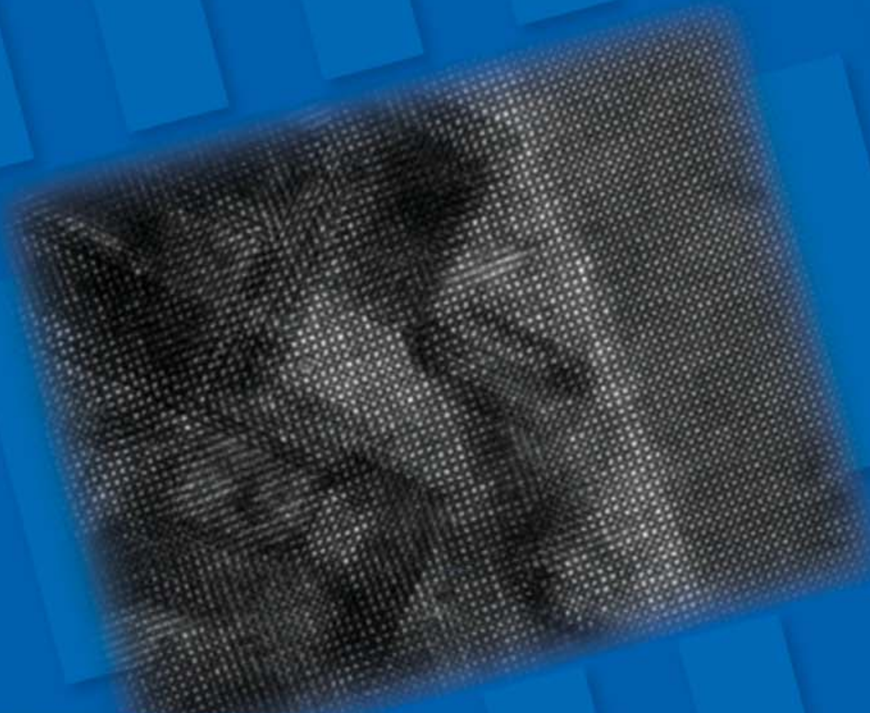


Model 1020
Plasma Cleaner

Effectively cleans specimens for electron microscopy

FISCHIONE INSTRUMENTS

EXCELLENCE...MAGNIFIED

Model 1020 Plasma Cleaner

- ***Simultaneously cleans specimen and specimen holder.***
- ***Cleans highly contaminated specimens in 2 minutes or less.***
- ***No change to the specimen's elemental composition or structural characteristics.***
- ***Oil-free vacuum system.***
- ***Easy-to-use front panel controls.***
- ***Readily accepts side-entry specimen holders for all commercial TEMs and STEMs.***
- ***For SEM as well as other surface science techniques.***
- ***Handy for evacuating specimen holder Vacuum Storage Containers.***

Model 1020 Plasma Cleaner removes organic contamination from physical science specimens and specimen holders.

Electron microscopy requires clean specimens

Clean, well-prepared specimens are imperative for imaging and microanalysis in electron microscopy.

Transmission electron microscopy (TEM) analysis requires that specimens be prepared without altering their microstructure or composition.

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. Unfortunately, as probe size decreases and beam current density increases, 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 typically comes from several sources: inadvertent touching of specimens or specimen holders, backstreaming of oil from an oil diffusion pumped ion milling system, electron microscope column contamination, and adhesives or solvents used in the preparation process. Even when great care is taken to clean the specimen, standard cleaning methods are often not 100% successful.

Model 1020 for final cleaning

The Model 1020 Plasma Cleaner cleans specimens immediately before they are inserted into the electron microscope. Plasma cleaning both 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.

The Model 1020 readily accepts side-entry specimen holders for all commercial TEMs and STEMs and can accept bulk specimens for cleaning before conducting scanning electron microscopy (SEM) or surface analysis.

Plasma cleaning

In a nonequilibrium, high frequency plasma, free electrons are accelerated to high velocities by an oscillating electromagnetic field that excites gas atoms and creates the plasma. The plasma ions impinge upon the surface with energies of less than 12eV, which is below the specimen's sputtering threshold.

Cleaning is solely by reactive gas compounds formed by the plasma chemically reacting with carbonaceous material on the specimen and specimen holder.

To optimize cleaning, a mixture of 25% oxygen and 75% argon is generally recommended. An oxygen plasma is highly effective in removing organic (hydrocarbon) contamination.

The reaction yields H_2O, CO, and CO_2 that are evacuated by the vacuum system.

Clean specimens and holders

A wide variety of materials, prepared by a number of preparation techniques, can be plasma cleaned by the Model 1020 and yield identical results — no contamination of the sample under the electron beam.

The plasma chamber configuration makes it possible to clean specimen holders for any commercially available TEM or STEM. The specimen holder is inserted through a single port into the plasma chamber. The port contains a vacuum-sealing surface compatible with the specimen holder's o-ring.

Ports are available for side entry specimen holders for FEI/Philips, Hitachi, JEOL, Zeiss/LEO, and Topcon TEMs. Ports are easily interchangeable without tools in as little as 10 seconds.

continued

Model 1020 Plasma Cleaner

Clean specimens and holders *(continued)*

For cleaning specimens containing significant amounts of carbon or those mounted onto carbon support grids, shielded specimen holder ports are available that optimize the cleaning action of the plasma.

The large diameter plasma chamber and specially designed holders accommodate bulk specimens for SEM as well as other surface science techniques. Likewise, aperture strips, specimen clamping rings, and SEM specimen holders can also be cleaned.

Typical TEM Specimen Holder Port

Shielded Specimen Holder Port

Multiple Specimen Holder; SEM stubs

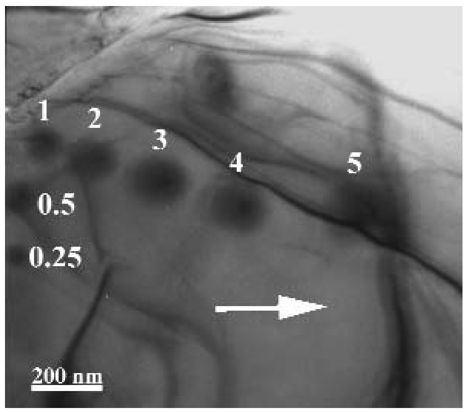
Clamp-Grip Holder for retaining bulk materials

Multiple Specimen Holder for TEM specimens

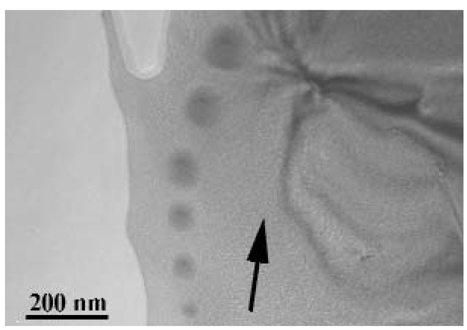
Imaging can proceed with confidence

Plasma cleaning is an essential final step in the preparation of specimens for electron microscopy. Using the Model 1020 Plasma Cleaner ensures confidence that carbonaceous contamination will not interfere with imaging or analysis, even during fine probe microanalysis for extended periods.

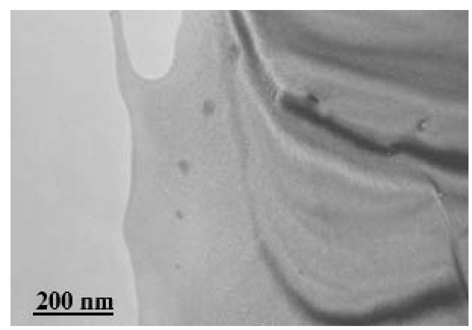
Plasma cleaning results: TEM



TEM image of a stainless steel (grade 304) specimen after one minute of plasma cleaning. The black spots are where a 5nm probe from a Hitachi HF-2000 cFEG-TEM was converged on the specimen before plasma cleaning. The numbers next to the spots correspond to how long (in minutes) the probe was held in that position. After plasma cleaning with a process gas mix of 25% oxygen in argon, the probe was converged on the specimen and held for five minutes at the position indicated by the arrow. No contamination of the specimen is evident.



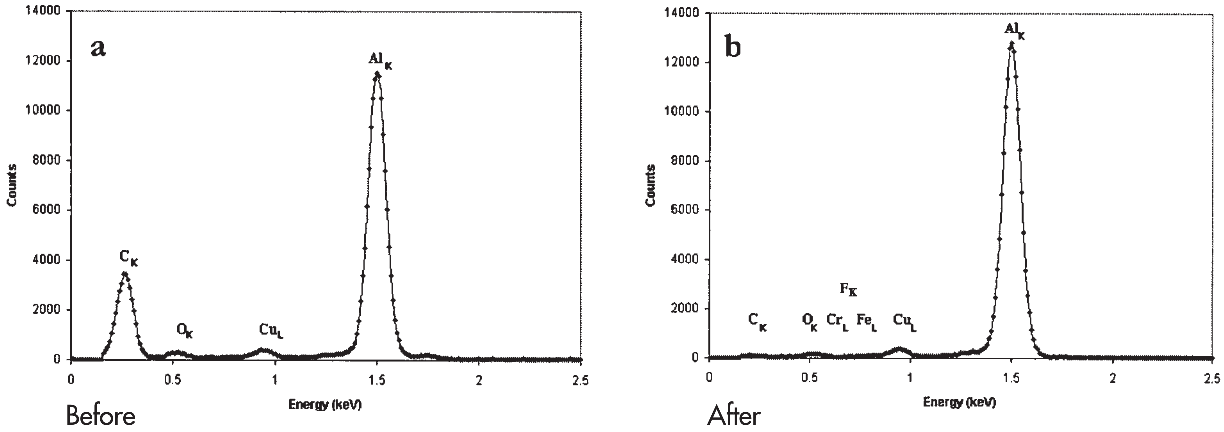
TEM image of a strontium titanate ($SrTiO_3$) bicrystal after 1 minute of plasma cleaning. This was done in the same manner as the stainless steel specimen above. Contamination spots are from fine probe microanalysis performed on the specimen prior to the initial plasma cleaning. Again, note the lack of contamination at the position indicated by the arrow. Also note the hole to the upper left of the image showing an edge of carbonaceous contamination.



TEM image of a strontium titanate ($SrTiO_3$) bicrystal after an additional 4 minutes of plasma cleaning. The contamination spots are greatly diminished or even removed. After 5 minutes of total plasma cleaning, the edge of contamination on the hole has also been removed.

Model 1020 Plasma Cleaner

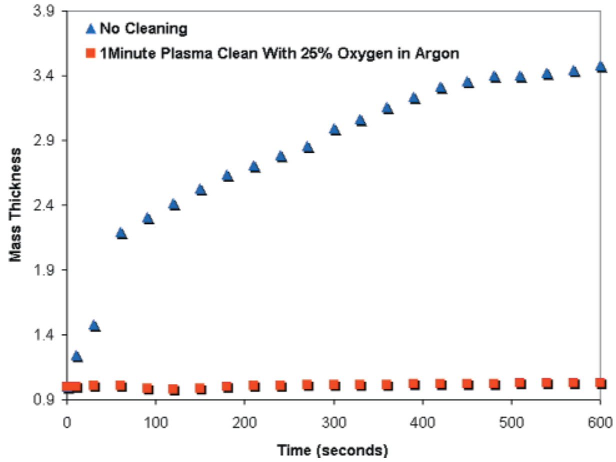
Plasma cleaning results: STEM (EDX)



Energy dispersive x-ray (EDX) spectra of a Cu-Al alloy before and after one minute of plasma cleaning. After plasma cleaning, no carbon is detected by EDX. Furthermore, note the lack of argon or oxygen, as would be detected if the Model 1020 implanted ions into or oxidized the specimen. Likewise, no iron, chromium, nickel, fluorine, or silicon are detected by EDX, indicating that no sputtering of plasma chamber constituents occurred.

Plasma cleaning results: EELS

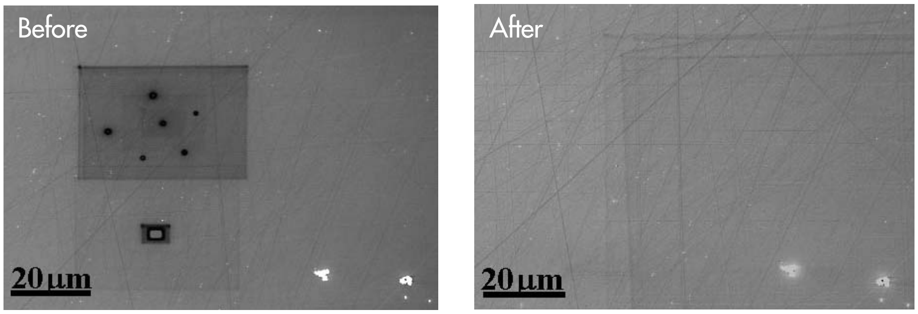
A measure of specimen contamination rate can be analytically determined by electron energy loss spectroscopy (EELS). The mass thickness of a specimen can readily be established as a function of time. If the specimen is contaminating, this thickness will increase as the electron probe is held on the specimen.



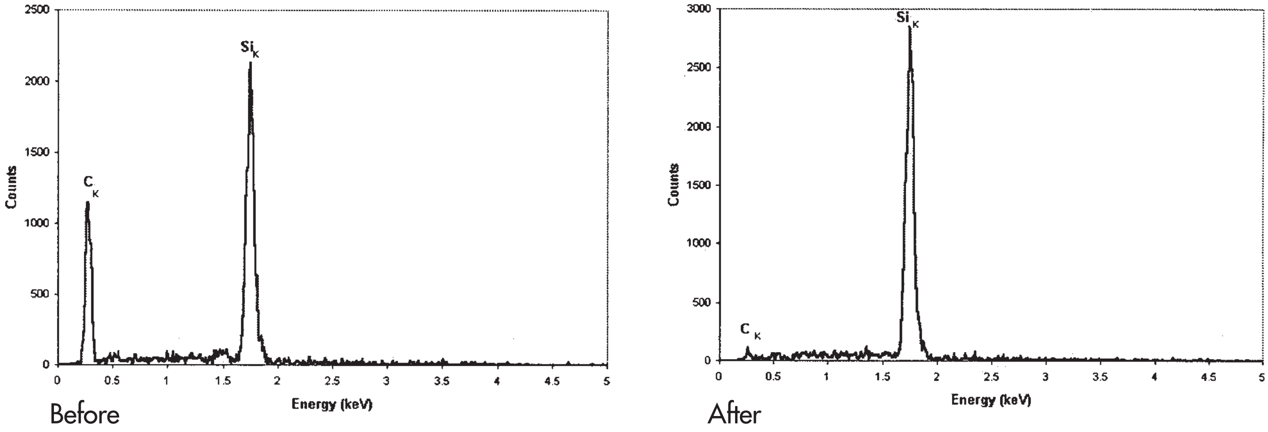
Electron energy loss spectroscopy plotting mass thickness versus time for a Si specimen. Before plasma cleaning, the specimen contaminates quickly, and continues to do so after the probe has been held in place for 10 minutes. After only one minute of plasma cleaning, holding the probe on the specimen for 10 minutes results in no change in the specimen thickness — indicating the absence of specimen contamination.

Plasma cleaning results: SEM (EDX)

The benefits of plasma cleaning are not solely applicable to TEM. By removing the TEM specimen holder port and replacing it with an appropriate holder, bulk specimens can be introduced into the plasma for cleaning. Thus, specimens for surface science and SEM analysis can be cleaned as well as SEM specimen holders, aperture strips, tweezers, specimen clamping rings, and anything else that can be placed into the plasma chamber.

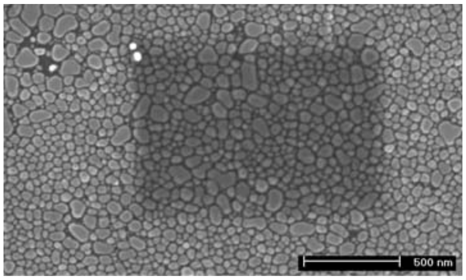


SEM images of silicon acquired in the secondary electron (SE) imaging mode before and after 5 minutes of plasma cleaning. Before cleaning, the image shows black carbonaceous spots that were formed by converging the electron beam in those positions for EDX analysis. The black boxes were formed by rastering the probe at those positions while the specimen was being imaged at a higher magnification. The image of the same specimen region after cleaning shows that all black spots and the black boxes have been eliminated by plasma cleaning and no contamination effects appeared subsequently.



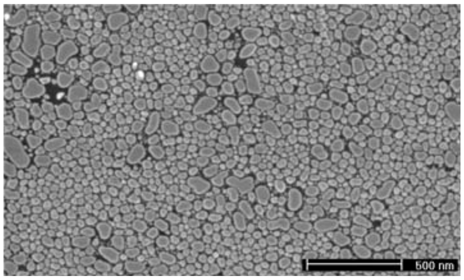
SEM (EDX) spectra of a bulk silicon specimen before and after 2 minutes of plasma cleaning. The SEM was operated in spot mode at an accelerating voltage of 5kV with an accumulated live time of 300 seconds. Before cleaning, the image shows a large carbon peak on the spectrum. After plasma cleaning for two minutes using the gas mixture of 25% oxygen in argon, the carbon peak is greatly reduced.

Model 1020 Plasma Cleaner



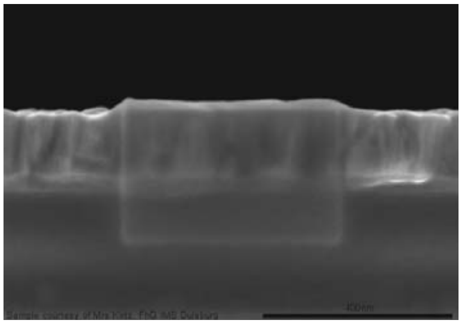
SE image (1kV) of gold on carbon, before plasma cleaning.

Image courtesy of M. Hayles, FEI Company (The Netherlands)



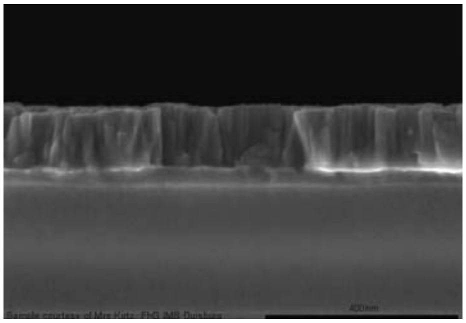
SE image (1kV) of gold on carbon, after 30 seconds of plasma cleaning.

Image courtesy of M. Hayles, FEI Company (The Netherlands)



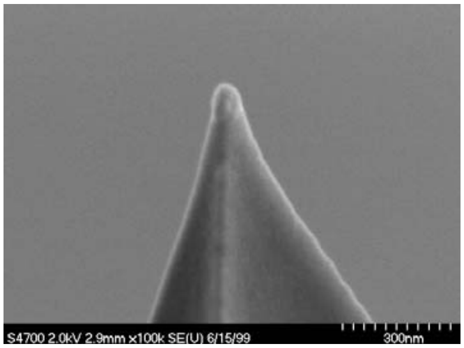
SE image (3kV) of TiN coated Si before plasma cleaning.

Image courtesy of R. Kirtz, FhG IMS (Germany)



SE image (3kV) of TiN coated Si after 30 seconds of plasma cleaning.

Image courtesy of R. Kirtz, FhG IMS (Germany)



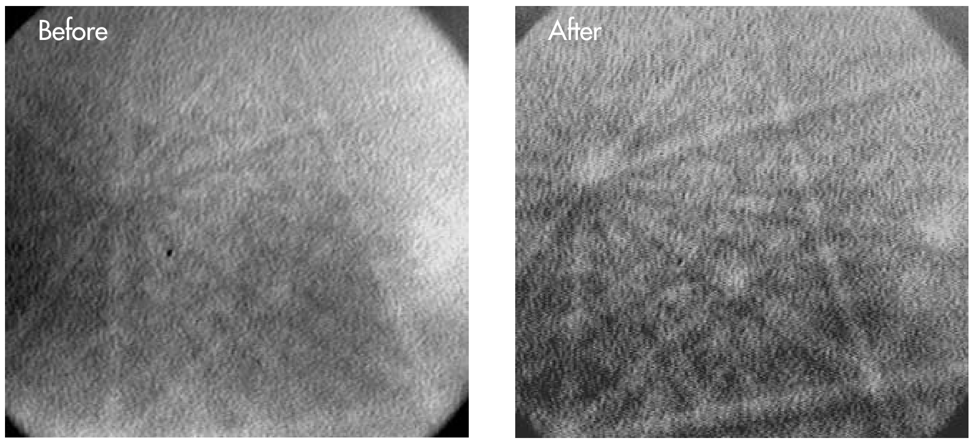
AFM tip following two minutes of plasma cleaning.

Plasma cleaning results: EBSD

Plasma cleaning works well for electron backscatter diffraction (EBSD) analysis, which is a highly surface-sensitive technique.

The first requirement is a properly prepared surface. Subsurface damage caused by mechanical or chemical polishing has to be removed, which is easily achievable with the Fischione Model 1010 Ion Mill. The Model 1010 accelerates argon ions toward the specimen surface. Through momentum transfer, material is sputtered from the specimen, resulting in the removal of surface artifacts.

The final treatment by plasma cleaning removes contaminants from the ion milled surface.



EBSD before and after two minutes of plasma cleaning on a silicon specimen.

Plasma chamber

The plasma is created in a cylindrical chamber made of quartz and stainless steel. Sophisticated gas dynamics ensure that the plasma is evenly distributed within the chamber to clean the specimen with negligible heating. A high frequency (HF) antenna, located outside the chamber, couples the oscillating field to the chamber. No components are located within the chamber other than the specimen and specimen holder that are being cleaned.

Vacuum system

An oil-free vacuum system is essential to prevent contamination and ensure optimal processing. The vacuum system for the Model 1020 consists of a turbomolecular drag pump and a multi-stage diaphragm pump, an ideal combination for establishing suitable vacuum characteristics to activate and sustain the plasma. Rapid chamber pump-down times allow plasma cleaning to begin almost immediately after a specimen is inserted.

The Model 1020 can also be used to evacuate individual specimen holder Vacuum Storage Containers and cyrotransfer TEM specimen holder dewars.

A port plug seals the chamber under vacuum when the instrument is not in use.

Model 1020 Plasma Cleaner

Power supply

The high-frequency (13.56 MHz) oscillating power supply can initiate and sustain a low-energy, inductively-coupled plasma. A matching network ensures effective coupling of the high-frequency field to the plasma chamber and ensures compatibility with specimen holders produced for diverse TEMs.

Effective shielding prevents the emission of high frequency interference, and fully complies with FCC guidelines.

Front panel controls and display

The control panel is especially easy to use. Dedicated keys control the vacuum pumping and venting sequence. LEDs indicate when the system is venting, at atmospheric pressure, pumping, and has achieved a high vacuum condition. A numeric touchpad is used to enter the plasma processing time.

Once the control system determines that appropriate conditions have been established for effective processing, the plasma is automatically activated. A digital display continuously indicates the remaining processing time. Cleaning ends automatically when the specified time has elapsed. A visual indicator notifies the user that plasma cleaning is complete and the specimen is ready for imaging and analysis.

Model 1020 Plasma Cleaner control panel

Front access to services

A service panel located on the front of the instrument's enclosure provides access to a potentiometer that controls the gas flow metering valve, the HF power control, and function switches used for diagnostic purposes. A digital display indicates the vacuum level and an hour meter tracks the vacuum system's total operating time so that routine maintenance can be scheduled accordingly.

Model 1020 Plasma Cleaner Accessories

Model 9010 Vacuum Storage Container

Model 9010 Vacuum Storage Container. After cleaning, specimen holders can be inserted into Model 9010 Vacuum Storage Containers so they can be stored or transported in a vacuum. A sight glass gives a clear view of the specimen area of the specimen holder.

Model 9020 Vacuum Pumping Station

Model 9020 Vacuum Pumping Station installed on the Plasma Cleaner. The Pumping Station simultaneously stores five specimen holders under vacuum. It includes a heavy-duty metal base with non-skid feet, five independently valved Vacuum Storage Containers, a vacuum pumping manifold, and all necessary components for connecting to the Model 1020 Plasma Cleaner.

Model 1020 specifications

Plasma system	High frequency (13.56 MHz) oscillating field system coupled to a quartz and stainless steel plasma chamber
	Ion energies less than 12eV
	Compatible with TEM specimen holders for FEI/Philips, Hitachi, JEOL, Zeiss/LEO, and Topcon
Vacuum system	Oil-free turbomolecular drag pump backed by a multi-stage diaphragm pump
	Ultimate vacuum of 1×10^{-7} mbar
Gas	25% oxygen and 75% argon
	Nominal 10 psi (200 kPa) delivery pressure
	Flow rate is factory set and can be adjusted via a potentiometer located on the service panel
Dimensions	23" (584mm) W x 20.5" (521mm) H x 22.3" (566mm) D
Weight	133.5 lb (60.6 kg)
User interface	Single control panel to initiate vacuum, plasma, and timer
	Process timer for automatic termination
	Service panel is easily accessible
Power requirements	100/120/220/240VAC, 50/60Hz, 660 watts
Warranty	Two year

U.S. Patent Number 5,633,502, Dated May 27, 1997
U.S. Patent Number 5,510,624, Dated April 23, 1996 (licensed from the Argonne National Laboratory)

Cover image: ADF-STEM (Gaussian smoothed) image of $LaTiO_3$ multilayers in $SrTiO_3$. Atomic columns of La, Sr, and Ti can be seen.

Image courtesy of D.A. Muller, J. Grazul, A. Ohtomo, H. Hwang, Lucent Technologies (U.S.A.)

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Part No. 011-0549, Rev. 1