Electron Microscopy

MODEL

PC2000

Plasma Cleaner

For removing hydrocarbon contamination from specimens and specimen holders prior to electron microscope analysis.

SBT

SOUTH BAY TECHNOLOGY, INC.

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It has been well documented that low energy plasmas can be used to reactively etch or remove organic materials found on the surface of inorganic materials. This technology has been used by the industrial community to clean semiconductor wafers and optical materials for many years. A related technique is now being implemented in the field of electron microscopy, where specimens can become contaminated during the preparation process or from other sources. Current analytical instruments use tightly focused, intense beams that create carbon deposits on the specimen surface due to organic contamination. The PC2000 is designed to simultaneously clean the specimen and specimen stage, which minimizes, and in many cases, eliminates contamination of the specimen being analyzed. The specimen holder and specimen are subjected to reactive gas plasma prior to electron microscope analysis.

Procedure

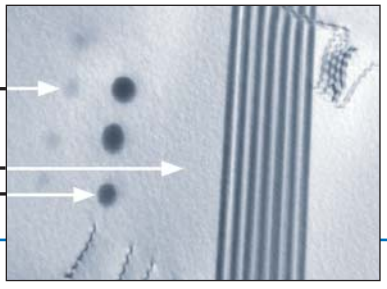
Plasma cleaning with the Model PC2000 involves subjecting the specimen and/or specimen stage to a reactive gas plasma that efficiently removes organic contamination from the surfaces exposed in the plasma. The procedure may be carried out prior to inserting the specimen and specimen stage into the EM by either mounting the specimen holder in one of the supplied ports or by inserting the entire sample or sample assembly through the hinged upper port and into the 8"OD x 4" high chamber. The system can be used with any side entry TEM holders, top entry TEM holders, or with any sample that will fit inside the chamber. For most applications nominally pure argon followed by nominally pure oxygen is used for plasma cleaning. However, the PC2000 allows a variety of gas species to be used, allowing the user to tailor specific reactions for a given specimen material. Cleaning times are typically a matter of minutes, which generally allows specimens to be studied for several hours.

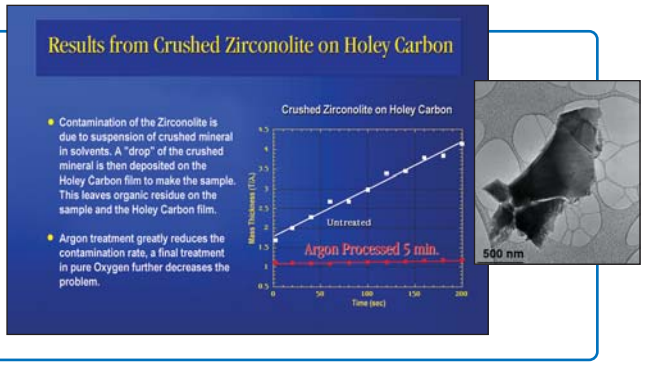
Control Panel

The PC2000 control panel allows the user to control and monitor every aspect of the plasma cleaning process directly from the front panel. The control panel integrates display and control of critical process related data with a straightforward and intuitive interface. Independent front panel controls for 2 gas inlets, forward power, tuning, RF Power and process time along with a DC bias display provide complete control over the entire process. While the RF, gas and vacuum controls are fully interlocked for user and lab safety, the system parameters can be easily changed from run to run or during a process run to optimize the desired effect. While complete control over every aspect of the process can be changed on the front panel, repetitive process runs can be easily duplicated using a simple 3 button sequence.

While the PC2000 offers all of the flexibility and functionality required in a research instrument, it does so with a thoughtfully designed interface that makes the system ideal for multi-user environments.

TEM micrograph of 304 stainless steel specimen





Data courtesy of Dr. Nestor J. Zaluzec, Argonne National Laboratory

Special Features

- Suitable for all side entry and top entry TEM holders. Large chamber also accommodates specimens up to 7" diameter and 3" high for cleaning tweezers, specimen mounts, etc.
- Digital LCD displays, indicate forward power in watts, reflected power, DC bias and vacuum level.
- Compatible with Argon, Oxygen, CF_4, and other gas mixtures.
- System comes standard with 3 ports which allow simultaneous cleaning of multiple holders or insertion of analytical tools into the plasma chamber.
- Power level can be optimized for each specimen type and gas species to maximize cleaning rate without risk of etching the specimen stage or the chamber[2].
- Viewport allows easy monitoring of the process.
- Safety interlocked controls simplify operation – ideal for multi-user environments.

Specimen Storage and Transfer

Once the samples have been cleaned, they can be inserted into a Vacuum Storage Container for transport to the microscope. The specimen holders can also be stored in these containers for an indefinite period preventing further contamination. The vacuum storage containers can also be mounted on the Vacuum Pumping Station manifold for evacuating multiple holders. Once evacuated, the containers can be maintained under vacuum or backfilled with dry nitrogen or other suitable gas while mounted on the Vacuum Pumping Station.

Plasma Trimming™

Plasma Trimming™ is a technique by which material is removed from a TEM specimen by use of a moderate energy Ar plasma discharge. By using an Ar gas plasma, the sample can be thinned in a very slow and controlled manner. This allows further thinning of specimens that have been previously ion or FIB milled to remove surface damage, ion milling defects or surface artifacts[5].

Vacuum Pump

The PC2000 uses a corrosive series 2-stage rotary vane pump charged with an inert vacuum fluid that is safe for use with oxygen, resistant to attack by corrosive gas species and has a low vapor pressure. Studies have shown that the proper use of a rotary pump combined with an inert vacuum fluid provides a safe and contamination free environment for plasma cleaning[1].

1. ***Surface Science Aspects of Contamination in TEM Sample Preparation,***
   J.T. Grant, S.D. Walck, F.J. Scheltons, A.A.Voevodin,
   Materials Research Society Vol. 480 1997.

2. ***Application of Reactive Gas Plasma Cleaning in Mitigating Contamination of Specimens During Transmission and Analytic Electron Microscopy,***
   Shane P. Roberts, Scott D. Walck, John T. Grant, Nestor J. Zaluzec,
   Materials Research Society Vol. 480 1997.

3. ***Reactive Gas Plasma Specimen Processing for use in Microanalysis and Imaging in Analytical Electron Microscopy,***
   Nestor J. Zaluzec, Bernard J. Kestel, David Henriks,
   Microscopy & Microanalysis 1997.

4. ***Simultaneous Specimen and Stage Cleaning for Analytical Electron Microscopy,***
   David Henriks, *Microscopy Today* issue 96-8 October 1996 pgs. 16-17.

5. ***In-Situ Transformation of a Zinc TEM Lift-Out Specimen***
   B.I. Prenitzer*, S. Collins+, and L.A. Giannuzzi*
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   Mechanical Materials & Aerospace Engineering
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PC2000 is shown with hinged upper lid open and 3 specimen stages inserted. Top entry stages and small parts can be inserted in the chamber through this hinged upper lid.

Vacuum Pumping Station

Vacuum Storage Container for specimen stage storage and transfer.

Built under license from Argonne National Laboratory
Inventor, Nestor J. Zaluzec
Pursuant to U.S. Patent No. 5,510,624

Control Panel:

Readouts:	Digital LCD displays for forward power in watts
Forward Power Control:	1-150 watts, adjustable
Reflected Power Control:	Manually adjustable,180 degree potentiometer
DC Bias:	Digital LCD display
Electronics:	Solid state, crystal controlled, 100kHz switchmode DC power converter to RF generator
Termination:	Digital interlocked timer or manual termination
Interlocks:	RF, gas and vacuum fully interlocked for user & lab safety

Vacuum System:

Mechanical Pump:	Corrosive series 2-stage rotary vane pump
Base Pressure:	$2x10^{-2}$ torr
Time to Reach Base Pressure:	30 seconds
Operating Pressure:	User variable (200 millitorr typical)
Pump Access:	Fully accessible,remote mount, KF-25 connection, all hoses & fittings provided
Activation:	Front panel LED "ON" indicator
Vacuum Sensor/Readout:	Capacitance manometer and digital LCD readout
Gas Delivery and Control:	Dual independent needle valves with safety interlock solenoid
System Vacuum Vent:	Independent solenoid interlocked with connection for dry nitrogen

Chamber:

Size:	8"OD x 4" high stainless steel electropolished cylinder with viewport
Electrode:	Immersed stainless steel
Specimen Interface:	"No Tools" quick connect for all side and top entry TEM holders Top entry holders mounted through hinged top port

System Specifications:

System Weight:	Less than 60 lbs. (without vacuum pump)
Dimension:	22"W x 14"D x 18"H
Power Requirements:	110/220 VAC single phase, 5 amps (at 120VAC)
Cooling:	Water cooled
RF:	13.56 MHz frequency, 150 watts forward power
WARRANTY:	**[2] Year Limited Warranty**

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DESIGNED & MANUFACTURED IN THE UNITED STATES

Cover image courtesy of Dr. Christian Kisielowski,
Lawrence Berkeley National Laboratory
REF: C. Kisielowski et al., Ultramicroscopy 89 (2001) 243.
Dumbbells in silicon [110] prepared by low energy (200v) ion milling.