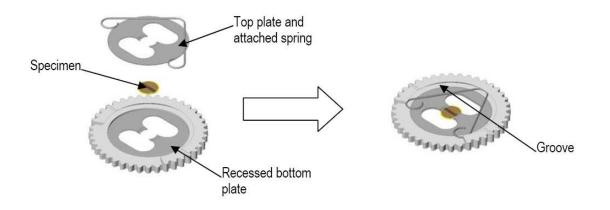
Instructions for using the Fischione 1010 Ion Mill

Before using the Ion Mill check the **log-in book** to make sure there is no trouble with the instrument as noted by the previous user.

Loading TEM Specimens

Note: Specimen thickness should be less than 100 µm.

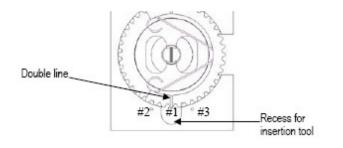
1. Place the TEM specimen into the recessed bottom plates (attached to the specimen gear) with manual or vacuum tweezers. See Below



2. Position the top plate and attached spring over the bottom plate.

3. Compressing the spring with manual tweezers, insert the left, top, and finally right leg of the spring into the available groove.

4. If an XTEM specimen is to be oscillated and milled, position the double line on the gear so it points to the position 1 shown below.



Specimen Cooling

1. Make sure the Dewar conductor rod is engaged. To engage rotate to dewar knob clockwise located behind the dewar clockwise as viewed from the instruments rear panel. Rotate until its pin contacts the pin stop. When the specimen stage is heated above 10 degrees C and the vacuum is less than 8x10-4 torr, the Dewar valve opens.

2. Fill the Dewar flask with liquid nitrogen.

Starting the Program

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

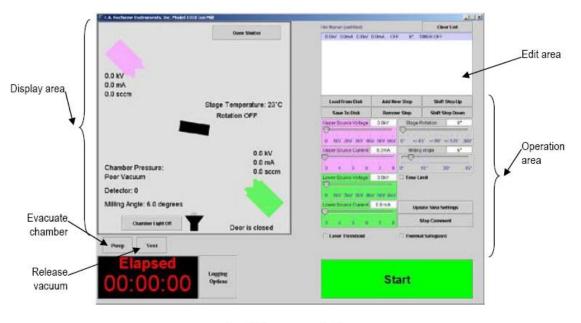
Note: "Click" means to press the left button of the touch pad on the computer.

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.

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.

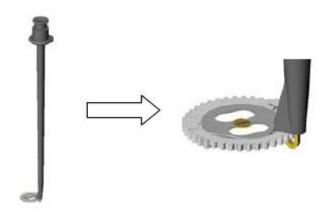
Click **Vent** to release the vacuum.

The chamber will not vent until the temperature rises to $(+10^{\circ}C)$.

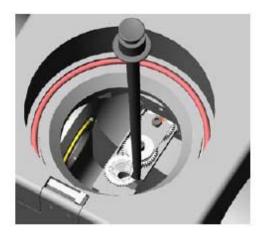
Inserting the specimen

1. If either the **laser** or the **microscope** has been left in place, remove them. Removing the microscope requires that you first <u>raise the</u> <u>microscope</u> by turning the focus knob clockwise until it clears the chamber cover, then loosen the knob at the rear of the microscope mount and gently swing it away from the cover.

2. Grasp the specimen gear with insertion tool.



3. Click **Open Shutter** to open the shutter below chamber insert.



4. Insert the specimen into the specimen chamber after removing the chamber insert.

<u>Microscope</u>

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

1. Turn **ON** the illuminator and the monitor.

2. Center the high magnification microscope with respect to the chamber insert by rotating the knobs of the X-Y translation stage.

3. Slowly lower the microscope until its lens is approximately 1 in. (25.4 mm) above the viewing window in the chamber insert. The microscope should be grasped while the height adjustment knob is rotated counterclockwise, and then lowered. Once the microscope is in position, the knob can be rotated clockwise.

4. Choose the magnification level by rotating the zoom adjustment.

5. Focus the microscope by rotating the coarse and then the fine focus knob.

6. Select an area of interest by further adjusting the knobs of the X-Y translation stage. Readjust the illumination level, magnification and focus as required.

Endpoint determination (Laser)

1. If the endpoint determination capability is used, install the laser.

2. Close the shutter on the front viewing window and click **Open Shutter**.

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.

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

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

Start milling

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

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

2. Close the shutter on the front window. Click **Close Shutter** if the laser is not used.

3. Wait until a vacuum of 2x10-5 torr has been achieved.

4. Click Start button.

Removing the Sample

1. Once the sample is complete vent the chamber by clicking on the **vent** button.

2. Remove the microscope or the laser from the chamber port.

3. Using the insertion tool, remove your specimen.

4. Click **Pump** in the Ion Milling program. Verify that that the vacuum level is 2X10-5 torr.

5. Sign out in the log book.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000 Room / facility emergencies: Contact Public Safety (609) 258-1000 Issues related to the instrument:

- 1. Contact IAC Staff.
- 2. If unsure, leave system as is.

3. Try to power down the system.

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

Nan Yao: Office (609)258-6394; Cell (908) 922-2236 Email: <u>nyao@princeton.edu</u> John Schreiber: Office(609)258-0034; Cell (215) 431-4670 Email: <u>js51@princeton.edu</u> Paul Shao : Office (609)258-3851; Cell (847) 721-086 Email: <u>pshao@princeton.edu</u>