State University (U.S.A.)

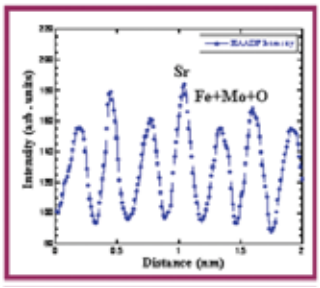


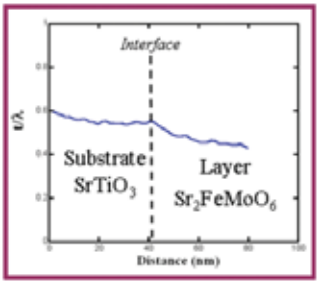


HAADF STEM image after a 500eV Ar NanoMilling℠ process shows that the interface is distinct, with a sharp transition in the intensity plot. The ion sites are well defined, as evidenced by the periodic variation in contrast superimposed on the brightness level.

Image courtesy of Professor Hamish Fraser and Dr. Arda Genc, The Ohio State University (U.S.A.)







Probe aberration-corrected Z-contrast STEM image of the complex interface between the Sr_2FeMoO_6 and $SrTiO_3$ phases. This specimen was initially prepared by FIB operated at 30keV and then subsequently reduced to 5keV. The NanoMilling[sm] process was conducted at 500eV. The position of the interface and major lattice sites within the spinel are located precisely, as indicated in the intensity plots.

Image courtesy of Professor Hamish Fraser and Dr. Arda Genc, The Ohio State University (U.S.A.)

NanoMill® system specifications

Ion source	Filament-based ion source combined with electrostatic lens system
	Variable voltage (50eV to 2kV), continuously adjustable
	Beam current density up to 1mA/cm^2
	Beam diameter as small as 2 microns
	Faraday cup for ion beam current monitoring with a range of 1 picoamp to 2,000 picoamps
	Field-replaceable apertures
Specimen stage	Load lock allows specimen exchange in less than 10 seconds
	Transfer rod for specimen exchange
	Milling angle range of 0° to ±10°
Vacuum system	Turbomolecular drag pump backed by an oil-free diaphragm pump
	Chamber vacuum measurement with a combination cold cathode and Pirani gauge with a range of atmosphere to 1 x 10^{-8} mbar
	System base vacuum of 3 x 10^{-7} mbar
	Operating vacuum of 1 x 10^{-4} mbar
Gas	Automated using mass flow control technology
	Flow rate up to 2 sccm
	Integral particulate filter
	Inert gas, typically argon, with recommended purity of 99.999%
Specimen targeting	Ion beam capable of being targeted at one spot on the specimen surface or scanned within a selected area
User interface	Menu-driven interface
	Programmable milling cycles with system status displayed
Chamber illumination	User-controlled chamber illumination to facilitate specimen exchange
Specimen cooling	Liquid nitrogen conductive cooling with automatic temperature interlocks
	Stage temperature to –175°C
	System cooldown time less than 20 minutes
	Specimen cooldown time less than 5 minutes
	Dewar hold time up to 6 hours
	Integral load lock heater ensures rapid specimen warming times to ambient temperature
Automatic termination	Process termination by time or temperature
Imaging	SED-based imaging technology
	3mm field of view
	Everhart-Thornley detector
	Specimen image displayed on Main View window
Enclosure	Dimensions: 39" (991 mm) W x 58" (1,474 mm) H x 31" (788 mm) D
	Weight: 507 lb (230.5 kg)
Power requirements	110/220 VAC, 50/60Hz, 1,000 watts
Warranty	One year
Service Contract	Available upon request

FISCHIONE INSTRUMENTS

EXCELLENCE...MAGNIFIED

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

Cover image: High resolution Bright Field TEM image of a Si specimen initially prepared by FIB. The specimen was final-thinned using the Model 1040 NanoMill® TEM specimen preparation system operated at 200eV for 10 minutes with a beam current of 170pA and a milling angle of 5 degrees. An area of at least 800nm by 800nm was available for analysis. The insert is the lattice image showing the removal of amorphization.

Part No. 009-1303, Rev. 3