

# MODEL 1010 ION MILL Standard Magnification Version INSTRUCTION MANUAL

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# Contents

SAFETY	1
BACKGROUND Ion Milling Theory Model 1010 Features Ion Sources Basic Procedure for Sample Preparation and Ion Milling	2 2 2 3 3
SYSTEM OVERVIEW Sample Chamber Vacuum System Gas Flow Water Flow Sample Cooling Capability (Optional) Endpoint Detection Capability (Optional) Visual Monitoring Control System and Software Specifications	4 5 6 7 7 7 7 8 8 8
INSTALLATION AND SETUP  Warnings, Cautions, and Notes Assessing initial condition Unpacking and Inspecting Accessories Connections Process gas Exhaust gas Water Microscope Installation Process Control Computer Laser Installation (optional) Power Connections For US application For non-US application	9 9 9 10 11 11 12 12 12 13 14 14 14
To change operating voltage  OPERATION  Basic Startup Procedure  Dewar Bakeout  Laser Calibration  Loading TEM Specimens  Specimen Cooling (optional)  Ion Milling  Insert specimen  Microscope  Endpoint determination (optional)  Set milling parameters  Set data logging options  Save recipe  Start milling  View Data	15 16 16 17 18 19 20 21 22 22 22 22 24 24 25 27
Basic Shutdown Procedure  MAINTENANCE	29 30



Preventive Maintenance Schedule	30
Cleaning the Air Filters	30
Changing the Fuses	31
Opening the Enclosure	32
Enclosure removal	32
Window Cleaning	33
Front access door window	33
Bottom light window	34
Cleaning o-rings	36
Lamp Replacement	36
Ion Source Maintenance	38
Disconnect gas line	39
Remove ion source cartridge	39
Remove cathode	41
Remove anode and extractor	41
Inspect components	42
Clean components	43
Service ion source body components	43
Ultrasonically clean and inspect components	45
Reassemble ion sources	45
Verify electrical connections	45
Replace ion source	46
Attach gas and electrical connections	46
Check leaks	46 47
Start up ion source Ion Source Alignment	47
Adjust source alignment	47
Verify alignment	48
Dewar Maintenance	48
Remove Dewar	48
Change Zeolite® desiccant	51
Install Dewar	52
Evacuate Dewar wall space	52
Chamber and Specimen Stage Cleaning	52
Stage maintenance	52
Ion source flange	56
Pirani gauge tube	58
Bottom plate	59
Mass Flow Controllers	60
Verify electronics	60
Evaluate flow	61
Replace mass flow controller	61
Leak Detection	62
High pressure	62
Vacuum	63
Turbomolecular Vacuum Pump	64
Reinstall Enclosure	64
Back Panel	64
TROUBLESHOOTING	65
Diagnosis chart	65
Error messages	67
Laser	68
SPARE PARTS AND CONSUMABLES	69
	09
REFERENCES	71





# Safety

The following hazards are associated with the Model 1010 Ion Mill.

**ELECTRICAL HAZARD.** High voltages can cause severe injury or death. Do not connect the power cord until after the initial inspection and after making gas connections. Do not attempt to operate the instrument with the cover removed.

Before disconnecting the power cord from the instrument, wait one minute to ensure that any charged electronic component will discharge.

**HAZARDOUS MATERIALS.** Toxic, reactive, or radioactive materials can cause severe injury or death. If hazardous materials will be inserted into the Model 1010 for processing, the ion mill must be connected directly to an exhaust system capable of accommodating the hazardous material. If radioactive materials will be milled, the instrument must be enclosed in protective shielding.

**LASER HAZARD.** Laser beams can cause severe eye damage. Do not point the laser beam into your eye or into any other person's eye. The ion mill contains a 670 nm red (visible) Class II laser.

**COMPRESSED GAS.** High-pressure gas stream can injure exposed skin or eyes. Point tubing away from personnel while connecting or disconnecting gas cylinders.

**MECHANICAL HAZARD.** Moving parts can injure fingers and hands. Do not attempt to operate the instrument with the cover open.



# Background

For many of today's advanced materials, ion milling is an excellent preparation technique for specimens that require TEM analysis. Electron microscopy needs samples that are clean, representative of the bulk state, and free of physical or chemical artifacts. To prepare them for microscopy, samples are first mechanically sectioned and ground and then polished or milled to remove additional material.

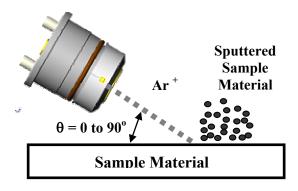
**Electropolishing** requires that a sample material possess a certain level of electrical conductivity.

**Ion milling** is effective for all materials, including those that are relatively nonconductive such as semiconductors and insulators as well as those that are conductive (metallic or semimetallic). Ion mills bombard the substrate surface with an energetic ion beam.

E.A. Fischione's Model 1010 Ion Mill is a state-of-the-art, compact, tabletop precision ion milling/polishing system that consistently produces high-quality TEM specimens with large electron transparent areas.

## Ion Milling Theory

Ion milling uses an impinging incident HAD (Hollow Anode Discharge) ion to remove ("sputter") ions from the surface of a sample.



If an inert gas (for example, Ar or Ar+) is used for milling, sputtering is the result of momentum transfer between the incident ion and the sample surface. If a reactive gas is used, sputtering is enhanced by chemical reactivity.

#### Model 1010 Features

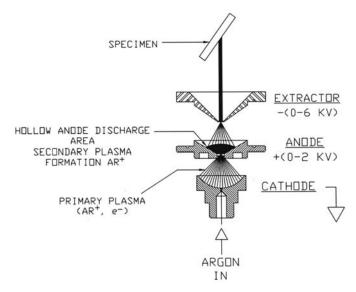
The Model 1010 is fully programmable, with features including two independently adjustable Hollow Anode Discharge (HAD) ion sources which permit either rapid milling or more gradual specimen polishing, automatic gas control, an oil-free vacuum system, a milling angle range of 0 to 45°, specimen rotation or rocking, an optional liquid nitrogen cooled specimen stage and optional automatic termination.



The choice of single or dual ion source operation allows milling from either one or both sides of the specimen. When using the Model 1010, total ion milling time is typically less than two hours depending on the specimen material and its initial thickness.

#### Ion Sources

The Hollow Anode Discharge (HAD) ion sources operate over user-selectable ranges of extractor voltage (0.5 kV to 6.0 kV) and current (3 mA to 8 mA), and are capable of producing ion beam currents up to 400 microamps. To a first approximation, varying voltage varies the average ion energy, while varying current varies the ion flux. The milling rate for a given material generally increases as either parameter is increased. They are fabricated from a combination of aluminum, brass, ceramics, polymers, and stainless steel.



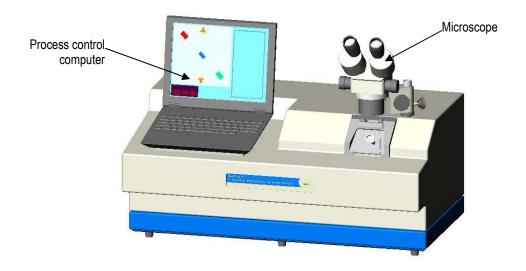
Hollow Anode Discharge (HAD) ion source.

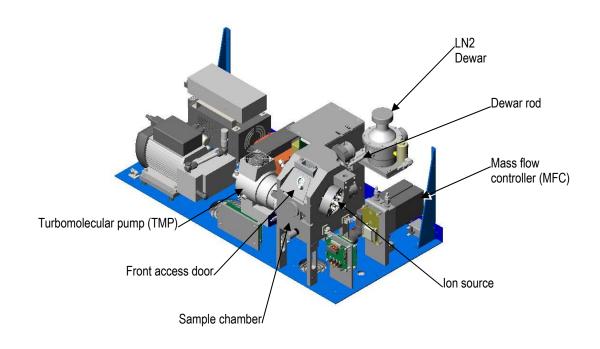
# **Basic Procedure for Sample Preparation and Ion Milling**

- 1. Mechanically section (cleave, saw) the sample.
- 2. Ultrasonically cut disks (3 mm diameter by <500 μm thick).
- 3. Rough grind (3 mm diameter by <75 µm thick).
- 4. Dimple grind (3 mm diameter by <10 μm center thickness).
- 5. Ion mill the top and bottom surfaces until they are electron transparent (3 mm diameter by <100 nm thick).
- 6. Plasma clean.

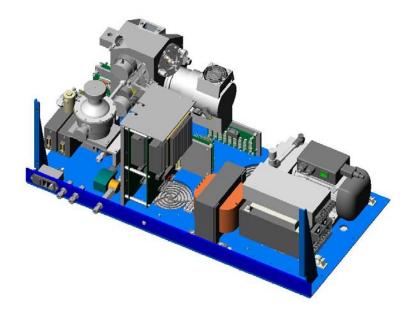


# System Overview



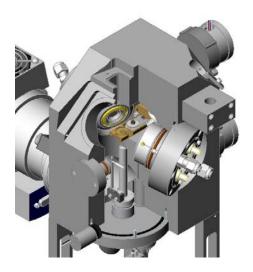






## Sample Chamber

The sample chamber is fabricated from a single block of aluminum. The ion sources are connected to the chamber via mounting and alignment flanges. Radial o-ring seals ensure the vacuum integrity between the ion sources and the chamber.



**Bottom:** A vacuum flange mounted on the chamber bottom contains:

- The Pirani gauge for sensing vacuum.
- The bottom lamp assembly for observing the specimen using transmitted light.
- The detector for milling termination.

**Rear:** The rear of the chamber includes:

- The specimen stage rotator assembly.
- A series of electrical connections.



- The specimen rotation drive motor.
- The angle adjustment drive motor.
- The vacuum pumping port for the liquid nitrogen Dewar flask.
- The optical pick-up and encoder, which provides the milling angle indication.

**Top:** The top chamber surface includes:

• The top light assembly

**Front:** The front of the chamber includes:

- The electro-mechanical leak valve
- The chamber access door

## **Vacuum System**

The Model 1010 is equipped with a 70 lps (minimum) turbomolecular vacuum pump. A fan creates a constant air flow across the turbo pump cooling vanes to minimize the heat associated with maintaining a vacuum under gas flow conditions. The Model 1010 also includes an external rough pump. This is a diaphragm pump that provides backing vacuum for the turbomolecular pump.

With no gas flow, ultimate chamber vacuum is  $1 \times 10^{-6}$  torr. Under normal milling conditions, the system vacuum is between  $1 \times 10^{-4}$  and  $2 \times 10^{-5}$  torr. Total pump down time is typically less than four minutes, although this time may vary based on ambient temperature and relative humidity as well as the length of time that the chamber front access door was open exposing the chamber to ambient conditions. Pump down times are also increased following ion source cleaning or chamber maintenance.

The turbomolecular pump is directly mounted to the chamber by two bolts. An inlet protection screen prevents debris from entering the pump and damaging the turbine blades.

An electro-mechanical vent valve is installed directly onto the chamber and all venting is controlled through the computer. Vacuum is sensed by a Pirani gauge and is continuously indicated in the Ion Milling Program screen. In order to improve ion source performance, argon flows through the ion sources during the venting process.

The specimen chamber vents through the normally closed solenoid valve located on the front of the chamber. When the vacuum system is de-energized, both the diaphragm and the turbomolecular pumps are turned off and voltage is applied to the vent valve. The vent valve slowly bleeds ambient air into the chamber over approximately 20 seconds to minimize stress on the rotary turbine blades in the turbo pump.

When liquid nitrogen is being used, venting will not occur until the specimen stage temperature reaches +10°C, to prevent contamination of the specimen.

If the main instrument power switch is turned off while the vacuum system is energized, the vacuum will be maintained, keeping the chamber sealed and free from ambient contamination.



#### Gas Flow

The Model 1010 contains an internal, automatic gas control system. Process gas (usually argon) is connected to the bulkhead fitting located on the rear of the instrument. The purity of argon should be equal to or greater than 99.998% and supplied at a pressure of 10 psi (69 Mpa).

Two independently controlled mass flow controllers, located in a feedback control loop, supply the appropriate amount of argon flow to the ion sources (between 0.4 sccm and 1.0 sccm). An internal pressure regulator is pre-set to provide the proper supply pressure to the mass flow controllers.

The computer continuously monitors ion source performance and provides the appropriate control signal to the flow controllers. The controllers are supplied with an inlet voltage of +15 vdc and the control signal is 0-5 vdc proportional.

#### **Water Flow**

The high ion energies associated with the HAD (Hollow Anode Discharge) ion source benefit from water cooling which both enhances performance and increases the time interval between routine source maintenance. Cooling water circulates through the alignment flange, which contains and is in thermal contact with the ion source cartridge.

Water enters the instrument though a bulkhead fitting on the rear panel. A solenoid valve located within the instrument enclosure is normally closed. When the ion sources are energized, the solenoid valve opens allowing water flow into the alignment flanges, thus cooling the ion sources. A solenoid valve conserves water by only permitting flow when required to cool an energized ion source.

The temperature of the inlet water is not critical but should be maintained between +5°C and room temperature. Water flow of approximately 0.5 lpm is recommended. In the unlikely event of the drain line becoming obstructed, the Model 1010 will not sense the loss of flow. Therefore, periodically during operation, confirm that water is being drained from the instrument.

# Sample Cooling Capability (Optional)

A sample can be milled in the Model 1010 while actively being cooled to temperatures approaching that of liquid nitrogen. The source of this cooling capability is an insulated vessel (or "Dewar") containing liquid nitrogen. A thermal path is provided by a conduction rod passing through the Dewar to the rear of the sample stage. The maximum temperature allowed during milling is selectable. It is selectable through the "Thermal Safeguard" in the Ion Milling program.

To optimize the sample cooling capability, a vacuum must be maintained in the space between the walls of the Dewar. Periodically the sample stage heater may need to be activated to remove absorbed moisture and regenerate the Zeolite® desiccant. These processes are controlled by the Dewar Bakeout program.

# **Endpoint Detection Capability (Optional)**

The Model 1010 can be programmed to automatically de-energize the ion sources when either a perforation in the sample is achieved or when the sample permits the transmission of laser light. The detector circuitry is designed with a variable sensitivity



range. The scale is 1 to 10, with 1 being the most sensitive. These values are selectable in the Ion Milling Program.

Note: Termination thresholds vary greatly based upon material and the desired perforation size. Experimentation with varying parameters is necessary to obtain the optimum termination point for different types of materials.

## **Visual Monitoring**

The progress of an ion milling operation is monitored visually by using the standard magnification stereo microscope. The magnification range with 10X eyepieces is 7 – 45X.

## **Control System and Software**

All operations are controlled using four independent software programs.

Ion Milling Program	Controls the milling operation. Allows the user to select milling parameters and create the conditions necessary for manual or automatic operations.
Dewar Bakeout Program	Controls the heating process for maintaining the Dewar insulation and Zeolite® desiccant. Opens the vacuum connection, raises the temperature, and controls the time at temperature
Laser Detector Calibration Program	Calibrates the laser detector according to the transmissive properties of the window protecting the laser sensor. Allows the user to choose the minimum and maximum sensitivity values.
Data Logging Program	Controls logging, retrieval, and display of milling parameters.

# **Specifications**

Ion source type	Hollow Anode Design		
Ion source settings range	Voltage: 0.5 – 6 kV, Current 3 – 8 mA		
Specimen motion	Full rotation (360°) or oscillation (0 to +/- 179°)		
Milling rate	Varies with material		
Control platform	PC compatible		
Dimensions 33 in. wide by 19.5 in. deep by 26.5 in. high			
Weight	115 lb (52 kg)		
Power requirements	60 Watts		
Water requirements	Temperature of the inlet water is not critical but should be maintained between +5°C and room temperature. Water flow should be approximately 0.5 lpm.		
Gas requirements	Argon equal to or greater than 99.998% and supplied at a pressure of 10 psi (69 MPa)		
Microscope capability 7-45X stereo zoom microscope			
Turbomolecular vacuum pump 70 lps (minimum). With no gas flow, ultimate chamber vacuum is 1 x 10 Under normal milling conditions, the system vacuum is between 1 x 10-4 x 10-5 torr			



# Installation and Setup



## Warnings, Cautions, and Notes

WARNING! LIFTING HAZARD. Do not try to lift the Model 1010 from the box

with only one person. The ion mill weighs about 115 pounds (52 kg).

WARNING! ELECTRICAL HAZARD. High voltages can cause severe injury or

death. Do not connect the power cord until after making gas and water connections and after installing the microscope and computer.

Before disconnecting the power cord from the instrument, turn off the instrument. Wait one minute to ensure that any charged

electronic component will discharge.

WARNING! COMPRESSED GAS HAZARD. High-pressure gas stream can

injure exposed skin or eyes. Point tubing away from personnel while

connecting or disconnecting gas cylinders.

CAUTION: Do not attempt to open the instrument enclosure while the

microscope or laser is installed. Remove the microscope, laser, and post assembly before initiating service. Opening the Model 1010 while these components are installed could damage the microscope

and the enclosure.

**CAUTION:** Do not energize the ion sources unless Argon gas is supplied.

CAUTION: Do not energize the ion sources without a specimen in the specimen

holder. Failure to follow this caution will result in the ion beams

milling the outer components of the opposing ion source.

The Model 1010 is recommended for operation in an ambient temperature range of 12°C to 40°C, and a relative humidity range of 10% to 70%.

The ion mill requires about 1 square meter of bench space.

# **Assessing initial condition**

- 1. If the shipment has been received in good condition, proceed with unpacking.
- 2. If the shipment has been received with obvious damage, contact E.A. Fischione Instruments before proceeding.

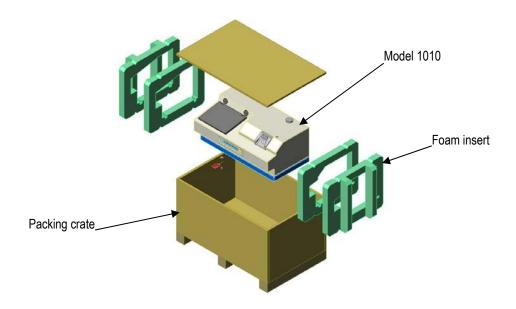
# **Unpacking and Inspecting**

- 1. Remove the top of the crate.
- 2. Remove the accessory carton.

WARNING! LIFTING HAZARD. The ion mill weighs about 115 pounds. Use two people to lift the ion mill.

3. Use two people to lift the Model 1010 from the base plate.





- 4. Place the Model 1010 on a firm surface where it can be accessed from front and back.
- 5. Tilt the Model 1010 and remove the foam inserts.
- Remove the Model 1010 from the plastic shipping bag.
   Note: Save the shipping crate and packing materials in case the Model 1010 needs to be returned to the factory for repair or maintenance.
- 7. Examine the outside of the Model 1010 for visible damage.
- 8. If there is evidence of damage, contact both the freight carrier and E.A. Fischione Instruments before proceeding.
- 9. Place the Model 1010 on the surface where it will be operated.

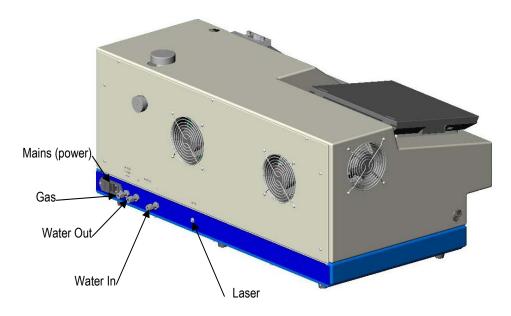
#### **Accessories**

- 1. Open the box containing the accessories. This box includes:
  - Microscope, microscope and spacer
  - Specimen loading station and accessories
  - Tool set
  - Optional laser, Dewar cap, and / or exhaust kit if ordered
- 2. Inspect all components in this box for signs of damage.
- 3. If there is evidence of damage, contact both the freight carrier and E.A. Fischione Instruments before proceeding.



## **Connections**

WARNING! ELECTRICAL HAZARD. High voltages can cause severe injury or death. Do not connect the power cord until after making gas and water connections and after installing the microscope and computer.



#### Process gas

Note: For plasma processing, E.A. Fischione Instruments recommends using argon with a grade equal to or better than 99.998% purity.

WARNING!

COMPRESSED GAS HAZARD. High-pressure gas stream can injure exposed skin or eyes. Point tubing away from personnel while connecting or disconnecting gas cylinders.

- 1. Position the process gas supply so that it is located within five feet of the ion mill.
- 2. Mount a suitable pressure regulator (Victor HPT 270 or equivalent) onto the process gas supply.
- 3. Connect the ¼ in. tubing (supplied) between the pressure regulator on the process gas supply and the gas inlet quick-connection on the rear of the Model 1010.
- 4. Tighten the connection 1½ turns after finger tight.
- 5. Open both the cylinder and the process gas supply valves.
- 6. Adjust the cylinder pressure regulator to a pressure of 10 psi (69 MPa).
- 7. Close the process gas cylinder valve and observe the cylinder pressure regulator gauge. The indicated pressure should remain constant for a minimum of five minutes. This action ensures that the process gas connection is properly sealed.
- 8. Open the process gas cylinder valve.



## **Exhaust gas**

If hazardous materials will be inserted into the Model 1010 for processing, the ion mill must be connected directly to an exhaust system capable of accommodating the hazardous material.

- 1. If the optional exhaust installation kit was purchased (P/N 009-0285) from E.A. Fischione Instruments, it includes the fitting and length of tubing required for making this connection.
  - Using gas tubing and fittings other than those specified is not recommended and will void the warranty.
- 2. Remove the small plastic muffler from the pump exhaust.
- 3. Thread the connector into the pump exhaust.
- 4. Press the included Viton® tubing over the barb connector.
- 5. Connect the exhaust of the diaphragm pump to a suitable fume hood or equivalent venting system.

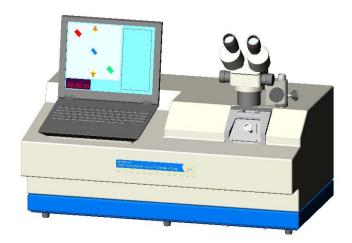
#### Water

The Model 1010 Ion Mill requires a minimum water flow of 0.5 liters per minute (lpm). Water temperature should be maintained at or below room temperature.

- E.A. Fischione Instruments recommends using an in-line filtering system.
- 1. Connect ½ in. tubing (not supplied) between the water inlet and outlet and the two quick-connections on the rear of the Model 1010.
- 2. Tighten the connections 1½ turns after finger tight.

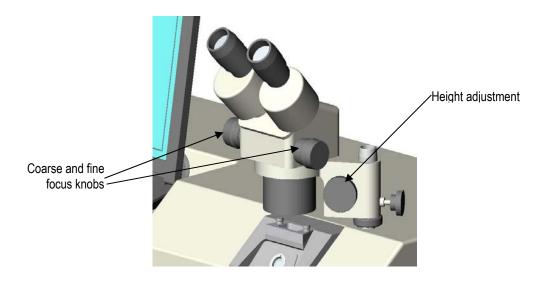
## Microscope Installation

1. Place the spacer over the end of the microscope post.





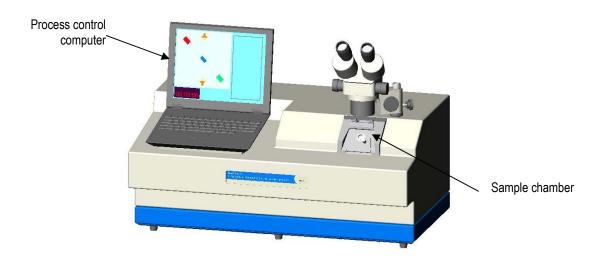
- 2. Insert the microscope post through the cutout on top of the enclosure, into the threaded hole
- 3. Rotate the microscope post clockwise until tight
- 4. Assemble the stereo microscope. Install the 10X eyepieces after removing their individual lens caps, and then remove the lens cap on the microscope's lens
- 5. Install the microscope onto its post, lowering its mount until it contacts the spacer



High magnification microscope installed

## **Process Control Computer**

The process control computer is shipped already installed. The connections to the rear of the computer should be checked before operation.

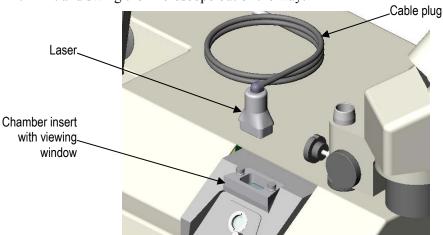




## **Laser Installation (optional)**

Note: The laser will not be illuminated while conducting this procedure. The laser is turned on during milling operations.

1. Lift and swing the microscope out of the way.



- 2. Connect the male connector of the laser cable to the receptacle on the rear of the instrument.
- 3. Insert the laser into the receptacle above the top viewing window.
- 4. When not being used, insert the laser into the receptacle located on top of the microscope post.

#### **Power Connections**

#### For US application

- Verify that the operating voltage matches the requirements of the Model 1010.
   The power entry module is equipped with two 6-amp fuses for 110 VAC operation and two 3-amp fuses for 220VAC operation.
- 2. Connect the power cord between the power entry module and a suitable receptacle.

#### For non-US application

1. Attach a suitable connector to the bare wires of the power cord (supplied) using the following color-coding:

Blue = Neutral (0V)
Brown = Phase (Line, AC)
Green and yellow striped = Ground (earth)

- 2. Verify that the operating voltage matches the requirements of the Model 1010.
  - 110, 220, or 240 VAC can be used with the appropriate fuse.
- 3. Connect the power cord between the power entry module and a suitable receptacle.



## To change operating voltage

Model 1010 has been configured to operate with the operating voltage of the country of the original purchase order. If the Model 1010 is to be used under other power conditions, contact E.A. Fischione Instruments for more information. The instrument must be reconfigured by a qualified service technician or returned to factory.



# Operation

CAUTION: Do not install any program onto the Model 1010 Ion Mill computer.

Installing other programs, including word processing and data management programs, could interfere with the real-time control of

the ion mill and will void the warranty.

CAUTION: Never attempt to force or pry open the chamber insert. To insert a

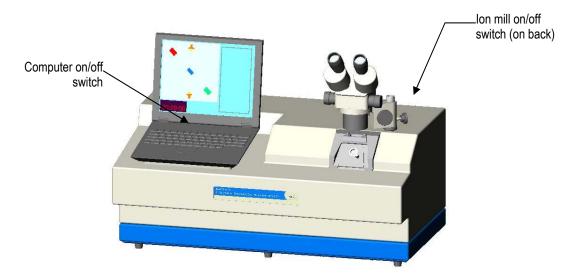
specimen, the vacuum must be off and the stage temperature must

be at least 10°C.

## **Basic Startup Procedure**

1. Verify that the ion mill is supplied with electricity, gas, and water.

2. Press the switch on the lower rear instrument panel (mains) to **ON**.



3. Press and hold the power button on the computer until the computer boots up.

A user identification dialog box will appear.

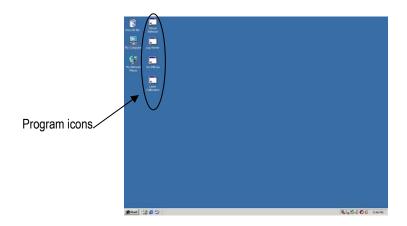
4. Click OK.

Note: "Click" means to use the touch pad on the computer to move the pointer into position then press the left button of the touch pad.

The desktop screen will appear with icons for four Model 1010 programs:

- Dewar Bakeout
- Log Viewer
- Ion Mill
- Laser Calibration (if applicable)



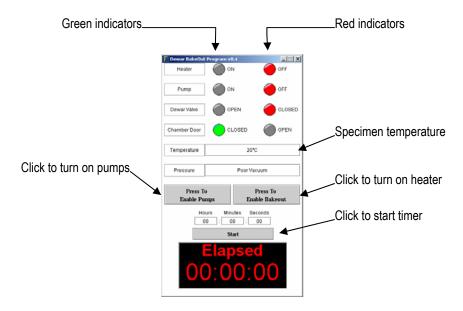


#### **Dewar Bakeout**

Good practice dictates that you heat the Dewar flask for 10 minutes once a day (or more often depending on use) to maintain the integrity of the desiccant.

- 1. Close all open programs.
- Remove all specimens from the stage.
   Heat from the bakeout could damage the specimen.
- 3. Double click the **Dewar Bakeout** icon on the desktop to initiate the Dewar flask heating program.

Note: "Click" means to use the touch pad to move the pointer into position then press the left button of the touch pad on the computer. "Double click" means to click twice.



- 4. Click **Press to Enable Pumps** to turn on the pumps.
- 5. Engage the Dewar conductor rod until thermal contact is made with the specimen stage. Rotate the Dewar knob clockwise, as viewed facing the instrument's rear



panel. Rotate the Dewar knob until its pin contacts the pin stop, preventing further rotation.

When the specimen stage is heated to above  $10^{\circ}$ C and the vacuum is less than  $8x10^{-4}$  torr, the Dewar valve opens.

6. Click **Press to Enable Bakeout** to turn on the heater.

The high temperature setpoint is 100°C. When the temperature exceeds the setpoint, the heater will be turned on and off intermittently to maintain this setpoint.

To operate with the low temperature setpoint (10°C), do not click the **Enable Bakeout** button.

7. Enter a time (hrs:min:sec).

Ten minutes is usually sufficient for daily, routine maintenance. If the dewar performance is not optimal after this step, the bakeout should be repeated for a time of 12 hrs

8. Click Start.

Note: To manually turn off the heater, click **Disable Bakeout**.

When the time has expired, the heater and pumps will turn off, the Dewar valve will close, and a dialog box appears indicating the procedure is finished.

Click OK.

- 9. Open the **Ion Mill** program (see page 21)
- 10. Click **Vent** to release the vacuum.

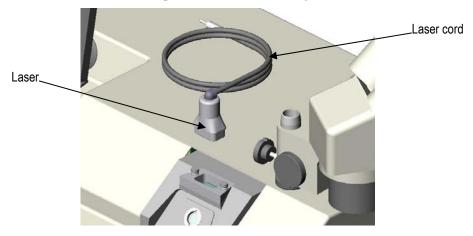
If the temperature is below 10°C the chamber cannot be vented.

11. Disengage the Dewar conductor rod by rotating its knob counterclockwise, as viewed from the rear of the instrument. Rotate the Dewar knob until its pin contacts the pin stop, and no further rotation is possible.

Note: A yellow message box will appear in the Display Area, to remind the user to disengage the conductor rod.

#### Laser Calibration

1. Install the laser into the receptacle above the viewing window.

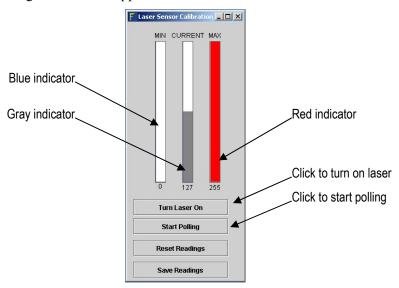




2. Connect the laser cord to the back of the ion mill.

WARNING! LASER HAZARD. Laser beams can cause serious damage to eyes. Do not turn on the laser until it is installed in the ion mill.

- 3. Close all open programs.
- 4. Double click the **Laser Calibration** icon on the desktop to initiate the laser sensor calibration program.
- 5. The following window will appear:



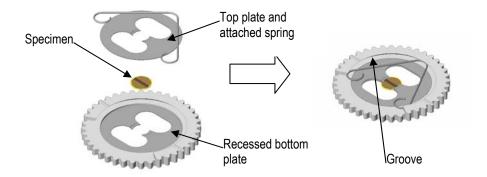
- 6. Click Start Polling.
- 7. The program will begin checking the laser detector readings.
- 8. Click Turn Laser On.
- 9. Minimum reading should remain low, about 0. Maximum reading should go up to about 255.
- 10. Click Save Readings.
- 11. If the readings are unusual or otherwise unacceptable, click **Reset Readings** and start the calibration again.
- 12. Close the program by clicking the **X** in the upper right corner of the program window.

# **Loading TEM Specimens**

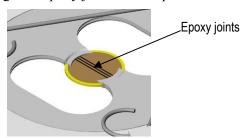
Note: Specimen thickness should not exceed 150 µm.

1. Place the TEM specimen into the recessed bottom plates (attached to the specimen gear) with manual or vacuum tweezers.



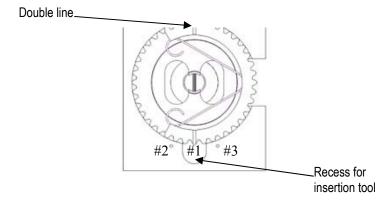


2. For an XTEM specimen, align the epoxy joints of the specimen as shown below:



- 3. Position the top plate and attached spring over the bottom plate.
- 4. Compressing the spring with manual tweezers, insert the left, top, and finally right leg of the spring into the available groove.

If an XTEM specimen is to be oscillated and milled using both ion sources, align the double line on the gear so it points to the position 1 shown below. If only the top source is used, align the double line so it points to position 2. If only the bottom source is used, align the double line so it points to position 3.



# **Specimen Cooling (optional)**

- 1. Bake out the Dewar. (See page 17.)
- 2. Make sure the Dewar conductor rod is engaged.
- 3. Fill the Dewar flask with liquid nitrogen.



## Ion Milling

1. Click the **Ion Mill** icon on the desktop to start the Ion Milling Program.

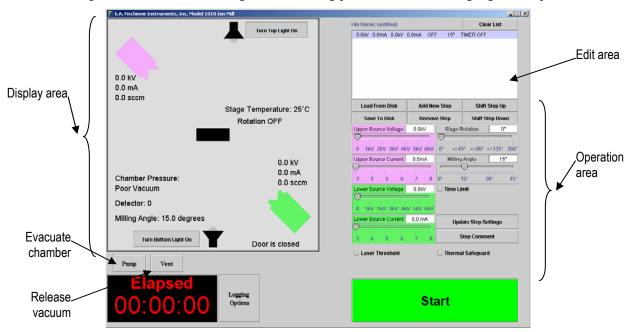
Note: "Click" means to press the left button of the touch pad on the computer.

The Ion Milling program window will appear. The window is divided into three general areas:

**Display area** for real time monitoring of the ion milling parameters. Buttons to turn on the lights are accessed within this space.

Edit area for building the ion milling program steps

**Operation area** for entering the ion milling parameters and editing a given step



Ion Mill program window

The program goes through several initialization procedures including checking the tilt of the stage, making sure the stepper motor is engaged, checking the pressure and temperature, and calculating some behind-the-scenes calibrations.

2. Wait until the initializing phase is completed.

The **Start** button will appear at the bottom of the window. Default values will appear for the first step.

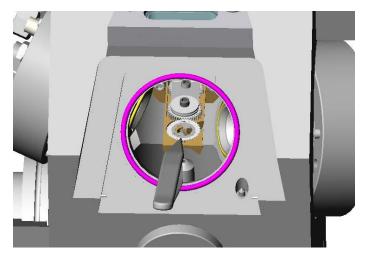
3. Click **Vent** to release the vacuum.

The chamber will not vent until the temperature rises to (+10°C).



#### Insert specimen

- 1. Grasp the specimen gear with tweezers.
- 2. Insert the specimen into the specimen chamber after opening the specimen access door.



#### Microscope

Omit this procedure if the endpoint determination option is being used.

- 1. Turn **ON** the top and bottom lights
- 2. Center the standard magnification microscope with respect to the specimen by rotating the microscope within its mount and /or the mount with respect to its post
- 3. Choose the magnification level by rotating the zoom adjustment.
- 4. Focus the microscope by rotating the focus knob.
- 5. Select an area of interest by further adjusting microscope with respect to its mount and/or post

#### **Endpoint determination (optional)**

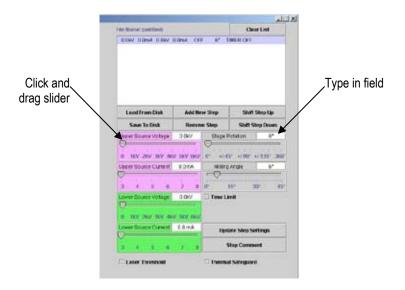
- 1. If the endpoint determination capability is used, install the laser (see page 18).
- 2. Close the shutter on the front viewing window
- 3. Make sure the chamber lights are turned off.

The lights might cause the laser to give a false reading.

#### Set milling parameters

1. Edit the default parameters for the ion sources (voltage and current), stage rotation, and milling angle.





Some values may be edited by typing in the fields, others by dragging the sliders with the pointer. (Hold the left touch pad button while dragging the pointer.)

- 2. To specify a duration for a particular step,
  - a. Select **Time Limit**.
  - b. Type a time (hr:min:sec).
- 3. Click Update Step Settings.
- 4. If additional steps are required, click **Add New Step** and repeat the above operations.
- 5. To change the order of the steps in the program,
  - a. Select a step.
  - b. Click Shift Step Up or Shift Step Down.
- 6. To remove a step,
  - a. Select the step.
  - b. Click Remove Step.
- 7. If the endpoint detection capability is used, click Laser Threshold.

The **Laser Threshold** is the sensitivity of the laser detector, defined in arbitrary units of 1 to 10. This sensitivity decreases as the threshold increases. A threshold of 1 is optimal for specimens that do not transmit laser light, such as metals. A higher value may be chosen for specimens that partially transmit such as bulk Silicon. The precise value is a function of specimen thickness, and must be determined by experience. An effective threshold cannot be chosen for specimens that fully transmit. An example is a XTEM specimen that is held together by epoxy joints.

8. Type a value between 1 and 10.



9. If the specimen cooling capability is used, the conductor rod must be engaged and the Dewar filled with liquid nitrogen. After the minimum desired temperature has been achieved, click **Thermal Safeguard**.

The **Thermal Safeguard** is the maximum specimen temperature permitted during ion milling. A value between -120 and 60°C may be selected. If the specimen temperature exceeds this value, process termination will occur automatically and a heat sensitive specimen will be protected.

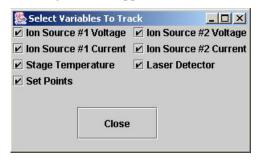
10. Type a value for the maximum temperature between −120 and 60°C.

#### Set data logging options

Selected real-time ion milling parameters may be recorded by using the data logging program. The information will be automatically saved to the computer's hard drive, and can be displayed in graphical form.

1. Click Logging Options.

The tracking selection dialog box will appear.



Tracking selection dialog box

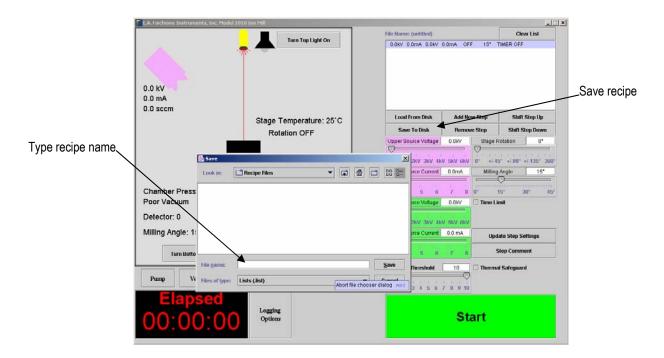
- 2. Click to select or deselect variables as desired.

  Selected variables will appear in the data logging program (see page 27).
- 3. Click Close.

#### Save recipe

1. Click **Save to Disk** to store the edited ion milling recipe on the computer's hard drive.





Save recipe

2. Type a name. When **Save** is clicked, the program will automatically add the required .rcp extension.

Note: To retrieve the program, click Load from Disk.

#### Start milling

Note: Before ion milling each specimen, start the pump, wait until the minimum pressure  $(2 \times 10^{-5})$  torr) is achieved, and then check the specimen temperature.

1. Click **Pump** to evacuate the specimen chamber.

Note: Many operators start the pump before setting milling parameters to save time.

- 2. Close the shutter on the front window.
- 3. Wait until a vacuum of  $2x10^{-5}$  torr has been achieved.
- 4. Engage the conductor rod by rotating the Dewar knob clockwise, as viewed from the rear of the instrument. Rotate until the pin on the knob contacts the pin stop.
- 5. If the specimen is to be cooled, fill the Dewar with liquid nitrogen.
- 6. Wait for the temperature to reach the minimum desired value, often −120°C.

  Omit steps 4 through 6 if the specimen is not to be cooled.
- 7. Click Start.

The following dialog box will appear asking whether to create a log of this run.



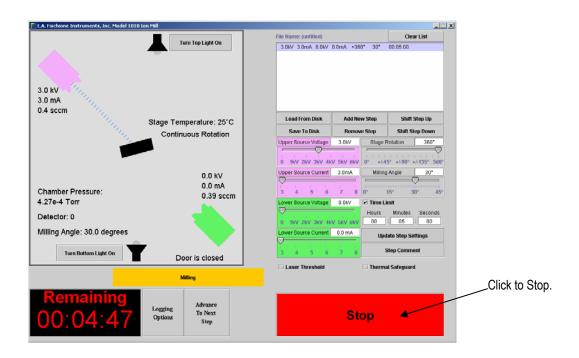


8. To create a log file, click **Yes**.

Note: Creating a log file is not required for performing a milling operation.

9. Provide a file name.

The ion milling process will begin. The display area of the ion mill program window shows the progress of the operation, including stage temperature, rocking angle, ion source voltage and current, milling angle, and status of shutter, lights, and chamber insert.



Milling operation underway

The Start button changes to a "Stop" button, which allows the user to manually terminate the milling operation at any time.

At the end of the ion milling program, the system initiates a resetting chamber sequence, which includes purging the gas line, returning the stage to horizontal, and recentering it. An audible signal notifies the user that the resetting chamber sequence is complete.

#### 10. Click **OK**.



- 11. If specimen cooling was used, disengage the conductor rod by rotating the Dewar knob counterclockwise, as viewed from the rear of the instrument. Rotate it until the knob's pin contacts the pin stop.
- 12. Click **Vent** to release the vacuum.

The venting sequence begins with the powering off of both the turbomolecular and rough pumps. After a delay of 20 seconds, the vent valve is opened and the chamber is brought to ambient pressure in approximately 10 additional seconds.

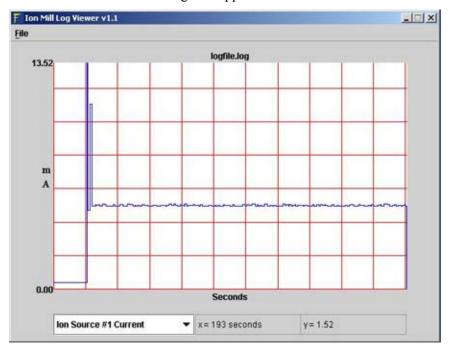
13. Open the chamber access door and retrieve the specimen gear and specimen..

#### **View Data**

- 1. Click the **Log Viewer** icon on the desktop to start the data-viewing program.
- 2. Select a file to view.
- 3. Select a parameter to view.

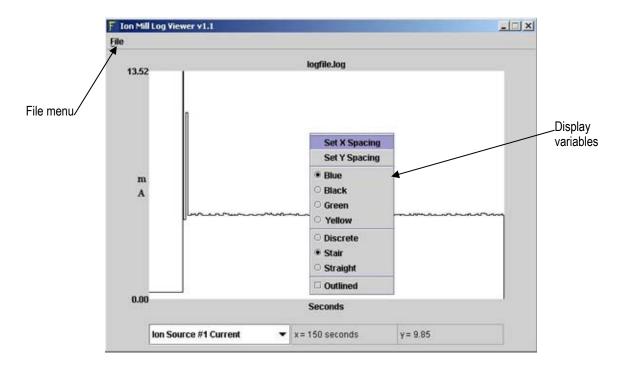
Possible parameters are stage temperature, current and voltage for both guns, laser detector, and the raw data.

A window similar to the following will appear:



4. To access display variables, use the right button to click anywhere on the window. The following dialog box will appear:





Display variable	Action
Set X spacing, Set Y spacing	Sets grid of X and Y axis
Blue, Black, Green, Yellow	Sets color of graph
Discrete	Displays results as individual data points
Stair	Displays curve as level steps between points
Straight	Displays curve as straight lines between points
Outlined	Displays points with boxes around them

- 5. Select the display variables desired.
- 6. To change the view, use the following keys:

Viewing option	Key
Zoom in on location of pointer	i
Zoom out	0
Recenter and rezoom (to starting point)	r
Move view of graph up, down, left, or right	Arrow keys

7. To print the data, save and then copy the file to a CD, and transfer to another computer that is connected to a printer.

The file can be read by a text-editing program.



## **Basic Shutdown Procedure**

- 1. If the instrument is vented, click **Pump** in the Ion Milling program. Verify that that the vacuum level is  $2x10^{-5}$  torr.
- 2. Close the **Ion Mill** program by clicking **X** in the top right corner of the program window. The instrument will remain under vacuum.
- 3. Log off the computer.
- 4. Shutdown the computer through Microsoft® Windows.
- 5. Press the switch on the lower rear instrument panel (mains) to the **OFF** position.
- 6. Shut off the valves that supply gas and water to the instrument.



## Maintenance

## WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the power entry module located on the back of the instrument.

#### **Preventive Maintenance Schedule**

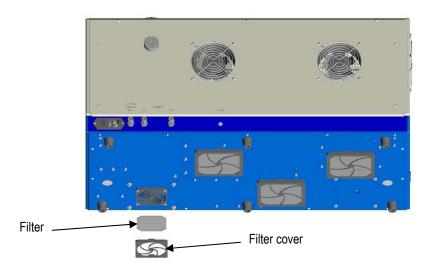
Component	Frequency	Maintenance action	Refer to
Dewar	1 week	Bakeout	Page 17
Air filters	6 months	Inspect, clean if needed	Page 30
Water filter	6 months	Inspect, clean if needed	Manufacturer's instructions
Top specimen chamber window	As required	Inspect, clean if needed	Page 33
Chamber access door window	As required	Inspect, clean if needed	Page 33
Top and Bottom light windows	As required	Inspect, clean if needed	Page 34
Turbo-drag pump	1 year	Lubricate	Manufacturer's instructions
Diaphragm pump	1 year	Inspect diaphragms	Manufacturer's instructions

# **Cleaning the Air Filters**

The Model 1010 is equipped with three ventilation fans that circulate the air through the instrument enclosure. Air is drawn in through four filtered ports located in the instrument's base plate. Because of the high volume of air flow through the enclosure, the filters should be cleaned once every 6 months.

- 1. Use one person to stabilize the instrument and another to remove the filters.
- Carefully position the instrument at the edge of a workbench.
   Do not allow the instrument project over the edge of the bench by more than required to access the filters.
- 3. Snap the filters off their mounts by applying steady downward pressure.





- 4. Wash the filters in warm, soapy water then let air dry.
- 5. Reinstall the filters by snapping them onto their respective mounts.

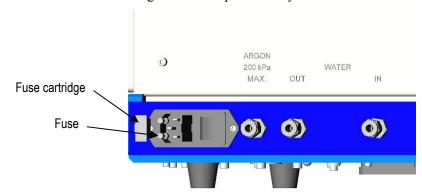
## **Changing the Fuses**

WARNING! ELF



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 1. Turn the Model 1010 power switch to the off position.
- 2. Disconnect the power cord.
- 3. Using a small-blade screwdriver, gently remove the plastic cover from the power entry module on the back of the instrument.
- 4. Pull the fuse cartridge out of the power entry module.



5. Replace the fuses.

For 110 VAC operation, replace two 6-amp fuses. For 220 VAC operation, replace two 3-amp fuses.



6. Reinstall the cartridge into the power entry module.

## **Opening the Enclosure**

To service most components, the instrument enclosure must be opened.

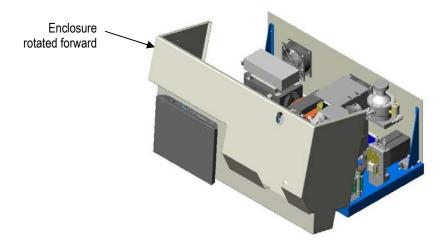
#### WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 1. Disconnect the laser, and remove it from the top of the instrument.
- 2. Remove the standard magnification microscope, microscope post and spacer
- 3. Remove the three screws located at the top of the rear panel.
- 4. Firmly grasp the bottom of the hinged enclosure portion and lift upward.

Because the instrument is hinged in the front, the enclosure will rotate forward.



#### **Enclosure removal**

For a major cleaning, remove the top instrument enclosure. This will provide easy access to the various system components.

- 1. Vent the instrument
- 2. Turn the Model 1010 power switch to the off position.

## WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 3. Disconnect the power cord.
- 4. Remove the standard magnification microscope, microscope post and spacer.
- 5. Open the instrument enclosure.
- 6. Remove the fan wire from the fan by pulling the molded plastic plug off the pin contacts on the fan.



- 7. Once all of the electrical components have been disconnected, remove all of the wires from their plastic clamps.
- 8. With the electrical cabling disconnected, remove the six hex nuts that attach the upper portion of the hinge to the instrument enclosure.
- 9. Remove the enclosure.

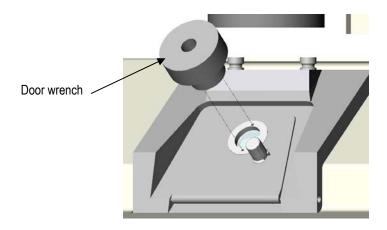
## **Window Cleaning**

The Model 1010 contains four sapphire windows, which permit both viewing and illumination of the specimen. They are positioned on top of the specimen chamber, within the chamber access door, and in the top and bottom light assemblies.

#### Chamber access door window

Although protected by a shutter assembly, this window will eventually need to be cleaned.

- 1. Ensure that the instrument is vented and not powered ON
- 2. Engage the pins on the door window removal wrench with the holes in the bezel.



3. Rotate the wrench counterclockwise until the bezel and window can be removed.

CAUTION: The sapphire window is permanently adhered to the bezel to assist in handling. Attempting to remove the window from the bezel will damage both components. If the window is broken, return the bezel and window to E.A. Fischione Instruments for repair.

- 4. Remove sputtered material from the window surface using any type of metal or glass polish.
- 5. Use ultrasonic cleaning for final cleaning.

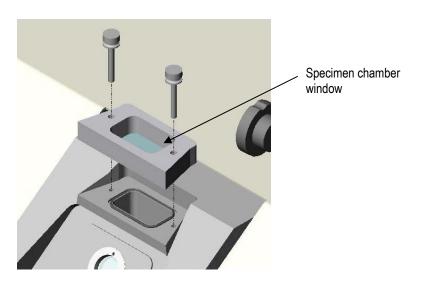


#### Specimen chamber window

Caution: This window is located on the top chamber surface and on the specimen axis of rotation, and requires periodic cleaning. Failure to properly maintain this window will result in diminished sensitivity of the laser detector.

- 1. Ensure the instrument is vented and not powered ON.
- 2. To remove, firmly grasp the knurled portion of the bezel, then rotate counterclockwise. The sapphire window is permanently adhered to the bezel to assist is handling. Attempting to remove the window from the bezel will result in damage to both components. Should the window become broken, return the bezel/window to E.A. Fischione Instruments for repair.
- 3. Remove sputtered material from the window surface using any type of metal or glass polish.
- 4. Use ultrasonic cleaning for final cleaning.

### Top light window



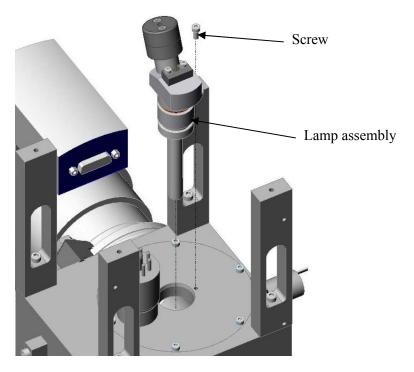
- 1. Ensure that the instrument is vented and not powered ON.
- 2. Remove the 8-32 socket head cap screw and angled bracket that press the top light assembly into its receptacle
- 3. Open the front chamber access door
- 4. Placing a gloved finger within the specimen chamber, gently press upward on the top light assembly until it exits its receptacle.
- 5. Locate the window shield, and engage the pins the wrench with the holes in the shield.
- 6. Rotate the wrench counterclockwise until the shield is removed.



- 7. Remove the window that is positioned below the shield and above an O ring.
- 8. Remove sputtered material from the window surface using any type of metal or glass polish.
- 9. Use ultrasonic cleaning for final cleaning.

### **Bottom light window**

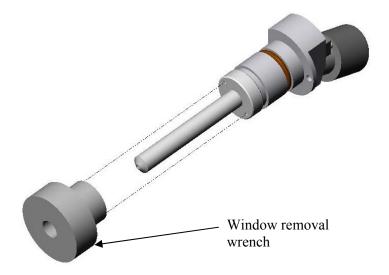
1. Ensure the instrument is vented and not powered ON



- 2. Remove the single screw which fastens the lamp assembly to the chamber flange.
- 3. Gently slide the light/laser detector assembly downward to the instrument's base plate.

**CAUTION**: The bottom light/laser detector contains a sputter shield which extends from the chamber bottom flange to the specimen stage. Because of the length involved in this shield, extreme care should be taken when removing or re-installing the assembly from the chamber. Bumping or rubbing of the shield on the chamber flange could cause nicks or scratches which may result in vacuum leaks.





4. To clean the window once the light/laser detector has been removed, slide the door window removal wrench over the sputter shield and engage the pins on the tool into the holes on the sputter shield/cap assembly and rotate counterclockwise. This window is not attached to the sputter shield/cap assembly, therefore, care should be taken when removing the window so that it is not dropped or broken during the disassembly procedure.

### Cleaning o-rings

Before reinstalling any window, E.A. Fischione Instruments recommends removing and cleaning the sealing o-ring.

1. Remove the o-ring using a wooden toothpick from the groove around the chamber insert.

CAUTION: The sealing surface is easily damaged. Never use a metal object to remove an o-ring.

2. Clean both the o-ring and its corresponding seat area with ethanol or isopropyl alcohol.

CAUTION: Use only ethanol or isopropyl alcohol to clean an o-ring. Acetone or other solvents will cause permanent damage necessitating o-ring replacement.

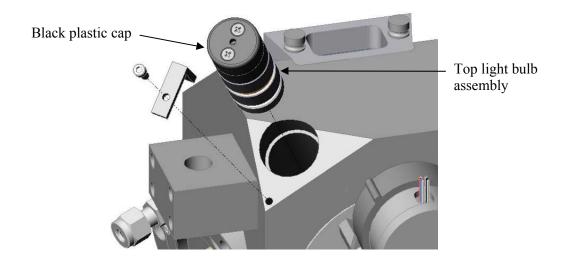
3. Apply a thin film of Fomblin® grease to the o-ring before reinstalling.

### Lamp Replacement

### **Top Light**

1. Ensure the instrument is vented and not powered ON

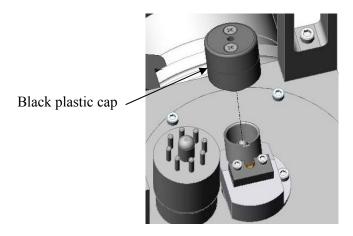




- 2. Remove the top light assembly (See page 34.)
- 3. To remove the light bulb from the light assembly, rotate the black plastic cap located on the rear of the light assembly counterclockwise. The light bulb will drop out of the assembly. Because the lamp is a halogen bulb, care should be taken not to touch the glass bulb surface. Any oil or other contamination will diminish the life of the bulb. Should inadvertent contact with the bulb occur, it must be wiped with methanol and a lint free tissue.
- 4. Place the new light bulb into the housing and thread on the screw cap. Install the light housing into the chamber and replace the "L" clamp

### **Bottom Light**

1. Ensure the instrument is vented and not powered ON



2. To replace the light bulb, unscrew the black plastic cap until it becomes free from the assembly at which point the bulb will drop out. Because the lamp is a halogen bulb, care should be taken not to touch the glass bulb surface. Any oil or other contamination will diminish the life of the bulb. Should inadvertent contact with the bulb occur, it must be wiped with methanol and a lint free tissue. Place the new light bulb into the housing and thread on the screw cap.



3. Replace the black plastic cap

#### Ion Source Maintenance

The life of the ion source components and the interval between their cleanings may vary greatly based on the energies at which the ion mill is operated. Ion source maintenance is needed when it exhibits difficulty in achieving a plasma, is unable to sustain a stable ion beam, or produces a weak beam as determined visually.

- 1. Remove the laser.
- 2. Remove the microscope, microscope post and spacer.
- 3. Remove the three screws on the top back surface of the instrument.
- 4. Rotate the hinged upper enclosure forward.

# WARNING!

 $\wedge$ 

COMPRESSED GAS HAZARD. Compressed gas can injure skin or eyes. Before beginning the ion source maintenance procedure, vent the instrument to atmosphere.

5. Vent the instrument.



Ion Mill program window

- 6. Shut down the computer.
- 7. Turn the Model 1010 power switch to the off position.



### WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

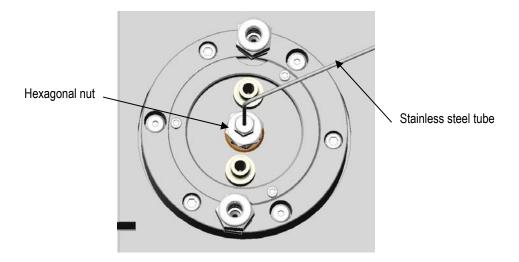
8. Disconnect the power cord.

### Disconnect gas line

1. Disconnect the gas inlet to the ion source by firmly grasping the hexagonal nut that connects the gas line to the cathode and rotating it counterclockwise.

A stainless steel ferrule on the stainless steel gas tube maintains the vacuum integrity between of the gas tube and the fitting.

Allow the stainless steel ferrule to remain on the stainless steel gas tube.



### Remove ion source cartridge

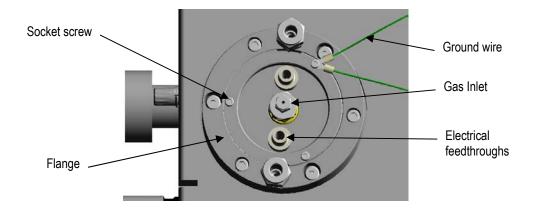
Note: The ion source functional components are all contained in the ion source cartridge.

CAUTION: Ion source components are easily damaged. Work in a clean area. The individual doing the work should be knowledgeable in the polishing and assembly of precision mechanical components.

1. Remove the three socket screws that attach the cartridge to the flange.

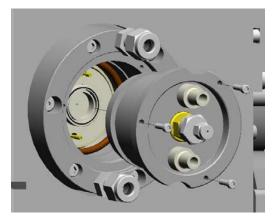
Note that the ion source ground wire (green) is connected to one of the screws.





CAUTION: Avoid contaminating ion source components. E.A. Fischione Instruments strongly recommends using talc-free rubber gloves when handling the ion source or source components to avoid contaminating source components.

- 2. Don talc-free rubber gloves.
- 3. With one hand, reach into the chamber and begin pushing the ion source cartridge outward.
- 4. With the other hand, both pull and direct the ion source cartridge out of the alignment flange.



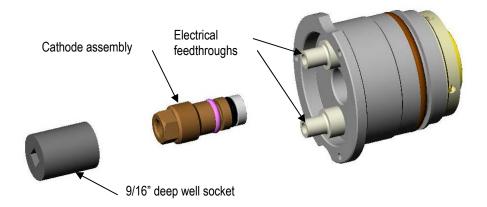
5. Remove the gun cover by sliding it off the cartridge.





### Remove cathode

1. Remove the cathode assembly from the gun body by rotating it counterclockwise.

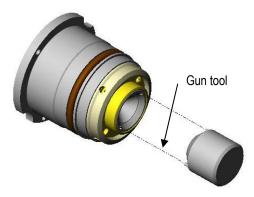


The cathode body is fitted with radial sealing o-rings that resist being un-threaded. A 9/16 in. deep well socket should be used to gently unscrew the cathode assembly so that the fragile electrical feedthroughs are not disturbed or damaged.

2. Once removed, set the cathode assembly aside.

### Remove anode and extractor

1. To disassemble the remainder of the components, insert the pins located on the end of the gun tool into the holes on the contact nut.



- 2. Rotate counterclockwise to remove.
- 3. Invert the cartridge (with the cathode port facing upward).
- 4. Cover the end of the ion source from which the beam is projected with one hand.

CAUTION: Components may be damaged if dropped. When removing ion source components from the gun body, make sure that they do not drop onto the workbench or the floor. Severe damage to the components, and in particular the ceramic insulators will result.

5. Insert the anode removal tool into the cathode port to apply pressure to source components.

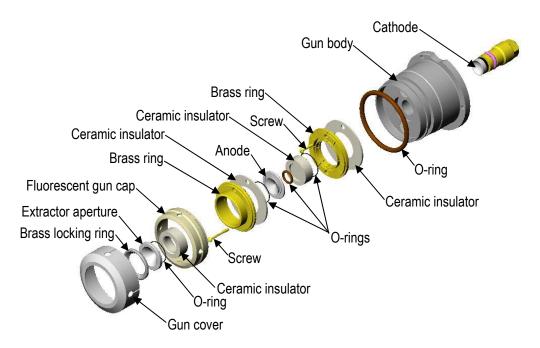




While rotating the threaded anode removal tool clockwise, the ion source components should now drop freely and fall into the hand covering the anode portion of the gun cartridge.

6. Remove all o-rings and set aside.

CAUTION: Do not allow o-rings to contact acetone. If they are contaminated with acetone, they must be replaced.



### **Inspect components**

1. Inspect the concave surface of the cathode.

With prolonged usage the concave surface of the cathode will become both discolored and pitted. This is a result of material being sputtered from this surface during the milling process.



2. Inspect the hole in the center of the cathode.

The hole in the center of the cathode becomes enlarged over time. Excessive hole diameters result in the ion source requiring additional gas. During operation, the gas flow approaching 1.0 sccm is an indication that the cathode needs to be replaced. The hole diameter of a new cathode is nominally 0.0135 in. (0.3429 mm).

3. Inspect the anode and the extraction aperture.

Both the anode and the extraction aperture will exhibit discoloration and a possible flaky build-up. These situations are normal.

Note: The extraction aperture should be replaced if the diameter of the central hole increases from a nominal 0.060 in. (1.52 mm) to more than 0.070 in. (1.78 mm).

4. Inspect the jeweled sapphire orifice in the anode.

A jeweled sapphire orifice is positioned in the anode to both resist wear and to precisely control hole size.

5. If the sapphire is cracked or shows excessive hole erosion, replace the anode.

### Clean components

With ion source usage, the ceramic components will become coated with material sputtered from the cathode. Because the sputtered material is electrically conductive, the ion source will short circuit when the sputter build-up becomes excessive. Shorting of ion source components greatly decreases ion source efficiency and could lead to electronic control problems.

Sputtered material must either be removed from the ceramic insulators or the insulators must be replaced. The cleaning procedure for the anode, cathode, and extraction aperture are virtually identical.

- 1. To facilitate handling and to avoid distortion of ion source components, secure them lightly in a collet and rotate them in a small lathe.
- 2. If the surface of the cathode is extremely pitted, polish using 1000 grit sand paper.
- 3. Scrape any sputtered material from the gun cap.
- 4. Polish all metal surfaces using either a fine grit (<3 micron) diamond abrasive compound or a polishing compound such as Wenol® or Pol®.
- 5. Bead blasting with fine glass beads may be used to clean the ceramic (but not metal) components.

### Service ion source body components

The parts remaining in the gun body require minimal service.

1. Unscrew both the anode and the extractor contact screws.

CAUTION: Take care when removing the brass rings to avoid damaging the ceramic insulators that separate the brass rings.

- 2. Unscrew the fluorescent gun cap from the gun body.
- 3. Detach the brass rings from the gun body. Ensure that they remain aligned, especially before the reassembly procedure is undertaken.



4. Inspect the electrical components for burn marks or other forms of deterioration.

Note: The electrical connections for the ion source are not field serviceable.

- 5. Following a thorough inspection, ultrasonically clean the gun body in methanol.
- 6. Inspect for electrical continuity by placing one probe of an ohmmeter into the receptacle for the high voltage wire, ensuring that it is touching the phosphor bronze pin contact.
- 7. Touch the other meter lead to the corresponding screw receptacle.
  - The meter should indicate < 1 ohm.
- 8. Repeat the procedure for the other electrical contact.
- 9. To reassemble the brass rings, position the components as shown on page 41 through on page 42.

### Note: The ceramic insulating rings have differing internal diameters.

- 10. Ensure that the ceramic ring with the smaller diameter is installed between the gun body and the brass anode-contacting ring.
- 11. Install the ceramic ring with the larger internal diameter between the two brass contacting rings.
- 12. Be sure that the anode and extractor contact screws are appropriately positioned but remain loose.
  - The anode contact screw is the inner most screw whose access is through the hole in the extraction brass contact ring.
- 13. Thread anode contact screw into the electrical contact that corresponds to the anode connection, which is marked with an "A" on the outside of the gun body.
  - The extractor contact screw is the outer most screw whose head is accessible on the face of the extraction brass contact ring.
- 14. Thread the extractor screw into the electrical contact that corresponds to the extraction connection, which is marked with an "E" on the outside of the gun body.
  - Failure to follow this instruction will result in incorrect voltage potentials being supplied to the ion source components.
- 15. Insert and thread the fluorescent gun cap into the gun body until resistance is felt to further rotation.
- 16. To align the brass contact rings and the ceramic insulators, insert the gun tool into the bore in the rings. The brass contact rings serve as alignment devices for the active source components.
- 17. Ensure that the gun tool is fully seated in the gun body, and that the groove in the tool coincides with the surface of the brass ring found under the gun cap.
- 18. While slowly rotating the tool, gradually tighten the anode and extractor contact screws less than a turn each. If significant resistance is felt, unthread the fluorescent gun cap less than a turn, and realign the brass rings with respect to the tool. Retighten the contact screws a turn each or until resistance is again felt. Using an iterative approach, readjust the fluorescent gun cap and thread in the contact screws until both screws have been tightened.



CAUTION: Do not overtighten these screws. Doing so may damage the ceramic insulating rings.

### Ultrasonically clean and inspect components

- 1. After cleaning and polishing the active source components (anode, extractor, ceramic insulators), ultrasonically clean them in methanol.
- 2. Allow them to dry on a lint-free cloth.
- 3. Inspect the components for burns, cracks, or other significant damage.
- 4. Replace components that exhibit excessive wear.

### Reassemble ion sources

- 1. When reassembling the ion source make sure no foreign objects are introduced onto any ion source components.
  - E.A. Fischione Instruments recommend using nylon or talc-free rubber gloves during the assembly procedure.
- 2. Remove foreign substances on source components with pressurized, inert gases such as nitrogen or argon.

CAUTION: Do not use vacuum grease on any ion source component. Grease or any other form of contamination diminishes ion source performance.

- 3. Stack the source components in their respective positions.
- 4. Hold the ion source cartridge in the inverted position (the cathode port facing upward and insert the component stack.
- 5. Make sure that all components are properly seated in their respective positions.
- 6. Rotate the ion source cartridge 180° until the extraction aperture is facing upward.
- 7. Verify that the extractor is centered in the brass ring and its face is both parallel and below the surface of the brass ring by approximately 2 mm.
- 8. Carefully thread the contact nut into place and tighten with the gun tool.
- 9. Replace the cathode by rotating it clockwise into place.

### Verify electrical connections

Before inserting the ion source cartridge into the alignment flange, confirm that the source components are not shorting. The following procedure verifies that there is no shorting of the anode to the extractor.

- 1. Place a standard ohmmeter probe into the anode connector ensuring that the probe is in electrical contact with the phosphor bronze pin.
- 2. Touch the other lead to the gun body.

The ohmmeter should indicate that there is no continuity.

3. Repeat the procedure with the extraction contact.



4. Place the leads of the ohmmeter into the electrical contacts ensuring continuity with the phosphor bronze pins.

The meter should again indicate that there is no continuity.

Any resistance measured during this testing (even mega-ohm indications), indicates that a short circuit condition exists within the ion source, which means it must be disassembled, inspected, and possibly re-cleaned.

### Replace ion source

The ion source cartridge is now ready for installation into the alignment flange/chamber.

- 1. Clean the o-ring that seals the ion source cartridge to the chamber with isopropyl alcohol, then apply a thin coating of Fomblin® vacuum grease.
- 2. Place the ion source cartridge into the alignment flange
- 3. Push inward until the o-ring contacts the chamber.
- 4. Rotate the cartridge until the grooves that clear the water tubing connections and the three attaching screw holes are properly aligned with their corresponding parts of the alignment flange.
- 5. Apply firm, even force to the ion source cartridge, pushing it into the chamber until the back of the gun body flange is flush with the outer surface of the alignment flange.
- 6. Install the three screws.
- 7. Be sure that the ion source ground wire is attached to one of the screws.

#### Attach gas and electrical connections

- 1. Reconnect the gas supply to the ion source.
- 2. Ensure that the stainless steel ferrules are positioned on the gas tube.
- 3. Position the gas tube in the gas inlet fitting and tighten the nut.

Note: To re-tighten an existing ferrule, make it finger-tight, then rotate it 1/4 turn. To tighten a new ferrule, make it finger-tight, then rotate it 1-1/4 turns.

- 4. Reconnect the high voltage cables.
- 5. Ensure the following:
  - a. The cable marked "anode" is connected to the high voltage terminal on the ion source marked with an "A".
  - b. The high voltage cable marked "extractor" is connected to the high voltage terminal on the ion source cartridge marked "E".

### **Check leaks**

After installing the ion source into the chamber, do a vacuum leak check. See page 63.



### Start up ion source

Once it has been confirmed that the instrument maintains vacuum, start up the Model 1010 using the procedure on page 16.

## Ion Source Alignment

The HAD ion source produces a beam comprised of both ions and fast neutrals. Because of the beam composition, it has been found that an optical-mechanical alignment of the ion sources provides the best results.

The ion sources are fixed in position with respect to the specimen stage; however, fine adjustment of the ion sources makes sure their precise position is correct with regard to the center of specimen rotation. Ion source alignment is normally not required except after the source position was accidentally moved, or after major service of the ion source.

If the specimen produced with the Model 1010 shows little or no milling on one or both specimen surfaces, or if perforations occur consistently off-center, it is an indication that adjustment of the ion sources may be required. This can be confirmed quickly by utilizing a beam alignment disk.

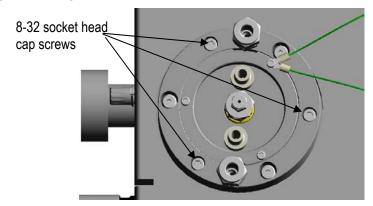
This disk is a 3 mm diameter and 150  $\mu$ m thick section of glass chosen so that a well-defined ion beam could be observed with the unaided eye. A 5X loupe may be placed over the viewing window in the chamber insert as a further aid.

### Adjust source alignment

1. Prepare a 3 mm disk cut from a glass coverslip to be used in place of a specimen. Load the alignment disk into the specimen gear.

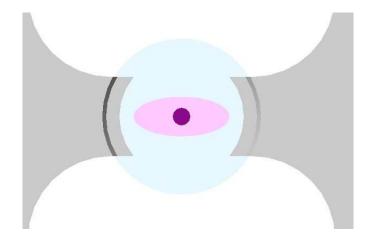
Such disks can be made using an ultrasonic disk cutter, such as the E.A. Fischione Instruments Model 170.

- 2. Make sure that the laser has been removed.
- 3. Open the instrument enclosure. (See page 32.)
- 4. Activate the vacuum system.
- 5. Energize the top ion source using parameters of 5kV, 5mA.
- 6. Change the milling angle to 15°.
- 7. Locate the three 8-32 socket head cap screws recessed beneath the surface of the alignment flange.





8. Observe the position of the ion beam.



- 9. If the beam is centered on the disk, de-energize the top ion source.
- 10. If the beam is not centered on the alignment disk, insert a 9/64" ball driver into the adjusting screws.

Note: The alignment flange is spring loaded to facilitate the alignment procedure.

- 11. While observing with the unaided eye or through a 5X loupe, rotate the screws clockwise and counterclockwise until the beam is centered on the alignment disk.
- 12. Repeat the procedure for the bottom ion source.

### Verify alignment

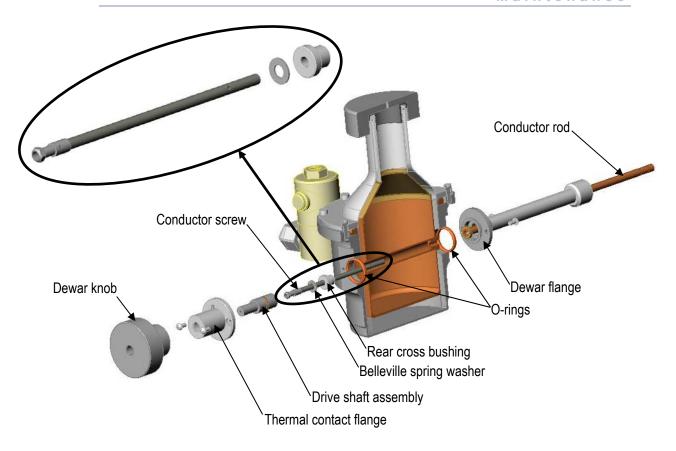
- To verify alignment, ion mill a thin (<25 micron) sheet of Cu or Al.</li>
   Copper or Aluminum is suggested because of their rapid milling rates, however, any type of material can be used.
- 2. Make sure that the beams are properly aligned and the perforation occurs either at or near center. For Aluminum foil, the time to perforation is typically 1 hour for the following conditions: 5kV, 5mA (both sources), 25° milling angle, 360° rotation.

### **Dewar Maintenance**

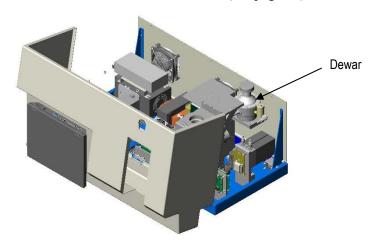
#### **Remove Dewar**

After prolonged use, it may become necessary to change the Zeolite® desiccant contained in the liquid nitrogen Dewar flask. This procedure should only be performed if repeated bakeout procedures are not successful and the Dewar performance cannot be optimized.





- 1. Ensure that the instrument is turned off and the power cord disconnected.
- 2. Open the instrument enclosure and locate the Dewar (see page 34).

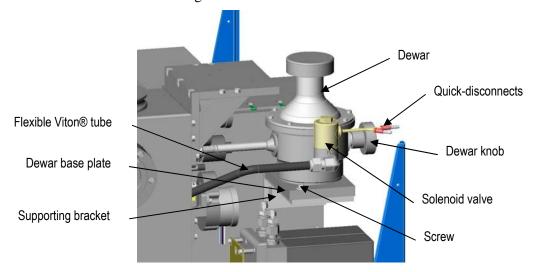


3. Before removing the Dewar knob, make sure that the orientation of the knob in relation to the shaft is clearly marked.



CAUTION: When re-installing the Dewar knob, it must be re-oriented to its original position. Failure to follow this instruction could result in Dewar damage or poor specimen cooling.

- 4. Remove the knob which projects through the instrument's back panel.
- 5. Disconnect the wires leading to the Dewar solenoid valve.



This connection includes a quick-disconnect mechanism that permits the unplugging of the wires. The Dewar is interlocked through several different means to maintain its vacuum integrity and avoid inadvertent venting.

- 6. Remove the four screws that attach the rear panel.
- 7. Remove the rear panel.
- 8. Remove the flexible Viton® tube that connects the Dewar valve to the specimen rotator assembly.
- 9. Remove the two screws in the Dewar base plate that attach the base plate to the supporting bracket.
- 10. With one hand, firmly grasp the Dewar housing.
- 11. With the other hand, stabilize the junction where the tube projecting from the Dewar meets the tube projecting from the stage.

This junction contains a radial o-ring seal that maintains the vacuum integrity in the Dewar.

12. SLOWLY move the Dewar toward the back of the instrument.

CAUTION: When the integrity of the vacuum seal is broken, the Dewar will move freely. It is important to have a firm grasp of the Dewar to avoid dropping it when vacuum is lost.

The plastic Dewar base serves as a receptacle for the Dewar.

13. Remove the Dewar from the base.

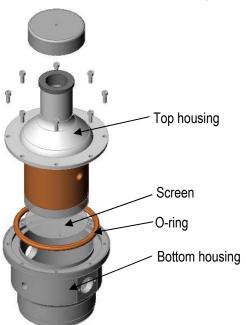
The base should remain with the instrument.



14. Take the Dewar to a clean work area.

### Change Zeolite® desiccant

- 1. Remove the tube that projects from the Dewar housing. This flange assembly protects the Dewar rod, which is made of a copper alloy.
- 2. Remove the thermal contact flange from the opposite side of the Dewar.
- 3. Remove the conductor screw by rotating it counterclockwise.
- 4. Remove the Belleville spring washer.
- 5. Remove the rear cross bushing.
- 6. Remove the eight screws that hold the top of the Dewar housing onto its bottom.
- 7. Lift and remove the top of the Dewar housing from the bottom.
- 8. Remove the four wires that attach the desiccant screen to the inner vessel.
- 9. Remove the desiccant and dispose of it properly.
- 10. Wipe the bottom of the inner vessel with methanol and allow to dry.



- 11. Replace the desiccant.
- 12. Install the desiccant screen.
- 13. Remove the o-ring from the Dewar bottom housing, and wipe it with isopropyl alcohol.
- 14. Clean both the Dewar bottom housing, and especially the o-ring seat.
- 15. Lightly coat the o-ring with Fomblin® vacuum grease
- 16. Reposition the o-ring with respect to the bottom housing.
- 17. Replace the bottom housing on the Dewar.



### **Install Dewar**

- 1. To reinstall the Dewar onto the instrument, replace the plastic Dewar base on its support bracket.
- 2. Place the Dewar bottom into the recess in the plastic support.
- 3. Remove, clean, re-grease, and reinstall the o-ring on the specimen stage seal tube.
- 4. Direct the end of the copper conductor rod into the end of the seal tube.
- 5. The end of the conductor contains two slots, which create a spring effect for the conductor.
- 6. Gently apply force to engage the copper rod into the specimen stage.
- If resistance is encountered, re-position the copper conductor rod and attempt again.
   When pressing the conductor into the stage, a minimal amount resistance will be encountered.
- 8. Continue to apply steady pressure until the fitting on the end of the Dewar tube covers the o-ring located on the specimen stage seal tube.
- 9. Replace the two screws that fasten the plastic Dewar base to its support.
- 10. Re-connect the electrical connections to the Dewar switch.
- 11. Connect the flexible Viton® tube to the rotator assembly.
- 12. If no further maintenance is required, replace the instrument's back panel and reconnect the Dewar valve.

#### **Evacuate Dewar wall space**

The volume between the inner vessel and outer housing of the Dewar needs to be evacuated in order to ensure proper vacuum for insulating purposes. Use the Dewar Bakeout function to evacuate the volume. See page 17.

## **Chamber and Specimen Stage Cleaning**

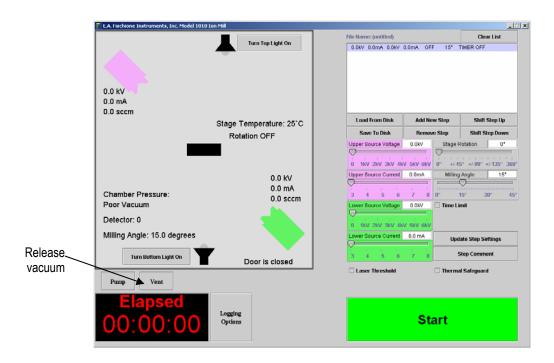
Due to the nature of ion milling, the entire chamber/specimen stage will need to be periodically cleaned to remove excessive amounts of sputtered material. Cleaning frequency depends on the usage of the instrument. However, E.A. Fischione Instruments recommends complete cleaning at least once per year.

- 1. For a thorough cleaning of the chamber, remove the components listed in the sections on pages 32 to 52 to provide full access to the internal chamber surfaces.
- 2. Wipe the chamber clean with acetone.
- 3. Remove sputtered material should with an abrasive pad then thoroughly wiped clean with acetone.

### Stage maintenance

1. Vent the instrument.





2. Turn the Model 1010 power switch to the off position.

# WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 3. Disconnect the power cord.
- 4. Remove the Dewar and the instrument's back panel as described on page 48.

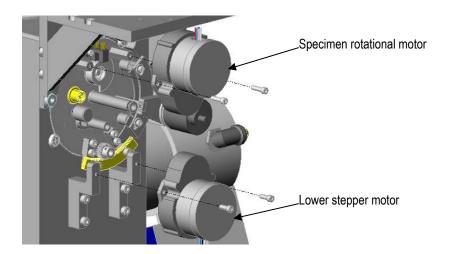
#### **Disconnect electrical connections**

- 1. Disconnect the electrical connections to the temperature sensor.

  This connector simply pulls straight out of its socket.
- 2. Remove the stage ground wire.
- 3. Unthread the bezel surrounding the connector to the stage heater, and then gently remove this connector from its socket.



### **Remove motors**

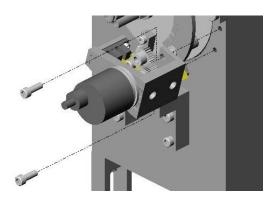


- Remove the lower stepper motor, which is used to provide stage angle movement.
   Two socket screws attach the motor to its mounting posts.
- 2. Remove the specimen rotational motor in the same manner.

Note: Do not discard the plastic component of the flexible coupler.

### Remove angle encoder

1. Remove the optical encoder and zero-position optical pick-up by removing the two screws that fasten the bracket to the chamber.



Once these screws are removed, TAKE CARE TO NOT DAMAGE THE OPTICAL PICK-UP.

2. Slide the encoder/pick-up assembly clockwise to free it from the gear.

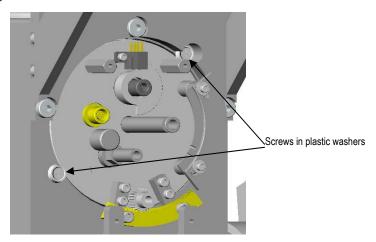
It may be necessary to rotate the entire rotator assembly by hand in order to position the gear for the removal of the encoder/pick-up assembly.



#### Remove rotator

- 1. Remove the two screws located within the plastic washers that are positioned at the chamber-rotator plate interface.
- 2. Firmly grasp the rotator assembly and pull toward the rear of the instrument.

It may be necessary to rotate the assembly so that the lower gear clears the motor mounts.



CAUTION: Never set the stage so that it is being supported by the rotator plate and the end of the specimen stage. Damage to the specimen stage may result.

3. Take the specimen stage rotator assembly to a clean work area for inspection, cleaning, and any necessary repairs.

CAUTION: Extreme care must be taken when handling or conducting service on the rotator plate. Any scratches or nicks on the face of the rotator plate will lead to vacuum leaks.

#### **Install rotator**

- 1. Prior to installation, remove the o-ring from the chamber and clean thoroughly with methanol or isopropyl alcohol.
- 2. Wipe the chamber surface with acetone.
- 3. Allow to dry.
- 4. Apply a light film of Fomblin® grease to the o-ring and position the o-ring into the chamber.
- 5. Install the rotator assembly into the chamber.

Make sure that the o-ring does not slip out of its groove during the installation of the rotator.

There is a precise fit between the chamber and the bearing and the outer diameter of the rotator plate. The rotator plate should be installed as straight as possible. Any angling of the rotator plate in relation to the chamber will prevent full insertion of the rotator plate leading to the inability to achieve a vacuum.



- 6. Install the two screws and plastic washers to secure the rotator plate to the chamber.
- 7. Rotate the rotator shaft to ensure smooth movement.

#### Replace angle encoder

1. Carefully position the encoder/optical pick-up on the angle gear.

CAUTION: The optical pickup is easily damaged. Be careful while installing the encoder/optical pick-up.

2. Fasten the encoder bracket to the chamber with the two screws.

### Replace motors

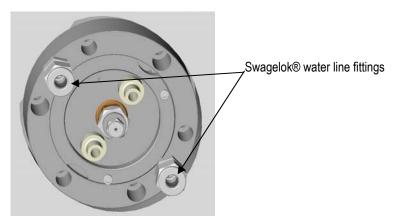
- 1. Locate the lower stepper motor containing the gear on its shaft.
- 2. Position this motor onto the standoffs and secure with the two screws.
- 3. Make sure that the motor gear meshes freely with the rack gear mounted to the rotator plate.
- 4. To install the rotation motor, make sure that the plastic component of the flexible coupler is in place in one half of the coupling.
- 5. Mate the shafts together with the plastic component in between.
- 6. Secure the motor with the two screws.

#### Attach electrical connections

- Attach the electrical connection for the heater and temperature sensor.
   The connector on the end of the cable is keyed, which ensures its correct orientation.
- 2. Attach the stage ground wire and secure it by tightening the bezel.

### Ion source flange

When conducting major maintenance, it may be advantageous to remove the ion source alignment flanges from the chamber. The flanges contain the water cooling connections for the ion sources.



#### **Disconnect water line**

1. Disconnect the water supply lines from the alignment flanges by loosening the Swagelok® connection.



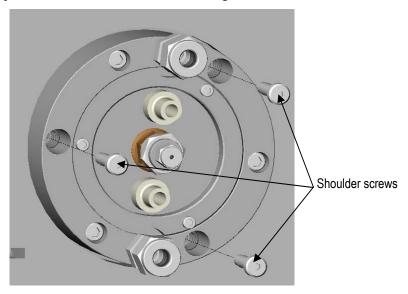
Note: The water lines are not equipped with check valves, that would prevent water spillage from the water contained within the alignment flange and water lines running to the flange.

2. Repeat this procedure for all four tubes.

### Remove flange

1. Locate the three shoulder screws.

These screws are positioned between the ion source alignment screws.



2. Remove the three shoulder screws and their springs.

The alignment flange is now free from the chamber.

### **Install flange**

- 1. Following the service to the chamber, reinstall the alignment flange to the chamber with the three shoulder screws.
- 2. Be sure to place a compression spring onto each screw prior to insertion into the alignment flange.

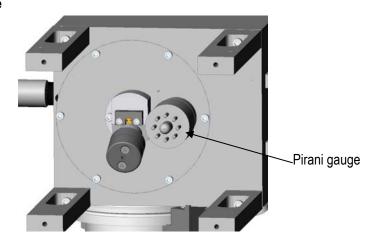
### **Connect water line**

1. Reconnect the water lines as described on page 12.

CAUTION: For proper water flow and function of the pressure/flow switch, the water lines must be connected properly. Failure to do so could result in overheating of the ion sources.



### Pirani gauge tube



The Pirani gauge is mounted in the bottom chamber plate directly behind the bottom laser light detector assembly.

1. Turn the Model 1010 power switch to the off position.

#### WARNING



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 2. Disconnect the power cord.
- 3. Detach the electrical connections from the Pirani gauge.
  The plug connector pulls off the end of the gauge tube.

#### Remove gauge tube

- 4. Loosen the knurled bezel nut that fastens the Pirani gauge into the vacuum bulkhead fitting on the bottom plate.
- 5. Slide the gauge tube out of the bulkhead fitting.
- 6. Clean and inspect the tube.

#### Install gauge tube

- 1. Remove the bezel nut, o-ring and ferrule.
- 2. These parts should be cleaned and the o-ring should be inspected.
- 3. If signs of damage exist, replace the o-ring.
- 4. Thoroughly wipe the bulkhead fitting with acetone and allow to dry.
- 5. To re-assemble the Pirani gauge, place the bezel nut over the smaller diameter end of the gauge tube with the threaded side of the nut facing the open end of the gauge tube.
- 6. Place the ferrule over the gauge tube.
- 7. Coat the o-ring with a light film of Fomblin® and slide over the gauge tube.



- 8. Insert the gauge tube into the bulkhead fitting.
- 9. Tighten the bezel nut hand-tight.
- 10. Reconnect the electrical connection.

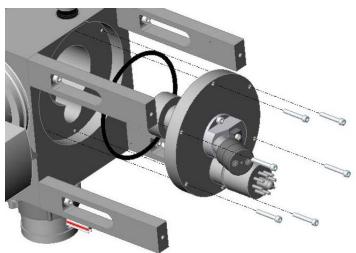
Note: The connector and gauge tube are keyed to ensure that the connector is installed in the correct orientation.

### **Bottom plate**

The bottom plate contains both the Pirani gauge tube and the laser light detector. Both of these components may be serviced without removing the bottom plate. However, when a major cleaning of the chamber is required, the bottom plate should be removed to provide suitable access to the bottom chamber area.

### Remove bottom plate

1. Remove the light source/photo detector (refer to page 34) and the Pirani gauge tube (page 58).



CAUTION: Once the screws holding it are removed, the bottom plate drops out of the chamber. Be sure to support the bottom plate when removing the screws to prevent damage.

- 2. Remove six socket head cap screws holding the bottom plate.
- 3. Remove the o-ring.
- 4. Soak and then clean the plate in acetone and allow to dry.

Depending on the amount of sputtered material on the plate surface, it may be necessary to polish the surface using a fine polishing paste prior to cleaning. Do not use an ultrasonic cleaner because a vacuum epoxy joint may be degraded.

- 5. Carefully inspect the bottom plate for scratches with extra emphasis focusing on the bore into which the light assembly fits.
- 6. If scratches appear on any o-ring sealing surface, remove them and re-clean the plate.



- 7. Wipe the inner bore and horizontal surface of the chamber bottom with acetone or isopropyl alcohol, and allow to dry.
- 8. Inspect the horizontal surface for scratches and nicks.

#### **Install bottom plate**

- 1. Clean the o-ring with methanol or isopropyl alcohol and then apply a thin film of Fomblin®.
- 2. Place the o-ring into the groove in the bottom plate.
- 3. Position the bottom plate into the chamber bottom with the Pirani gauge tube bulkhead fitting facing toward the rear of the Model 1010.
- 4. Insert and tighten the six securing screws.
  - Note: When tightening the screws, apply uniform torque to each screw, drawing the plate evenly upward. The premature tightening of only one screw could result in the plate surface not fully mating with the chamber surface resulting in vacuum leaks.
- 5. Install the Pirani gauge tube (page 58) and transmission light/photo detector assemblies (page 34).

#### Mass Flow Controllers

Two mass flow controllers supply the appropriate argon gas flow to each ion source. Argon flow is automatically controlled through a feedback control loop. There are no internal or external adjustments required for the flow controllers. They must, however, be checked to make sure that they are leak tight, both on the high pressure and vacuum sides.

#### Verify electronics

The functionality of the flow controllers can be evaluated electronically while the instrument is operational (ion sources energized).

- 1. Energize the ion sources.
- 2. Verify the controller functionality using the following:

```
Pin 7 = +15 vdc
```

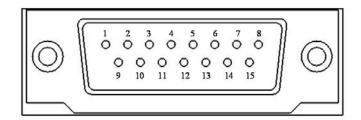
Pin 4 = -15 vdc

Pin 2 = control (0 to 5 vdc proportional)

Pin 5 = actual output (0 to 5 vdc proportional)

Zero volts indicates zero flow. +5 vdc indicates 1 sccm of flow. These values should be approximately equal to the flow indication on the computer's display.





#### **Evaluate flow**

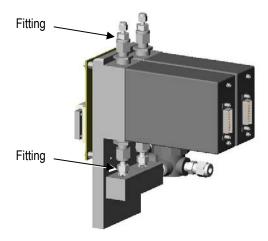
- 1. To evaluate the flow of the mass flow controllers, remove the gas line Swagelok® fitting from the ion source.
- 2. Cap the ion source by replacing this 1/16" Swagelok® fitting with one made not to accommodate a gas inlet (a blank).
- 3. Turn on the power to the instrument.
- 4. Allow the unit to warm up for 30 minutes.
- 5. Initiate the vacuum and wait until the vacuum indication reaches  $0.15 \times 10^{-3}$  torr.
- 6. Submerge the free end of the gas tube in a beaker of methanol.
  - There should be no bubbles emitted from the end of the gas tube.
- 7. To initiate gas flow, enter no value for the ion source voltage.
- 8. Do not enter an ion source current value. This procedure results in a flow of 0.5 sccm.
- 9. Submerge the end of the gas tube in the beaker of methanol.
  - Approximately two bubbles per second should emerge from the end of the tube.
- 10. If excessive bubbling occurs, replace the flow controller.
  - Excessive bubbling is an indication of a failed flow controller, which must be replaced. Such a flow condition would result in the inability of the instrument to achieve a proper operating vacuum.

### Replace mass flow controller

- 1. Shut the valve on the argon cylinder.
- 2. Remove the outlet Swagelok® fitting from the flow controllers.
- 3. Remove the Swagelok® fitting that connects the flow controller manifold to the inlet supply gas tube.
- 4. Slide the instrument to the edge of the counter and locate the screws in the base of the instrument that secure the flow controller bracket.
- 5. Remove the three screws.
- 6. Lift the flow controller assembly from the instrument.
- 7. Remove the two screws that attach the flow controller to the bracket.



- 8. Remove the Swagelok® fitting from the manifold.
- 9. Remove the fittings from the defective controller and re-position them in the appropriate position of the new controller.



- 10. Position the new controller on the bracket and connect its inlet to the manifold.
- 11. Tighten the Swagelok® fitting finger tight then tighten with a 9/16" wrench 1/4 turn.
- 12. Install the two screws that secure the controller to the mounting bracket.
- 13. Place the bracket with the flow controllers into the instrument and secure it from below to the base plate with the three corresponding screws.
- 14. Re-connect both the inlet line to the flow controller manifold and the outlet lines to the ion sources.
- 15. Leak check the instrument, both on the pressure and vacuum sides using the procedure in the following section.

Note: When reconnecting the argon line to the instrument, follow the purge procedure described on page 11.

### **Leak Detection**

#### High pressure

To evaluate the instrument for argon leakage on the pressure side:

1. Turn the Model 1010 power switch to the off position.

# WARNING!



ELECTRICAL HAZARD. High voltages can cause severe injury or death. Turn off the instrument. Wait one minute to ensure that any charged electronic component will discharge before disconnecting the power cord from the instrument.

- 2. Disconnect the power cord.
- 3. Close the cylinder valve and observe for a pressure drop.



- 4. Should a drop exist, open the cylinder valve and apply a soap bubble solution to all of the joints from the initial connection to the cylinder regulator to the inlet to the mass flow controllers.
- 5. Remove any component that exhibits a leak.
- 6. Thoroughly clean the fitting and, where appropriate, remove any remaining Teflon® tape.
- 7. Re-tape the fitting.
  - Wrap the tape clockwise, in the direction of the threads.
- 8. In the unlikely event that a Swagelok® fitting develops a leak, it should be removed and the ferrules carefully inspected.
- 9. Replace any damaged ferrules.
  - Note: To re-tighten an existing ferrule, make it finger-tight, then rotate it 1/4 turn. To tighten a new ferrule, make it finger-tight, then rotate it 1-1/4 turns.
- 10. Re-evaluate the instrument for signs of leakage until the pressure indicated on the cylinder regulator does not drop for five minutes.

#### Vacuum

If the instrument will not pump down to  $2 \times 10^{-5}$  torr or if the instrument is extremely slow in reaching this vacuum, it is an indication of a possible vacuum leak.

NOTE: If the instrument has not been used for or has been open to atmosphere for prolonged periods of time, pumping times will be longer that usual. Pump the instrument repeatedly before concluding that a vacuum leak exists.

- 1. To vacuum leak check the instrument, open the enclosure. (See page 32.)
- 2. Locate the black Viton® tube that connects the Dewar to the chamber.
- 3. Confirm that the Dewar is not being evacuated or baked out before proceeding.
- 4. Remove the Viton® tube from the rotator plate.
- 5. Connect the tube from the helium leak detector to the Dewar pumping port located on the rear of the rotator.
- 6. Parallel pump the instrument in accordance with the recommendation of the helium leak detector manufacturer.
  - Spray helium around the various seal locations (following the leak detector manufacturer's recommended procedure for leak testing) and observe the leak detector for indications of a change in leak rate. A leak rate greater than  $5 \times 10^{-6}$  torr-l/s for helium is an indication that a leak path may exist. Attempt to identify the leak location
- 7. If any leak is detected, disassemble, clean (ensure all seal surfaces are free of dirt, oil, hair and dust) and repair the component.



### **Turbomolecular Vacuum Pump**

To check that the pump is operating correctly, locate the green and red LED's on the pump housing. If the green LED is lit, the pump is operating correctly. If the red LED is lit, the pump may need to be serviced or replaced. Consult the manufacturer's manual or E.A. Fischione Instruments for more information

### **Reinstall Enclosure**

At the conclusion of major service, reinstall the enclosure.

- 1. Position the enclosure in front of the instrument.
- 2. Insert the six studs projecting from the instrument enclosure through the holes in the hinges.
- 3. Fasten with the six hex nuts.
- 4. Re-connect the audible alarm, fan and computer.
- 5. Slowly lower the enclosure into the closed position, making sure that no wires or other objects are caught, trapped, or pinched.

#### **Back Panel**

If the back panel has been removed, it should be replaced at this time.

- 1. Attach the wires that connect the fans.
- 2. Insert the four screws into the back panel, leaving them loose.
- 3. Secure the enclosure to the back panel with three screws then tighten all seven screws on the back of the instrument.
- 4. Install the Dewar knob in accordance with the procedure in on page 48.



# Troubleshooting

# **Diagnosis chart**

**Computer problem** 

Problem	Possible causes and solutions	
Error message on computer	See descriptions on page 67.	
Computer won't start	Check power connection to computer	
Computer won't control ion mill	Check serial (RS232) connection to computer	

Milling problem

Problem	Possible causes and solutions
Difficulty in achieving a plasma	Gas leak (See page 62.) or clean the ion source. (See page 38.)
Unable to produce an ion beam	Gas leak (See page 62.) or clean the ion source. (See page 38.)
Weak ion beam as determined visually	Check the high voltage connections and ion source power supplies (after contacting E.A. Fischione Instruments)
Little or no milling on one or both specimen surfaces	Align the ion source. (See page 47.)

Maintaining temperature problem

Within turning temperature problem		
Problem	Possible causes and solutions	
Insufficient cooling	Check for disengaged or damaged conductor rod. If neither condition applies then bakeout the Dewar. (See page 17.)	
Insufficient cooling after repeated Bakeout	Change the Zeolite® desiccant in the Dewar. (See page 51.)	

Vacuum problem

vacuum problem			
Problem	Possible causes and solutions		
Inability to achieve or maintain a vacuum	Rotator plate is scratched or damaged. (See page 55.)		
	O-ring or Teflon® component associated with the rotator is damaged.		
	Mass flow controller is malfunctioning. (See page 60.)		
	Vacuum leak (See page 63.)		
Takes too long to achieve a vacuum	Check and / or service vacuum pumps		



Overheating problem

Over nearing problem		
Problem	Possible causes and solutions	
Excessive heat build up in cabinetry	Check operation of fans, then clean the air filters as require (See page 30.)	
Overheated ion sources	Check water connections. (See page 12.)	
	Insufficient water flow or malfunctioning pressure/flow switch. (See page 61.)	
	Inoperable water inlet solenoid. Service and maybe replace solenoid. (See page 61.)	

Power problem

Problem	Possible causes and solutions
Ion mill won't turn on	No power, check the powercord. (See page 14.)
	Check the fuses and replace if necessary. (See page 31.)
	Safety interlock on lid is not engaged. Make sure the enclosure is closed. (See page 64.)
No light in the chamber	Check lamps and replace if necessary. (See page 36.)
	Check or service top and bottom light sources. (Consult illuminator manufacturer's manual.)

Leak problem

zum prostum		
Problem	Possible causes and solutions	
Water leak	Check water connections. (See page 12.)	
	Service water inlet solenoid. (See page 61.)	
Gas leak	High pressure argon gas leak. (See page 62.)	
Condensation forms on the Dewar	Bakeout. (See page 17.)	



### **Error messages**

Errors can occur at two levels of severity:

Warning occurs when a variable is slightly outside of the allowable range, but there is a good chance that

it can be brought back within acceptable limits or operation can proceed safely without it

returning.

**Fault** occurs when a variable is severely outside of tolerance and cannot safely return.

#### Warning and fault messages

Vacuum	Motor	Current	Voltage	Gas Flow	Temperature
Warnings	Warnings				
NA	NA	Gun X current is outside of expected range. Abort, Retry, Ignore?	Gun X voltage is outside of expected range. Abort, Retry, Ignore?	Gun X gas flow is outside of expected range. Abort, Retry, Ignore?	Stage temperature has exceeded safeguard setting. Sequence halted.
Faults					
Vacuum system failure. Please contact your service representative.	Motor X failure. Please contact your service representative.	Gun X current is outside of tolerance. Please contact your service representative.	Gun X voltage is outside of tolerance. Please contact your service representative.	Gun X gas flow is outside of tolerance. Please contact your service representative.	Stage temperature is outside of tolerance. Please power down the unit and contact your service representative.

For warnings that include Abort, Retry, Ignore options,

**Abort** will terminate the sequence but leave the program window running.

**Retry** will clear the message but continue normally until another warning occurs.

**Ignore** will continue operation but ignore that particular kind of warning for the rest of the sequence.

#### **Motor errors**

Any time a motor does not complete an assigned task within some time limit, a message will appear informing the user of the condition and the graphical user interface (GUI) will terminate after issuing the safe mode command.

### **Temperature errors**

Any time the stage temperature exceeds 50 degrees, the heater is turned off. The temperature is recorded and stored for later. If the temperature later exceeds the previously recorded value by ten degrees the Sonalert will sound and a message will appear informing the user of the condition and the program window will terminate after issuing the safe mode command.

If the thermal safeguard is enabled and the temperature exceeds the set value during milling, the sequence will be terminated and the user informed.



### Laser

The laser operates independently from the ion mill. It turns on whenever the checkbox for laser threshold is checked and turns off whenever the box is unchecked. This allows the user to test the light penetration of the sample without actually milling it.



# Spare Parts and Consumables

Part No. Description	Reference Drawing	Find Number
341-0001 Fan; Tubeaxial Cooling; AC & DC; 115V; 4.690"	009-0712	7
341-0002 Fan; Filter Assembly; 4.690"	009-0710	19
341-0005 Fan; Filter Assembly; 3.125"; Plastic; 45 PPI	009-0710	18
009-0206 Ion Source	009-0708	45
009-0755 Vent Valve	009-0708	27
322-0004 Clamp; Single Claw Flange; ISO NW 63; Bolt Size M8	009-0708	8
341-0013 Fan; Turbo	009-0708	6
387-0021 O-RING; Viton®; -018	009-0708	15
387-0026 O-RING; Viton®; -235	009-0708	18
407-0018 Pump; Turbo	009-0708	5
445-0005 Vent Valve; 12 VDC	009-0708	10
475-0037 Fitting; O-seal Straight Thread; 5/16-24; Male; 0.062" O.D.	009-0708	46
475-0247 Fitting; 1/4 Straight Thread to 8mm; Elbow Push Lok	009-0708	7
009-0002 Anode Contact Ring	009-0206	12
009-0003 Anode Insulating Ring	009-0206	11
009-0007 Extractor	009-0206	6
009-0010 Extractor Insulating Ring	009-0206	10
009-0016 Anode Insulator	009-0206	14
009-0019 Extractor Screw	009-0206	7
009-0024 Anode Screw	009-0206	8
009-0272 Anode	009-0206	13
009-0276 Extractor Spacer Set	009-0206	15
009-0283 Cathode	009-0206	2
387-0002 O-RING; Viton®; -011	009-0206	18
387-0009 O-RING; Viton®; -224	009-0206	20
387-0014 O-RING; Buna; .722 x .024	009-0206	17
387-0028 O-RING; Buna; .650 x .024	009-0206	19
500-0073 Power Supply; +24V, 300W	009-0679	2
500-0105 Power supply, 100W, +5,+15,-15V	009-0679	3
009-0696 PCB Assembly; Stepper Motor Network Module	009-0679	4
009-0694 PCB Assembly; Network Backplane	009-0683	4
	-	



Part No. Description	Reference Drawing	Find Number
009-0700 PCB Assembly; Ion Gun Network Module	009-0678	7
009-0750 Cable, Ion Gun PCB to Ion Gun	009-0678	13
372-0013 Lamp; Halogen; 5.2V; 0.85A	009-0765	7
387-0003 O-RING; Viton®; -014	009-0765	4
387-0024 O-RING; Viton®; -116	009-0765	8
372-0013 Lamp; Halogen; 5.2V; 0.85A	009-0764	5
387-0003 Lamp; Halogen; 5.2V; 0.85A	009-0764	2
387-0024 O-RING; Viton®; -116	009-0764	3
387-0027 O-Ring; Viton®; -238	009-0767	8
444-0001 Vacuum; Gauge Tube	009-0767	5
009-0762 Assembly, Vacuum Gauge Controller, 115VAC	009-0768	6
327-0002 Mass Flow Controller	009-0768	1
009-0378 Low Angle Gear	009-0381	2
458-0003 Tweezer; Dumont #5; Antimagnetic; Stainless Steel	009-0381	5
477-0018 Grid Storage Box	009-0381	6
009-0413 Low Angle Bottom Plate	009-0378	1
009-0414 Top low Angle Plate	009-0378	2
387-0004 O-Ring; Viton®; -015	009-0297	5
009-0081 Drive Shaft	009-0297	10
009-0360 Conductor Rod	009-0297	12
387-0063 O-Ring; Viton®; -337	009-0620	2



# **Applications**

Many different materials systems have been prepared for high resolution TEM or STEM analysis using a combination of mechanical thinning, ion milling using the Model 1010, and then plasma cleaning using the Model 1020 Plasma Cleaner. Plasma cleaning for a few minutes in an inductively coupled plasma of Ar/25% O<sub>2</sub> removes residual carbonaceous contamination that may interfere with subsequent analysis.

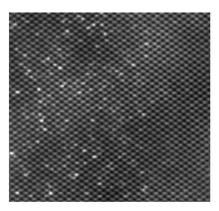
The achievable milling rate will be a function of both material type, specimen geometry and milling conditions. Extractor voltage and current should be chosen to avoid damaging a specimen through amorphization, ion implantation, or overheating. It is recommended that liquid nitrogen cooling always be used to avoid overheating. Typical instrument parameters for milling materials are summarized below.

Material	Extractor Voltage	Extractor Current	Temperature
	(kV)	(mA)	(Degrees C)
Polymers	<2	5	< -90
Metals	2 - 3	5	< -90
Silicon	3.5	5	< -90
Ceramics	4 - 6	5	< -90

After wedge polishing, a sample of Sb doped Silicon was ion milled using liquid nitrogen cooling and then plasma cleaned. Liquid nitrogen cooling during ion milling promoted retention of the dopant distribution. Annular dark field (ADF) STEM images of the electron transparent material were acquired with atomic resolution. These images could be used to quantify the dopant concentration.



### Quantitative Mapping of Sb in Doped Silicon



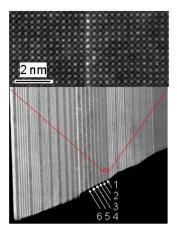
- JEOL 2010F
- Filtered image
- Gaussian smoothed
- Thickness corrected
- Sb brighter
- · Si darker

P. M. Voyles, D. A. Muller, J. L. Grazul, P. H. Citrin, H. - J. L. Gossmann,

"Atomic - Scale Imaging of Individual Dopant Atoms and Clusters in Highly n - type Bulk Si", Nature, Vol. 416, 25 April 2002, p. 826.

A similar protocol was used to prepare a specimen with multilayers of La and Sr titanates. The results are included in the following two images. After wedge polishing, ion milling with liquid nitrogen cooling and plasma cleaning; the electron transparent region yielded a  $0.4~\mu m$  field of view. Images were acquired at the interphase interface with resolution on the atomic scale, specifically 1.9~Angstroms.

### Imaging of LaTiO<sub>3</sub> Multilayers

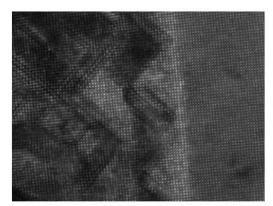


- LaTiO<sub>3</sub> multilayers in SrTiO<sub>3</sub> imaged in JEOL 2010 F
- Material (in box) thin enough for ADF lattice imaging over 0.4 µm field of view
- Specimen prepared by wedge polishing, followed by low-angle milling at LN2 temperature

Courtesy of D. A. Muller, J. Grazul, A. Ohtomo, H. Hwang of Bell Labs, Lucent Technologies



## SrTiO<sub>3</sub> / LaTiO<sub>3</sub> Interface Structure

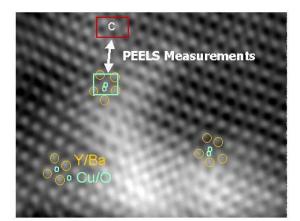


- JEOL 2010F
- 1K X 1K image
- 128 ms / pixel
- Gaussian smoothed
- · La brighter (left)
- · Sr darker (right)
- Resolution of Sr vs. Ti atoms is 1.9 Angstroms

Courtesy of D. A. Muller, J. Grazul, A. Ohtomo, H. Hwang of Bell Labs, Lucent Technologies

Another example involves wedge polishing, ion milling with liquid nitrogen cooling, and plasma cleaning of a specimen consisting of YBCO superconducting material. Atomic resolution ADF STEM images were acquired at grain boundaries within the electron transparent region. Situated at the boundaries, the Ca dopant acts to couple the superconducting current between adjacent grains. Liquid nitrogen cooling during ion milling is critical to retaining the distribution of Ca.

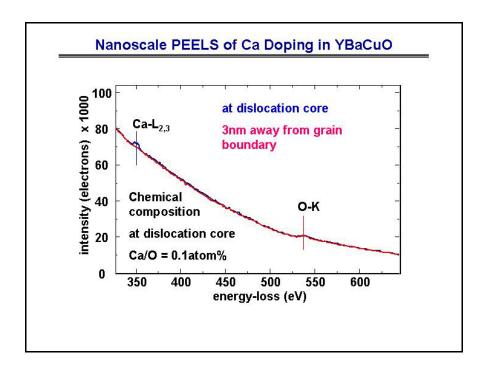
### ADF Imaging of Ca Doped YBaCuO Superconductor



Courtesy of Gerd Duscher and Julia Luck, ORNL



Once the grain boundary itself and an adjacent area 3 nm away were targeted, PEELS measurements were made of the Ca concentrations. An enrichment at the grain boundary was identified existing on the nanoscale.



## References

General references on ion milling theory and practice include:

- **P.J. Goodhew**, "Final thinning: ion beam thinning and mechanical techniques", in Thin Foil Preparation for Electron Microscopy, Elsevier, 1985.
- **D.B. Williams and C.B. Carter**, "Specimen Preparation" in Transmission Electron Microscopy: A Textbook for Materials Science, Plenum Press, New York, 1996.

