16 - Fischione Instruments

The Fischione range of electron microscopy specimen preparation equipment has been developed, refined and expanded over a number of years. Based on proven technology, Fischione products are renowned for their reliability and consistency in producing high quality specimens, from initial preparation with the model 160 specimen grinder to final processing for the production of high quality TEM specimens.

TEM examination and analysis requires clean, well-prepared specimens that are made without altering their microstructure or composition. The specimen needs to be thinned to electron transparency and the model 1010 Ion Mill is an excellent tool for creating such thin specimens. The model 1010 is fully programmable and has two independently adjustable Hollow Anode Discharge (HAD) ion sources with individual automatic ion source gas control. High or low energy operation is possible for either rapid milling or gentle specimen polishing. Furthermore, the Ion Mill has an oil free vacuum system so specimen contamination is minimised.

Modern electron microscopes with high brightness electron sources, such as LaB_6 filaments and field emission guns (FEG), combine a small electron probe for microanalysis with increased beam current density, yielding high resolution images as well as enhanced analytical data.

As the probe size is decreased and beam current density increased, specimens tend to become contaminated. As a result, the quality of the specimen and the cleanliness of both the specimen and the specimen holder are more important than ever. Contamination comes from many sources, including the sample preparation process and handling of both the specimen and TEM holder for example. The model 1020 plasma cleaner cleans specimens and TEM holders immediately before they are inserted into the electron microscope. Plasma cleaning removes existing carbonaceous debris from the specimen and also prevents contamination from occurring during examination.

The Fischione product portfolio has recently been improved and expanded to include a new generation of state-of-the-art instruments: the model 1030 ASaP automated sample preparation system is a powerful and flexible tool that can significantly enhance image quality and analytical data derived from SEM specimens; the model 1040 NanoMill® is a system in which a revolutionary ultra-low-energy, inert-gas, concentrated ion beam produces specimens free from amorphous and implanted layers, making it ideal for post-FIB processing.

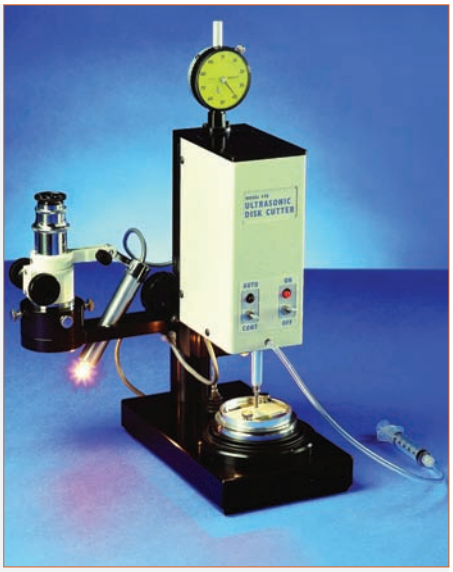
To assist with sample examination, interpretation and analysis, the model 2000 TEM tomography holder series enables 3-D construction of TEM images. This advanced series includes specimen holders that allow high tilt and extended field of view, and is available in single, dual and on-axis versions which are compatible with even the narrowest pole piece geometries.

Fischione model 130 specimen punch

This punch prepares high quality disc specimens using a precision ground punch and die plate to eliminate specimen stress and distortion. For convenience, a spring-loaded return plunger keeps the disc specimen on the die plate surface. The model 130 punch is available in standard sizes of 3.0 mm and 2.3 mm.

B8910 Model 130 specimen punch, 3.0 mm
B8911 Model 130 specimen punch, 2.3 mm

Fischione model 170 ultrasonic disc cutter

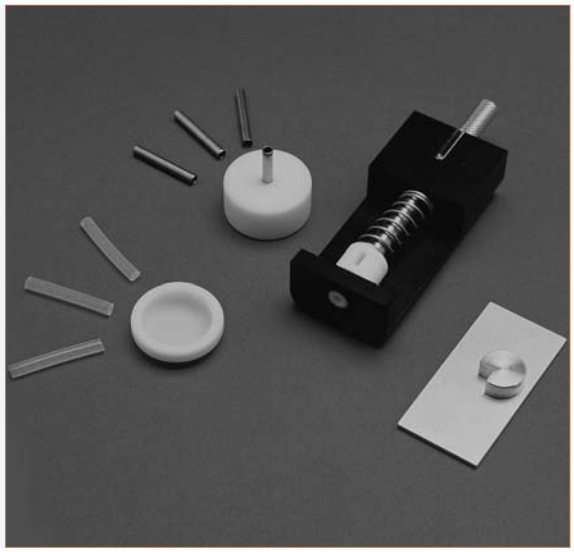


The ultrasonic disc cutter is ideal for preparing cross-section TEM specimens (XTEM) from materials which are traditionally difficult to machine by conventional means, eg. ceramics, semiconductors and geological minerals. The disc cutter machines samples into discs, cores or rectangular wafers, ranging from 10 µm to 10 mm in thickness, using an abrasive slurry of cubic boron nitride or silicon carbide. The cutting rate is optimised to achieve rapid machining while minimising thermal and mechanical damage. A dial indicator accurately displays the cutting depth of the tool, and an electrical continuity detector automatically senses when the cutting process is complete. The disc cutter comes with a 3 mm diameter tool as standard, but a range of other cutting tools is also available, including a 2.3 mm diameter cutter and rectangular cutters of 2 x 3 mm and 4 x 5 mm.

An optional optical microscope (x40 magnification) is available and highly recommended for both the observation of the specimen and as an aid to positioning. A precision mechanism establishes the angular positioning of the microscope and an alignment locking mechanism ensures that the registration between the microscope and cutting tool has a repeatability of better than 10 µm.

B8165 Model 170 ultrasonic disc cutter
B8166 Microscope attachment
B8168A Cutter, 2.3 mm dia
B8168B Cutter, 2 x 3 mm
B8168C Cutter, 4 x 5 mm

Fischione model 180 XTEM preparation kit



The XTEM preparation kit includes all components needed to prepare high quality, precise cross-section TEM (XTEM) specimens from rectangular wafers of an area of interest from a bulk sample. The XTEM preparation kit makes it easy to stack and bond the cut wafers together. These wafers can then be readily cut or cored by the model 170 ultrasonic disc cutter (**B8165**). The kit includes a vice assembly, a stack mounting plate assembly, a mixing dish, a Teflon® mould, glue sticks and brass tubes.

B8915 Model 180 XTEM preparation kit

Fischione model 160 specimen grinder

This specimen grinder provides an accurate and dependable means of mechanically pre-thinning specimens. The large diameter of this grinder provides excellent stability and, with the precise fit of the platen, specimens of uniform thickness and parallel sides are consistently produced. It has a graduated scale for accurate material removal and large mass, so no force need be applied.

If further thinning via dimpling is required, the platen is easily transferable from the specimen grinder to the model 200 dimpling grinder, eliminating the risk of damaging the specimen during demounting.

B8156 Model 160 specimen grinder

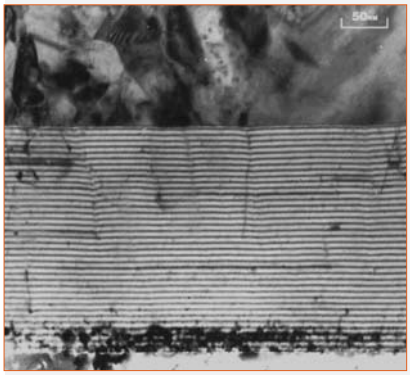
Fischione model 200 dimpling grinder

The model 200 Dimpling Grinder is a state-of-the-art mechanical grinder for preparing electron microscopy samples, which makes it indispensable when ion milling is used for final specimen thinning.

This ultra-precise and versatile tool is ideal for creating specimens free from uneven thinning, surface defects and irradiation damage. By simply changing tools, the dimpling grinder can be used to flat grind bulk specimens, dimple, and then finally polish the specimen to electron transparency. This easy-to-programme grinder includes precise specimen positioning, an indicator for precise specimen thickness and optimised grinding control resulting in vibration-free grinding. As both the grinding force and rate are controlled, specimens of exceptional quality are produced.

B8155 Model 200 dimpling grinder

Fischione model 1010 standard Ion Mill



XTEM specimen showing a polycrystalline copper film on an epitaxial $Ti_{0.5}W_{0.5}N/TiN$ superlattice on MgO.

Image courtesy of I. Petrov, University of Illinois (U.S.A.)

The Ion Mill is an excellent advanced microprocessor controlled bench top tool that creates the thin specimens needed for TEM imaging and analysis with minimal user intervention. It offers clean specimen processing and minimal maintenance, and is ideally suited to preparing cross section (XTEM) specimens from heterogeneous or layered materials. All milling parameters are programmed through an integrated keyboard and the menu driven system provides easy operation in manual or automatic modes. Milling is performed by two independently controlled hollow anode discharge ion sources which are focused on either side of the specimen. Voltages up to 6.0 kV can be selected for rapid milling or as low as 500 V and the beam impingement angle is programmable from 0° to 45°. The specimen stage can be liquid nitrogen cooled to minimise specimen damage.

Accessories include an increased capacity liquid nitrogen Dewar which can extend the cooling time up to 11 hours, an optical microscope for specimen viewing, automatic milling termination and chemically assisted etching.

B8900 Model 1010 Ion Mill
B8901 Low angle milling upgrade
B8902 Stereomicroscope
B8903 Autotermination
B8904 Liquid nitrogen cooling
B8906 Liquid nitrogen cooling, extended duration
B8905 Chemically assisted etching

Fischione model 1010 Ion Mill high magnification version

A high magnification version of the Ion Mill is also available. The model 1010 can be configured with a high magnification imaging system comprised of a high magnification microscope coupled to a CCD camera and video monitor for image capture and display. This system is ideal for preparing site-specific specimens.

B8920 Model 1010 Ion Mill, high magnification version
B8922 High magnification microscope/camera assembly for specimen observation
B8923 Autotermination system
B8924 Liquid nitrogen cooling
B8926 Liquid nitrogen cooling, extended duration

Fischione model 110 twin-jet electropolisher

The twin-jet electropolisher simultaneously thins and polishes both sides of a specimen, using two jets to direct electrolyte flow, and producing electron transparent specimens free from induced artifacts within just a few minutes. Constructed from electrolyte resistant materials, the polishing cell incorporates a photocell perforation detection system. The unit is supplied complete with jets, electrolyte pump, photocell and specimen holder. A digital (model 140) or analogue (model 120) power controller is required in addition.

B8410 Model 110 automatic twin-jet electropolisher
B8413 Specimen holder 3.0 mm
B8414 Specimen holder 2.3 mm

Fischione model 120 automatic power controller

The automatic power control unit provides complete electronic support for the model 110 twin-jet electropolisher. It controls the electrolyte flow, polishing voltage circuit, light source, detection sensitivity and photocell shutoff circuit. Two analogue meters indicate the polishing voltage and current levels. A switch selects whether or not current is applied, enabling either electropolishing or chemical etching. Audible and visual indicators are included.

B8411 Model 120 automatic power controller

Fischione model 140 digital power controller

The digital power control unit, like the model 120 analogue controller, is also designed for use with the model 110 twin-jet electropolisher. Digital displays allow precise setting and monitoring of voltage and current levels. The photocell circuitry detects the first sign of light penetration through the specimen and activates both audible and visual alarms independent of the position of the pump and polish switches. The amount of light required to activate the photocell shut-off can be adjusted to control the perforation size in the specimen. Various polishing methods are possible depending on whether pump, continuous or polish auto mode is chosen.

B8412 Model 140 digital power controller

Fischione model 220 low temperature container

The low temperature container is for use with the model 110 twin-jet electropolisher for applications at cryogenic temperatures. The electrolyte contained in the glass dish is cooled via conduction from a cooling medium such as liquid nitrogen and methanol. A double wall, highly insulated construction maintains temperature throughout the electropolishing process. A hole in the top plate provides access for a thermometer or thermocouple. The container is manufactured entirely from electrolyte resistant materials.

B8415 Model 220 low temperature container

Fischione model 1020 plasma cleaner

The model 1020 plasma cleaner cleans specimens immediately before they are inserted into the electron microscope. Plasma cleaning removes existing carbonaceous debris from the specimen and also prevents contamination from occurring during imaging and analysis.

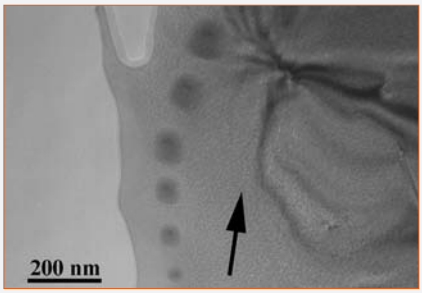
A low-energy, inductively-coupled high frequency (HF) plasma effectively cleans a specimen surface without changing its elemental composition or structural characteristics. Highly contaminated specimens can be cleaned in two minutes or less. To optimize cleaning, a mixture of 25 % oxygen and 75 % argon is generally recommended. An oxygen based plasma is highly effective in removing organic (hydrocarbon) contamination.

An oil free vacuum is essential to ensure optimal cleaning, in this case using a turbo molecular drag pump backed by a multi-stage diaphragm pump, which provides suitable vacuum characteristics to generate and sustain the plasma. The plasma is created in a quartz tube into which the specimen holder is inserted. Sophisticated gas dynamics ensure an even distribution of plasma within the chamber and ultimate downstream sample or holder positioning enables effective cleaning with minimal heating.

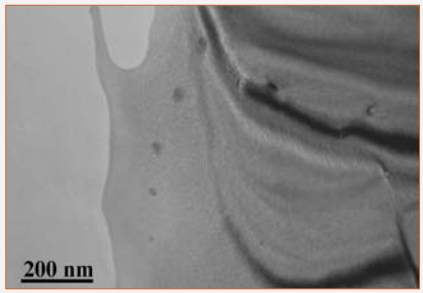
The specimen holder is inserted through a single port into the chamber. Ports are available for FEI/Philips, JEOL, Zeiss/LEO, Hitachi and ISI/ABT/Topcon side entry holders and are easily interchangeable. A port plug seals the chamber under vacuum when not in use.

The chamber can also be used for cleaning bulk SEM specimens, aperture strips and other microscope components. The instrument is operated via a simple keypad with manual or automatic operation. A portable pumping station (**B8951**) can be interfaced to the plasma cleaner to evacuate holder storage tubes.

B8950 Model 1020 plasma cleaner

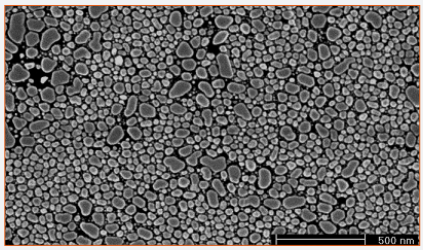


TEM (a)

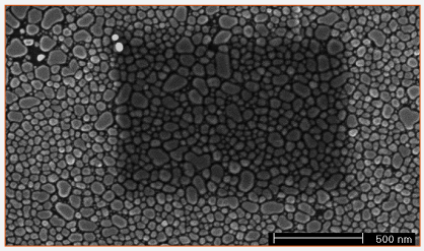


TEM (b)

TEM micrographs of $SrTiO_3$ taken (a) after 1 min and (b) 5 min of plasma cleaning.



SEM (a)



SEM (b)

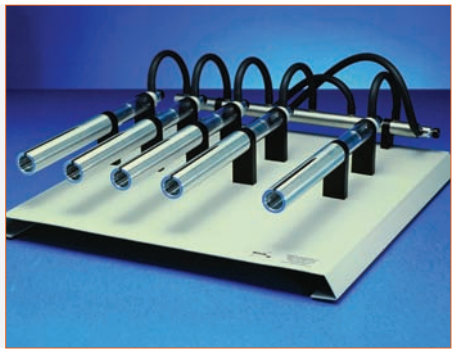
SEM micrographs of gold islands on carbon taken (a) before and (b) after plasma cleaning.

Please specify microscope and holder type.

Fischione model 9020 vacuum pumping station

The vacuum pumping station can be installed on the plasma cleaner to store five specimen holders under vacuum. The pumping station features a heavy duty metal base with non-skid feet, five independently valved vacuum storage containers, a vacuum pumping manifold and all the necessary components for connecting to the model 1020 plasma cleaner.

B8951 Model 9020 vacuum pumping station



Please specify microscope and holder type.

Fischione model 9010 vacuum storage container

After cleaning in the model 1020 plasma cleaner, specimen holders can be inserted into the vacuum storage container so they can be stored or transported securely in a vacuum environment. A sight glass gives a clear view of the specimen area and specimen holder.

B8952 Model 9010 vacuum storage container

Please specify microscope and holder type.

Fischione model 190 Cryo-Can

The Cryo-Can is designed to eliminate sample contamination during SEM operation, providing a clean environment for sample imaging and analysis. Contaminants within the chamber condense onto a removable, cold surface, helping to eliminate contamination from sample outgassing and other sources. It requires no separate vacuum or electrical interface, and can be used either before or during SEM operation.

The SEM can still be operated while the Cryo-Can is cooled, even on SEMs without airlocks. The Cryo-Can is ideal for high beam current applications, improving both imaging and analytical data quality.

B8436 Model 190 Cryo-Can

Fischione model 1030 automated sample prep (ASaP) system

The ASaP system is a powerful and flexible tool that enhances image quality and analytical data derived from samples. The system incorporates four functions (plasma cleaning, ion beam etching (IBE), reactive ion etching (RIE) and ion beam sputter coating (IBSC)) within a continuous, oil free vacuum.

Samples up to 25 mm (1") diameter and 25 mm (1") thick can be accommodated. Sample manipulation is assisted by a 5-axis stage and an automated load lock which facilitates rapid sample exchange.

- **Plasma cleaning**
  High frequency (HF) 13.56 MHz capacitively coupled plasma
  Autotuning and matching of RF power supply
  Ion energies of less than 12 eV

- **Ion beam etching**
  Ion source parameters:
  Variable voltage (0.5 to 6.0 kV) continuously adjustable
  Variable current 1 mA to 8 mA continuously adjustable
  Milling angle 0° - 90° adjustable in 1° increments
  360° sample rotation or rocking with 1° increments adjustable from 1° - 179°

- **Reactive ion etching**
  Adjustable RF power, chamber pressure, gas flow rate(s), plate distance and sample cooling
  Autotuning and matching of RF power supply
  Six process gas inlets

- **Ion beam sputter coating**
  Coating thickness uniformity on a 1 cm sample
  Amorphous coating
  Six user-selectable targets
  Target carousel interchangeable through load lock

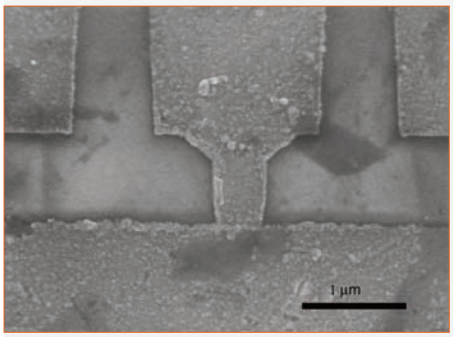
- **Supporting hardware**
  Load lock pump down time <20 seconds
  Auto sample height detection
  Auto sample positioning at individual processing locations
  In situ sample imaging with a microscope and CCD camera (x1000)
  Diagnostic maintenance software

These methods can be used to enhance a sample's surface characteristics, selectively removing material from the sample to either expose grain structures or provide topographical differentiation between layers or structures. IBE and RIBE achieve this by applying energetic ions normal to the sample surface, RIE through a combination of chemical and ionic bombardment created by a reactive plasma. Up to three process gases can be either selected or blended for etching the sample surface, depending on the sample material, for example specific concentrations of CF_4 and oxygen are extremely effective for processing many types of semiconductor samples.

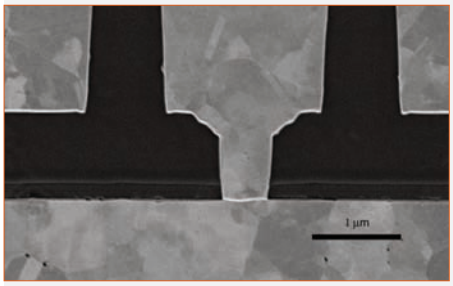
To eliminate the detrimental effects of charging on image quality, this instrument allows the selection of the desired high resolution coating material for a given application, including multiple coatings if required. The rate of deposition and coating thickness are programmable and typical coating materials include tungsten, chromium, platinum, iridium, tantalum and carbon, although others can be used if required.

The ASaP system is fully programmable and automatically adjusts all instrument parameters, producing consistent results for high throughput applications.

B8930 Model 1030 automated sample prep system



Before:
SE (2 kV) image of a mechanically ground (with a 0.1 micron diamond finish) and oxidized cross-section of a Cu-based microelectronic material.



After:
SE/BSE (2 kV) image of a Cu-based microelectronic material following plasma cleaning, ion beam etching, reactive ion etching, and ion beam sputter coating.

Fischione model 1040 NanoMill®

The NanoMill is an excellent tool for preparing the ultra-thin high quality specimens needed for advanced TEM imaging and analysis.

Incorporated in the NanoMill is an inert gas ion source capable of ultra low ion energies of 50 eV.

The concentrated ion beam (2 µm), with scanning capabilities, is perfect for the targeting of specific areas of a sample, allowing the removal of damaged, implanted or amorphised layers without the risk of re-deposition. This makes the NanoMill ideal for post-Focused Ion Beam (FIB) processing, where liquid metal (Ga) ion sources often result in amorphisation and Ga implantation, and these layers can be as much as 10 - 30 nm thick. The NanoMill is ideally suited to removing these damaged layers. The NanoMill is also capable of enhancing the results from conventionally prepared specimens.

A liquid nitrogen cooled cryo-stage is also a feature for samples that are predisposed to heating damage. Furthermore, a rapid specimen exchange solution is incorporated for high throughput applications.

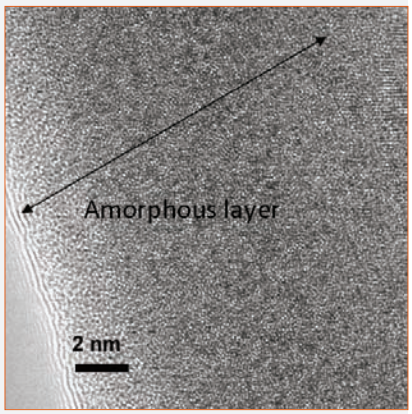
The NanoMill is computer-controlled, fully programmable and easy-to-use, with a contamination-free, dry vacuum system.

Targeting the beam to a specific area of interest is achieved via a secondary electron detector (SED) which creates a real-time image of the specimen aligned with the ion beam, displaying positions and dimensions in microns. The beam can be focused onto an exact point with the cursor or, alternatively, if a larger area is to be thinned, the cursor can be used to draw a box around the selected area.

The impingement angle of the ion beam is precisely programmable from 0° to ±12°. The ion source itself is fixed in position and the specimen stage tilts to achieve the programmed milling angle. A load lock feature allows rapid specimen exchange using a conventional transfer rod, and reduces specimen contamination from ambient conditions.

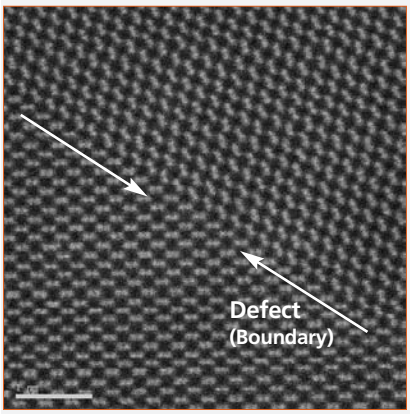
The NanoMill is fully programmable and flexible, requiring minimal user intervention. Conditions such as ion source parameters, milling angle, specimen position, temperature threshold and processing time are programmed via its easy-to-use graphical interface. A sequence of steps can be stored and recalled, creating identical recipes for given materials that result in highly reproducible results. There is selected access to the various controls according to a user's level of expertise, and shortcut keys, utility and maintenance menus speed up programming and routine operation. The NanoMill can also be networked.

B8940 Model 1040 NanoMill

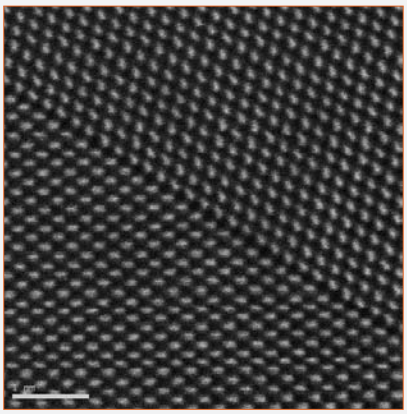


Before:

FIB Damage in Ti_6Al_4V alloy/α-Ti



HAADF

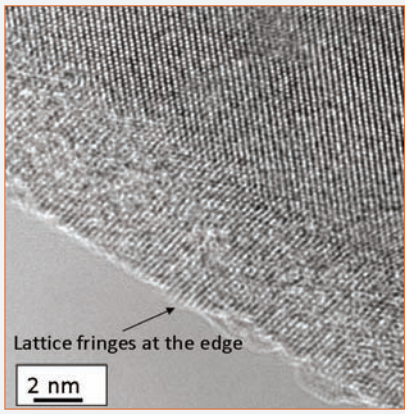


BF

HAADF and BF TEM images of diamond.

A NanoMilling℠ process of 900 eV followed by 500 eV at 150 picoamps.

Courtesy of Rhonda Stroud, Nabil Bassim, and Yoosuf Picard, Naval Research Lab



After:

After a 500 eV Ar NanoMilling℠ process, lattice fringes visible at the edge.

Courtesy of Hamish Fraser, The Ohio State University.

Fischione model 2000 series tomography specimen holders

The model 2000 TEM tomography holder series from Fischione brings TEM into the three dimensional world. This advanced series includes specimen holders that allow high tilt and extended field of view, and is available in single-, dual-, and on-axis versions that are compatible with even the narrowest pole piece geometries.

All Fischione advanced tomography holders come with a dedicated loading station for secure specimen handling, tools to assist in specimen clamping and a Fischione model 9010 vacuum storage container for storing the holder in a clean vacuum environment.

Fischione model 2020 advanced tomography holder

The model 2020 advanced tomography holder is ideal for applications in life and physical sciences, as well as those requiring high specimen tilt while maintaining a large field of view. It allows room temperature data collection over wide tilt and translation ranges, even in restrictive pole piece gap geometries. This holder's streamlined clamping mechanism is far superior to other clamping mechanisms that can limit the specimen size or interfere with viewing. The model 2020 eliminates the typical shadowing associated with most holders at high tilt angles.

This holder accepts specimen grids of standard 3 mm diameter, TEM specimens or FIB lamellae. Specimen thickness can be up to 250 µm.

B8431 Model 2020 advanced tomography holder

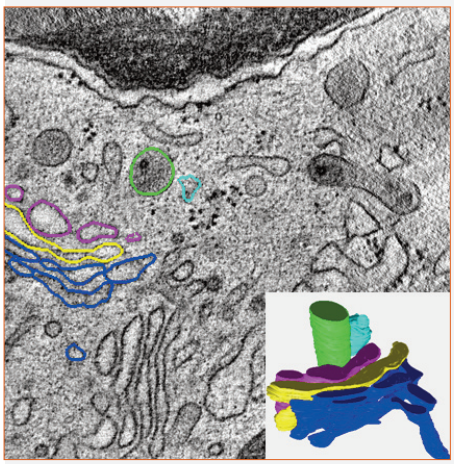


Image courtesy of University of Utrecht.

Please specify make and model of microscope.

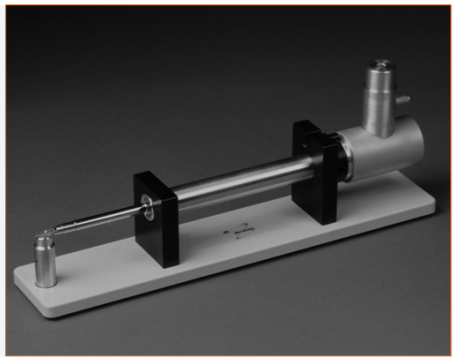
Fischione model 2030 ultra narrow gap tomography holder

The ultra narrow gap tomography holder is suitable for use with microscopes with narrow gap pole piece geometries (<3 mm). This holder accepts 1.5 mm square or round TEM grids, and includes a specimen loading station that allows easy positioning and clamping of the specimen into a cartridge. It is capable of tilting up to 90° while providing a maximised field of view. Optimised specimen protection is also maintained during insertion.

B8432 Model 2030 ultra narrow gap tomography holder

Please specify make and model of microscope.

Fischione model 2040 dual axis tomography holder



The dual axis tomography holder is suitable for room temperature imaging or analysis that requires *in situ* specimen rotation. It maximises specimen visibility, even at high tilt angles, featuring an optimal tilt angle range in narrow gap pole piece geometries (approximately 5 mm). A fully jewelled mechanism provides ultra-precise, in plane specimen rotation, while maintaining eucentricity; the specimen can be fully rotated through 360° to orientate either the grid bars or a specimen feature to the alpha tilt axis. A FlexiClamp spring-type, annular ring securely clamps the specimen into the specimen cup. A dedicated tool facilitates the use of the FlexiClamp.

B8433 Model 2040 dual axis tomography holder

Please specify make and model of microscope.

Fischione model 2050 on axis rotation tomography holder

The revolutionary on axis rotation tomography holder accepts both rod shaped and conically shaped specimens and rotates them fully through 360° about the axis of the holder, providing the maximum achievable amount of data from the specimen. This holder is ideal for specimens prepared by Focused Ion Beam (FIB) and for Atom Probe Field Ion Microscopy (APFIM) specimens.

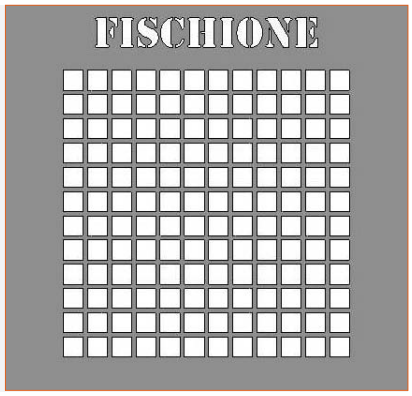
This innovative holder features a cylindrical specimen cartridge into which a sample post is inserted. Sample posts are available with a diameter of either 1.8 mm to accept common APFIM specimen mounts, or 1 mm to accept FIB-prepared specimens. The specimen cartridge precisely fits within the body of the holder and is accurately aligned with the eucentric plane of the microscope. The cartridge is rigidly affixed to a mechanism which both moves along and rotates about the axis of the holder. Initially, the specimen can be rotated through 360° to select the appropriate specimen orientation. A three-position precision indexing mechanism then allows accurate axial rotation of the specimen in order to maximise the amount of data obtained.

Following TEM imaging the specimen cartridge can be removed from the tomography holder and stored securely in the dedicated storage box. The storage box can also be used to transport the specimens safely.

B8434 Model 2050 on axis rotation tomography holder

Please specify make and model of microscope.

Tomography holder grids



Specifically designed for use with Fischione tomography specimen holders, the small size (1.5 x 1.5 mm) of this 300 mesh grid allows increased tilt for tomography use in TEMs with small pole piece gaps. The square shape and identifying mark provide a simple reference when rotating the grid through 90°.

G2396 Tomography grids, 300 mesh, copper. Tube of 50