PIPS II

Precision Ion Polishing System

Owner's Manual and User's Guide

Part Number 695.82001

Revision 4.0

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Safety Information

This chapter presents a summary of the safety symbols throughout this manual. Gatan, Inc., recommends following all safety precautions to prevent harm to yourself or the equipment. Please follow all warnings marked on the equipment as well.

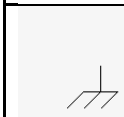
 **CAUTION - Documentation must be consulted in all cases where this symbol is marked.**

IMPORTANT - For Regulatory Compliance and Safety information and instructions please refer to the *Regulatory Pamphlet* provided with this product. Review this document in full before installing and operating this product.

Symbols and Attention Symbols

You must be aware of safety when you install and use this system. This Guide provides various procedures that require careful attention to precautions.

SYMBOL	REFERENCE	DESCRIPTION
	IEC 60417-5031 (2002-10)	Direct current
	IEC 60417-5032 (2002-10)	Alternating current
	IEC 60417-5033 (2002-10)	Both direct and alternating current
	IEC 60417-5017 (2006-08)	Earth (ground) TERMINAL

	IEC 60417-5019 (2006-08)	Protective Conductor Terminal
	IEC 60417-5020 (2002-10)	Frame or chassis TERMINAL
	IEC 60417-5007 (2009-02)	On (Power)
	IEC 60417-5008 (2009-02)	Off (Power)
		Caution, possibility of electric shock
	IEC 60417-5041 (2002-10)	Caution, hot surface
	ISO 7000-0434B (2004-01)	Caution - documentation must be consulted in all cases where this symbol is marked
		Caution, magnetic field.

Product Safety Information

Review the following precautions to avoid injury and prevent damage to this product, or any products to which it is connected. To avoid potential hazards, use the product only as specified. Read all safety information provided in the component product user manuals and understand the precautions associated with safety symbols, written warnings, and cautions before accessing parts or locations within the unit. Save this document for future reference. Follow all warnings and instructions marked on the equipment. Ensure that the voltage and frequency of your power source matches the voltage and frequency

inscribed on the equipment's electrical rating label. Never push objects of any kind through the openings in the equipment. Dangerous voltages may be present. Conductive foreign objects could produce a short circuit that could cause fire, electrical shock, or damage your equipment.

Danger: Disconnect power before replacing fuses and only use value specified on the product's rating label.

Do Not Operate Without Covers: To avoid electric shock or fire hazard, do not operate this product with any removed enclosure covers or panels.

To Avoid the Risk of Electric Shock: Do not operate in wet, damp, or condensing conditions. When supplying power to the system, always make connections to a grounded main. Always use a power cable with a grounded plug (third grounding pin).

Do not operate in wet, damp, or condensing conditions.

Disconnect all external power connections before servicing.

Should a leak occur, remove power from PIPS. Use paper towels or Kem wipe to clean up the spill.

Warning: To avoid electrical hazards (heat, shock and/or fire hazard), do not make connections to terminals outside the range specified for that terminal. See the product user manual for correct connections.

Electronic components on printed circuit boards are extremely sensitive to static electricity. Ordinary amounts of static electricity generated by your clothing or work environment can damage the electronic equipment.

When installing the board in a system, you must use anti-static grounding straps and anti-static mats to prevent damage due to electrostatic discharge.

To avoid injury, fire hazard, or explosion, do not operate this product in an explosive atmosphere.

Preface

Copyright and Trademarks

Disclaimer

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Support

Gatan, Inc. provides free technical support via phone, fax, and electronic mail. To reach Gatan technical support, contact the facility nearest you, or send electronic mail to help@gatan.com.

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Returns

If there is a need to return equipment to the factory, please call Gatan to obtain a Returned Merchandise Authorization Number (RMA #). This RMA number must appear on your shipping document, to help in tracking and to ensure that proper action will be taken to repair or replace your equipment.

Table of Contents

List of Figures

List of Tables

1. Overview

The Model 695 Precision Ion Polishing System (PIPS II ™) is a compact, bench-top system designed to produce high-quality TEM specimens with exceptionally large, clean, electron-transparent areas. This manual was written for PIPS II software version 1.6.

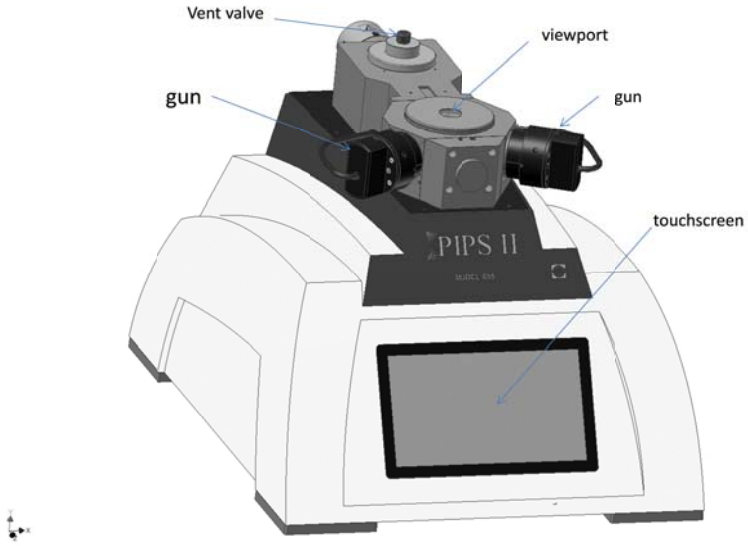

Figure 1-1 PIPS II, basic system, front view.

1.1. Features of the Precision Ion Polishing System

1.1.1. *Dual Ion Source*

Ion polishing is done by two variable-angle, miniature Penning ion guns (PIGs). The operating angle of each gun, ± 10°, is independent of one another and both have the ability to center the beam onto the specimen at any angle within this range. The PIGs incorporate powerful rare-earth magnets and are capable of very high thinning rates. Each gun is mounted in a universal joint so that the x- and z-alignment drives can be used to center the beams on the specimen. These features make it possible to thin specimens at very low angles in a reasonably short time. Motorized operation of beam tilt angle is available as an optional feature.

1.1.2. *Optimum Gun Design*

The gun's ion optics has virtually eliminated cathode-aperture erosion and, as a result, gun maintenance is reduced, specimen contamination from the ion

guns is minimized, and gun consumables have been eliminated. The new focus electrodes in each gun have improved the low energy spot size, keeping the spot size approximately constant across all beam energies. This results in dramatically faster milling rates at low energy, such that it is now practical to mill to perforation at energies as low as 100 eV.

1.1.3. *Gas Flow Optimization*

The optimum gas flow for all beam energies is calibrated at the factory, and may be selected by using the automatic gas flow option. The gas flow of each gun may also be set manually.

1.1.4. *Compact Vacuum System*

Specimen contamination is reduced with an oil-free vacuum system consisting of a molecular drag pump (MDP) backed by a 2-stage diaphragm pump (DP). Additionally, a liquid-nitrogen trap is available to further reduce contaminants and water vapor.

1.1.5. *Touch-screen Interface*

Operation of the system is controlled by the user via a touch-screen interface, which is customer selectable between several languages.

1.1.6. *Versatile Sample Holders*

The Gatan specimen post, for single-sided milling, and the DuoPost, for double-sided milling, eliminate transfer of material onto the specimen by secondary sputtering from the specimen platform and provide excellent thermal contact with the specimen to prevent specimen overheating. Lastly, both specimen posts allow the specimen to have an unobstructed view of the ion beam and thus ion polishing can be performed at angles approaching 0°.

In order to allow for centering the thin section at the rotation axis the sample mount includes a manual x-y stage. This allows the user to compensate for a sample that is mounted off-axis in the post.

1.1.7. *Stereo Microscope*

An optional optical stereo microscope is used to inspect the specimen in its working position at any time during the thinning process to achieve very precise control over the final stage of specimen thinning. This feature is especially important for insulators and semi-conductors since these materials are transparent to light and the interference-fringe technique can be used to control the final specimen thickness in the region of interest to an accuracy of about ±10 nm.

1.1.8. *Digital Zoom Microscope System*

The Digital Zoom Microscope is a system option. This system uses a digital camera connected to an external PC. The camera is mounted on the system and allows for in-situ observation of the sample. In addition, the supporting software allows for manual/automated acquisition of images, typically one automated image per rotation. The external PC can also be used for remote control of the PIPS II system, via remote desktop type software.

1.1.9. *Autoterminator*

This can be provided as an optional feature and uses the amount of light transmitted through the sample. Milling is stopped when this reaches a previously set value.

1.1.10. *Whisperlok™ Stage*

Quick specimen exchange (<30 sec) is achieved using a miniaturized version of Gatan's pneumatically controlled Whisperlok™. The specimen can be easily transferred and viewed at frequent intervals during the final moments of the thinning process.

1.2. Main Work Chamber

Figure 1-2 is a top view of the PIPS II main Work Chamber. The figure shows the right and left PIGs. The Airlock cover is removed to reveal the main Airlock O-ring and a top view of the specimen mount.

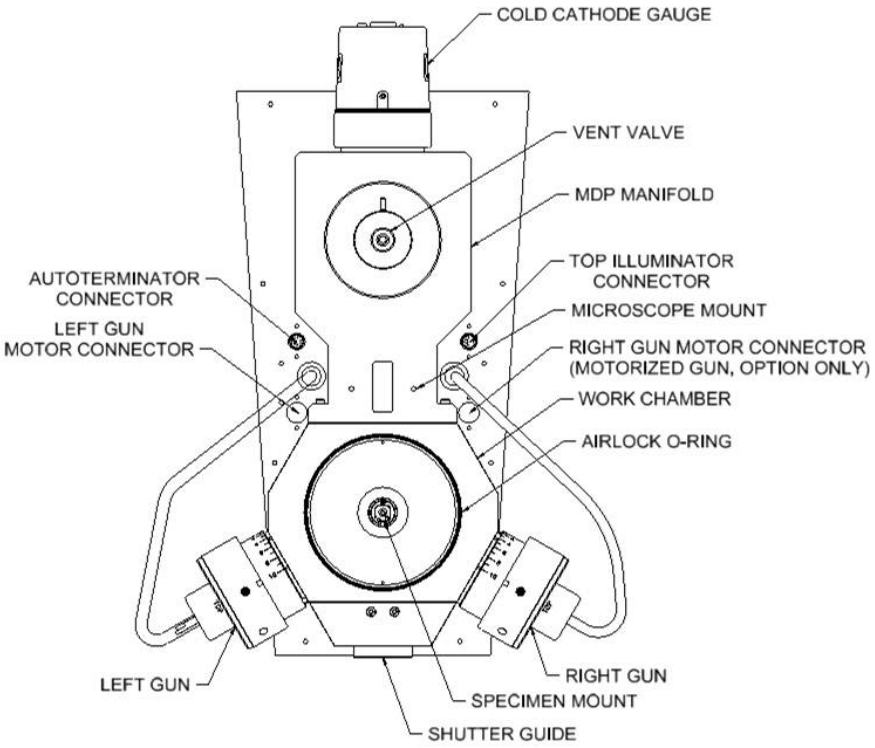

Figure 1-2 Work chamber, top view.

Figure 1-3 is a cross-sectional view through the main Work Chamber of the PIPS II. The Airlock cover is in place with the specimen in its working position at the center of the Chamber.

Specimens are mounted on posts that plug into the specimen mount and can be milled on both surfaces with proper orientation of the PIGs. One of the PIGs is shown inclined at a positive angle (+10°) to the horizontal (beam incident to the top surface of sample). By simply grasping the gun knob and rotating it, the gun angle can be reduced down through 0° continuing on to a negative angle (-10°, beam incident to the bottom surface of sample).

The Shutter is shown in its inserted position, which prevents sputtered material from depositing on the specimen Viewing Port.

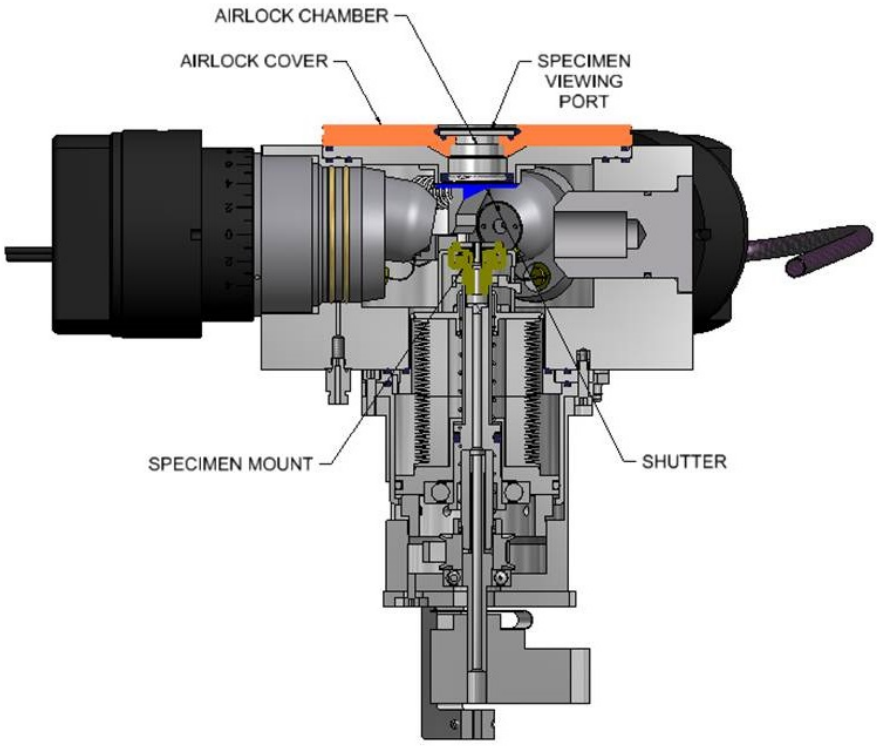

Figure 1-3 Work chamber, cross-section view.

1.3. Vacuum System

PIPS II has a compact, oil-free vacuum system consisting of a molecular drag pump (MDP) backed by a 2-stage diaphragm pump (DP). The vacuum system is designed to hold vacuum when the power is turned off. The working vacuum can be reached very quickly when the power is resumed.

1.3.1. *The Pumping system*

The MDP has an argon pumping speed of 80 L/sec. It is in series with a 2-stage diaphragm pump (DP) that maintains a backing pressure for the MDP of less than 10 Torr and a chamber base pressure in the 10^{-6} Torr range. The pumping time from atmosphere to near the base pressure is typically less than 15 min. The console is cooled by a single fan mounted on the rear panel that directs air onto the MDP.

1.3.2. *The Pumping Manifold*

The Pumping Manifold contains the cold-cathode gauge tube and the MDP, which is offset from the Work Chamber to minimize any possibility of debris

falling into the pump. Pressure is monitored by the cold-cathode gauge tube, which will not turn on unless the MDP is close to its normal running speed.

1.3.3. *Airlock Vacuum*

The Airlock vacuum is controlled by three solenoid valves (see Figure 1-4). The VAC valve in conjunction with the LL (loadlock) valve evacuates the Airlock. The Vent valve in conjunction with the LL valve vents the Airlock.

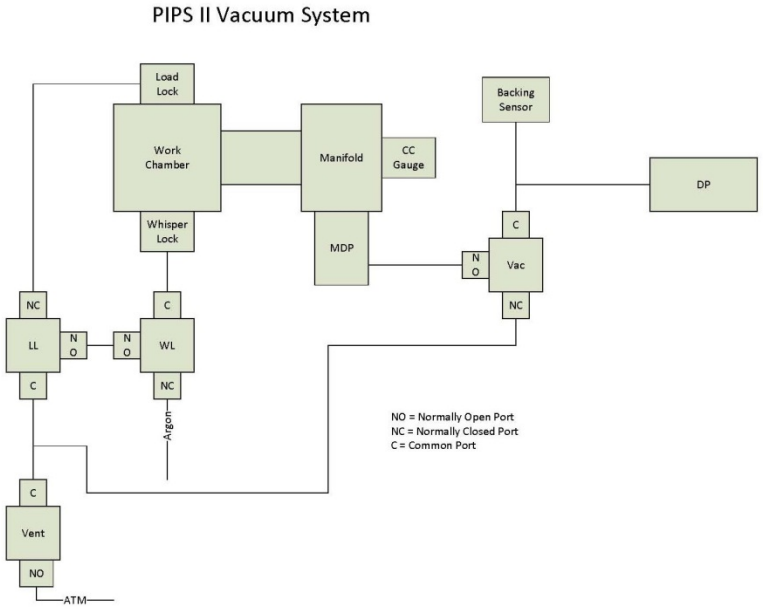

Figure 1-4 Vacuum system.

1.3.4. *Gas Manifold*

The gas manifold combines all of the solenoid valves except for the VAC valve onto a single manifold. This includes valves for pneumatic controls as well as airlock vacuum control. All of these valves are of the same type; 3-way normally closed, 15 mm valves from Clippard Minimatic. These valves have an LED which indicates when they are active. The mass flow controllers and a gas regulator are also mounted on the gas manifold.

Fittings with captive o-rings are inserted into tapped holes on the manifold and gas and vacuum tubing is connected to the fittings. It is critical that these fittings are not over-tightened, or the manifold material can be cracked. When tightening a compression fitting, be sure that a second wrench prevents the fitting from turning.

Energy Isolation

Prior to service, remove the AC line cord and attach a suitable lockout/tagout device, such as RS Hughes Co. part number 65674 or the like. Gas-control System

The Gas-Control system controls the Argon gas supply to the ion guns, an alternate gas input for the guns, the Whisperlok piston, and the pneumatic shutter. The Gas-Control System consists of a pressure regulator and four normally closed three-way solenoid valves. Figure 1-5 shows the Gas-control system. The gas supply to the guns is controlled by a regulator.

The alternate gas input for the guns may be used if a different gas is desired for the ion guns than is used for the pneumatic control. For example, Xenon may be used for the guns while Argon is used for pneumatic control. This minimizes the amount of the much more expensive Xenon that is used.

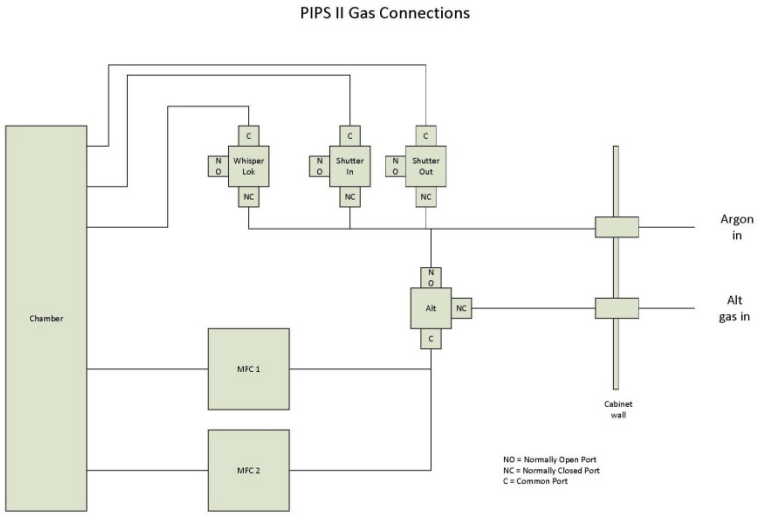

Figure 1-5 Gas-control system.

Gas Supply to the Guns

The PIPS requires a clean, high purity (99.998%) Argon supply at 25 psi (1.72 bar). The Argon gas for the ion guns is regulated by a pressure regulator that reduces the incoming gas supply from 25 psi down to about 7.5 psi. Two O-rings form vacuum seals in the gun housing and the ionizing gas is fed into the guns between the O-rings. The Alt valve (AG) when activated connects the alternate gas input to the guns, however, Argon gas must still be supplied to the Ar input to provide pneumatic control.

Gas Supply to the Whisperlock™

The Whisperlok assembly is controlled by a normally closed three-way solenoid valve (WL). When WL valve is energized, Argon pressurizes the Whisperlok assembly and lowers the piston. When the power to WL is

switched off, the gas pressure is cut and the piston is raised. This means that in the event of a power failure, the specimen will automatically be raised into the Airlock. In addition, the LL and the VAC valves are configured so that the DP evacuates the bellows. This increases the pressure raising the Whisperlok.

Gas Supply to the Pneumatic Shutter

The pneumatically-operated Shutter is designed to minimize sputtered material from depositing on the specimen Viewing Port and is controlled by the 3-way valves, SI and SO. When the power to SI is switched off, the shutter piston cylinder is vented and the Shutter is opened by the action of a coil spring mounted behind the Shutter piston. This means that in the event of a power failure the Shutter will automatically retract and allow the specimen to rise into the Airlock. When power to SO is activated, the shutter piston is pneumatically driven outward, in addition to the action of the spring.

1.4. Electrical system

The total power consumption of the PIPS II is relatively small. The beam energy has been limited to 8.0 keV as the best compromise between maximizing the specimen thinning rate and minimizing specimen radiation damage and heating effects.

1.4.1. *Air Flow*

The cabinet interior is cooled by a single fan, mounted on the rear panel directing air onto the MDP. The air flow to the fan and the slots on the rear panel should not be blocked since this may cause the MDP to overheat and shut down, possibly damaging other electrical components in the instrument.

1.4.2. *DC Power Supply*

All power to the system is supplied by a 24 VDC power supply connected to the power main input. It accepts universal power input (90-240 VAC, 50-60 Hz). This supply has an internal fan that is activated when the internal temperature exceeds its set point.

1.4.3. *HV Power Supply*

The high voltage (HV) power supply provides the ionization voltage, the acceleration voltage, and the focus voltage for the ion guns. The three voltages are programmed with a defined relationship to give the optimum beam parameters for each beam energy. This supply also provides for fast switching of the guns during sector milling.

The HV supply also measures the current to each electrode and the output voltages. This can be used to determine if the guns are operating properly.

1.5. The Standard Operating Mode

Gatan recommends the PIPS II be left running continuously 24 hr a day, seven days a week. This will insure optimum performance of the vacuum system and the ion guns and purge time will be minimized.

2. Installation

Although the PIPS II is a small, bench-top system, it is relatively heavy (38 kg) and should not be lifted by a single person. It can be lifted safely by two people who are experienced in the techniques of lifting heavy objects. Alternatively, proper laboratory lifting equipment should be used. The size of the PIPS is 20” (W) x 23” (L) x 30” (H). The size of the vacuum pump is 7” (W) x 9” (L) x 9” (H), and the weight is 15 lb.

2.1. Site Requirements

The PIPS II requires a sturdy bench top area approximately 1.2 m (48 in.) wide by 60 cm (23.6 in.) deep by 72 cm (28.3 in.) high, located near a power outlet and a source of 99.998% purity argon (Grade 4.8). If the cold stage option is installed, the dewar will often interfere with any cabinet mounted on the wall above the system, therefore, a bench space without a cabinet would be required. A desktop computer will be used with the camera system, and a 23” monitor, keyboard, and mouse will occupy desk space next to the PIPS II, and a small tower case will sit on the floor. A molded power cord is supplied with the PIPS II to fit the local standard power socket. If the power cord supplied is not suitable, the plug should be replaced with a suitable one. Before connecting the new plug, make sure the voltage requirement conforms to that specified on the label on the rear panel of the PIPS. The wiring color codes should conform as shown:

Live	Black or Brown
Neutral	White or Blue
Ground	Green or Green/Yellow

Electrical Ratings:
100-120/220-240 VAC
50/60 Hz
0.5/0.25 A

A 3 m nylon tubing with compression fittings (1/8 inch Swagelock) is supplied to connect the argon regulator to the gas input of the PIPS II, located on the rear panel of the console. The PIPS II is air cooled and does not require connection to a water supply.

The system typically uses about 150 Watts when in operation, 130 Watts when idle, and peak power usage is about 300 Watts.

2.2. Unpacking

Be sure to have the necessary personnel or use proper laboratory lifting equipment when unpacking the PIPS II.

1. Inspect the exterior and interior of the shipping box for damage.

Note or photograph any external visible damage.

Open the boxs and inspect for any internal damage. If any damage is observed, the Shipper should be informed immediately.

2. Remove the computer (if applicable) and accessory boxes.

If a camera system was purchased with the PIPS II, the computer and accessories are packed in a smaller box on tip of the main box. Lift the cover off of the top box and remove the computer and accessories.

Lift off the top layer of support foam and unfold the protective plastic cover.

3. Lift the box lid, then the internal box off of the PIPS II.

The internal box for the PIPS II is designed to lift off of the PIPS II, do not cut open the top of this box. Lift off the top layer of support foam and unfold the protective plastic cover.

4. Lift the PIPS II off of the support foam and place on a sturdy surface such as a bench top (follow lifting instructions above).

5. Remove the diaphragm pump from the small box, and the camera system (if applicable) from the larger box.

If a camera system was not purchased, the accessories will be in a smaller internal box.

6. Keep all packing material.

Replace all packing material into the shipping box and store in the event the instrument must be returned for factory repair or maintenance.

7. Verify accessory items.

Inspect the contents of the accessory boxes against the items ordered and those listed on the packing list.

If there are any discrepancies, inform your local Gatan Sales Office immediately.

2.3. Installation

Place the PIPS II on an appropriate work bench, close to a suitable power outlet and a cylinder of compressed argon. Then proceed with the setup.

Figure 2-1 View of connections on rear of cabinet.

2.3.1. *Setup of the Diaphragm Pump (DP)*

1. Connect the diaphragm pump cable to the pump connection at the rear of the PIPS II and to the two connectors on the diaphragm pump. Tighten screw on large connector on the diaphragm pump.

2. Connect the vacuum line between the rear of the PIPS II at the Quick Release Fitting and to the fitting on the DP.

2.3.2. *Connecting the cables*

1. Connect the Cold Cathode gauge cable (695.04203) from the CC gauge to the connector marked CC Gauge on the back of the instrument.

2. In the cold stage option is installed (selected models only), connect the dewar cable (695.04204) between the dewar and the connector marked Dewar on the back of the instrument.

2.3.3. *Connecting the Argon Source*

NOTE: Be sure the argon supply is properly secured.

1. Adjust your argon tank regulator to 25 psi (1.72 bar).

2. Connect gas-supply hose. Connect one end of the nylon gas-supply hose to the regulator on the cylinder bottle.

3. Purge the gas-supply hose. Crack open the main valve on the cylinder to purge the gas-supply hose.

4. Connect hose to the console. With the argon flowing, connect the hose to the gas-inlet port on the rear panel of the PIPS II. Do not over tighten the fitting as this may fracture the hose.

5. Check the pressure. Turn off the main gas valve and check that the pressure reading on the high pressure side of the regulator does not decrease over a 5min period. This will verify that the gas-inlet line is not leaking.

6. Turn on the main gas valve again to restore the argon supply.

2.3.4. *Setting up the Camera System*

If a camera system is included with the system, it needs to be mounted and connected to the PIPS II and the imaging PC.

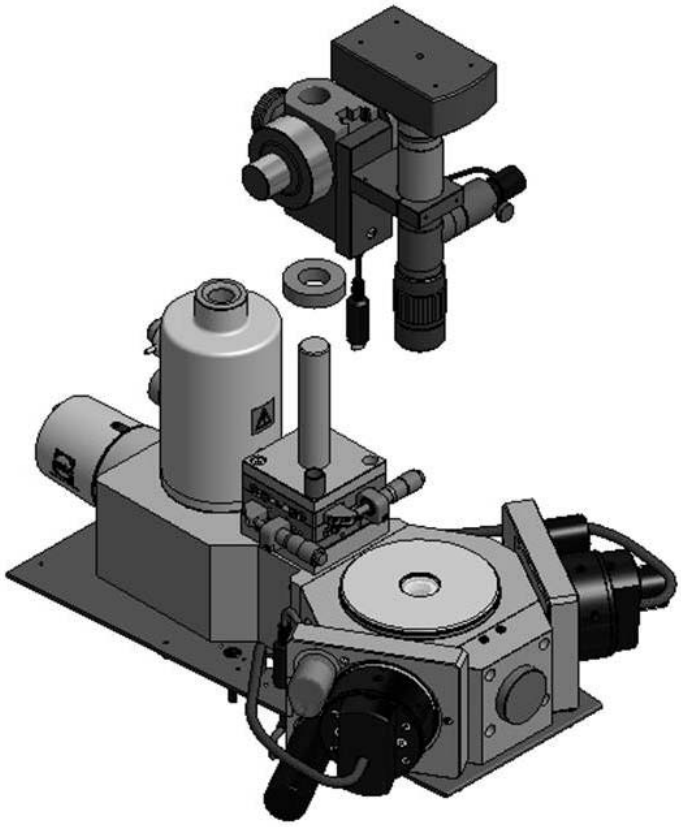

Figure 2-2 Camera system mount onto the PIPS II.

1. Unpack the camera system.

2. Place the plastic washer onto the vertical post on the Manifold.

3. Engage the hole in the bottom of the rack and pinion mount into the vertical post on the Manifold.

4. Lower the camera system to its working position.

5. Plug the illuminator cable into the top illuminator connector on the right hand side of the cabinet, and insert the illuminator into the port on the right side of the microscope column. Gently tighten the knob to secure the illuminator.

6. Unpack the imaging computer and monitor. Connect the monitor, keyboard, and mouse to the appropriate port on the back of the PC.

7. Connect the USB cable from the digital camera to an available USB port on the PC.

8. Connect the camera trigger cable from the digital camera to the camera trigger port on the rear panel of the PIPS II.

9. Connect the crossover Ethernet cable from the second Ethernet port of the PC to the Ethernet port on the rear panel of the PIPS II.

This port should be labeled "To Ion Polisher" on the back of the PC. Note that a standard Ethernet cable will not work, this must be a "crossover" type Ethernet cable (supplied with the system). The second Ethernet port on the PC is on a PCI add-on card. The Ethernet cable must be plugged in to the proper port of the PC in order for the PC to communicate with the PIPS II system.

10. Turn on the PC, wait for Windows to load.

11. Start DigitalMicrograph. The camera system is now ready to use.

2.3.5. *Mounting the Stereo Microscope*

If a microscope was purchased with the system, it needs to be mounted and centered.

1. Properly engage the microscope slide into the pivoting slide on the Manifold.

2. Lower the microscope to its working position.

Rotate the focus knob CCW to lower the microscope to its working position where it can pivot to the left or right rest position.

3. Plug the microscope illuminator into the Reflection Illuminator power jack.

4. Plug the PIPS II into the main power socket. Do not load a specimen post just yet.

5. Rotate the microscope objective turret to the 2x position.

Adjust the focus knob to clearly view the hex shape at the top of the piston.

2.3.6. *Aligning the Stereo Microscope*

The microscope is shipped pre-aligned so the hex shape at the top of the piston should appear concentric with the microscope field-of-view. Keep in mind the field-of-view is a true image such that if a gap exists between the post and the field-of-view at the 6 o-clock position, the microscope must be shifted toward the rear of the PIPS II for centering.

NOTE: Alignment should be performed only when the PIPS II is under vacuum and the piston can be lowered into the chamber.

If alignment is necessary, the tools required are a 1.5 mm and a 3.0 mm hex wrench and the small spanner wrench all supplied in the accessory kit.

To Setup for Alignment

1. **Insert a copper specimen post into the top of the piston to use as a target.**

2. **Turn on the Reflection Illuminator.**

3. **Determine the direction the microscope must move to properly center the target.**

Front-to-back alignment

1. **Loosen sufficiently the two socket-head screws on the pivoting slide.**

Use the 3.0 mm hex wrench to loosen the two screws. This will permit the microscope to slide back and forth with minimal side motion.

2. **Center the specimen post; tighten the two screws.**

Be sure the two socket-head screws are tight before proceeding.

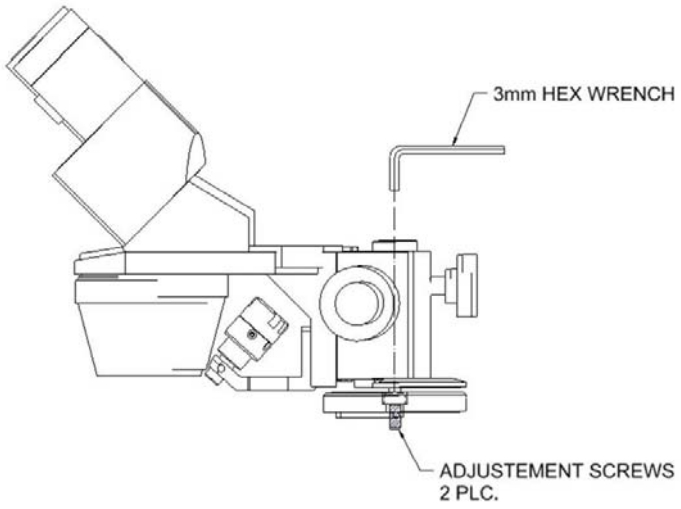

Figure 2-3 Microscope front to back alignment.

Left-to-right alignment

1. Loosen the socket-set screw on the pivoting slide.

Use the 1.5 mm hex wrench to loosen the socket-set screw (facing the rear on the pivoting slide itself).

2. Position the microscope.

Look between the microscope slide and the pivoting slide to find a brass cam. Engage the spanner wrench in the cam and rotate left or right in small increments to position the microscope. Rotate CW to move the microscope to the right. Rotate CCW to move the microscope to the left.

3. Once centered, tighten the set screw.

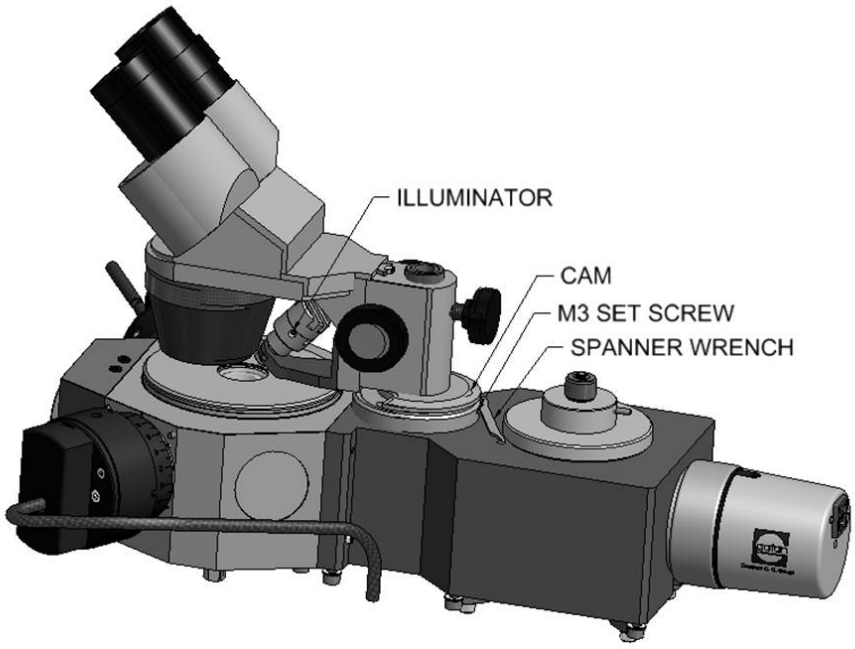

Figure 2-4 Microscope left to right alignment.

3. Operation

The PIPS II is relatively simple to operate. The ability of the operator to obtain good TEM specimens depends more on the quality of the starting specimen disks than on any other factor. Specimen-preparation times are relatively short if the starting disks are thin and well polished mechanically.

3.1. Graphical User Interface (GUI)

PIPS II is mainly operated using the touch screen interface. This interface contains:

- The Milling page
- The Recipes page
- The Alignment page
- The Camera or Viewing page
- The Settings Page
- The Maintenance page

3.1.1. *The Milling Page*

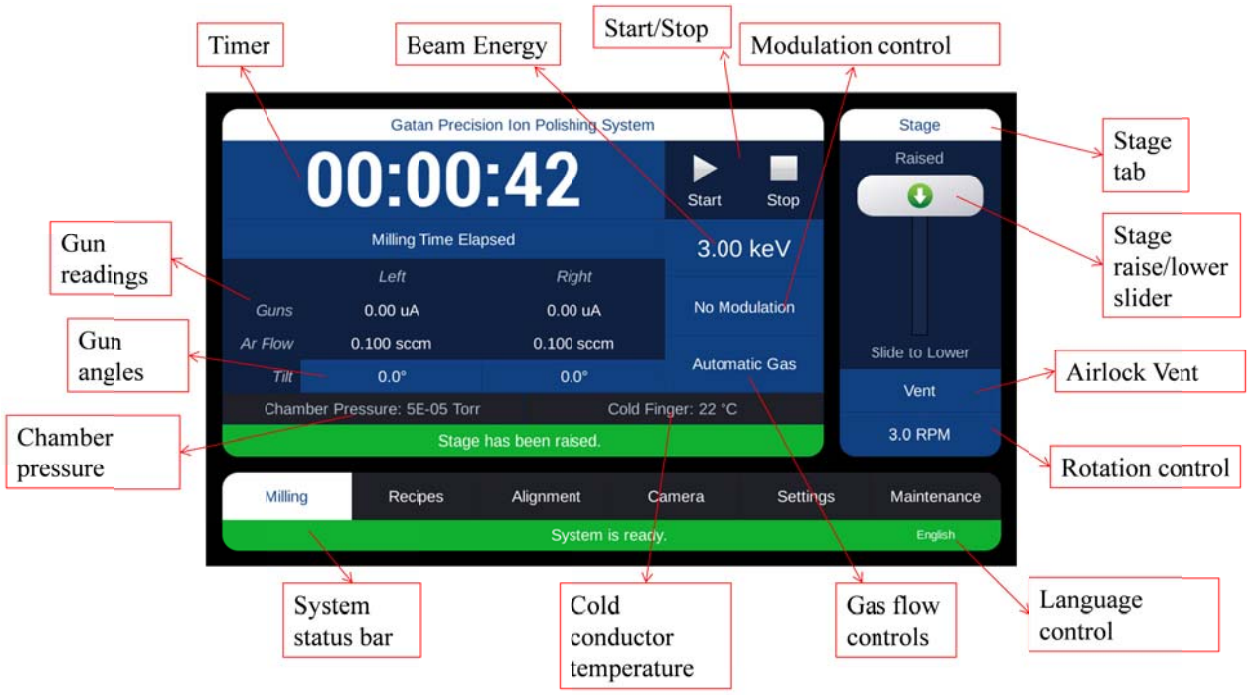

Figure 3-1 Milling page.

Timer: Use to define the milling duration. For this, touch the Timer and enter the milling duration in the window shown below (Figure 3-2), then touch Apply.

When the system is milling and the timer times out, the voltage is turned off, the stage rotation stops, the shutter is opened, and the user is notified by a buzzer.

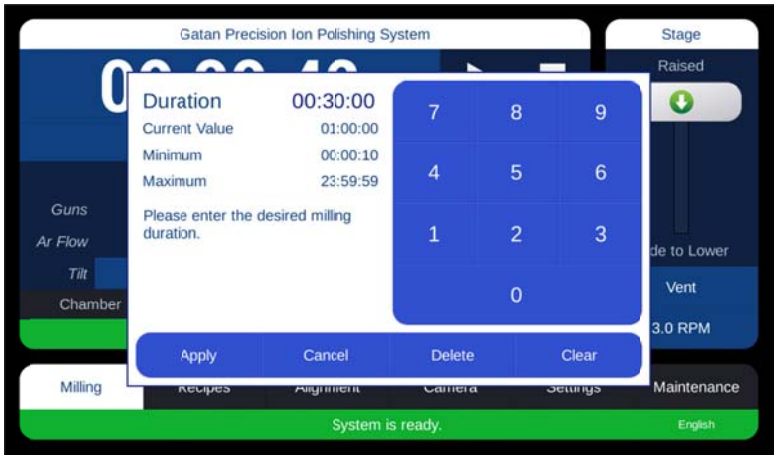

Figure 3-2 Setting the milling duration.

Gun controls: These are used to define the accelerating voltage (0-8.0 KeV), and to read the left and right gun currents. Note that the current read by this display is the current leaving the inner gun. Some of this current is neutralized and some is blocked by the focus electrode or the housing. This current value, then, is larger than would be measured by a Faraday cup. Since the sample does not block this current, it can be read at any time during milling.

Gas flow controls: The amount of gas flow is shown in sccm (standard cubic centimeter per minute) for each gun and can be varied between 0 to 1.0 sccm. The gas flow can be controlled automatically or manually. Note that when the Alternate gas is selected (Settings-General), the display will indicate Alt Flow instead of Ar Flow.

Automatic: The system uses a pre-set gas flow value that was determined at the factory. This optimium value for each accelerating voltage is stored in a table. This table is calibrated at the factory, but can be re-calibrated if necessary.

Manual: Each gas flow can be entered manually.

Tilt: Displays a dialog box with number entry to set the Gun Tilt of each gun. This option is only displayed if the system has the motorized guns option.

Cold conductor temperature: Shows the temperature at the cold conductor. This is an intermediate block between the dewar and the sample mount. This is not the sample temperature, but is proportional to the sample temperature. There is a time delay of approximately 15 minutes between the time the cold conductor reaches its minimum temperature and when the sample mount reaches its minimum temperature.

If you touch this display, the Options / Heaters screen will be displayed. The cold block and dewar heaters may be operated from this screen.

System status bar: Shows the status of the system (busy, ready, etc.)

Chamber pressure: Shows the pressure inside the work chamber and depends on the gas flow settings of the left and right guns. If you touch this display, the Maintenance / Vacuum screen will be displayed.

Rotation control: Used to set the rotation speed (rpm) during the milling process. It can vary between 1 and 6 rpm.

Modulation control: Used to set the modulation mode during milling process. Modulation controls the action of the guns as the sample is rotated. When modulation is set to No Modulation, both guns are on at all times. When modulation is set to Single Modulation, each gun is turned on only when the front of the sample mount faces that gun. When modulation is set to Dual Modulation, each gun is turned on only when the front or rear of the sample mount faces that gun. When modulation is set to Stationary Left, the left gun is on continuously, the right gun is off, and the sample does not rotate. When modulation is set to Stationary Right, the right gun is on continuously, the left gun is off, and the sample does not rotate.

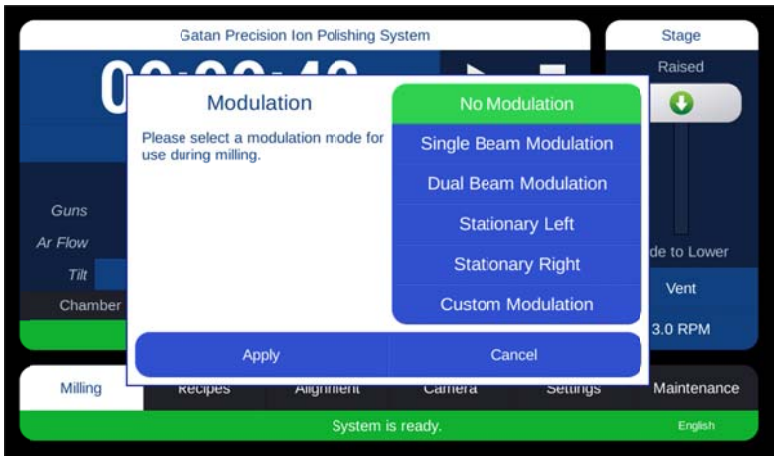

Figure 3-3 Setting the modulation mode.

Stage tab: Used for raising/lowering the stage, for venting the airlock when the sample is in raised position, and for opening the shutter and turning on the illuminators when the stage is in the lowered position.

NOTE: The airlock is automatically pumped down when the sample is lowered. After a sample is raised, the airlock must be manually vented to remove the sample.

Start/Stop: Start turns on the gas flow, starts stage rotation, turns on the beam, closes the shutter and starts the timer. Stop turns the voltage off, stops the time, rotates the stage to the home position and opens the shutter.

Language Control: Press this text to set the language of the user interface.

Figure 3-4 Setting the language.

3.1.2. *The Recipes Page*

Use this page to create, store, edit and run milling recipes. Milling parameters like voltage, duration, gun angle (if motorized guns are provided), rotation speed, beam modulation, top illumination and color, bottom illumination, camera mode, cold stage set-point and auto terminator can be varied for each step in a recipe.

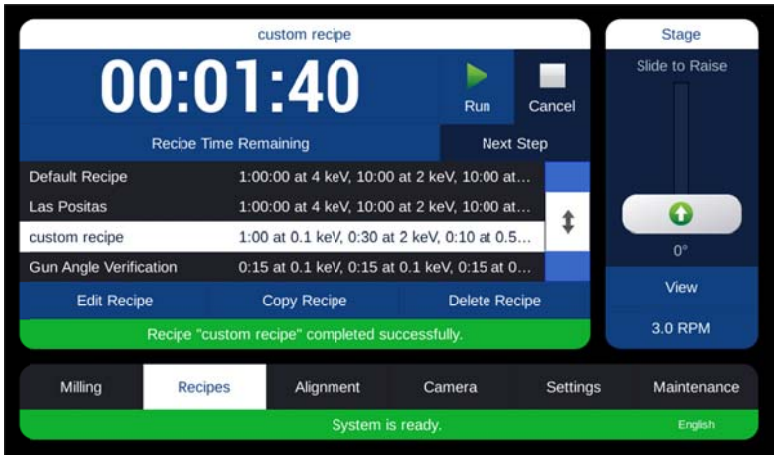

Figure 3-5 Recipes page.

Running a recipe: To run an existing recipe, select the recipe you want and touch Run. As a recipe runs, the remaining time and the current step number are shown on the top left corner of the page. Status bar will show the user that the system is busy running a recipe.

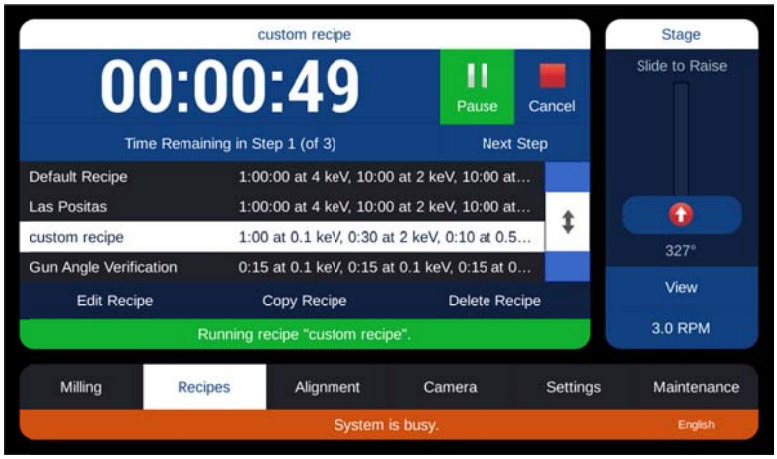

Figure 3-6 Status bar, showing a recipe is running.

Milling can be stopped or paused anytime during this process. In order to skip from one step to the next, touch Next Step. After all the steps are complete, the status bar will show the user that the recipe has been completed successfully.

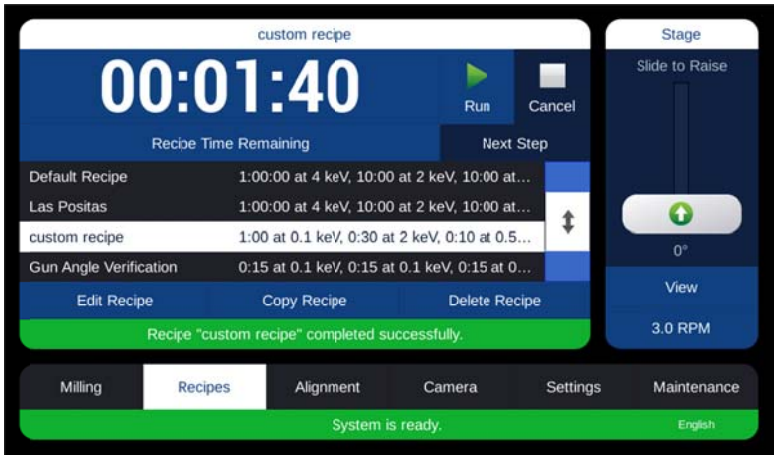

Figure 3-7 Status bar, showing a recipe is completed successfully.

Editing a recipe: To edit a recipe, select the recipe and touch Edit Recipe. This opens the Recipe Edit page, where you can edit the parameters in each step, add/delete a step, rename the recipe, revert (bring all parameters to what they were set to before editing started) and save. The horizontal slider allows more options to be viewed or edited. The vertical slider allows more steps to be viewed or edited.

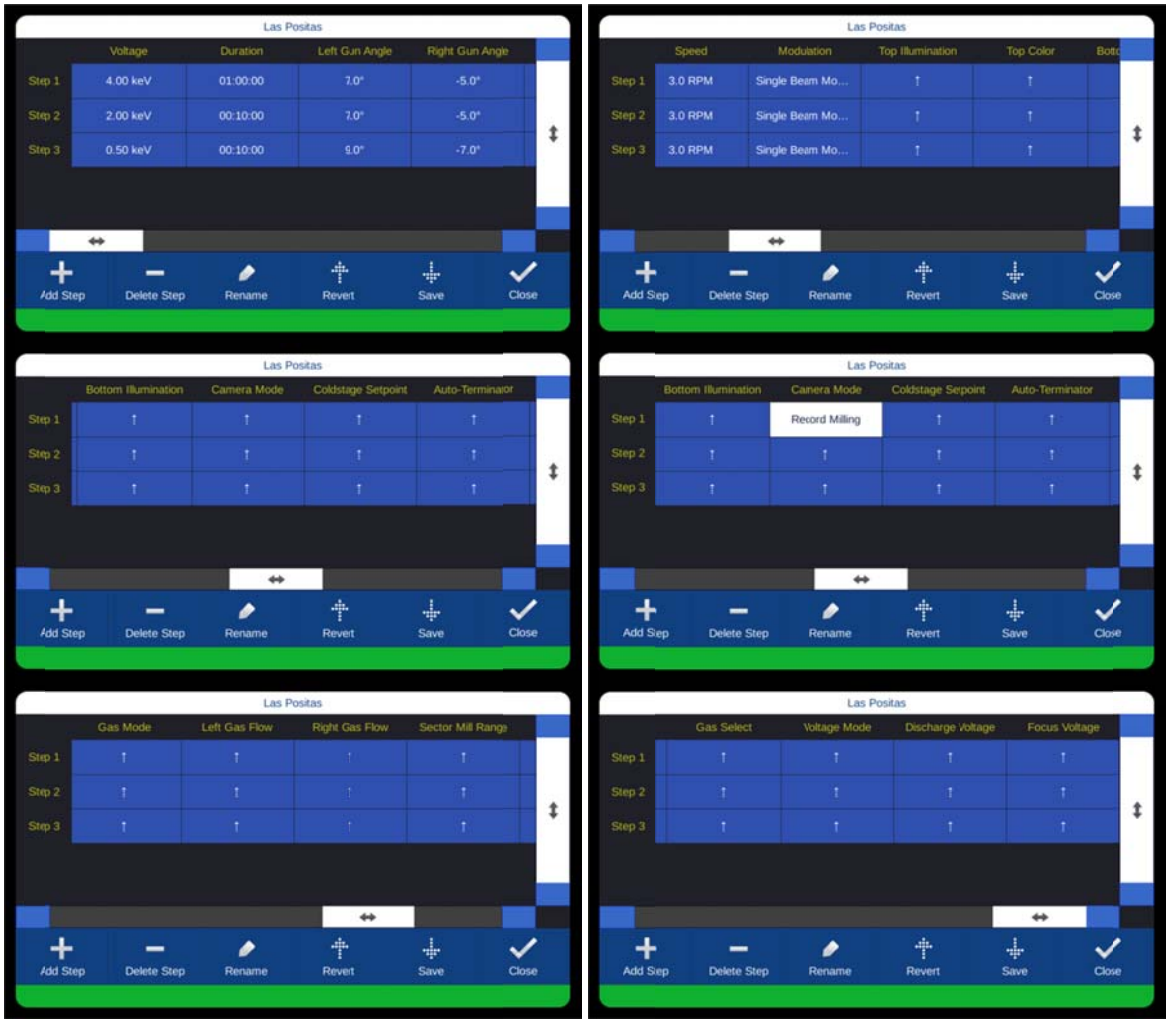

Figure 3-8 Edit recipe page.

In order to change each parameter in recipe steps, touch it. A window opens where you can enter the new value/setting and apply. An up arrow entry means that the value will not change from its previous setting. (see the final image in Figure 3-8) For instance, if you want to manually set the gas flow before starting a recipe, use the up arrow selection. Then when the recipe is started, the gas flow value will stay the same as it was set prior to starting the recipe.

To add a step, touch the Add Step button, enter the step number, and touch Apply.

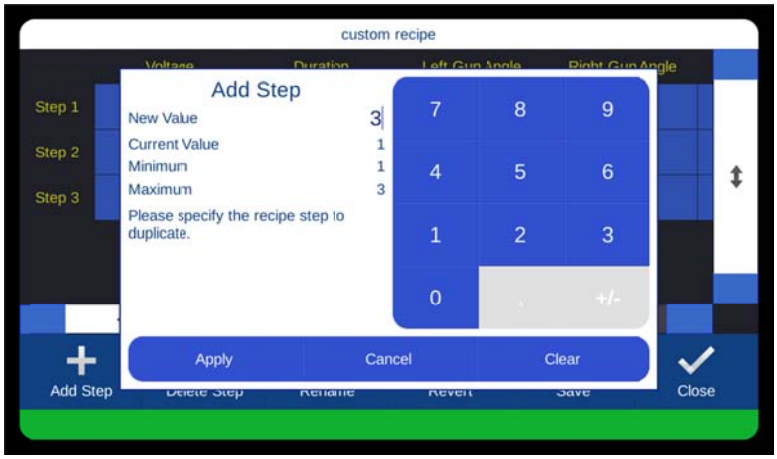

Figure 3-9 Adding a recipe step.

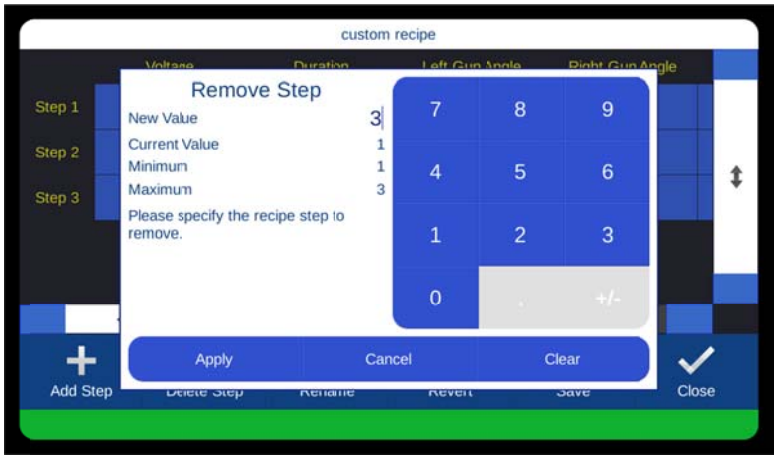

Figure 3-10. Deleting a recipe step.

After done editing, touch Save and then Close to go back to the recipes page.

Creating a new recipe: This is done by copying an existing recipe and then editing it. To create a new recipe, select the recipe you want to copy, then touch Copy Recipe. The user will then be asked to enter the name for the new recipe. Enter the name and touch Apply. After the new recipe is created, select it and go to Edit Recipe page to modify/delete the existing steps or to add more steps.

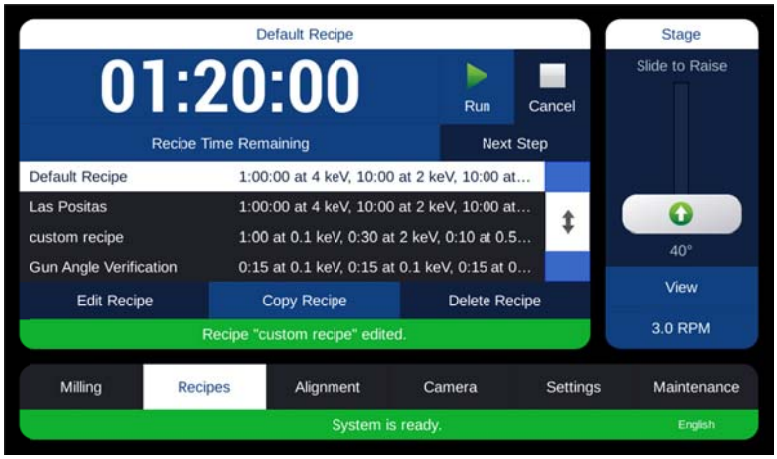

Figure 3-11 Copying a recipe.

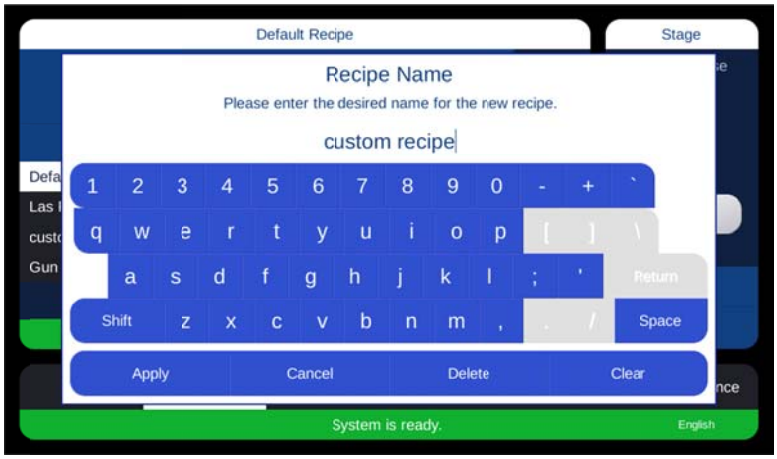

Figure 3-12 Creating a new recipe: enter the name.

3.1.3. *The Alignment Page*

Use this page to perform the beam alignment and x-y stage alignments. The sequence should be:

1. **Insert the beam alignment screen and center the x-y stage by bringing the center of the alignment screen to the center of rotation.**

2. **Align the guns to the center of the beam alignment screen.**

3. **Bring the point of interest to the center of rotation.**

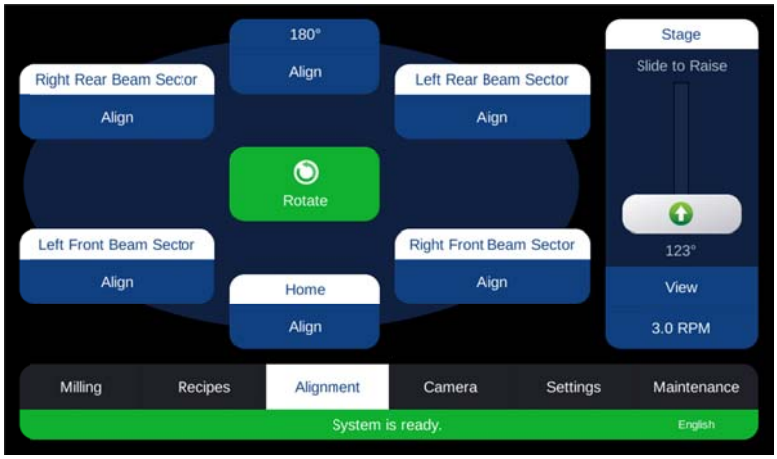

Figure 3-13 Alignment page.

Whenever "Align" is touched in this page, the stage will rotate to the specified position and will stay there until "Rotate" is touched again. The green color on Rotate, shows that rotation is on. Left Front Beam Sector causes the front of the specimen post to face the left gun. Left Rear Beam Sector causes the rear of the specimen post to face the left gun. Right Front Beam Sector causes the front of the specimen post to face the right gun. Right Rear Beam Sector causes the rear of the specimen post to face the right gun. Home causes the front of the specimen post to face the front of the instrument. The Align button with an angle displayed causes the stage to rotate to that specific angle. The angle may be set manually between 0 and 360 degrees.

The Angle display on the stage tab shows the current rotation angle. The stage tab can be used similarly to the milling page to lower/raise the sample and change the rotation speed.

The View button opens the shutter and turns on the illuminators which are set to Enabled.

3.1.4. *The Camera or Viewing Page*

The Camera page is used to control camera acquisition in the systems that have this option and to set the top and bottom illumination. In systems without a camera, this page is titled View and is used to open/close the shutter and turn on the illuminators.

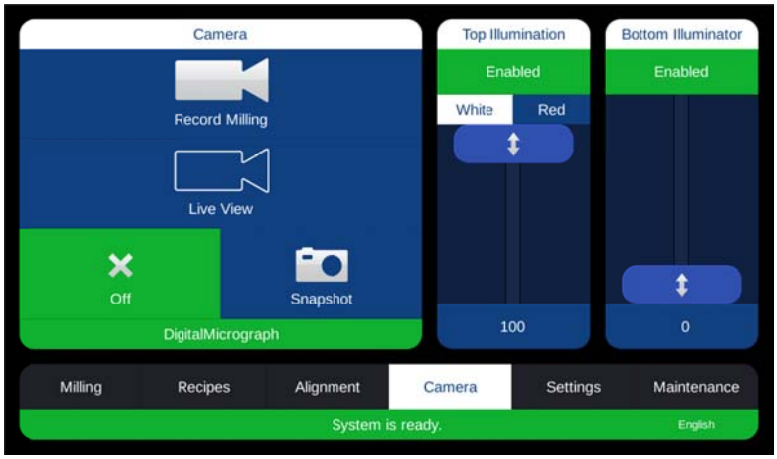

Figure 3-14 Camera page.

Camera page: Is visible when the Digital Zoom Microscope option is installed. It is used to set the imaging mode in DM and to control the illuminators.

Record milling: acquires an image every rotation. Image saving options can be changed in DM. This is a full resolution image.

Live view: keeps the camera on at all times and gives the user a live view of the sample. This is a VGA image, either binned by 1x, 2x, or 4x depending on zoom level. This mode also opens the shutter and turns on the enabled illuminators.
Off: stops camera, turns off illuminators and closes shutter.
Snapshot: takes a snapshot. This is a full resolution image. You should be in Live View mode prior to pressing this button so that the illuminators are active.

Top illuminator tab: Is used to enable or disable the reflection lights (red and white) and to change their intensities (use the sliders) independently. Pressing View or Live View opens the shutter and turns on the illuminators according to their settings. Some systems have a two-color illuminator. If you have this option, then the color of the top illuminator may be selected between white and red. If you do not have this option, then you must unplug the white illuminator and plug in the red illuminator to change color (keep the white color selected on this screen no matter which illuminator is plugged in). The intensity display button at the bottom of the slider may be touched in order to enter a specific intensity level (between 0 and 100%).

Bottom illuminator tab: Is used to enable or disable the transmission light and to change its intensity (use the sliders). Pressing View or Live View opens the shutter and turns on the illuminators according to their settings. The intensity display button at the bottom of the slider may be touched in order to enter a specific intensity level (between 0 and 100%).

Viewing page: Is visible when the Digital Zoom Microscope option is not installed. It is used to control the illuminators and open the shutter.

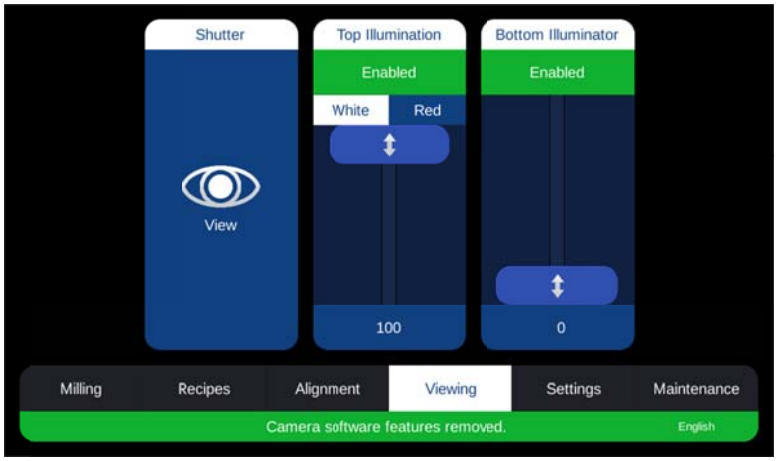

Figure 3-15. Viewing page.

Shutter tab: The View button is used to open the shutter and turn on the enabled illuminators.

Top illuminator tab: Is used to enable or disable the reflection lights (red and white) and to change their intensities (use the sliders) independently. Pressing View or Live View opens the shutter and turns on the illuminators according to their settings. Some systems have a two-color illuminator. If you have this option, then the color of the top illuminator may be selected between white and red. If you do not have this option, then you must unplug the white illuminator and plug in the optional red illuminator to change color (keep the white color selected on this screen no matter which illuminator is plugged in). The intensity display button at the bottom of the slider may be touched in order to enter a specific intensity level (between 0 and 100%).

Bottom illuminator tab: Is used to enable or disable the transmission light and to change its intensity (use the sliders). Pressing View or Live View opens the shutter and turns on the illuminators according to their settings. The intensity display button at the bottom of the slider may be touched in order to enter a specific intensity level (between 0 and 100%).

3.1.5. *The Settings Page*

General:

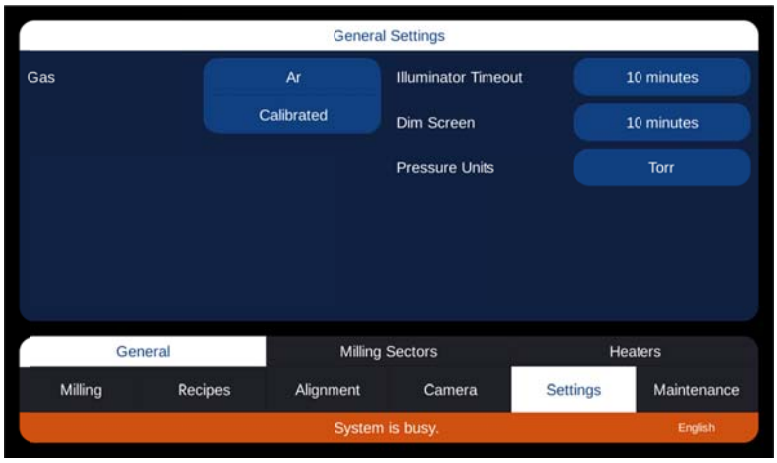

Figure 3-16 General Settings page.

Illuminator timeout: This timer starts when the user touches the View button. When it times out, the shutter closes and the illumination is turned off.

Dim Screen: When the touch screen has not been touched for this time, the screen will be dimmed.

Pressure units: Torr or Pascal.

Gas: allows the user to choose between Ar and Alternate Gas to be used for the guns. The default is Ar. Alternate is chosen by activating the Alternate Gas valve. If Alternate is selected, an alternate gas must be connected to the Alt input on the rear panel of the cabinet.

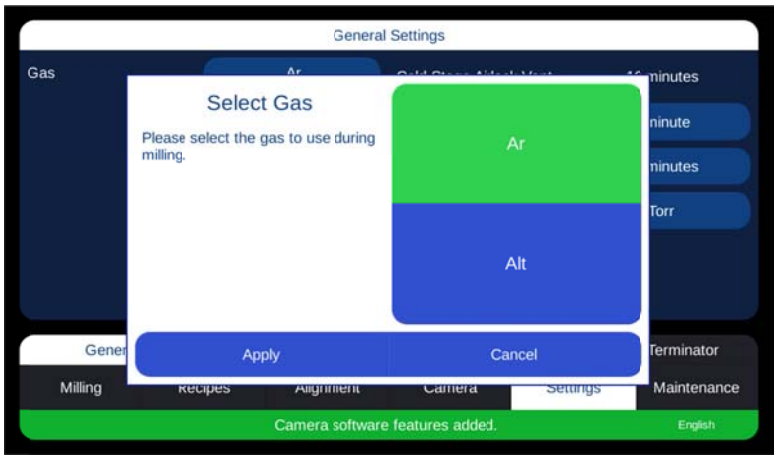

Figure 3-17 Setting the gas inlet used for the guns.

When Argon is chosen, the button just below is highlighted in blue. You may then choose between 3 calibration modes: Calibrated, Factory, and Safe. Calibrated mode uses the latest calibration values. These values are overwritten when the gas flow is calibrated (see section 3.1.6). By selecting Factory mode, the system is returned to the values calibrated at the factory. Selecting Safe mode sets the gas flow to a set of values that will generally work on any gun, but are not optimized for that gun. For instance, if the calibration routine is run when the guns are not fully degassed, then once the guns are degassed they will not operate properly. If the calibrated values are not working well, selecting the Safe mode will set the system to a mode where both guns operate at all voltages.

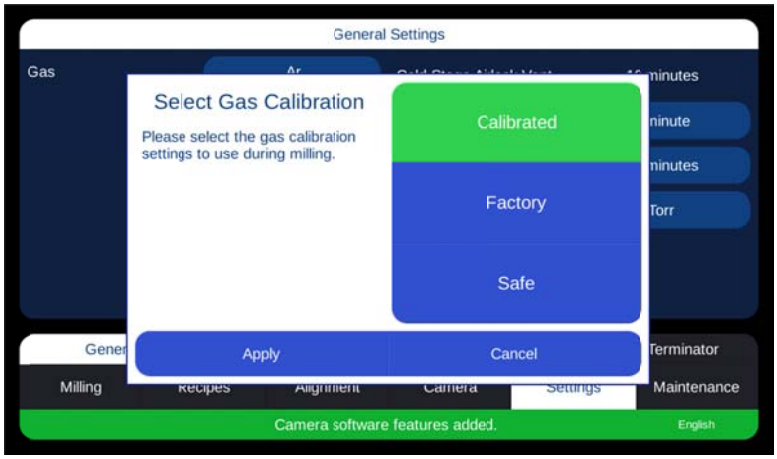

Figure 3-18 Choosing the calibration table used for the Argon gas inlet.

Milling Sectors:

Figure 3-19. Milling sectors.

Sector milling range: Sector Mill Range sets the angle through which the sector milling is on. Default is 60 degrees. Values can be changed from 90 to 10 degrees. This option may also be set in individual recipes.

Custom Modulation: This feature allows the user to define the start and stop angles for up to two custom defined sectors. This can be useful for cleaning up FIB samples that are mounted on the side of a grid-bar, where you may want front and rear sectors that are asymmetric. The angle entries are the stage angles when the guns should turn on and off. Only positive angle entries are allowed.

For example, the standard dual beam modulation settings would be:

Sector 1	**On**	**Off**
Left Gun	275	335
Right Gun	25	85
Sector 2	**On**	**Off**
Left Gun	95	155
Right Gun	205	265

Heaters:

This option is only functional in systems with a cold stage.

Cold Finger reading: Displays the temperature of the cold conductor.

Cold stage heater: Turns the heater on and off. If the heater is on and the temperature drops below the heater set point, the heater is turned on until the temperature is above the set point.

Dewar heater: Activates the dewar heater. This heater stays on continuously until the Cold Finger Temp reaches 25° C, then it shuts off. The stage heater must be off before starting this heater.

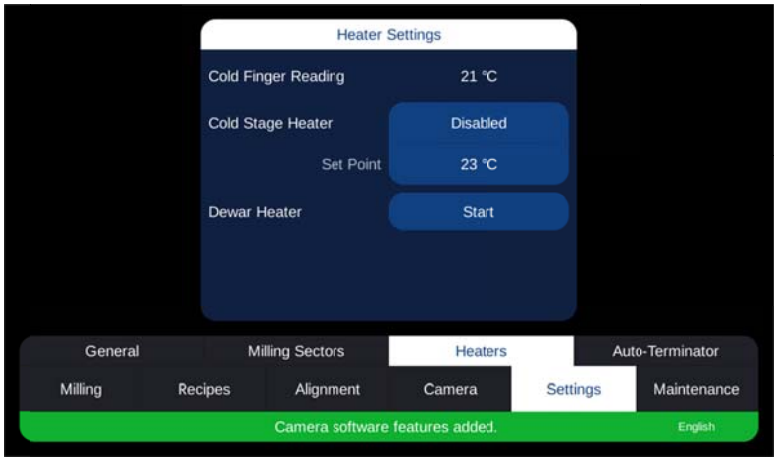

Figure 3-20 Heaters settings page.

Autoterminator:

An optional Autoterminator must be purchased to use this option.

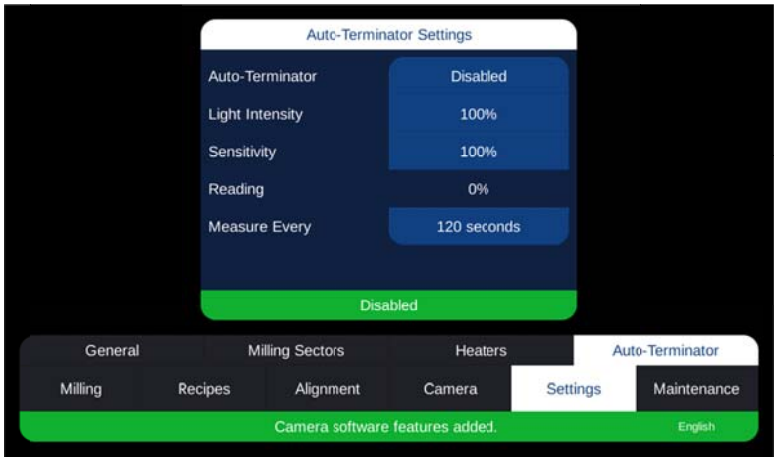

Figure 3-21 Auto-terminator page.

Reading: Displays the last signal read by the autoterminator.

Sensitivity: This control enables the operator to select varying termination hole sizes in the specimen and also to compensate for varying degrees of specimen transparency. See “Autoterminator” for further information.

Light intensity: Sets the intensity of the bottom illuminator while a reading is being made.

Measure every: Tells the system how often to take a measurement. It can be every X rotation or every X seconds. Once the reading reaches 15% of the trip point, readings will be taken every 16 seconds, instead of the interval set above

3.1.6. *The Maintenance Page*

Calibration:

Gas flow: This is performed at the factory and is not generally needed unless something changes dramatically in a gun. When Create All is selected, the optimum gas flow for each gun will be measured and saved at different beam energies. These values are used when "Automatic gas flow" control is selected. If the guns have been changed or if it is observed that the automatic gas flow settings do not produce good results, then this calibration may be run.

The bar at the top indicates if **Ar**gon or **Alt**ernate gas has been selected.

Figure 3-22 Gas flow calibration.

Individual settings in the calibration table may be set manually using this screen. First choose a Calibration Point: point 1 = 250 eV, point 2 = 500 eV, …, point 10 = 8 keV. Next select the sccm button for the left gun (left sccm button), and set it to the desired value. Then select the sccm button for the right gun, and set it to the desired value. Repeat for all Calibration Points that you would like to change.

An automatic gas flow curve for a single Calibration Point may also be run. First remove any sample posts, and lower the piston. Next select the Calibration Point. Next press Recalibrate.

Zero Controllers is a function to remove offset errors in the mass flow controllers. This is not normally needed unless requested by Gatan service.

Gun angles:

This is only available for systems with motorized guns:

1. **Touch Maintenance – Guns – Gun Tilt**
2. **Manually set both guns to +10 deg, write down the dac readings displayed.**
3. **Manually set both guns to -10 deg, write down the dac readings displayed.**
4. **Touch Maintenance – Calibrations – Gun angle**
5. **Enter the dac readings for the appropriate settings.**
6. **Touch Maintenance – Guns – Gun Tilt**
7. **Verify that both guns can be set within the full range of -10 to +10 deg.**

Figure 3-23 Motorized guns calibration.

Stage position: This window is used for calibrating the stage home position.

1. **Lower piston.**
2. **Go to the Alignment page.**
3. **Watch the stage rotate, when it gets to the home position press Rotate. If it does not stop exactly at the home position, repeat.**

4. **Go to the Maintenance | Calibrate | Stage screen. Note the Current Encoder Poition. Press the Index button. Enter the current encoder position noted above.**

5. **Go to Alignment screen, press Rotate, then press Home.**

6. **Repeat above steps until the adjustment screws of the stage are aligned toward the front and the inside edge of the top plate is parallel to the front of the system.**

Figure 3-24 Properly aligned stage in the Home position.

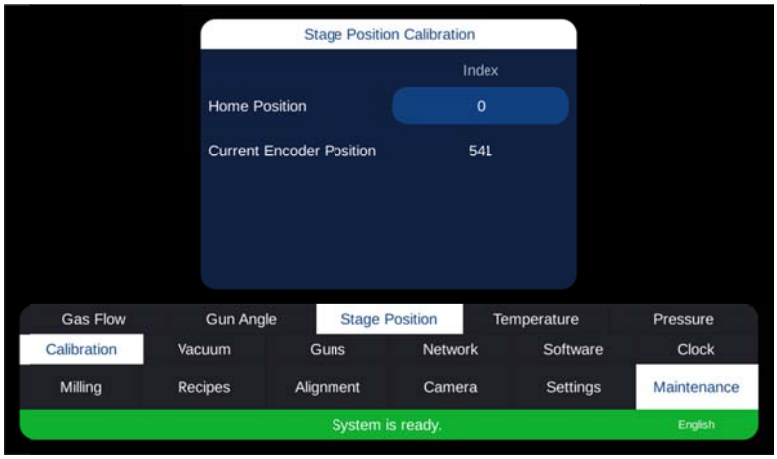

Figure 3-25 Stage home position calibration.

Temperature Gauge: This sensor measures the temperature of the cold conductor. To set, press the blue temperature button and enter the actual temperature of the cold conductor.

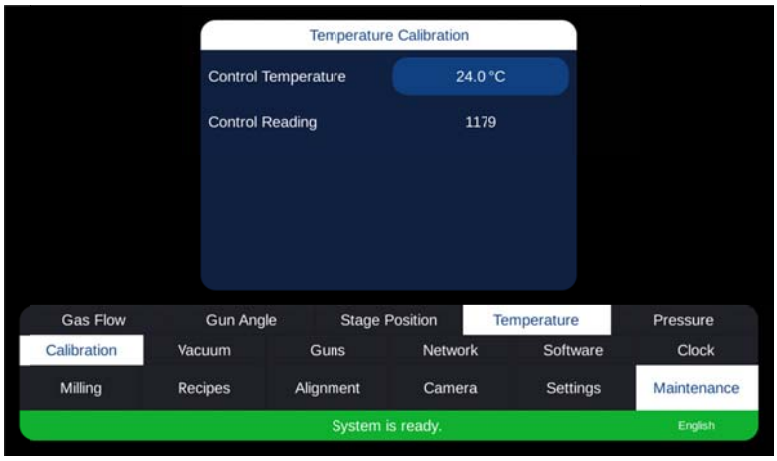

Figure 3-26 Temperature sensor calibration.

Pressure: The gauges in this unit are factory calibrated and ready to use. Calibration is a multi-step procedure requiring the connection of external control equipment and calibration should only be performed at the instruction of Gatan service. If gauges need to be calibrated in the field, it is best to set them to the default calibration.

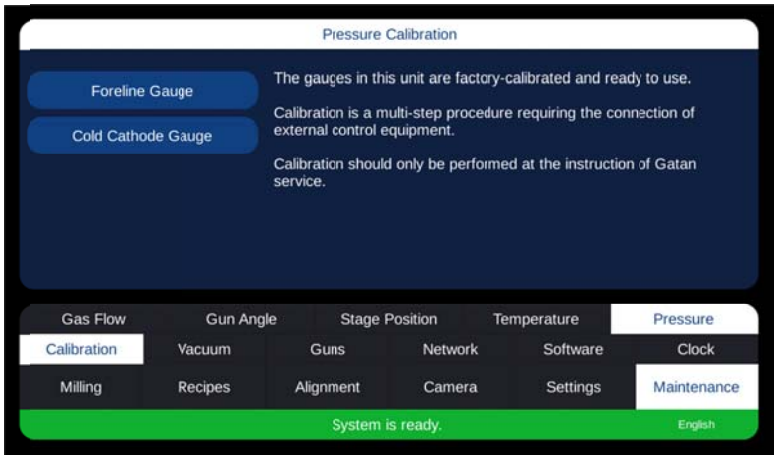

Figure 3-27 Pressure calibration.

Foreline Gauge: This gauge measures the pressure in the line leading to the diaphragm pump. It normally is measuring the backing pressure, but when the airlock is pumped out it measures the airlock pressure. This should not need to be calibrated in the field (Default | Apply).

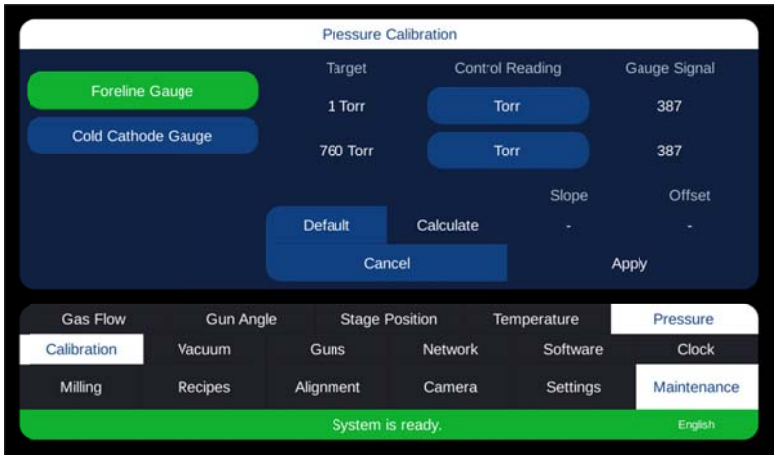

Figure 3-28 Foreline gauge calibration.

Cold cathode gauge: This gauge measures the work chamber pressure. It is turned on when the MDP speed is above 1250 rpm. This should not need to be calibrated in the field.

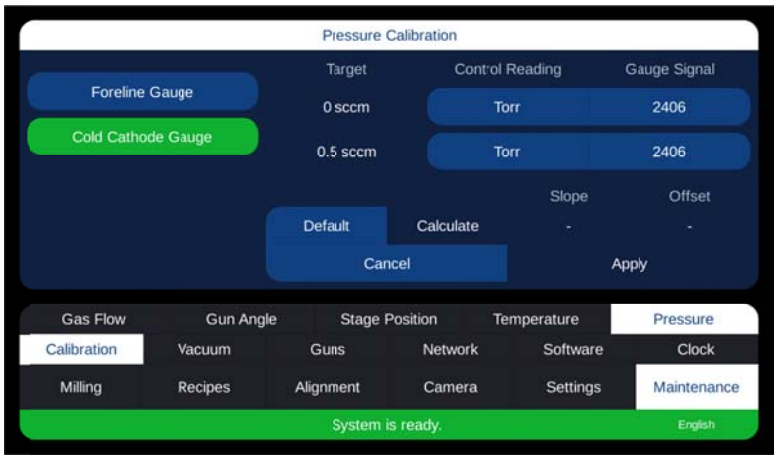

Figure 3-29 Cold Cathode gauge calibration.

Vacuum:

This screen is used to control and monitor the vacuum pumps and gauges.

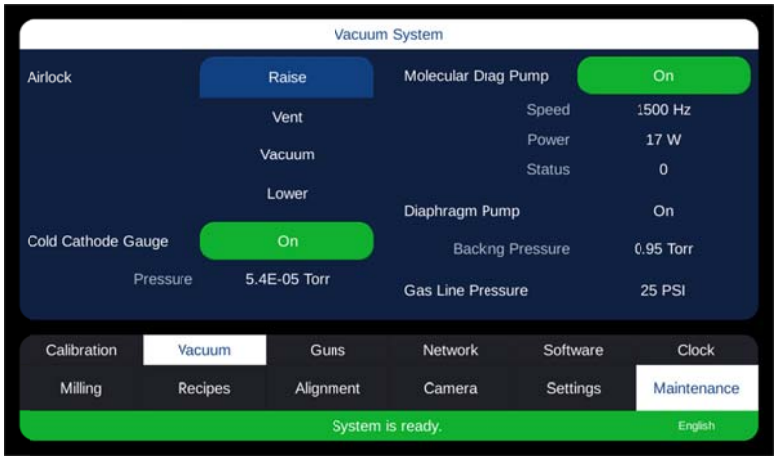

Figure 3-30. Vacuum page.

Airlock: Raise is used to raise the stage into the airlock. Vent is used to vent the airlock. Vacuum is used to pump out the airlock without lowering the stage. Lower is used to pump out the airlock and lower the stage.

Cold Cathode Gauge: On/Off controls the power to the CC gauge. The power is automatically turned off when the MDP speed is less than 1275 Hz. Pressure displays the pressure measured by the gauge.

Molecular Drag Pump: On/Off controls the pump. Speed displays the rotational speed of the pump, where the nominal speed is 1500 Hz. Power displays the power drawn by the MDP. Status shows any errors (0= none).

Diaphragm Pump: On/Off controls power to the pump. This control is disabled when the MDP is on, so the DP cannot be turned off if the MDP is on. Backing Pressure displays the pressure measured in the backing line. When this pressure is higher than 10 Torr, the DP is set to full speed, when it is less than 10 Torr the DP is set to half speed. This reading is used to determine when the airlock is pumped out sufficiently for the stage to be lowered.

Gas Line Pressure: Measures the pressure of the Argon gas inlet line. If the pressure is outside of the acceptable range, an error message will be displayed and the system will not mill.

Guns:

Gun readings: This page displays the high voltage power supply readings (Figure 3-31). The Adjust Flow buttons are visible when Manual Gas Flow is set. Touching the up or down arrow will increase or decrease the gas flow for the left or right gun.

Accelerating Voltage is the voltage on the anode. This is the beam energy. Discharge Voltage is the voltage between the anode and the cathode, this voltage sustains the plasma. The discharge voltage floats on top of the accelerating voltage. Focus voltage is used to focus low energy ions, and is only used when the accelerating voltage is lower than 2.5 kV. Focus current is the sum of the currents on the left and right gun focus electrodes. Accelerating current is proportional to the total beam current. It includes neutral ions in the beam and current lost to the focus electrodes and housings. Discharge current is the current through the plasma. Faraday Cup current is the current measured by the Faraday cups, but is not used in PIPS II. This current does not include neutral ions or current that misses the Faraday cup. Gas flow is measured by the mass flow controllers.

Accelerating current is a better approximation of the total Argon dose because it includes neutrals. The downside to this measurement is that it over-counts because it includes the part of the beam that strikes the housing or misses the sample. Faraday cup current is not used in PIPS II and should read close to 0.

The gun maintenance screen can be used to help determine if a gun is shorted. If the discharge current in microamps is approximately equal to the discharge voltage in volts, and the accelerating current is unusually low; then the gun is likely shorted. For example, when the beam voltage is 6 kV, the discharge voltage is approximately 1100 V. If the discharge current is approximately 1100 uA and the accelerating current is significantly lower than normal for 6 kV beam voltage, then the gun is likely shorted. Note that these same conditions apply during beam modulation when the guns are between milling sectors.

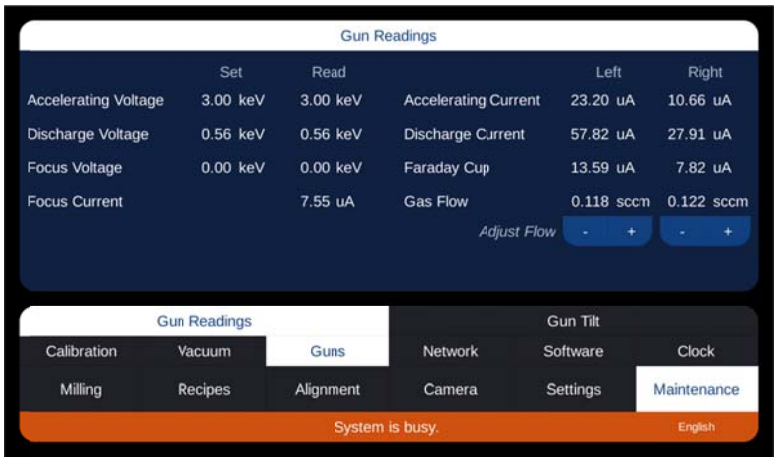

Figure 3-31 Gun Readings.

Gun tilt: This option is only functional for systems that have motorized guns. Left/Right Gun Angles set the tilt angle the left/right guns should be set to,

respectively. The position reads the current gun angle position in degrees and dac values. The remainder of the readings are for service personnel diagnostic purposes.

Figure 3-32 Gun Tilt page.

Network:

This displays the network settings for the PIPS II. These setting are set at the factory and can not be adjusted.

Figure 3-33 Network page.

Software:

Software Version:

This page shows the latest software version and the date it has been last updated.

In order to update the software, plug in the USB drive that is provided for this purpose in the back of the PIPS II. This drive should have 2 files in its root directory, ending in .swimg and .fwimg. Wait a few moments for it to load and then touch "Update System". After successful installation of the updated version, a message will appear on the screen to notify the user to restart the PIPS II. At this time turn the power off, wait a few seconds, then turn the power on. The power switch is located on the back of the instrument.

The latest software and firmware revisions can be found at: ftp.gatan.com\public\software\SpecimenPrep Download the zip file and unzip it to the root of a USB flash drive.

Not all flash drives are supported. It may take some experimentation to find a drive that works.

The "Export Support Files" button copies configuration files to a USB flash drive (gatan_export directory) that must first be inserted into the USB port on the back of the system. These files may be useful to service personnel when troubleshooting.

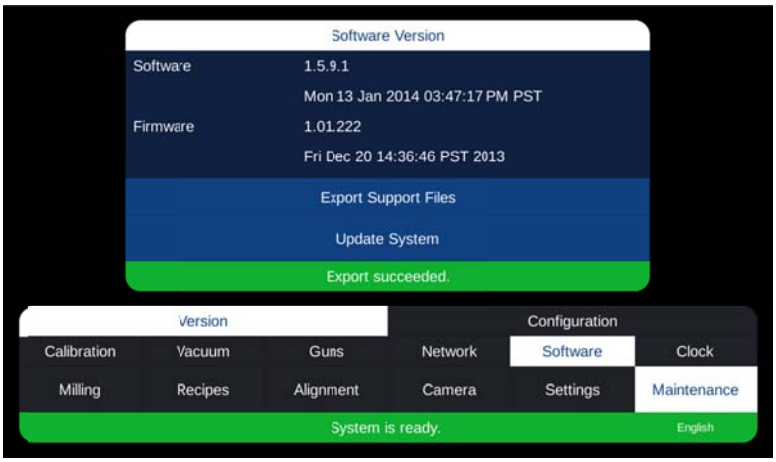

Figure 3-34 Software maintenance page.

Software Configuration:

This page configures the software for system options. If the system has any of these options, the button to the right of that option should be set to Included. Certain pages in the user interface change depending on which options are included.

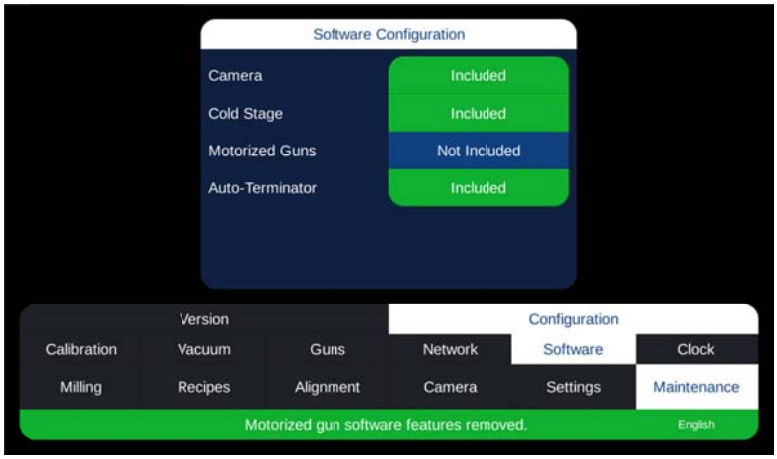

Figure 3-35. Software configuration page.

Clock:

Set the date and time on this page. Note that the system clock does not automatically adjust for daylight savings time.

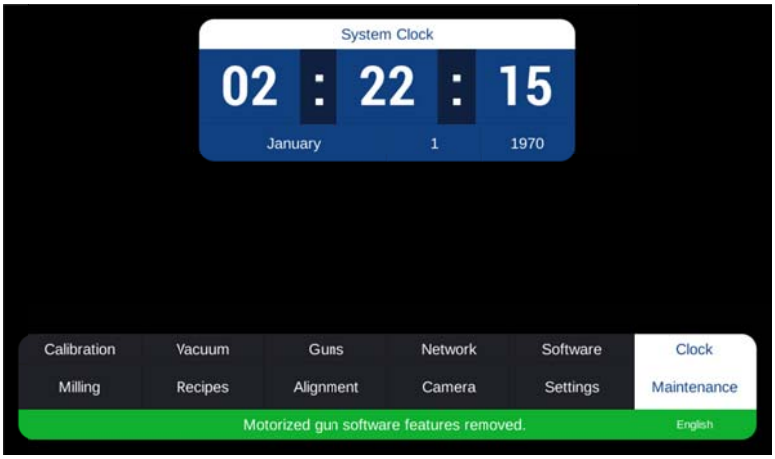

Figure 3-36 Clock page.

3.2. Start-up Procedure

Turn on the power to the PIPS II. The molecular drag pump (MDP) will start immediately and the diaphragm pump (DP) will start after about 30 seconds.

Once the operating system has booted up, the milling page will be displayed. The cold cathode pressure reading will turn on when the MDP is at about 85% of full speed. Wait for the MDP to spin up fully before lowering the stage.

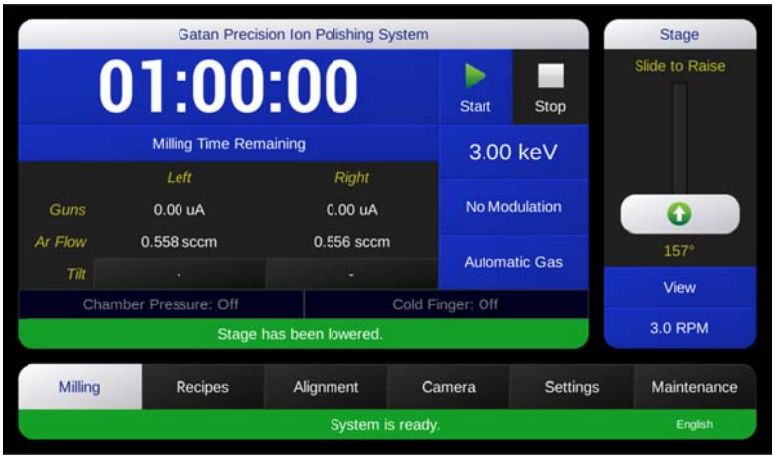

Figure 3-37 Milling page.

3.2.1. *Ion-gun Purging*

The PIGs are very efficient and operate with an extremely low gas throughput. However, even when the argon gas flow to the guns is turned off, small amounts of out-gassing from materials in the ion guns will produce significant ion currents (discharge current >10 µA). In extreme cases, out-gassing will result in sudden bursts of ionization that make the PIGs unstable in operation. To minimize this effect, the PIGs must be purged with dry argon. Typically, this is necessary whenever the gun components have been exposed to a poor vacuum, i.e., whenever the PIPS II has been switched off for more than 4 hours or the chamber has been vented. In addition, the automatic gas flow settings are valid only after the guns have been purged thoroughly.

The gas flow settings are set to 0.3 sccm on start-up in order to facilitate purging. Once the guns have been operated, the gas flow will be reset to their automatic values. If further purging is needed, it is recommended that the gas flow of both guns be set to 1.0 sccm using the Manual mode. Gas flow will not likely be stable above 0.3 sccm, but this is acceptable during purging. Once purging is complete, set the gas flow back to Automatic mode.

To Purge the Guns Manually

Switch the gas flow to manual, and set both guns to 1.0 sccm. Purge for about 15 min if the guns have been under vacuum. Purge for 4 hours minimim if the system has been vented to atmosphere. In any case, purging should be continued until a gun current of <10 µA is obtained with an accelerating voltage of 5.0 keV and the gas flow turned off to both guns (manual gas flow = 0 sccm). For best results, it is recommended that maintenance be performed at the end of the day, and the guns be purged overnight.

Guns may be purged at up to 1.0 sccm, however, gas flow will not likely be stable above 0.3 sccm, but this is acceptable during purging.

3.3. Specimen Loading and Unloading

Specimens are mounted either on a Gatan DuoPost or a regular specimen post. The specimen post is inserted into a specimen mount located at the top of the Whisperlok piston. The following procedure assumes the piston/specimen mount is in the Work Chamber.

3.3.1. *To Raise the Specimen Mount/piston:*

1. **On the Milling page, go to the Stage tab and slide the bar up.**

This will raise the specimen mount/piston into the Airlock to facilitate specimen loading. The piston will not rise immediately but waits for the specimen mount to rotate to its reference or home position. The piston then rises and seals off the Work Chamber from the Airlock chamber.

2. **Vent the Airlock chamber by touching the Vent button.**

Once vented, the Airlock's cover can be removed and a new specimen post can be inserted or an old one removed. A special pair of angled tweezers is supplied to facilitate this operation. Be careful not to apply a sideways force when inserting a sample, as this can change the position of the xy specimen stage.

NOTE: Be sure not to rotate the specimen mount when exchanging specimens. Any rotation of the mount will displace the home position of the specimen and will cause misalignment for modulated milling.

NOTE: When loading a specimen post, make sure it is properly seated in its lowest position; the height of the post is critical if the ion beams are to polish at the center of the specimen.

3.3.2. *To Lower the Specimen Mount/piston*

1. **Replace the Airlock cover.**

2. **On the Stage tab, slide the stage down to start evacuation and to lower the stage.**

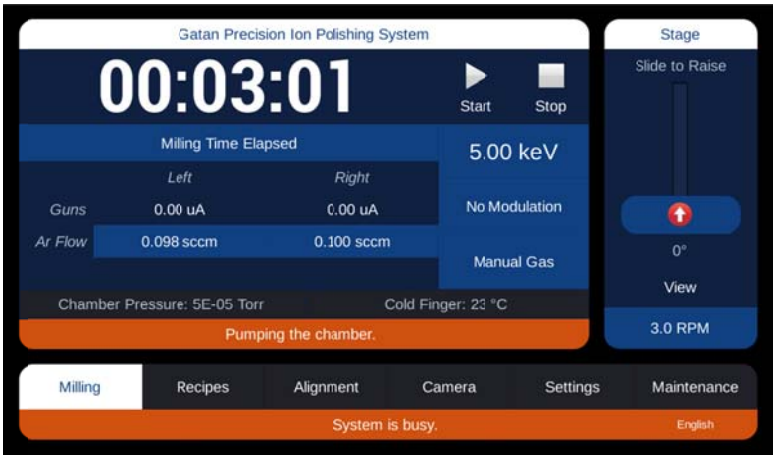

Figure 3-38 Lowering the stage.

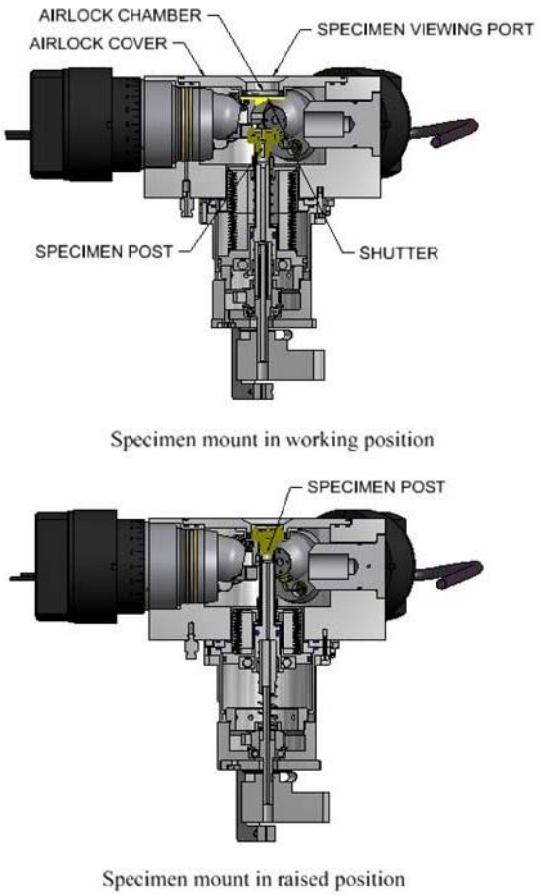

Figure 3-39 Specimen mount in raised and working positions.

3.4. Specimen Viewing

The PIPS II has been designed so that the specimen is clearly visible both with the naked eye or with the stereo microscope or camera either raised (in the Airlock) or in the lowered position (in the Work Chamber). The wide-angle view with the naked eye is necessary when aligning the PIGs using the Beam Alignment Screen. The microscope/camera view is essential when one uses interference fringes to control the final stages of the thinning process and when observing the final stages of polishing of specimens that do not show interference fringes.

3.4.1. *Illuminators*

The Reflection (top) and Transmission (below) illuminators are controlled by the GUI. Each illuminator is enabled or disabled using the button at the top of the slider. Turn the illuminators on/off by touching the View button on the Milling, Recipe, or Alignment pages; the Live View button on the Camera page, or the View button on the Viewing page. Increase/decrease the intensity of each light by lowering/raising the sliders. Finer control over the intensity may be achieved by touching the numerical display tab below the slider, then entering a number between 0 and 100%. The illumination intensity is intentionally non-linear over the range of 0 to 100%. Note that the Camera page will be active if the system has a camera, and the Viewing page will be active if it does not have a camera.

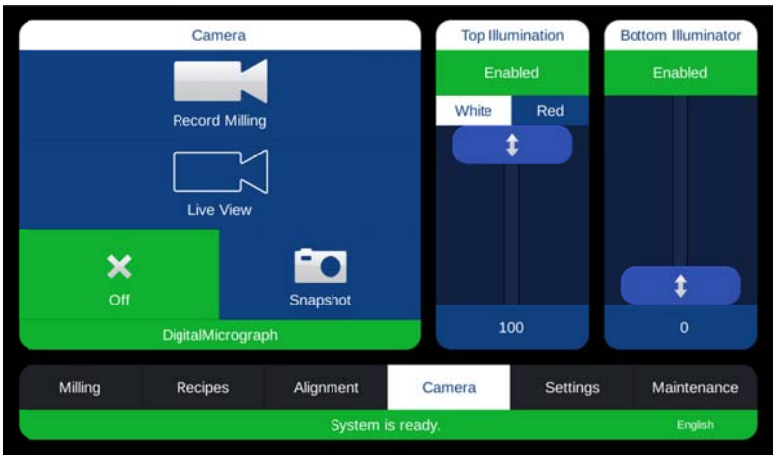

Figure 3-40 Camera page.

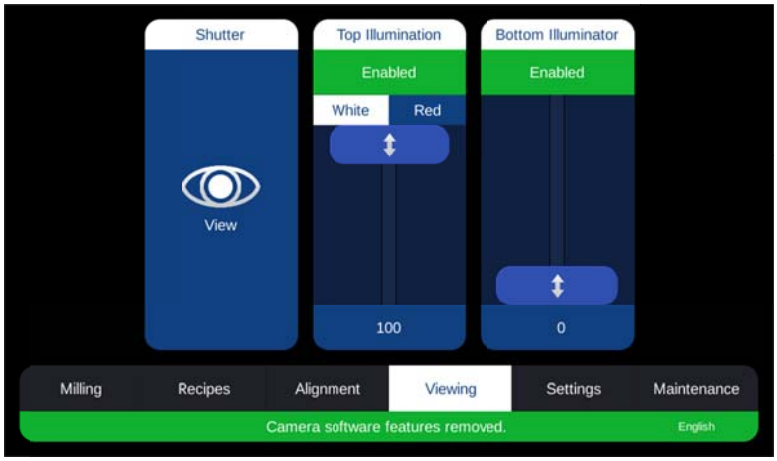

Figure 3-41 Viewing page.

3.5. Shutter Control

The Shutter protects the specimen Viewing Port from sputter deposits, operates automatically, and is keyed to the Start and View buttons in the Milling and Recipe tabs and the View and Live View buttons in the Alignment and Camera tabs respectively. It closes when milling starts and retracts when milling stops. The Shutter will also retract when the piston is raised. Similarly, the shutter retracts when View is turned on and it closes when View is turned off. When the camera is in Record Milling mode, the shutter will open once per rotation so that an image may be acquired.

NOTE: The Autoterminator incorporates a special shutter-control feature to minimize the amount of sputtered material accumulating on the Viewing Port. When the Autoterminator is in place and the guns are operating, the Shutter will open for about 1 sec every defined seconds/rotations for sampling of the transmitted light intensity. This sampling rate is maintained until the reading reaches 15% of the trip point, whereupon the sampling rate is automatically increased to about 1 sec every 16 sec with this rate maintained until termination.

3.6. Specimen Rotation

The specimen is rotated in a CCW direction by a variable-speed DC motor. The rotation speed can be varied from 1 through 6 rpm using the Rotation Speed control on the GUI. Rotation can be stopped by the Rotate button on the Alignment page. The motor drives a timing belt mounted to the Whisperlok piston.

An optical encoder is mounted to the drive shaft which allows for recognizing the home position as well as sector milling angles. The home position is

calibrated in the Maintenance section of the GUI. The stage advances to the home position prior to raising the stage into the airlock.

Figure 3-42 Specimen mount raised and in the Home position.

3.7. Centering the x-y Stage

The x-y stage should be centered with respect to the center of rotation before beam alignment is performed. This can be accomplished using the stage alignment tool, which is a small clear plastic piece, with a small hole in the center and two larger holes near the outside.

1. **Insert the alignment screen.**

2. **Install the Stage alignment tool. Align the two outside holes to the adjustment positions of the x-y stage.**

3. **Turn on bottom illumination.**

4. **Using the multipurpose tool, or a 1.5 mm hex wrench, adjust the position of the x-y stage until the hole in the alignment screen is aligned to the hole in the stage alignment tool.** Be careful that your eye is directly above the hole in the stage alignment tool during adjustment. The 5x loupe can be used to aid this alignment, however, it is very important that it is aligned exactly between your eye and the hole.

3.7.1. *To Verify the Alignment:*

1. **Lower the sample and start the rotation.**

2. **Find the center of rotation by looking through the microscope or at the camera live view.**

3. **Verify that the hole in the alignment screen does not move by more than about 125 um.** This is sufficient for aligning the guns, since it is significantly smaller than the beam diameter.

3.7.2. *To Fine Tune the Alignment:*

1. **Bring the stage to home position and note the position of the center of rotation with respect to a known feature on the alignment screen.**

2. **Raise the stage and vent.**

3. **Use the hex key and move the center of the alignment screen to the center of rotation.**

4. **Repeat 2-6, for fine adjustments.**

3.8. Centering the Point of Interest

After the alignment screen is centered, the center point of the alignment screen is the same as the center of rotation. Ion guns can then be aligned with respect to this same point. This means that if the x-y stage is moved, and any specific point in the sample is brought to the center of rotation, that point will be milled. This is specifically useful when a sample is imaged in TEM and needs further milling in the PIPS, or when ion gun damage in FIB samples need to be removed.

In order to move the point of interest on the sample to the center of rotation:

1. **Insert the sample.**

2. **Lower the stage and start the rotation.**

3. **Find the center of rotation by looking through the microscope or at the camera live view.**

4. **Bring the stage to home position and note the position of the center of rotation with respect to a known feature on the sample.**

5. **Raise the stage and vent.**

6. **Use the hex key and move the point of interest to the center of rotation.**

7. **Repeat 2-6, for fine adjustments.**

If the PIPS II has a camera system, Digital Micrograph (DM) can be used to aid this process:

1. **Insert the sample, with the front of the specimen post facing the front of the system and the two arms facing the left and right sides.** The sample will be milled from the front.

2. **Lower the stage.**

3. **Activate Live View mode at 1x zoom in DM.**

4. **Find an easily recognizable feature on the sample that stays in the field of view of the camera during the entire rotation, but is not near the center of rotation.** We will refer to this as the alignment feature.

5. **Go to the Alignment tab on the PIPS II GUI.**

6. **Touch the Align Front Left Beam Sector button.** The stage will rotate and stop when the front of the specimen post is facing the left gun.

7. **Using the line tool in DM, place the start of a line at the alignment feature and the end of the line on the other side of the sample.** It is not important where the end of the line is place, as you will move it shortly.

8. **Touch the Align Rear Left Beam Sector button on the Alignment tab.** The stage will rotate and stop when the rear of the specimen post is facing the left gun. Align the end of the line with the alignment feature.

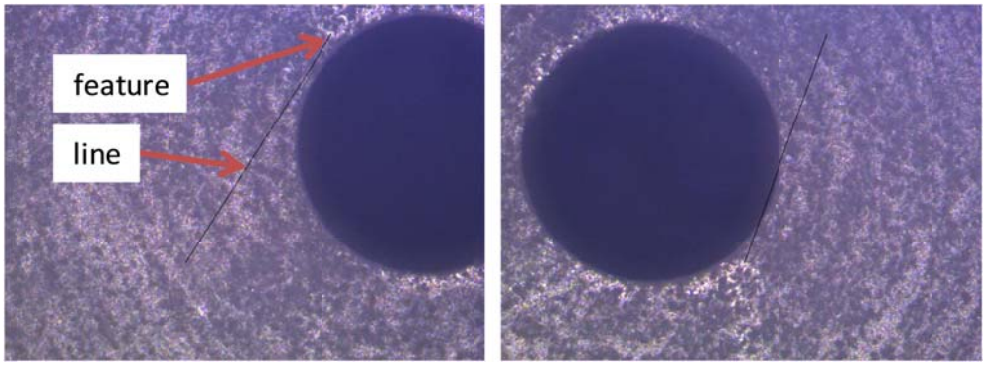

Figure 3-43 x-y alignment, steps 7 (left) and 8 (right).

Images are of a beam alignment screen.

9. **Touch the Align Front Right Beam Sector button.** The stage will rotate and stop when the front of the specimen post is facing the right gun.

10. **Using the line tool in DM, place the start of a new line at the alignment feature and the end of the line on the other side of the sample.** It is not important where the end of the line is place, as you will move it shortly.

11. **Touch the Align Rear Right Beam Sector button on the Alignment tab.** The stage will rotate and stop when the rear of the specimen post is facing the right gun. Align the end of the line with the alignment feature.

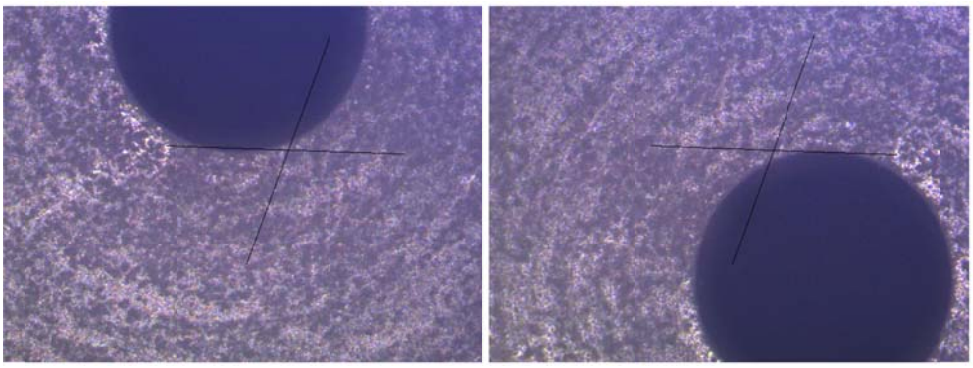

Figure 3-44 x-y alignment, steps 10 (left) and 11 (right).

Images are of a beam alignment screen.

12. **Touch Align Home. After rotation stops, use the line tool in DM to place a line that starts at the place where the two previous lines cross and ends at the part of the sample that you want to be at the center of rotation.** In the example shown, we want the center of rotation to be in the center of the hole of this alignment screen.

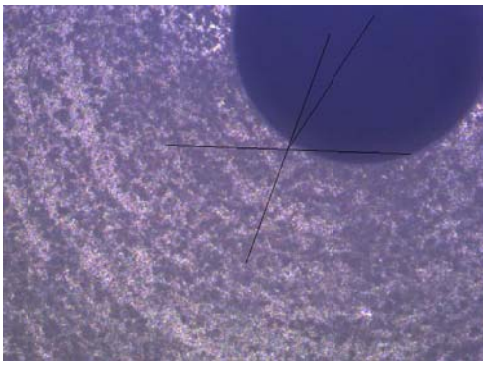

Figure 3-45 x-y alignment, step 12.

13. **Raise the stage. Remove the airlock cover. Raise the microscope and focus on the sample.** The position of the sample will have moved from where it was in the lowered position.

14. **Click and hold on the center of the last line you created. Drag the line, until the end of the line is at the part of the sample that you want to be at the center of rotation.** If this part of the sample is not in the field of view of the microscope, move the microscope x-y stage until it is, then move the line.

15. **Using the multipurpose tool, or a 1.5 mm hex tool, adjust the position of the x-y stage until the hole in the alignment screen is aligned to the other end of the line.**

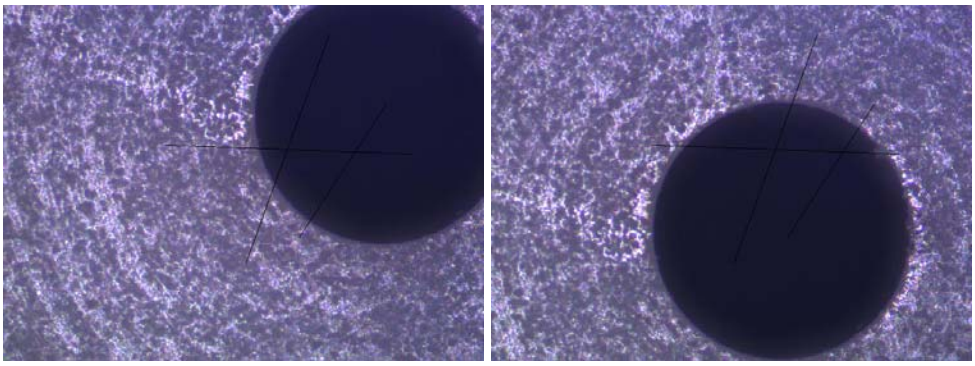

Figure 3-46 x-y alignment, steps 14 (left) and 15 (right).

5. **Repeat for finer adjustment if necessary.**

3.9. Gun Gas-flow Adjustment

The Automatic gas flow mode is designed to set the gas flow to an optimized value. Gatan recommends using this mode.

NOTE: The gun current readings displayed on the Milling tab is not a Faraday cup reading, as was used in previous versions of Gatan PIPS. This reading is now a measure of the current leaving the anode in the gun, as measured at the high voltage power supply. The best operating mode is for this current to be at its peak value.

NOTE: The optimum operating gas flow must be obtained once the guns have been thoroughly purged. If performing manual gas flow adjustment, adjust the flow one gun at a time.

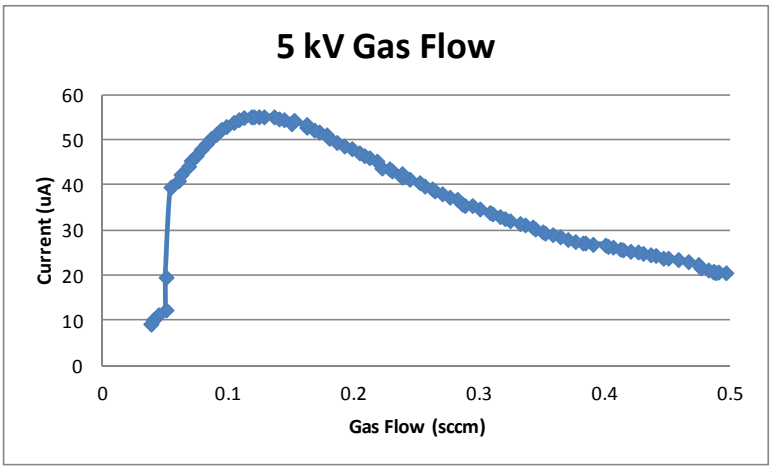

Figure 3-47 Operating characteristics of the PIG.

The gas flow can be adjusted either automatic or manually. For consistent and best performance, Gatan recommends setting the gas flow to automatic.

3.9.1. *To Adjust the Gas Flow Automatically*

1. **On the Milling page, select Automatic for the Gas Mode.**

2. **Set the milling energy.**

3. **Start milling.**

3.9.2. *To Adjust the Gas Flow Manually*

Note that as guns warm up, the beam current can drift. The guns should be warmed up for 10-20 minutes before setting the gas flow manually. The optimum gas flow changes with accelerating voltage, and it is necessary to adjust the gas flow whenever the accelerating voltage is changed. The curve in Figure 3-47 shifts to the right and increases in height as a gun warms up. If the curve shifts so that the operating point is far to the left of the peak, then it can be in an unstable region of the curve or the current may drop to an unusable level.

1. **On the Milling page, select Manual for the Gas Mode.**

2. **Set the Rotate Speed control to 3 rpm (Milling page, Gas flow controls).**

3. **Be sure Beam Modulator is turned off.**

4. **Lower the stage by sliding the slider on the Stage panel on Milling page.** This lowers the piston to its working position.

5. **Adjust the Ion Gun voltage to 5.0 keV.**

6. **Set the timer to 30 min and touch start.**

7. **Adjust the right gun gas flow.** Find the gas flow correlating to the peak current. A typical curve relating gas flow to ion current is shown inFigure 3-47. The operating point indicated has been chosen because it gives the most focused beam and the highest milling rate.

8. **Adjust the left gun gas flow.** Find the gas flow correlating to the peak current.

NOTE: Variations of ±20% in the performance of the two ion guns are typical and are caused by small differences in the properties of the rare-earth magnets used to enhance the gas-ionization rate.

3.10. Aligning the Beam

The ion beams produced by the PIGs contain both ions and fast neutrals. Electrostatic beam alignment does not work with the fast neutrals and the ion guns in the PIPS II must be aligned mechanically. This is done with the aid of the Beam Alignment Screen.

This Screen inserts into the standard specimen mount and is precisely positioned at the standard specimen height. It consists of a 7 mm diameter fluorescent screen with a 0.5 mm diameter hole at its center. After lowering the Screen to its standard working position the guns are turned on. The ion beams will be seen through as two blue lines intersecting at 120° on the fluorescent screen. A 5x loupe is provided to aid is viewing the beams.

NOTE: In older PIPS models it was common to align both guns at + 10° whether the guns were used for Top or Bottom milling. Currently we recommend the users to align:

- The gun that is being used for top milling, at + 10° using the beam alignment screen. Then tilt the gun to +5°, and verify that the alignment is still accurate. If necessary adjust the alignment.
- The gun that is being used for bottom milling, at - 10°, using a glass slide mounted on a glue type duo post. Then tilt the gun to -5°, and verify that the alignment is still accurate. If necessary adjust the alignment.

3.10.1. *To Align a Beam for Top Milling*

1. **Insert the alignment screen and lower the stage**.

2. **On the Milling page:**

    a. Set the stage rotation speed to 6 RPM.

    b. Set the gun voltage to 5 KeV.

    c. Set the gas flow control to Automatic.

    d. Set the modulation to Off.

    e. Set the timer to 30 min.

    f. Start milling.

3. **Go to Alignment page and bring the stage to the Front Beam Sector of this gun.**

4. Rotate the gun to 10° Top. This is done by manually turning the guns to 10° Top position or setting the Gun tilts to + 10° on the Milling page, when motorized guns are available.

5. Turn off the beam for the other gun (set the other gun to Disabled).

6. Adjust the z-alignment drive screw (vertical adjust). While viewing the beam crossing the screen, use the multipurpose tool or a 2 mm hex tool to adjust the z-alignment drive screw (Figure 3-42).

Note that a portion of the line crossing the screen has a higher intensity than the rest. The higher-intensity zone is elliptical in shape and several millimeters in length. Adjust the drive screw to center this zone over the center hole in the screen.

Figure 3-48 X and Z-alignment device screws.

7. Adjust the x-alignment drive screw (horizontal adjust). Adjust the drive screw to center the beam over the center hole in the screen (Figure 3-43).

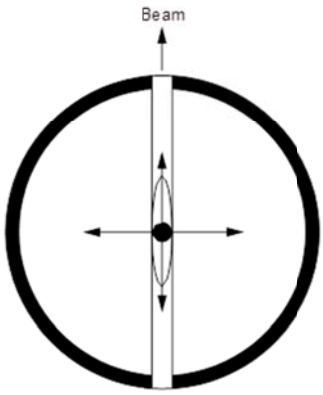

Figure 3-49 Alignment ellipse observed in the beam.

8. Start rotating the stage (touch Rotate) and watch the center of the alignment screen. Make sure the center is not wiggling too much (>~125 um). If it does, align the beam to center of the movement.

9. Rotate the gun angle to + 5° and watch the position of the beam as you are rotating the gun. Adjust the alignment if needed. When the gun is rotated, the beam should not move too far away from the center of the alignment screen.

10. Turn on the beam for the other gun (set the other gun to Enabled).

3.10.2. *To Align a Beam for Bottom Milling*

1. Insert the glue top duo post that has a piece of glass slide mounted on it. This is used to view the beam that is milling from bottom.

2. On the Milling page:

a. Set the stage rotation speed to 6 RPM.

b. Set the gun voltage to 5 KeV.

c. Set the gas flow control to Automatic.

d. Set the modulation to Dual.

e. Set the timer to 30 min.

f. Start milling.

3. Go to Alignment page and bring the stage to the Front Beam Sector of this gun.

4. Rotate the gun to -10° bottom. This is done by manually turning the guns to -10° position or setting the Gun tilts to - 10° on the Milling page, when motorized guns are available.

5. Adjust the z-alignment drive screw (vertical adjust). While viewing the beam crossing the glass slide, use the tool provided to adjust the z-alignment drive screw.

Note that a portion of the line crossing the glass slide has a higher intensity than the rest. The higher-intensity zone is elliptical in shape and several millimeters in length. Adjust the drive screw to center this zone over the center point of the glass slide.

6. Adjust the x-alignment drive screw (horizontal adjust). Adjust the drive screw to center the beam over the center point of the glass slide.

7. Go to Alignment page and bring the stage to the Rear Beam Sector of this gun. Adjust the beam alignment if necessary and make sure the beam is centered as this position as well (repeat 6 and 7).

8. Start rotating the stage (touch Rotate) and watch beam at front and rear positions. Make sure the gun is aligned at both positions. IF the alignment is different for the front and rear positions, it may be that the glass slide is not horizontal. In this case find a compromise between the front and rear positions.

9. Rotate the gun angle to - 5°. As you are rotating the gun watch the position of the beam (for both front and rear beam sectors. Adjust the alignment if needed. When the gun is rotated, the beam should not move too far away from the center of the alignment screen.

Note that narrowing the beam (by increasing the gas flow to the gun) decreases the amount of the beam incident upon the specimen. (The reverse is true for a smaller ion current.) Some flickering of the beams across the screen is normal and is due to electrostatic charging and discharging of the screen, which is an insulating material.

Once the guns have been aligned, it should not be necessary to realign them for several samples. When polishing the first specimen, it is recommended that the ion current and the beam angle be noted. When polishing subsequent specimens, adjust the parameters to these values to provide a good degree of beam calibration.

3.10.3. *Installing gun alignment knobs*

The set screws used for alignment can be replaced with optional gun alignment knobs. This allows for faster alignment since a tool is not necessary. Since the knobs are easily accessible, they may also be accidentally bumped, which could cause misalignment. Be very careful not to bump the knobs during operation.

To install gun alignment knobs:

1. Using the alignment tool, remove the set screws from the gun knob.
2. Install the gun alignment knobs. The shorter version should be installed in the hole closest to the “TOP” label.
3. Align guns as described above.

Some systems include gun alignment knobs as part of the accessory kit. They may also be ordered as a kit: 695.09816 Kit, Alignment Knob; which includes 2 of each.

Figure 3-50 Gun knob with gun alignment knobs installed.

Caution: Gun alignment knobs MUST NOT be used with motorized guns. Doing so will result in damage to the system that is not covered by warranty.

3.11. Ion-beam Modulation

Ion-beam modulation is used primarily for polishing cross-sectional TEM materials that are "glued" together or have interfaces of materials of different hardness. Beam modulation consists of fast on/off electronic switching of the guns with variable specimen-rotation speeds within polishing sectors to minimize differential thinning rates of specimens. Variable rotation speeds within the sector of up to 6 rpm are achieved while outside the polishing sector, the speed is fixed at 12 rpm to reduce total specimen preparation time. With this feature, the ion beam is turned off when the support arms of the DuoPost enter the path of the ion beam. This effectively reduces specimen contamination of sputtered material from these parts and extends the life of the post in addition to providing higher quality specimens. On particularly fragile specimens, it is preferable to simultaneously work the top and bottom of the specimen to prevent stresses that could break the specimen.

NOTE: When loading a cross-sectional specimen, it is important to insert the specimen such that the cross-sectional interface (glue line) is parallel to the front panel (home position). This procedure ensures that the polishing sectors will be ±30° normal to the glue line during operation of the Beam Modulator. It is also important to note that the specimen will automatically rotate to this home position before the piston will rise into the Airlock.

The default sector size is ±30°. This can be changed for all samples on the Options tab, or for specific samples by using a custom recipe.

3.11.1. *Ion-Beam Modulation Selection*

This panel enables selection of single- or double-sector mode:

Single Modulation: The system is operating in the single-sector mode. This activates each gun during the polishing sector when the front of the specimen post is facing that gun. The stage rotates at the milling speed during the polishing sectors and at 12 rpm between the polishing sectors to reduce total specimen-preparation time.

Dual Modulation: The system is operating in the double-sector mode. This activates each gun during the polishing sector when the front and rear of the specimen post is facing the gun. The stage rotates at the milling speed during the polishing sectors, and at 12 rpm between sectors.

No Modulation: Beam modulation is disabled and there is continuous milling.

Stationary Left: The stage does not rotate, and the left gun is on continuously. The right gun is off. The desired stage rotation position must be set prior to pressing Start.

Stationary Right: The stage does not rotate, and the right gun is on continuously. The left gun is off. The desired stage rotation position must be set prior to pressing Start.

Custom Modulation: This feature allows the user to define the start and stop angles for up to two custom defined sectors. This can be useful for cleaning up FIB samples that are mounted on the side of a grid-bar, where you may want front and rear sectors that are asymmetric. The angle entries are the stage angles when the guns should turn on and off. Positive angle entries must be used.

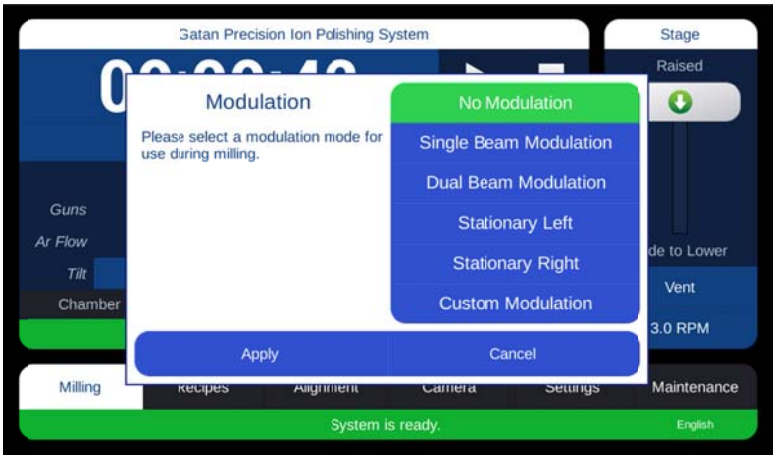

Figure 3-51 Modulation modes.

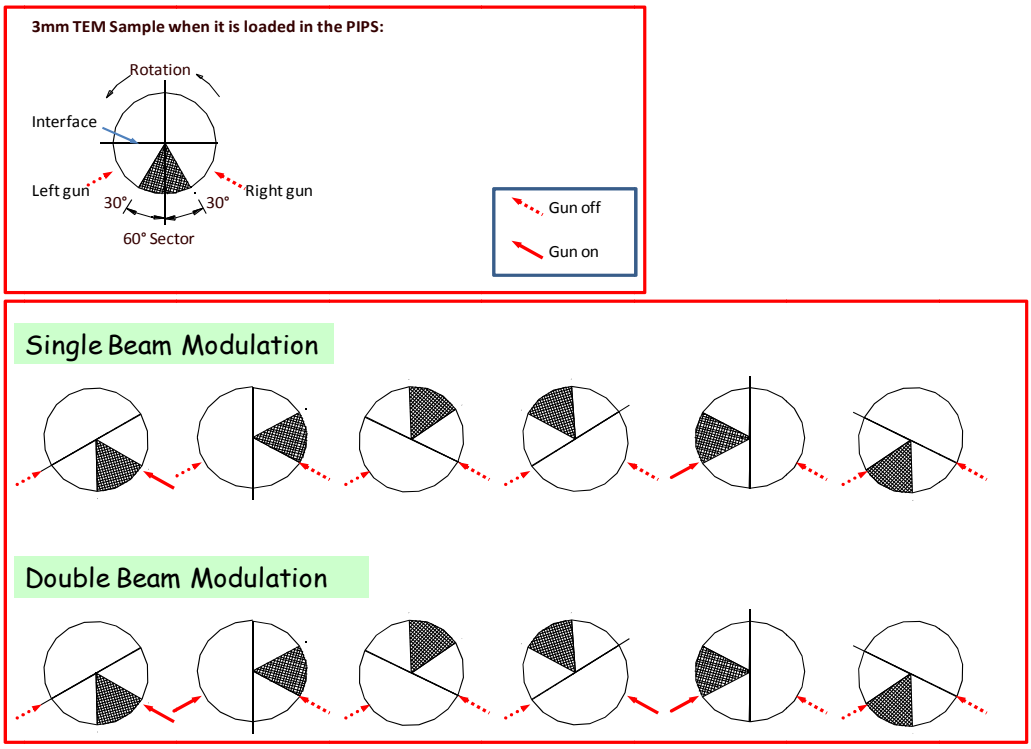

Figure 3-52 Beam modulation.

3.12. Manual Shutdown Procedure

Main power to the PIPS II (power consumption of less than 100 W) should be left on at all times to provide for more efficient trouble-free operation. The vacuum will be continuously maintained resulting in a cleaner system with shorter pump downs and minimum purging requirements of the PIGs.

Prior to shutting down the system, the stage should be raised and any sample removed from the specimen mount. Once the power is turned off, the airlock control will not function.

To shut down, simply turn off the power switch on the rear panel of the instrument.

4. Specimen Preparation

4.1. Disk Preparation

All TEMs manufactured today require samples that are 3.05 mm in diameter. The following methods are suggested to prepare plan view 3mm discs from bulk material:

Polish, slice, or cleave the starting bulk material to obtain a slab about 500 µm thick.

NOTE: For brittle materials (ceramics, geological materials and semiconductors), the Gatan Model 601 Ultrasonic cutting tool is ideal to core or cut 3 mm discs. For ductile materials (metals and alloys), use the Model 659.00001 Disc punch to punch discs without mechanical damage to the central region or tearing of the edges.

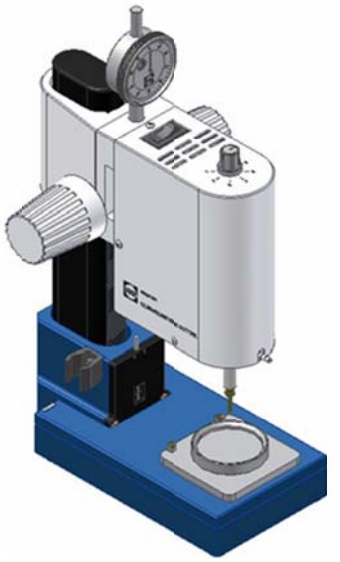

Figure 4-1 Gatan model 601 ultrasonic cutting tool.

Figure 4-2 Gatan model 659.00001 disk punch.

4.1.1. *Mechanical Pre-thinning*

The importance of pre-thinning prior to ion polishing cannot be overemphasized. Pre-thinning is usually done by mechanical grinding and polishing but chemical polishing can sometimes be used to advantage. Ion polishing is a relatively slow process and pre-thinning can greatly reduce the time required to make a TEM specimen. There is, however, a tendency to develop a surface roughness during ion polishing and the shorter the ion-polishing time, the smoother the TEM specimen.

It is now possible to thin specimens mechanically all the way to electron transparency so that ion-polishing can be avoided altogether. Unfortunately, mechanically-thinned specimens are difficult to clean and usually show artifacts due to mechanical damage that can make them unsuitable for most TEM work. For this reason, our recommended procedure is to mechanically pre-thin as much as possible but not to less than about four times the depth of the mechanically damaged layer.

The damage depth depends on many factors, i.e., the material being thinned, the polishing compound used, and the polishing force applied. For semiconductors, ceramics, and minerals, it appears that TEM specimens free from mechanical damage can be obtained if mechanical pre-thinning is taken down to about 5 µm. For metals, the pre-thinning thickness is generally greater than this and usually the optimum value must be determined on a trial and error basis by pre- thinning to a smaller and smaller thickness until artifacts start to appear in the TEM specimens.

TEM cross-sectioned samples are essential for studying the microstructure of multilayered materials. Preparation of cross sections is somewhat more involved than the preparation of regular plan view discs. The preparation of

cross sections is greatly facilitated by the use of the Gatan Model 601.07000 Cross-section kit. Follow these basic steps, described in detail in the following sections, to prepare cross-sectional samples:

Disk Grinding

Use the Gatan Model 623 Disc Grinder to mechanically thin the previously prepared 500 µm thick sample discs down to about 40-70 µm.

Figure 4-3 Gatan model 623 disk grinder.

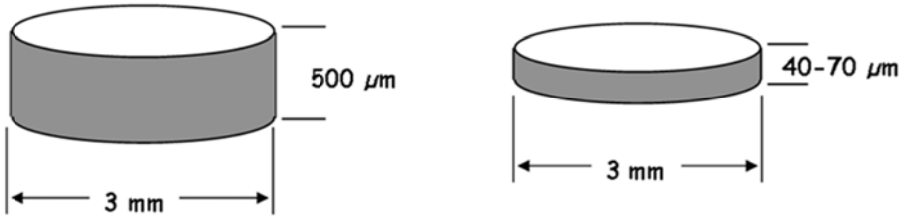

Figure 4-4 Disk grinding: initial and final.

Dimple Grinding

Dimple grinding produces a large amount of thin area in the center of the sample disc surrounded by a thick rim (Figure 4-5). This provides the mechanical strength required for subsequent sample handling. Dimple grinding is achieved using the Gatan Model 656 Dimple Grinder.

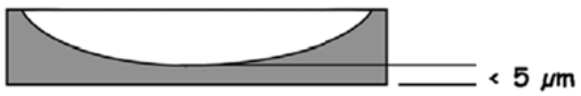

Figure 4-5 Sample disk after dimple grinding (not to scale).

Figure 4-6 Gatan model 656 dimple grinder.

Dimple diameter and depth

Since the beam angle for ion polishing may be very small, one must be careful that the rim around a dimpled specimen does not cast a shadow over the central region. The relationship between the diameter of the dimple grinder wheel (D), the diameter of the dimple (2r), and the dimple depth (d) is given by the approximation, $d = r^2/D$.

Typically, the width of the rim around a 3mm diameter specimen is 0.4 mm and hence the dimple diameter, 2r, is 2.2 mm. In this case, the maximum dimple depth without forming a shadow at the center of the specimen under an ion beam at 4° to the horizontal is 1.1 x tan 4° = 77 µm. The table below shows the dimple depth for various diameters of polishing wheel (dimple diameter is 2.2 mm).

Table 1 Polishing wheel diameter versus dimple depth

Polishing wheel diameter (mm)	Dimple depth (µm)
10	121
15	80
20	60
25	48

The table indicates that a 10mm diameter polishing wheel is not suitable for making PIPS II specimens since the dimple depth is greater than 77 µm and the center of the dimple will be shadowed. The 15mm wheel would seem to be border line but can produce good results in practice since the edge of the rim gets milled away very quickly during the initial stages of polishing.

Optimum initial specimen disc thickness

Table 2 uses the above data to estimate the optimum initial specimen disc thickness for specimens dimpled from one side. As a rule-of-thumb, for a 2.2 mm dimple diameter, the initial disc thickness equals dimple depth plus 4 times the thickness of the damaged layer.

Clearly, as the diameter of the wheel increases, the initial thickness of the specimen disc should decrease. Large diameter wheels have the advantage of producing large thin areas of specimen, but they also produce more fragile specimens. A good compromise is to use flat dimple grinder wheels that give a larger flat central area without reducing the strength of the surrounding support rim. Of course, if a flat wheel is used, the initial dimple depth should not be greater than approximately 77 µm; otherwise shadowing effects will occur.

Table 2 Optimum initial specimen disc thickness

Polishing wheel diameter (mm)	Damage layer thickness (µm)	Thickness at the base of dimple (µm)	Optimum initial disc thickness (µm)
15	1	4	84
15	2	8	88
15	2	12	92
15	4	16	96
20	1	4	64
20	2	8	68
20	2	12	72
20	4	16	76
25	1	4	52
25	2	8	56
25	2	12	60
25	4	16	66

Specimen disc geometry

Figure 4-7 shows the recommended geometry of PIPS II specimens produced by dimpling. Note that specimens are normally dimpled from one side only. The flat side of the PIPS II specimen is normally prepared using a disc grinder followed by rough polishing with a dimple grinder using 3µm diamond paste on a felt wheel and final polishing with 0.25µm diamond paste or 0.05µm alumina suspension. It has been found that cubic boron nitride (CBN) pastes instead of diamond paste can be successfully used for dimpling ductile materials, e.g., metals and alloys. This is due primarily to the fact that CBN particles do not become easily embedded in the materials being polished.

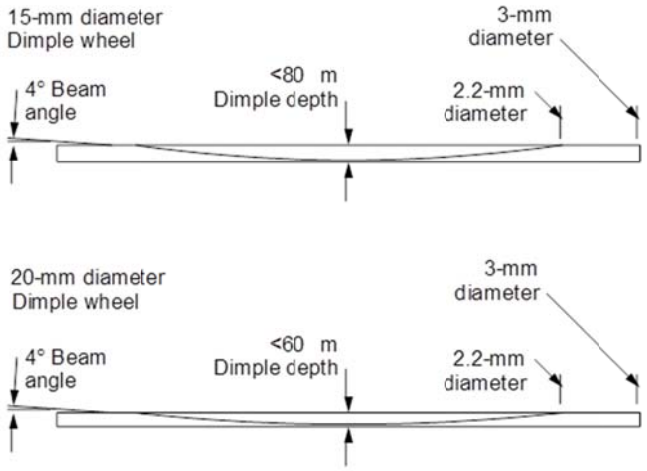

Figure 4-7 Specimen disc geometry.

The PIPS II does not use any specimen clamps and hence it will accept a large variety of specimen shapes. Rectangular TEM tensile specimens can be mounted directly on a modified specimen post and dimpled in place prior to ion polishing.

To obtain best results with dimple grinding, follow the steps described below (Refer to the Gatan Model 656 Dimple Grinder User's Guide for detailed operating instructions.)

1. After disc grinding to 40-70 µm, coarse dimple grind the sample with the 15 mm phosphor bronze wheel to a thickness of 20-25 µm using 2-4 µm diamond paste, 15-20 g load and low to medium speed.

2. **Dimple polish with felt wheel for 3-5 min using 2-4 µm diamond paste, 20-25 g load and low to medium speed.** (This is equivalent to a thickness of about 10 µm when measurable, and is possible with transmitted light for certain ceramics and semiconductors.)

3. Dimple polish with a new felt wheel for 5-6 min (or to a thickness of 6-8 µm) using 0-2 µm diamond paste, 20-25 gm load and medium speed.

4. Dimple polish with a new felt wheel for 8-10 min (or to a thickness of about 5 µm) using 0.05 µm Alumina suspension, 20-30 gm load and medium speed.

NOTE: Use different felt wheels for different abrasives to avoid cross-contamination.

Please note that the loads and speeds suggested above work best for silicon and silicon-based materials. For materials that are more brittle, lower loads

and speeds are preferable. For most metals and alloys, slightly higher loads and speeds work better.

Also note that samples are normally dimpled from one side only. However, it is extremely important that the side chosen to be the flat side must first be fine polished (0.1µm abrasive or lower) prior to dimple grinding.

4.1.2. *Chemical Pre-thinning*

Pre-thinning of specimen discs is sometimes carried out by electrolytic or chemical jet polishing. Subsequent ion polishing is performed in order to better control the final thickness of the electron-transparent specimen or to reduce the effects of differential chemical attack in multiphase specimens. Chemically pre- thinned specimens have the advantage of being free from mechanically induced artifacts and can be made extremely thin prior to ion polishing. Ion polishing is also useful for cleaning up electro-polished specimens that have become contaminated during storage or have areas that are a little too thick for satisfactory TEM imaging or analysis.

4.1.3. *Cross Sections*

Ion-milled cross-sectioned TEM specimens are used extensively for studying the microstructure of multilayered materials. Usually, the layers in such specimens have differing mass thicknesses requiring different milling rates. By milling at low angles, the PIPS II can minimizes differential thinning effects of cross-sectional TEM specimens. However, milling at low angles alone is insufficient to prevent the preferential thinning that occurs when the ion beams pass along the cross-section interface. This preferential thinning is reduced by using Ion-Beam Modulation.

The initial preparation of cross-sectional discs is greatly facilitated by the use of a special kit developed by Gatan:

1. Cut rectangular wafers. The pre-sliced specimen is glued face down with mounting wax onto a glass slide. The glass slide is secured to the specimen table of the Gatan Ultrasonic Disc Cutter with mounting wax. The disc cutter is fitted with a 4 × 5 mm cutting tool to cut out rectangular wafers (Figure 4-8, Step 1).

2. Make the specimen stack. A specimen stack is made consisting of six 4 × 5mm wafers and stacked as illustrated in Figure 4-8, Step 2.

The wafers having the surface or interface of interest should be located face to face in positions 3 and 4 in the middle of the stack. In many cases, the original specimen slice is only large enough to obtain one or two wafers so the remaining four or five wafers are dummy ones of pure silicon. One great advantage of using silicon for the dummy wafers (even for cross sectioning other materials) is that their interference fringes can be used to accurately gauge the specimen thickness at the 2-5 µm level during dimpling. The

dummy and specimen wafers are coated with a thin layer of G-1 epoxy. G-1 has characteristics that are similar to M-Bond 610 epoxy but offers the following advantages: Fast curing time (5-10 min at 130 °C), Ease of filling both thick and thin gaps between wafers, Long shelf life (1 yr without refrigeration), High temperature stability (heated in a TEM hot stage up to 1000 °C)

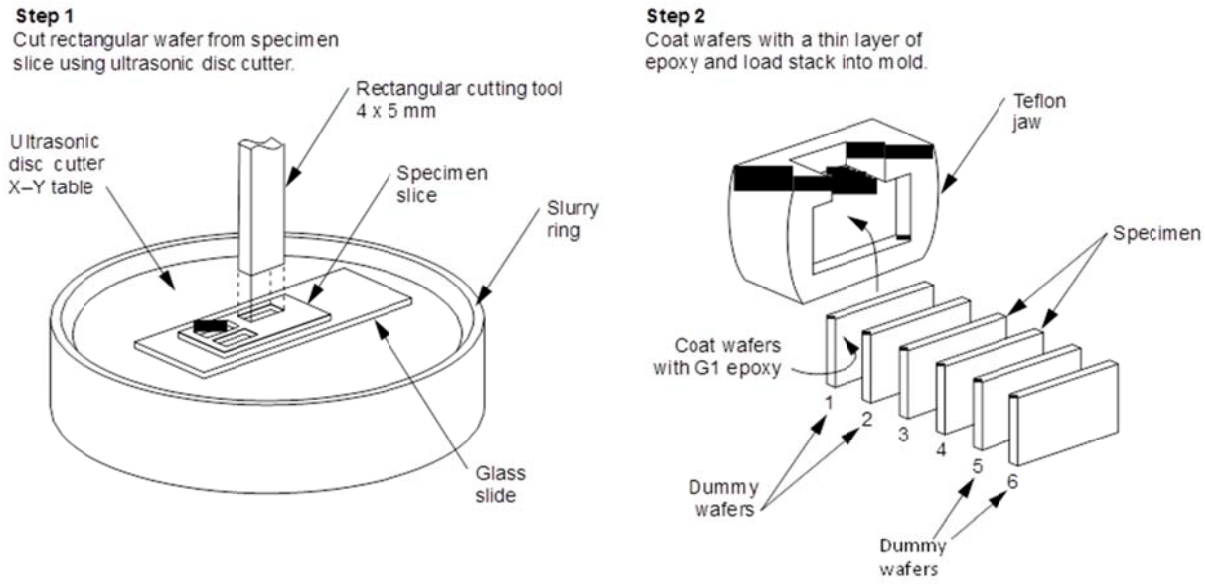

Figure 4-8 Cross section specimen preparation.

3. Pressure bond the specimen stack. A spring-loaded vise is used to bond the specimen stack together and the stack is cured under pressure for 10 min at 130 °C on a hot plate to obtain a strong bond with minimum glue thickness. When the curing process is complete, the assembly is cooled to room temperature (Figure 4-9, Step 3).

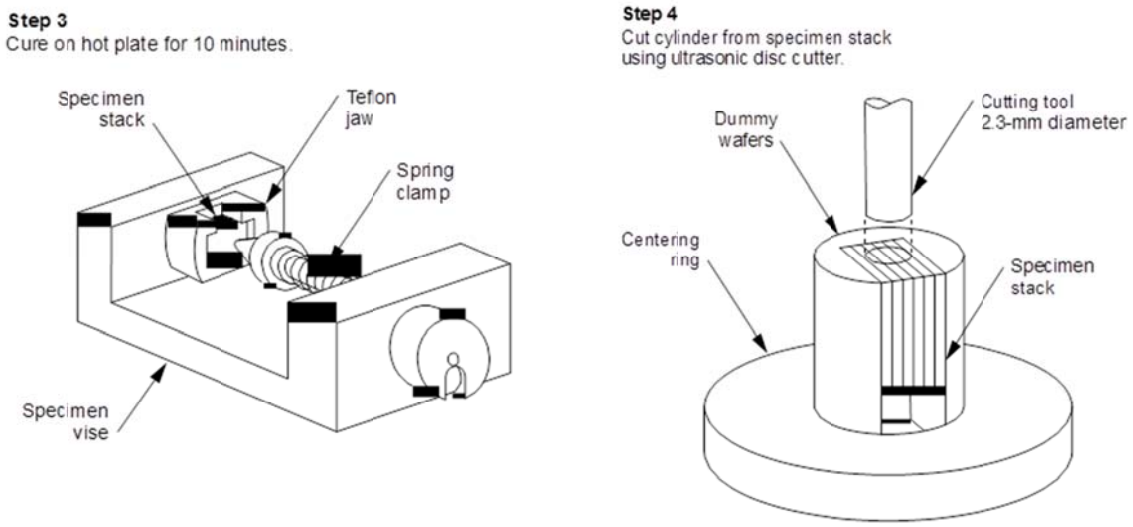

Figure 4-9 Cross section specimen preparation.

4. Cut the cylindrical specimen stack. The stack is glued into a slotted specimen mount with mounting wax. A 2.3 mm diameter cutting tool is used to cut a cylinder from the middle of the stack (Figure 4-9, Step 4)

5. Strengthen the cylindrical specimen stack. The 2.3 mm cylinder is now glued with G-1 epoxy inside a 3mm diameter metal reinforcing tube and cured for 10 min on a hot plate at 130 °C (Figure 4-10, Step 5). The tube holds the fragile cross-sectioned structure together during grinding, dimpling, ion milling (especially low-angle milling), and subsequent clamping in the TEM specimen holder.

6. Slice the specimen discs. The metal reinforcing tube containing the specimen cylinders is sliced into a series of 250-400 µm thick discs with a thin blade diamond saw (Figure 4-10, Step 6). The sliced disc is then ground flat from both sides to a thickness of approximately 80 µm using the Gatan Precision Disc Grinder and dimpled according to the procedure described in Section 4.2.

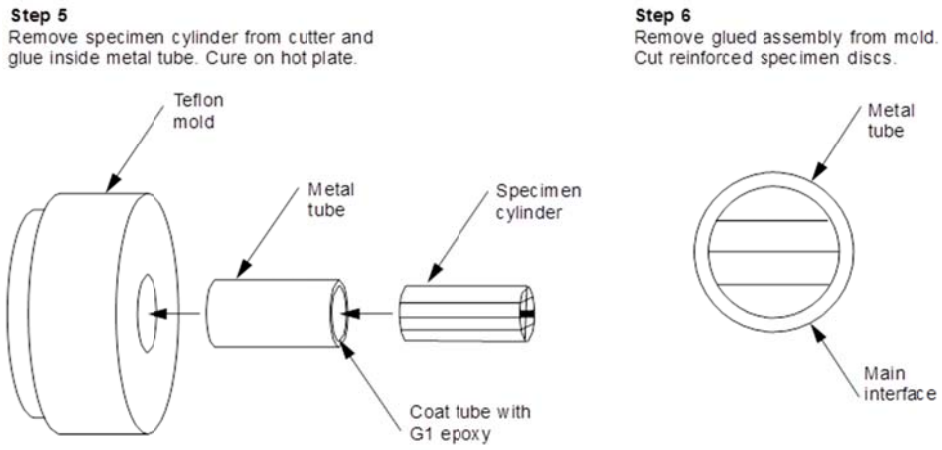

Figure 4-10 Cross section specimen preparation.

Dimpling is generally performed on only one side of the specimen and that side of the disc is mechanically polished using felt polishing wheels on the dimple grinder as a micro-polishing device.

4.2. Specimen Mounting

After pre-thinning, the sample discs are mounted on a PIPS II sample holder and placed into the PIPS II for ion milling to electron transparency. The sample holder plugs into the sample-mount assembly located in the airlock chamber and can be lowered pneumatically to the working position for ion polishing. The PIPS II does not use any sample clamps and hence it will accept a large variety of sample shapes.

Five kinds of sample holders are available with the PIPS II:

- DuoPosts (clamp-type and glue-type)
- Graphite holder
- Molybdenum rimmed post
- Copper sample post

4.2.1. *DuoPosts*

DuoPosts are available in two kinds: glue-type and clamp-type. The glue-type DuoPost is used in conjunction with wax to secure the sample. The clamp-type DuoPost employs spring loaded arms to secure the sample in the recess and is used if securing with wax is undesirable. However, using a glue-type holder has its advantages in that the heat transfer rate is much greater due to increased contact area. Both posts allow the use of the transmission illuminator and the Auto- Terminator.

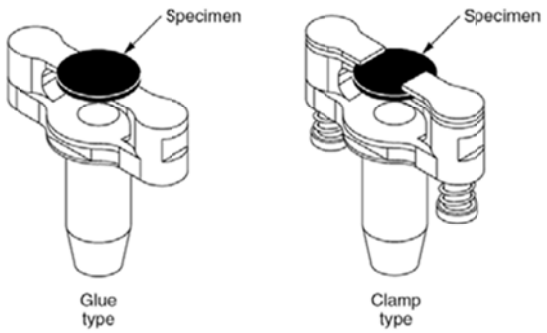

Figure 4-11 DuoPosts, glue type and clamp type.

An important aspect of the DuoPost is its use in conjunction with the beam-modulation feature of the PIPS II. When mounting cross-sections, the main interface of the sample should be aligned parallel to the support arms of the post and the post then inserted into the sample-mount assembly with the arms parallel to the front panel. This procedure ensures the polishing sectors will be ±30° normal to the interface when using the beam modulator (see Section 2.9 on page 30), thereby effectively minimizing differential milling.

Clamp-type DuoPost

The clamp type DuoPost offers fast and easy sample loading. A loading dock is available for use with the clamp-type DuoPost to facilitate sample loading and unloading. Follow the procedure described here for use with this loading dock.

NOTE: This procedure is best performed under a stereo microscope.

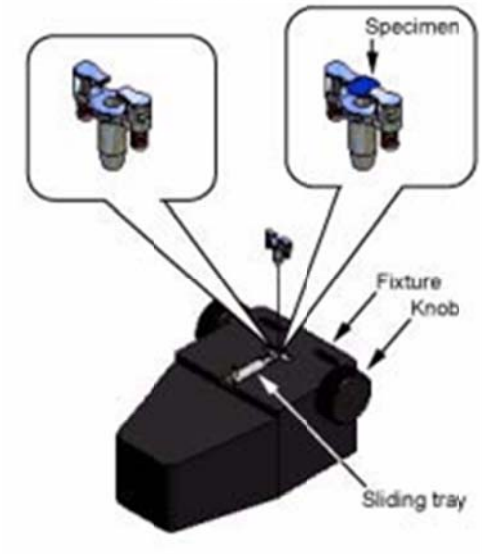

Figure 4-12 Clamp-type post and loading dock.

To mount a sample on a clamp-type DuoPost:

1. **Slide the tray back completely.**

2. **Place the clamp-type DuoPost into the recess of the loading dock. Use the grooves provided on the post to hold with tweezers.**

3. **Rotate the knob to open up the gap in the clamp.**

4. **Rotate one of the knobs on the side of the fixture to open the gap between the upper and lower arms of the clamp.**

5. **Place the sample into the recess of the sliding tray and slide the tray forward. This will locate the sample in the center of the clamp.**

6. **Rotate the knob in the opposite direction from step 2, until you hear one click. This lifts the lower arms of the post and lifts the sample from the tray.**

7. **Carefully retract the tray from between the arms.**

8. **Rotate the knob in the same direction until you hear another click. This step makes the arms of the posts firmly clamp the sample.**

9. **Rotate the knob in the same direction again, till you hear a third click. The post is now raised and ready to pick up with a pair of tweezers.**

To remove a sample from a clamp-type DuoPost:

1. **Transfer the DuoPost to the loading dock and slide the empty tray beneath the sample.**

2. **Rotate the knob on the side of the fixture to open up the gap between the upper and lower arms of the clamping post.** This motion will place the sample onto the tray.

3. **Retract the tray and carefully lift the sample from the tray.**

Glue-type DuoPost

The glue-type holder requires the use of wax to secure the sample. As a result of the increased contact area, heat transfer during ion milling is greater. Therefore, the glue-type DuoPost is preferred when the sample is heat sensitive.

To mount a sample on a glue-type DuoPost:

Prepare the mounting wax.

1. **Using a clean Petri dish, dissolve a small amount of mounting wax in an equal volume of acetone.**

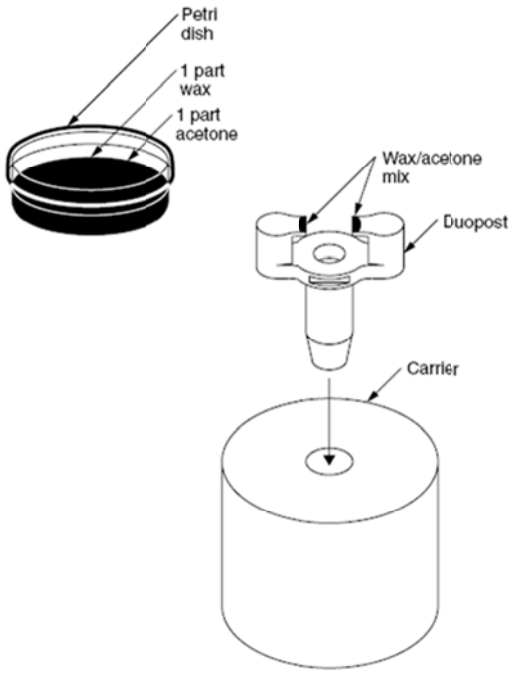

Figure 4-13 Mounting a sample, steps 1 and 2.

2. **Place the sample post in the carrier provided and use a tooth pick to place a thin layer of the diluted wax around the recess edge of the post.**

   NOTE: This operation is best performed under a stereo microscope. Do not allow any wax to flow into the central hole of the sample post.

3. **Evaporate the acetone in air or use a hot plate to form an ultra-thin layer of dry wax. Position a sample (dimpled side up) on the sample post.**

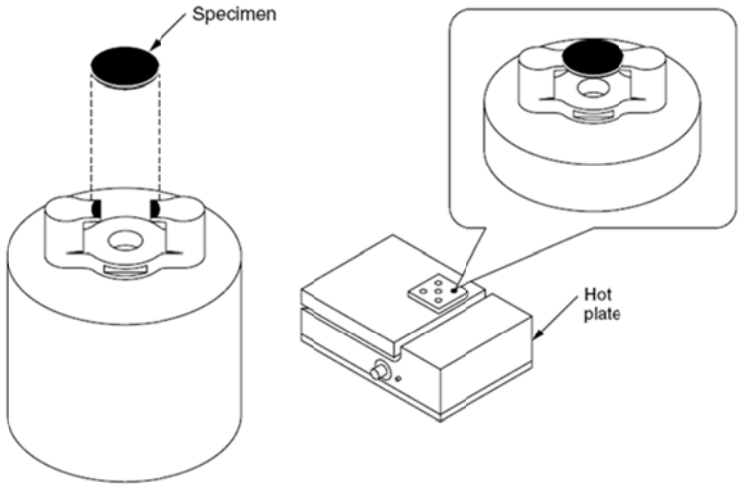

Figure 4-14 Mounting a sample, Steps 4 and 5.

4. **Carefully center the sample over the dry wax. Bond the sample to the sample post.**

5. **Transfer the complete assembly to a hot plate to melt the wax and bond the sample to the sample post.**

6. **The final position of the sample on the post can be set by quickly transferring the sample post in the carrier from the hot plate to a stereo microscope while the wax is melted.**

7. **Air cool to bond the sample. Transfer the sample post to the PIPS II.**

8. **Inspect the sample under the microscope to make sure the center region of the sample is clean, then transfer the sample post to the PIPS II for ion polishing.**

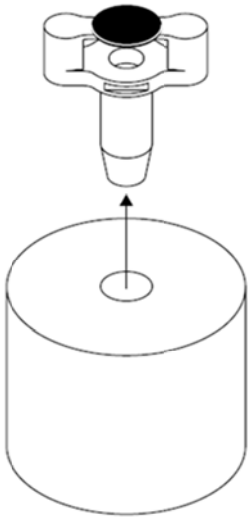

Figure 4-15 Mounting a sample, step 8.

To remove a sample from a glue-type DuoPost:

1. **Dissolve the wax in acetone.** Place the post with sample on a piece of lint-free tissue in a Petri dish containing clean acetone. After a few minutes, the sample will fall off the post onto the tissue.

2. **Transfer to a second clean acetone bath.** Pick up the wet tissue with the sample and post on it and transfer them to a second dish of clean acetone.

3. **Dry off excess acetone. Transfer the wet tissue with sample onto a clean, dry tissue to soak up any excess acetone.**

4.2.2. *Graphite Holder*

The graphite holder offers quick and easy sample loading/unloading (Figure 4-16). The sample is secured between two independent slides that grip the sample at edge. The graphite holder is most useful for very brittle samples. If heat transfer is a concern, the graphite holder can even be used with low melting point wax. A loading dock, with a built-in high intensity transmission illuminator, is available for use with the graphite holder.

Follow the steps described here for use with this loading dock. This procedure is best performed under a stereo microscope.

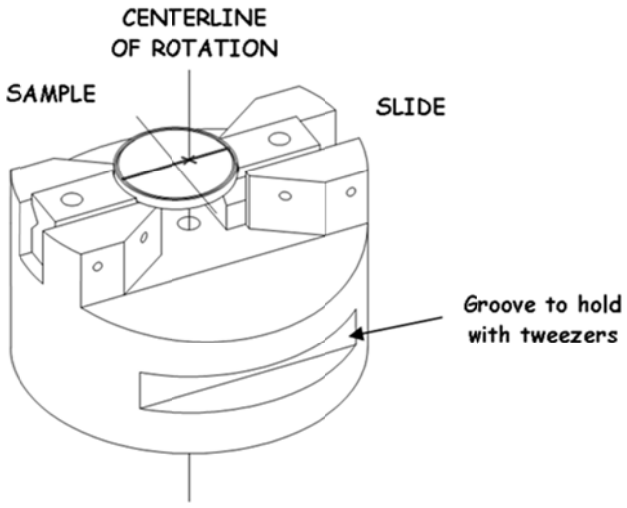

Figure 4-16 Graphite holder.

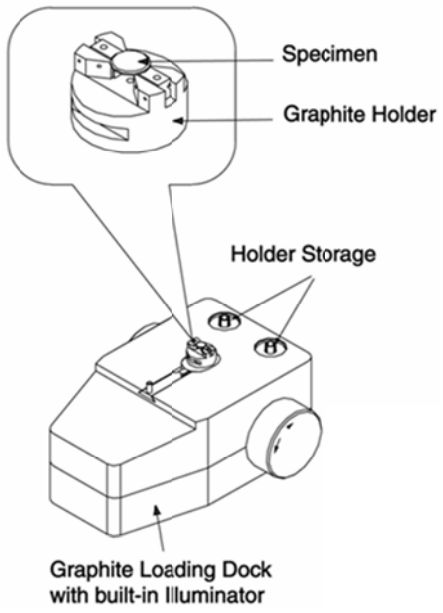

Figure 4-17 Graphite holder loading dock.

To Mount a Sample on a Graphite Holder:

1. **Completely retract the loading dock tray (Figure 4-17).**
2. **Rotate the knob forward completely to raise the post for the graphite holder.**
3. **Use the grooves on the graphite holder to hold with tweezers and gently plug the holder onto the designated post on the loading dock.**
4. **Rotate the knob back to completely lower the graphite holder.**
5. **Place the sample into the recess of the sliding tray and slide the tray forward.**
6. **Rotate the knob forward one click. This allows the slides of the graphite holder to lift the sample off the tray.**
7. **Carefully retract the tray completely.**
8. **Use the holder slides and the transmitted light (for materials that transmit light) to center dimple or align a specific area on the sample to the center of rotation in the PIPS II.**
9. **Rotate the knob forward two clicks. The post is now raised and ready to pick up with a pair of tweezers.**

NOTE: The transmitted light comes on and stays on for 3 minutes each time the knob is rotated.

4.2.3. *Molybdenum Specimen Post*

For specimens mounted without wax, Gatan has the molybdenum specimen post with a raised rim within which the specimen is placed. However there are heat transfer considerations with this post.

4.2.4. *Copper Sample Post*

Unlike the molybdenum post, the copper post does not have a raised rim, therefore sample mounting requires wax. Wax mounting, coupled with good heat conductivity of copper, makes this post very useful for milling extremely heat sensitive materials. However, ion milling is possible only from the top. Also, since copper sputters easily, if the ion guns are misaligned or continue to run after a perforation occurs, re-deposition can be a concern.

4.2.5. *Heat Transfer*

It should be understood that when a specimen is under vacuum and simply “rests” on a support post, the rate of heat transfer between the specimen and its surroundings is minimal. At high thinning rates, the ion beams from both guns can direct as much as 300 mW of power to the specimen and the

temperature rise in the specimen can be considerable. Cooling the specimen post to liquid nitrogen temperatures has very little effect on the specimen temperature since heat flow is dominated by the large thermal resistance at the minute points of contact between the specimen and its support.

In order to improve heat flow, a specimen must either be clamped mechanically to a support platform or attached to a single-sided post using a thin layer of mounting wax. The latter method is preferred because the heat-transfer rate is much greater due to increased contact area, low risk of specimen damage, and lack of shadowing of the beam at low angles.

In order to improve heat flow, a specimen must either be clamped mechanically to a support platform or attached to a single-sided post using a thin layer of mounting wax. The latter method is preferred because the heat-transfer rate is much greater due to increased contact area, low risk of specimen damage, and lack of shadowing of the beam at low angles.

4.3. Ion-beam Milling

Once the sample disc is pre-thinned and mounted on a sample post, it is then ready to be ion-milled to electron transparency. Follow the steps outlined below:

1. **Adjust the ion beam energy on the GUI Milling page (keV).**
2. **Check gun gas flow adjustment and beam alignment.**
3. **Load the sample holder into the sample mount inside the airlock chamber and lower it to the working position for ion milling.**
4. **Check if the point of interest is at the center of rotation.**
5. **Set the gun angles for each gun (either manually or by changing the Gun tilts on the GUI Milling page).**
6. **Set the rpm using the rotation speed control on the Milling page.**
7. **Set the desired time interval on the timer.**
8. **Set the beam modulation to “Single,” “Dual,” or “Off”.**
9. **Press the "Start" button on the Milling page.**
10. **After milling is complete, unload the sample holder from the airlock chamber.**

4.3.1. *Milling Rates*

Milling rates depend on the relative masses of the ion and sample atom, ion energy, atomic density and crystalline structure of the sample and the angle of incidence of the ion beam. The higher the beam energy and the beam angle, the faster the milling rate.

Shown below are some typical milling rates at 4° obtained for various materials using two ion guns operating at 5.0 keV, a gun current of 25 µA, and specimen rotation off. This is not a guarantee of performance, rather it is typical data noted on previous machines.

Table 3 Typical Milling rates

Material	Milling Rate (µm/hr/gun pair)
Copper	22
Silicon	24
Silicon Carbide	16
Stainless steel 316	14
Tantalum	8

4.3.2. *PIPS II Milling Parameters*

As mentioned earlier, higher beam energies (keV) and milling angles lead to higher milling rates. However, they also lead to relatively more damage (on the order of about one nanometer per keV) to the surface of the sample. Lower energies and milling angles produce not just a lower amount of surface damage but also a larger amount of thin area. Therefore, it is essential to arrive at a reasonable compromise between the amount of damage and the milling time.

While setting the milling angles, one must be careful that the rim around the dimpled sample does not cast a shadow over the central region. Table 4 shows the minimum milling angle that can be chosen, depending on rim/initial bulk thickness.

NOTE: Values are for samples dimpled down to 5 µm with a 15 mm wheel.

Table 4 Bulk/rim thickness vs. minimum milling angle

Bulk/rim Thickness	Minimum Milling Angle
40	3°
50-70	4°
70-100	5°
100-150	6°
150-200	7°

Use the sample recipe below as a starting guide to choosing PIPS II milling parameters. This recipe works best for a silicon-based sample of bulk thickness 60 µm top side dimpled to 5 µm with a 15 mm wheel. Note that because the milling rates have increased dramatically at low energy compared to the original PIPS, Gatan recommends obtaining a perforation at low energy (e.g. 0.5 keV) for samples that exhibit interference fringes.

Table 5 PIPS II milling parameters

Gun KeV	Gun angle	Beam Modulation	Rotation	Time
4.0	5° Top 3° Bottom	Dual Beam	3 rpm	Until clearly defined fringes appear
2.5	4° Top 2° Bottom	Dual Beam	3 rpm	Until inner-most fringe changes color
0.5	4° Top 2° Bottom	Dual Beam	3 rpm	Until perforation reaches area of interest

5. Routine Maintenance and Servicing

The maintenance operations listed in Table below should be carried out on a routine basis.

Table 6 Maintenance Operations

Operation	Frequency	Symptom
Clean Viewing Port.	Weekly	Specimen viewing becomes difficult.
Clean Airlock vacuum seals.	Monthly	Piston will not fully rise into Airlock.
Clean Specimen-Mount assembly.	Every 3 months	XY stage does not move freely.
Clean Cold-Cathode gauge tube.	As required	Erratic reading or no reading.
Clean Shutter.	Every 3 months	Sputtered material falling onto specimen.
Dry clean the PIGs.	As required	Gun shorted.
Wet clean the PIGs.	Once a year	Excessive sputtered material.
MDP maintenance.	Once a year	Required servicing.
Diaphragm Pump maintenance.	Every 4000 hr	Backing pressure above 12 Torr.
Argon leak detection	As required	Excessive argon usage.
Clean Work Chamber.	Once a year	Excessive flaking of sputtered material.
Replace stage motor.	As required	Stage piston does not turn.
Replace stage encoder.	As required	Angle position does not register.
Replace sample mount.	As required	Excessive scratches or bent pins.
Replace bellows assembly.	As required	Chamber vents when stage is lowered.
Set Specimen height.	As required	Specimen height incorrect.
Replace gas manifold.	As required	Ar or vacuum leak or valve malfunction.

Caution: Do not use acetone as a cleaning agent. It will cause irreparable damage to instrument parts.

5.1. Cleaning the Viewing Port

The Viewing Port should be cleaned on a weekly basis with regular use.

NOTE: This operation can be performed without requiring the PIPS II to be shut down and vented.

1. **Raise the stage and vent the Airlock chamber (Milling Screen).**
2. **Lift off the Viewing Port capsule.**
3. **Check the capsule O-rings. Clean them. If necessary, replace.**

4. **Clean the window.** Use a nonabrasive cleaner or a 2-4 µm diamond polishing compound. Replace the window if deposits are too difficult to remove.

5. **Replace the window into the capsule O-rings.**

6. **Replace the Viewing Port capsule.**

7. **Evacuate Airlock chamber.** Slide down the slider on the Stage panel, while pushing down on the window to properly seat it.

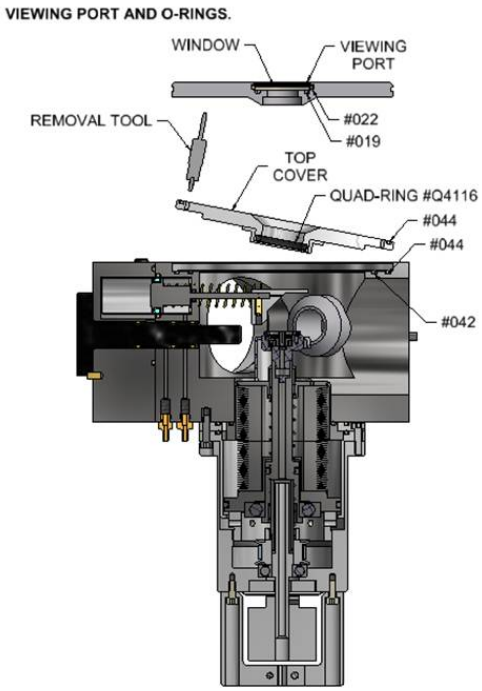

Figure 5-1 Viewing port and o-rings.

5.2. Cleaning the Airlock Vacuum Seals

The Airlock vacuum seals should be cleaned on a monthly basis with regular use.

NOTE: This procedure is necessary when the piston cannot be completely raised to its upper position due to buildup of sputtered material on the Airlock O-ring.

1. **Raise the Stage and vent the airlock chamber.** This allows the airlock cover to be removed after system power is off.

2. **Shut down the power to the PIPS II.** Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve.

3. **Lift off the Viewing Port.** Press the Airlock piston down into the Work Chamber if it hasn't already lowered itself.

4. **Remove top cover plate.** Using the pin end of the Specimen Mount Removal Tool, insert the pin into one of the holes in the top cover plate, push gently and tilt the plate up and out for removal.

5. **Remove the large O-ring from the top cover.**

6. **Remove the smaller O-ring from its groove in the cover.** Use a wooden toothpick or o-ring removal tool to remove the O-ring. Never use a metal tool to remove an O-ring.

7. **Clean the underside of the plate and the O-ring grooves with a grease solvent.**

8. **Clean the O-rings on the chassis with a lint-free cloth and replace.** It is usually not necessary to lubricate these o-rings, however, if they do not seal properly after cleaning they may be lubricated with Krytox GPL-206 vacuum grease (supplied with the system).

9. **Clean the Airlock quad-ring with a grease solvent and lubricate with Krytox GPL-206 vacuum grease.**

10. **Replace the Top Cover plate, the Viewing Port, and close the Vent valve.**

11. **Consider cleaning the guns, if they have not been cleaned lately.** Venting the system can cause particles to flake off the inner walls of the anode cup and create a gun short.

12. **Turn on the power.** Pump down to keep the system free of moisture and minimize oxidation of sputtered materials around the guns. The guns will need to be purged before use.

5.3. Cleaning the Specimen-mount Assembly

Milling the bottom surface of specimens will sputter material directly onto the window shield of the specimen-mount assembly, and onto the XY stage assembly. This deposit will reduce the light intensity transmitted by the Transmission Illuminator and may become a problem particularly when using the Autoterminator. In addition, sputtered material can interfere with the motion of the XY stage. For this reason, the specimen-mount assembly can be removed to provide unobstructed access for cleaning purposes. The XY stage may not need to be cleaned as often as the window shield.

It is good practice to clean the Airlock quad-ring any time the system is vented.

Caution: Do not mill samples without the specimen mount window in place. Sputtered material would then be deposited directly onto the rotate shaft, which cannot be cleaned.

1. **Raise the stage into the Airlock chamber and vent Airlock.**

2. **Shut down the power to the PIPS II.** Wait at least 10 min to allow the MDP to come to a complete stop.

3. **Remove the Viewing Port and the specimen post, if any.**

4. **Remove Top Cover plate.** Using the pin end of the Specimen Mount Removal Tool, insert the pin into one of the holes in the top cover plate, push gently and tilt the plate up and out for removal.

5. **Remove the specimen mount.** Rotate the specimen mount counter-clockwise until it is free from the rotate shaft.

NOTE: If the specimen mount only rotates but does not unscrew, remove the cover from the PIPS II cabinet and manually restrain the motor belt at the bottom of the Whisperlok to prevent it from rotating while the specimen mount is being unscrewed.

6. **Remove the specimen mount window.** The specimen mount window may fall out of the specimen mount assembly when it is removed from the rotate shaft. If not, push it out of the specimen mount assembly with a tooth pick.

7. **Clean top and bottom surfaces of the specimen mount window.** The top window surface may be cleaned with a small amount of 2-4 µm diamond paste on a cotton swab. The under side of the window should only require cleaning with a tissue.

8. If necessary, clean the XY stage parts. This should be performed if the stage parts are covered with excessive sputtered material or if the stage does not move freely.

1. **Remove the screw from the top of the specimen mount assembly and lift off the clamp plate.**

2. **Carefully remove the XY stage parts using tweezers.** Remember how the parts were assembled.

3. **Clean the parts with a lint-free cloth.** A solvent may be used on the metal parts if necessary. Gear teeth may be cleaned with a toothpick if necessary.

4. **Carefully re-assemble the XY stage assembly.** Be careful not to bend the pins.

5. **Replace the clamp plate and screw**.

9. Insert the specimen mount window back into the specimen mount assembly. A small amount of Krytox vacuum grease may be applied to the outside of the window shield so that it will stay in place during installation.

10. Screw the specimen-mount assembly back into the rotate shaft. If necessary, hold the motor and belt at the bottom of the Whisperlok to prevent the specimen mount from rotating while it is being screwed back in (CW rotation).

11. Replace the Top Cover plate, View Port, and turn on the power to the PIPS II. Note that the guns will need to be purged before use.

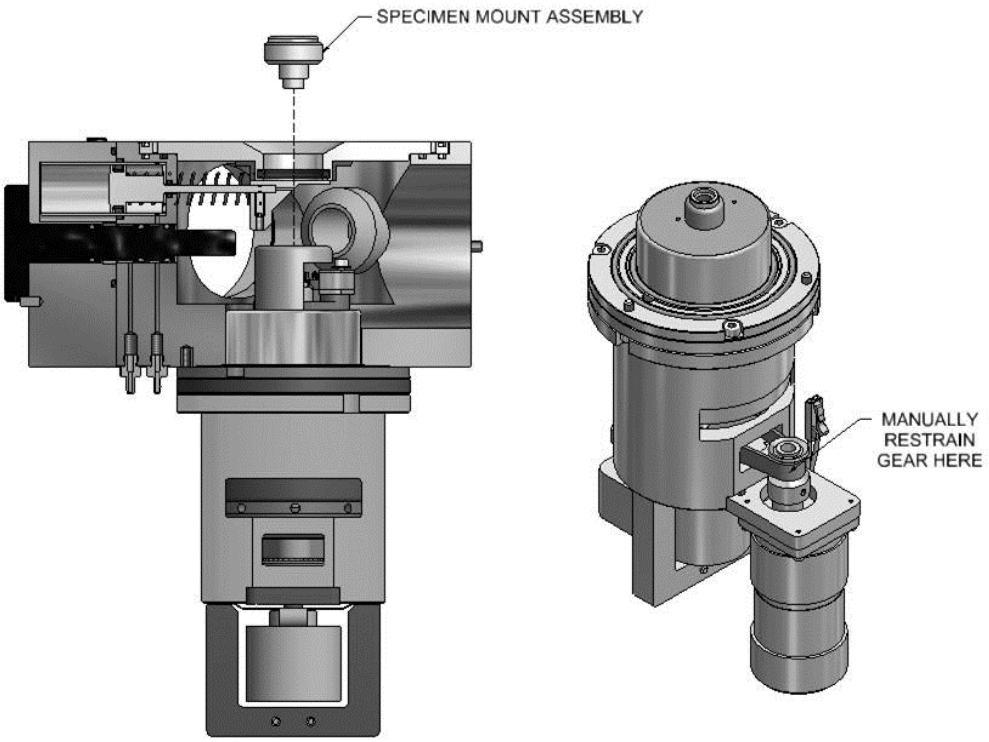

Figure 5-2 Specimen mount removal.

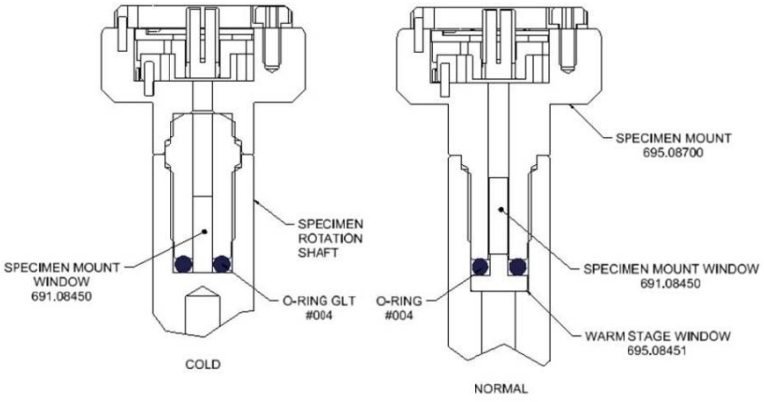

Figure 5-3 Specimen-mount and window assembly.

Caution: Cold Stage version: do not remove the o-ring from the specimen rotate shaft unless absolutely necessary. The material below this o-ring scratches very easily. It is best to remove the o-ring using tweezers, and to not touch the rotate shaft in the process.

5.4. Cleaning the Cold-cathode Gauge Tube

Caution: The cold cathode gauge contains a very powerful permanent magnet. Pacemaker wearers should not clean this gauge.

Contamination of the measuring chamber within the tube will affect the pressure reading and generally produce an indication that the pressure is poor. If contamination becomes severe, instability may occur resulting in shorts that may cause pressure bursts. If this occurs, the gauge tube must be dismantled and cleaned.

Tools required: Hex wrenches (1.5 mm & 3.0 mm), open-end wrench (7.0 mm), and locking-ring or snap-ring pliers.

5.4.1. *To Disassemble the Gauge Tube*

1. **Shut down the power to the PIPS II.** Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve. Unplug the power cable from the back of the system.

2. **Unplug the connector from the gauge tube.** Unscrew the retaining screw at the center of the connector.

3. **Remove the gauge tube. Pull it straight out from the Manifold.**

4. **Remove the electronic module.** Use the 1.5 mm hex wrench to loosen the set screw on the side of the module and slide it from the gauge tube.

5. **Remove the retainer.** Use the 3.0 mm hex wrench to remove the two socket-head screws at the back of the tube and remove the retainer.

6. **Carefully remove the anode, support ring, and Viton O-ring.** These parts can be individually cleaned or replaced if necessary. Use compressed air to blow out loose particles from within the gauge tube. If the inside of the gauge tube must be cleaned with an abrasive, continue with Steps 7 and 8.

7. **Separate the anode assembly from the magnet.** Use the 7.0 mm wrench to remove the hex-head screw from the magnet and slide off the anode assembly from the magnet.

8. Remove the locking ring and the pole insert from the front of the measuring chamber of the anode assembly.

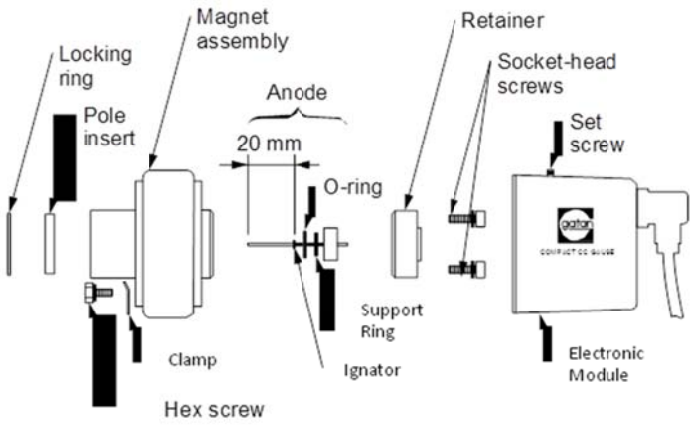

Figure 5-4 Cold-cathode gauge tube.

5.4.2. *To Clean Gauge Tube Parts*

1. Clean the inside of the tube and the front pole insert. Use a "Scotccbrite" pad or polishing cloth (500 grain).

2. Rinse both parts with methanol. Dry with compressed air or nitrogen gas.

3. Carefully clean the anode and ignitor with a polishing cloth.

The ignitor can be moved on the anode by sliding it up or down. The ignitor is fragile and can easily be damaged, use extreme care when moving or cleaning it. Do not bend the anode pin or damage the ceramic part since it forms the vacuum seal.

5.4.3. *To Reassemble the Gauge Tube*

1. Position the ignitor 20 mm from the end of the anode pin.

2. Insert the O-ring and support ring into the tube. The sealing surface, O-ring, and ceramic part must be clean.

3. Carefully insert the anode and ignitor into the tube.

4. Replace the retainer and tighten the screws uniformly until the stop position is reached.

5. Slide the pole insert into the front of the tube and mount the snap ring against the pole insert.

NOTE: Visually check that the anode pin is centered within the hole of the pole insert.

6. Mount the magnet onto the anode assembly. Lock it with the hex-head screw and clamp.

7. Carefully push on the electronics module until it stops.

8. Position the connector rotated 180º from the magnet retaining screw. Secure the module snugly in place with the socket-set screw.

Caution: Do not tighten down hard on the set screw.

9. Replace the gauge tube into the manifold. Locate the magnet retaining screw into the notch on the manifold.

10. Plug the connector into the gauge tube. Secure the retaining screw.

11. Close the Vent valve, restart the PIPS II, and pump down the system.

Caution: Do not allow the PIPS II to run for more than 1 hr with the cold-cathode gauge at pressures above 10^{-3} Torr since a glow discharge will occur in the tube causing it to become contaminated.

5.5. Cleaning the Shutter

The pneumatically operated Shutter is designed to operate for an extended period of time with only a minimal amount of maintenance. The Shutter reduces buildup of sputtered material on the viewing window and instead accumulates material on its underside.

Over a period of time, the accumulated material may crack, peel, and flake off onto the specimen. Venting to atmosphere also may cause the sputtered material to lose adhesion and to peel and flake. For these reasons, the underside of the Shutter should be examined and cleaned periodically, every 3 months or so with regular use.

1. Raise the stage and vent the Airlock chamber.

2. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve.

3. **Lift off the Viewing Port.**

4. **Press the Airlock piston down into the Work Chamber.**

5. **Remove the Top Cover plate with the Removal tool.**

6. **Pull out the Shutter Guide.** Grasp the Shutter Guide at the front of the chamber and pull it straight out.

7. **Rotate the Shutter manually 90°-180° to view the underside.** It may help to pull the shutter slightly forward into the chamber to allow it to rotate easily.

8. **Use a tissue saturated with freon or methanol and wipe off the underside.** If the shutter is relatively clean, it may only require manual wiping.

9. **Remove the Shutter for more thorough cleaning.** If a more thorough cleaning is required, the Shutter must be removed by unscrewing the M2 × 6 mm retaining screw.

10. **Clean the Shutter.** Sputter deposits on the Shutter should be removed with an abrasive cleaner after which the Shutter should be cleaned with alcohol and thoroughly dried before replacing in the chamber.

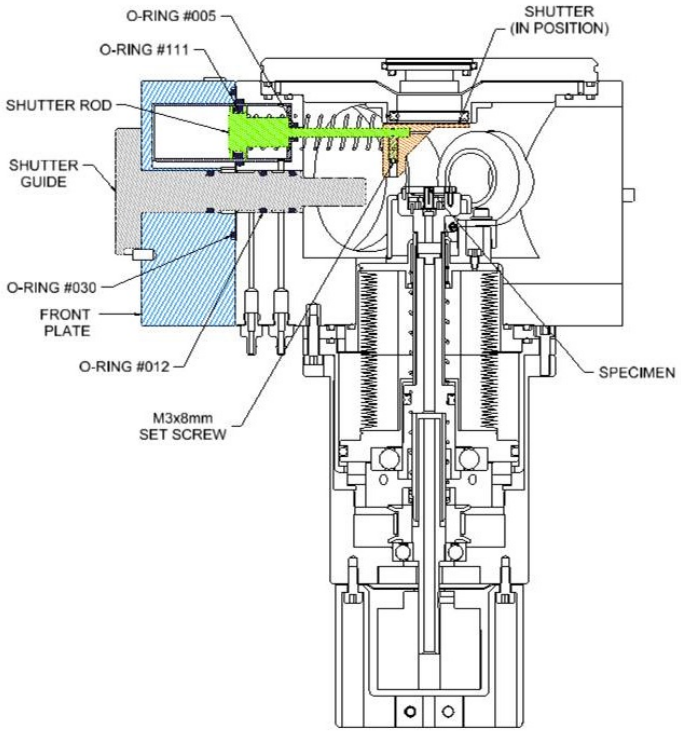

Figure 5-5 Cleaning the shutter.

Caution: The Shutter will not operate if the blade or shaft is bent by improper handling during cleaning.

5.6. Care of Penning Ion Guns

Caution: The Penning guns contain very powerful permanent magnets. Pacemaker wearers should not clean these guns.

Good care and maintenance of the PIGs are absolutely essential to obtaining good specimen thinning. There are two ways to clean the guns: dry method and wet method.

The gun maintenance screen can be used to help determine if a gun is shorted. If the discharge current in microamps is approximately equal to the discharge voltage in volts, and the accelerating current is unusually low; then the gun is likely shorted. For example, when the beam voltage is 6 kV, the discharge voltage is approximately 890 V. If the discharge current is approximately 890 uA and the accelerating current is significantly lower than normal for 6 kV beam voltage, then the gun is likely shorted. Note that these same conditions apply during beam modulation when the guns are between milling sectors, because the guns are shorted in the HVPS. Similarly, if the accelerating voltage read is significantly lower than the accelerating voltage set, then the gun is also likely shorted.

Caution: any time the work chamber is vented, the guns must be purged for 4-5 hours before milling samples (overnight is better). This ensures that the gas flow settings are correct and that the beams will be focused properly.

5.6.1. *Dry Cleaning the Penning Ion Guns*

The dry method of cleaning involves wiping the parts with a clean dry tissue, then using dry nitrogen or clean compressed air to remove any dust, lint, or metallic whiskers that are the primary cause of shorts in the guns. This method is preferred because the cleaning time and the actual time the gun parts are out of the vacuum is reduced to a minimum. Additionally, since no solvents are used, the required argon purging time for the guns after start-up is greatly reduced.

To Remove the Gun:

1. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Unplug the power cable from the back of the system.

Then vent the Work Chamber by opening the Vent valve. There is no need to unplug the HV cables nor remove any of the side covers from the PIPS II.

2. Remove the gun knob from the gun housing. Rotate the gun knob to the 10° Top position. Use the 3.0mm hex wrench to remove the two screws from the gun knob and pull the knob from the gun housing.

3. Withdraw the ion source from the gun housing. Use the 3.0 mm hex wrench to remove the single screw from the molded connector assembly. Slowly pull on the toggle stick to withdraw the ion source from the gun housing.

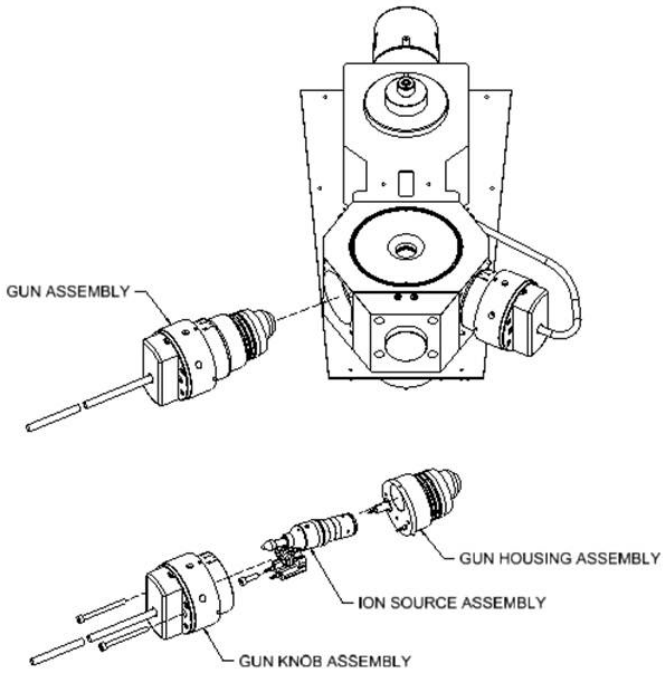

Figure 5-6. Removal and disassembly of ion guns.

NOTE: From this point, use nylon gloves to handle all parts. Special attention must be paid to the cleanliness of all the parts, especially the magnet

assembly. The disassembly and subsequent assembly should be done with the aid of a x10 stereo microscope.

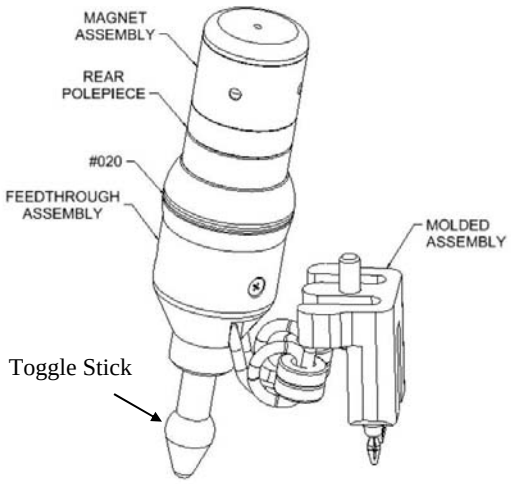

Figure 5-7 Ion source and magnet assembly.

To Disassemble the Gun:

1. Lift the magnet assembly off the rear polepiece.

Hold the ion source with one hand and grasp the magnet assembly with the other hand. Lift the magnet assembly off the rear polepiece by tilting it to one side.

NOTE: The rear polepiece can be cleaned directly on the HV connector (without disassembly) by dusting it off using dry nitrogen or clean compressed air. If any particles remain, use a tissue to remove them and dust again with compressed air.

2. Remove the anode cup assembly from the magnet.

Lightly tap the assembly on its edge until enough of the anode protrudes to be pulled out of the magnet.

3. Remove the anode cup insulator with the eraser end of a pencil.

4. Separate magnet from the front polepiece.

Holding the magnet in one hand, place the eraser end of a pencil into the magnet and push against the front polepiece to separate it from the magnet. Warning: The magnet is extremely powerful and requires careful handling to prevent it from attracting metallic whiskers and from being attracted to any other magnetic material that may shatter it.

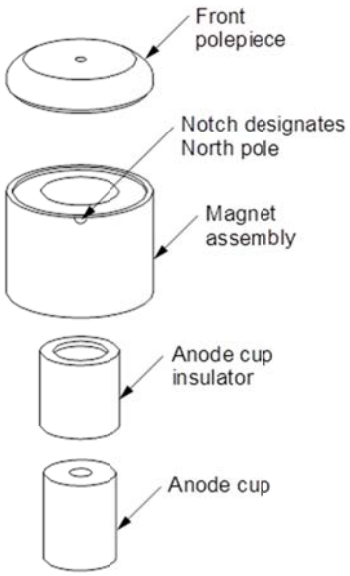

Figure 5-8 Removal of anode assembly and anode cup insulator.

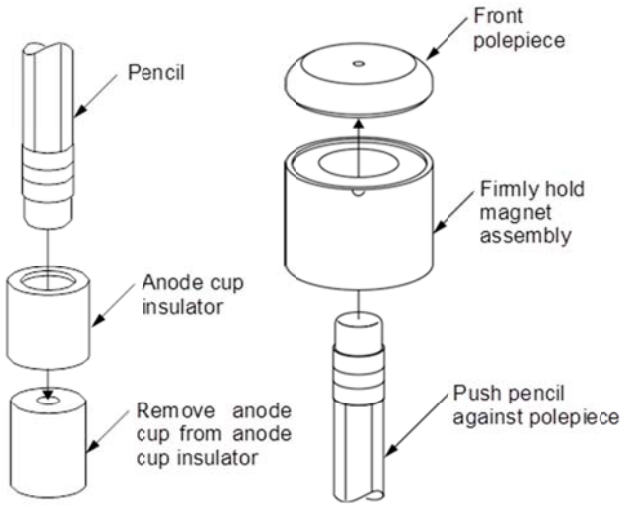

Figure 5-9 Removing anode cup assembly/front pole piece.

To Inspect and Clean the Gun:

1. Carefully examine the inside face of the front polepiece and the top of the anode cup.

Look for black or burnt spots that would indicate a short. Burn marks on the front polepiece may easily be removed using 600-grit emery paper. Depending upon the severity, burn marks on the anode cup insulator may also be removed with 600-grit paper. However, if burn marks are deep, replace the anode cup insulator.

2. Clean the anode cup.

Clean the anode cup by wiping with a clean dry tissue and dusting it with dry nitrogen or clean compressed air. Clean all the loose sputtered material on the inside surface and the face of the cup using an abrasive pad such as Scotchbrite. Wipe clean with a dry tissue and dust with dry nitrogen or compressed air.

3. Clean any particles or whiskers off of the magnet assembly. Use a lint-free cloth and clean compressed air. If there are stubborn particles that are difficult to remove, touch the surface with scotch tape to remove the particles.

4. Remove the O-ring from the ion source, if necessary.

Squeeze and push up from both sides with thumb and index finger to remove the O-ring from the ion source. Clean, apply vacuum grease (Krytox), and replace. Apply vacuum grease to the curved Rulon bearing that captures the o-ring. If this surface and the o-ring are not lubricated, then the beam alignment will not move freely. Also apply vacuum grease to the curved surface of the toggle stick, which mates with the drive screws in the gun knob. This helps the toggle stick slide freely against the drive screws.

5. Dust inside the gun housing and the inside face of the front polepiece.

Be sure the curved face on the inside of the gun housing that meets the o-ring is clean. This face can be lubricated if desired.

To Reassemble the Gun:

1. Insert the anode cup into the anode cup insulator (sliding fit).

2. Insert the anode assembly into the magnet assembly (loose sliding fit).

3. Carefully place the magnet assembly against the edge of the rear polepiece.

Slowly lower the magnet assembly in place until the rear polepiece is within the magnet shield. The parts will be concentric to one another.

4. Slip the ion source into the gun housing.

Pay particular attention that the O-ring is not damaged in the process. Carefully align the white reference dot at the back of the gun to the mating groove machined into the outside diameter of the gun housing.

5. Insert the screw into the molded connector assembly and tighten.

This assembly should be aligned relatively square with the chamber. Guide the knob over the toggle stick until it is firmly in place and screw in the two retaining screws.

Repeat this procedure on the second gun if necessary.

5.6.2. *Wet Cleaning the Penning Ion Guns*

As stated earlier, the dry method of cleaning the guns is preferred. However, once the guns have been used extensively, a more thorough cleaning is required. The wet method of cleaning involves the use of solvents such as freon or methanol with an abrasive material. A Scotchbrite pad or 600-grit emery paper can be used to remove all sputtered material. Once complete and assembled, the time required to pump down the chamber and to argon purge the guns is significantly longer when compared to the dry method of cleaning. If it is necessary to use this method, then this is a good time to also clean the Shutter and the inside of the Work Chamber to reduce overall down time.

To Remove the Gun

1. Shut down the power to the PIPS II.

Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve. Since the HV cables are not unplugged, there is no need to remove any of the side covers from the PIPS II. Unplug the power cable from the back of the system.

2. Remove the gun knob from the gun housing.

Rotate the gun knob to the 10° Top position. Use the 3.0mm hex wrench to remove the two screws from the gun knob and pull the knob from the gun housing.

3. Lift off the Viewing Port and the top cover plate from the chamber.

Place one hand at the back of the gun and with the other hand push on the gun housing from inside the chamber. Remove the gun.

To Disassemble the Gun

1. Withdraw the ion source from the gun housing.

Use the 3.0mm hex wrench to remove the single screw from the molded connector assembly then slowly pull on the toggle stick to withdraw the ion source from the gun housing.

2. Lift the magnet assembly off the rear polepiece.

Hold the ion source with one hand and grasp the magnet assembly with the other hand. Lift the magnet assembly off the rear polepiece by tilting it to one side.

NOTE: The rear polepiece can be cleaned directly on the HV connector by dusting it off with dry nitrogen or clean compressed air. If any particles remain, use a tissue to remove them and dust again with the compressed air. Special attention must be paid to the cleanliness of all the parts, especially the magnet assembly.

3. Remove the anode cup assembly from the magnet.

Lightly tap the assembly on its edge until enough of the anode cup protrudes to be pulled out of the magnet.

4. Remove the anode cup insulator with the eraser end of a pencil.

5. Separate magnet from the front polepiece.

Holding the magnet in one hand, place the eraser end of a pencil into the magnet and push against the front polepiece to separate it from the magnet.

Warning: The magnet is extremely powerful and requires careful handling to prevent it from attracting metallic whiskers and from being attracted to any other magnetic material that may shatter it.

To Inspect and Clean the Gun

1. With a low-power microscope, carefully examine the inside face of the front polepiece and the top of the anode cup.

Look for black or burnt spots that would indicate a short. Burn marks on the front polepiece may easily be removed using 600-grit emery paper. Depending upon the severity, burn marks on the anode cup insulator may also be removed with 600-grit paper. However, if burn marks are deep, replace the anode cup insulator.

NOTE: From this point, use nylon gloves to handle all clean parts. Special attention must be paid to the cleanliness of all the parts, especially the magnet assembly. The subsequent assembly should be done with the aid of a ×10 stereo microscope.

2. Clean the anode cup using freon or methanol.

Clean all the sputtered material on the inside surface of the cup using a Scotchbrite pad.

3. Wipe off the magnet and the rear polepiece with freon or methanol.

4. Clean and lubricate the O-ring and the bearing surface on the outside of the ion-source assembly.

Dust parts off using clean compressed air or nitrogen gas to remove any dust, lint or metallic whiskers that are the primary cause of shorting in the gun.

Squeeze and push up from both sides with thumb and index finger to remove the O-ring from the ion source. Clean, apply vacuum grease (Krytox), and replace. Apply vacuum grease to the curved Rulon bearing that captures the o-ring. If this surface and the o-ring are not lubricated, then the beam alignment will not move freely. Also apply vacuum grease to the curved surface of the toggle stick, which mates with the drive screws in the gun knob. This helps the toggle stick slide freely against the drive screws.

5. Clean the gun housing O-rings.

Remove the two O-rings from the gun housing and thoroughly clean all surfaces with freon or methanol, including the O-ring grooves.

Replace O-rings, if necessary.

Dry off all surfaces and parts with compressed air or freon gas.

To Reassemble the Gun

1. Insert the anode cup into the anode cup insulator.

2. Insert the anode assembly into the magnet.

Be sure the top of the anode is at the north face of the magnet.

3. Replace the front pole piece.

4. Carefully place the magnet assembly against the edge of the rear polepiece.

Slowly lower the magnet assembly in place until the rear polepiece is within the magnet shield. Properly assembled, the parts will be perfectly concentric to one another.

5. Place a light film of vacuum grease around the inside surface (first one centimeter) of the port for the ion source.

6. Slip the ion source into the gun housing.

Pay particular attention that the O-ring is not damaged in the process.

7. Align ion source in the housing.

Align ion source so that the moulded assembly is aligned to the screw hole in the housing.

8. Insert the screw into the molded connector assembly and tighten.

This assembly should be aligned squarely with the Chamber.

9. Test the gun before inserting into the Work Chamber.

Use an ohmmeter and test the gun to ensure that a short does not exist across the HV contacts. A direct short usually indicates the gun was not assembled properly. A higher resistance short up to 2MΩ indicates the presence of small conductive whiskers within the gun. If this is the case, the cleaning steps described above should be repeated.

10. Place a light film of vacuum grease around the Work Chamber gun port.

11. Insert the complete assembly into the Work Chamber.

Align the reference mark on the diameter of the gun housing to the mating mark on the Chamber.

12. Replace the gun knob in the gun housing.

Guide the gun knob over the toggle stick until it is firmly in place and screw in the two retaining screws.

Repeat this procedure on the second gun.

5.6.3. *Servicing the Focus Electrode*

The focus electrode should not normally require maintenance. Sputtered material may build up on the electrode or insulator and need to be removed.

1. Shut down the power to the PIPS II.

Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve.

2. Remove the gun knob from the gun housing.

Rotate the gun knob to the 10° Top position. Use the 3.0mm hex wrench to remove the two screws from the gun knob and pull the knob from the gun housing.

3. Remove the gun housing from the work chamber.

4. Remove the upper front housing electrode from the gun housing.

Rotate the electrode counter clock-wise using a spanner wrench.

5. Clean the ground electrode if necessary.

6. Remove the focus electrode insulator from the housing.

Gently pry the insulator from the housing. Be careful not to damage the flex cable or the gasket material.

7. Clean the focus electrode.

Clean the focus electrode cup by wiping with a clean dry tissue and dusting it with dry nitrogen or clean compressed air. Clean all the loose sputtered material on the inside and outside faces of the cup using an abrasive pad such as Scotchbrite. Wipe clean with a dry tissue and dust with dry nitrogen or compressed air.

8. Reassemble the gun housing.

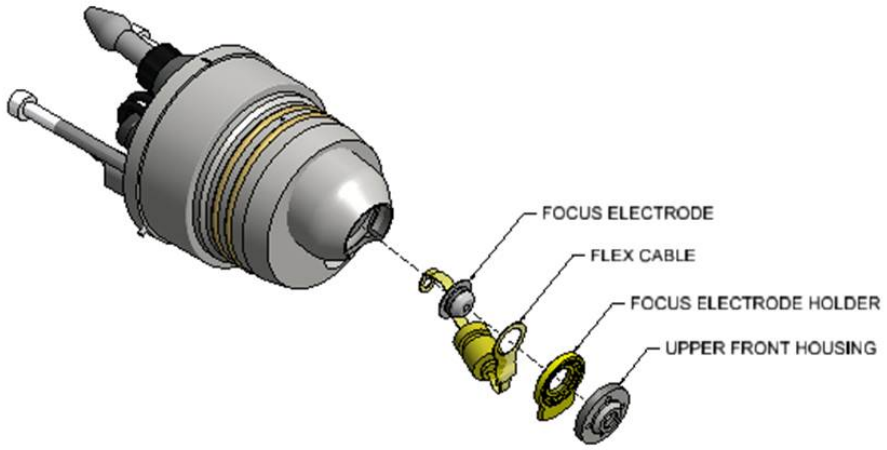

Figure 5-10 Focus electrode assembly.

5.7. Removing the Cover

To remove the cover:

1. Shut down the power to the PIPS II.

Unplug the power cable from the power entry module.

2. Remove the 4 M3 screws from the outside edges of the rear panel.

3. Pull the cover forward until the latches release from the frame.

4. Bend the sides of the cover slightly outward so that the cover may be completely removed.

Be careful not to bend the cover too far.

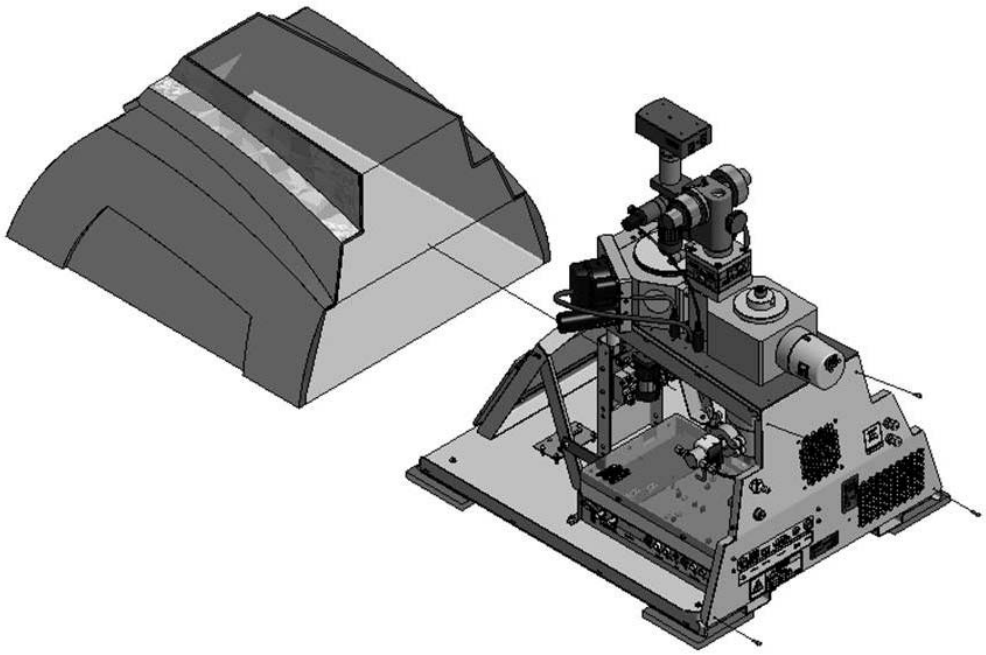

Figure 5-11 Cover removal.

5.8. Replacing the MDP Oil Cartridge

The MDP requires the oil cartridge to be changed at least once a year. However, if a high- pitched squeal begins, the oil cartridge should be changed immediately.

The oil cartridge consists of a stack of felt discs saturated with oil and is replaced as a unit. Changing the cartridge requires the Chamber to be vented to atmosphere and the MDP to be completely removed from the PIPS II. This provides an opportunity to service other parts of the vacuum system.

NOTE: The oil in the MDP is for lubrication of the bearings only, and does not come in contact with the vacuum chamber hence eliminating any concern for hydrocarbon contamination.

5.8.1. *To Remove the MDP*

1. **Shut down the power to the PIPS II.**

   Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve. Unplug the power cable from the power entry module.

2. **Remove the control cable from the MDP.**

   Loosen the 2 screws in the D-sub connector and unplug the cable.

3. **Remove the Vac-valve assembly.**

   Loosen and remove the compression fitting on the back of the Vac-valve assembly, this will separate the Vac-valve assembly from the tubing that connect the DP. Loosen and remove the KF clamp and centering ring from the exhaust port in order to remove the Vac-valve assembly. Carefully place the Vac-valve assembly on the electronics enclosure, being careful not to kink the nylon tubing.

4. **Loosen the 4 MDP mounting screws on the support plate.**

   These screws retain the flange clamps used to lock the MDP to the manifold. Remove two of the screws and clamps completely; then support the MDP from the underside with one hand while removing the other two screws and clamps.

5. **Lower the MDP and remove it from inside the cabinet.**

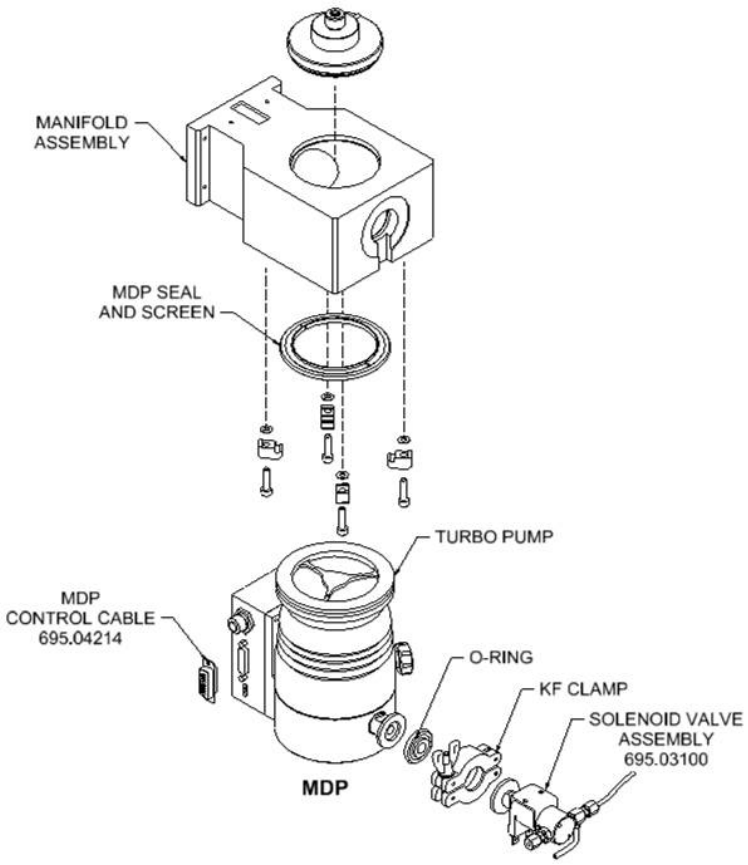

Figure 5-12 Molecular drag pump removal.

The oil cartridge can be replaced upon removal of the MDP from the cabinet.

5.8.2. *To Replace the Oil Cartridge*

Follow the directions in the Pfeiffer HiPace 80 TurboDrag Pump Operating Instructions manual (shipped with PIPS II).

After the MDP is reconnected to the PIPS, and the system is powered on, the guns must be purged for 4-5 hours before milling samples.

5.9. Diaphragm Pump Maintenance

Both diaphragms should be replaced after 4000 h of use. If either of them fails after 2000 h, replace both of them.

5.9.1. *To Disconnect the Diaphragm Pump*

1. **Shut down the power to the PIPS II.**

Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve.

2. Unplug the power cord from the power entry module (rear panel).

3. Unplug the two electrical connectors on the DP.

4. Disconnect the vacuum hose running from the pump to the back of the PIPS cabinet.

Press the latch of the quick disconnect fitting and remove the hose.

5.9.2. *To Replace Diaphragm*

Follow the directions in the Pfeiffer MVP 020-3 Diaphragm Pump Operating Instructions manual (shipped with PIPS).

After the DP is reconnected to the PIPS, and the system is powered on, the guns must be purged for 4-5 hours before milling samples.

5.10. Cleaning the Work Chamber

Clean the Work Chamber when you have vented the system for other maintenance to reduce overall down time.

5.10.1. *To Vent and Clean the Work Chamber*

1. Raise the stage and vent the Airlock.

2. Shut down the power to the PIPS II.

Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the Work Chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. Lift off the Viewing Port and the Top Cover plate from the Chamber.

4. Clean the Chamber. There is no need to polish the chamber. Just remove flakes of sputtered materials with a simple vacuuming and/or wiping with a Kimwipe. Methanol can be used but it will increase pump down time.

5. Replace the top cover plate and the Viewing Port.

6. Power up the PIPS II.

5.11. Cleaning the Shutter Piston

The shutter piston is controlled by two valves, SI and SO. The SI valve is on and the SO valve is off when the shutter is closed (covering the window). The SI valve is off and the SO valve is on when the shutter is open. Sputtered material can build up on the shutter piston shaft, and need to be cleaned off. If the speed of the shutter decreases significantly over time, or if the shutter no longer moves as far, it may need to be cleaned and inspected.

1. **Press the Airlock piston down into the Work Chamber.**

2. **Remove the Top Cover plate with the Removal tool.**

3. **Pull out the Shutter Guide.** Grasp the Shutter Guide at the front of the chamber and pull it straight out.

4. **Rotate the Shutter manually 90°-180° to view the underside.** It may help to pull the shutter slightly forward into the chamber to allow it to rotate easily.

5. **Remove the Shutter.** Loosen the M2 × 6 mm retaining screw. Remove the shutter from the shutter piston rod.

6. **Remove the front plate.** Loosen the 4 screws in the front plate. Pull the front plate straight forward. The shutter piston will be pushed out by the spring.

7. **Clean the shutter piston.** Remove any sputtered material from the shutter piston. Inspect the o-rings, replace if they are damaged. Clean and lubricate the o-rings with Krytox GPL-206. Clean and lubricate the inside of the cylinder where the o-ring contacts.

8. **Reassemble all parts.**

9. **Power up the PIPS II.**

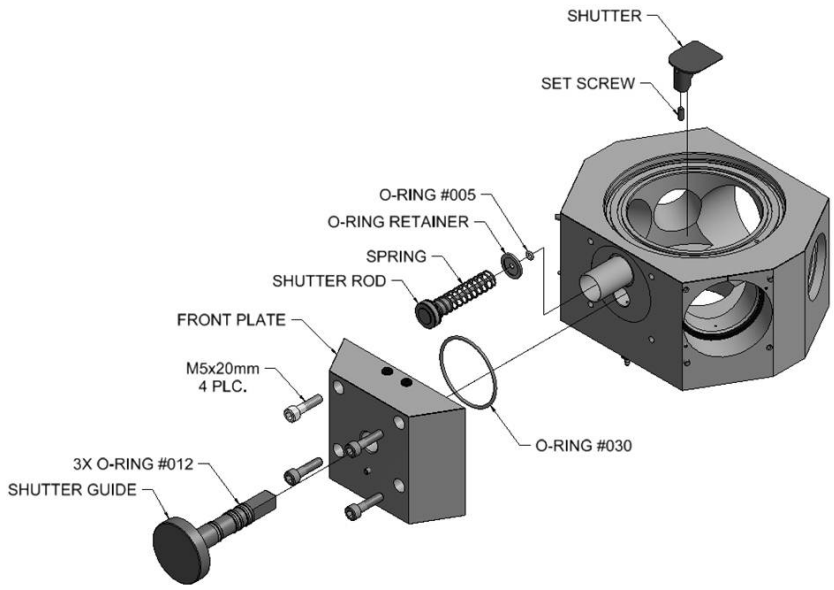

Figure 5-13 Shutter servicing.

5.12. Motor Drive Replacement

The specimen motor drive is located under the specimen chamber. It does not need to be replaced unless it fails, which typically does not happen during the lifetime of the instrument.

It can usually be replaced without removing the stage assembly, but if this proves too difficult the Whisperlok assembly can be removed from the work chamber prior to motor replacement.

1. Shut down the power to the PIPS II. Unplug the power cable from the back of the system.

2. Remove the cover.

3. Unplug the motor from the motor cable.

4. Loosen the set screws that hold the timing pulley to the motor.

5. Remove the four M2 socket head screws that hold the motor to the bracket. The motor has specific characteristics for the PIPS II and should only be replaced with the same type.

6. Remove and replace the motor. Insert the motor drive shaft into the timing pulley as the motor is installed.

7. **Tighten the set screws to the timing pulley.**

8. **Plug the motor into the motor drive cable.**

9. **Install the cover.**

10. **Turn on power to the PIPS II.**

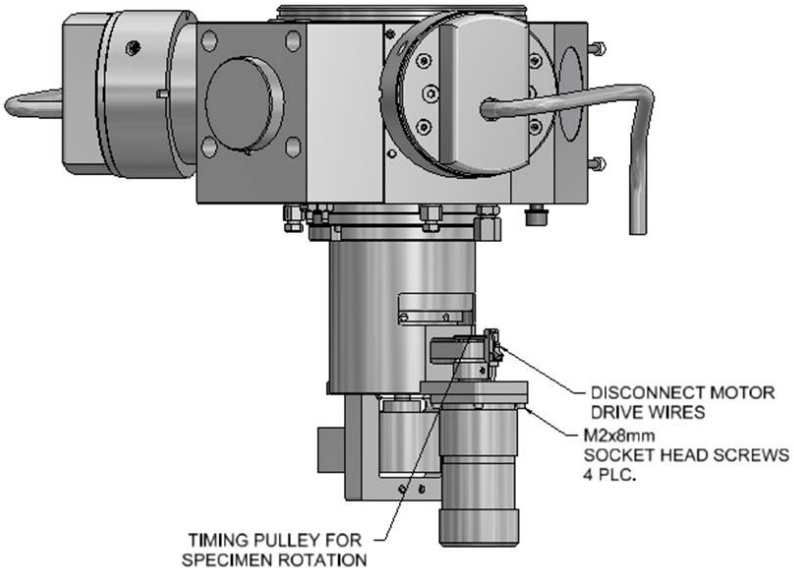

Figure 5-14 Motor Drive Removal.

5.13. Replacing the Stage Encoder

The stage encoder is located under the specimen chamber. It does not need to be replaced unless it fails, which typically does not happen during the lifetime of the instrument.

The stage encoder can be replaced without removing the stage assembly from the chamber, but it may be easier to remove the stage assembly first and then change the encoder on a bench.

1. **Remove the cover.**

2. **Shut down power to the PIPS II.** Unplug the power cable from the back of the system.

3. **Remove the cover.**

4. Mark the fiber cable with a piece of tape so you can reinstall it at the exact same place.

5. Loosen the 2 set screws that connect the encoder to the drive shaft.

6. Loosen the 2 screws on the fiber cable clamp.

Caution: The fiber cable must be installed at exactly the same position after removal. Because the stage is raised when the fiber cable is installed, it is possible to install it too deep within the piston. This would result in damage to the fiber cable and perhaps the piston.

Prior to removing the fiber cable, place a piece of electrical tape or similar on the cable just below the clamp. This will allow it to be re-installed at the same position. Gently pull the fiber cable downward and remove it.

7. Remove the 2 screws that secure the U-shaped clamp at the bottom of the stage assembly, and remove the clamp and the encoder.

8. Install the new encoder, securing it with the U-shaped clamp. Tighten the 2 set screws.

9. Re-install the fiber cable.

Be sure it is not inserted farther than previously, or it will interfere with stage lowering.

10. Re-install the cover. Turn the power on.

11. Calibrate the stage home position.

a) Lower the stage and set the home position. Alignment > Home.
b) Observe the actual rotational position of the stage. It will not be at the home position. Note how many degrees in rotation it is from the proper home position.
c) Adjust the home calibration setting to compensate. Maintenance > Calibration > Stage. The calibration setting is in 10ths of a degree, so that if the position is 10 degrees away from the proper position adjust the calibration setting by 100.
d) Repeat this process until the home position is correct.

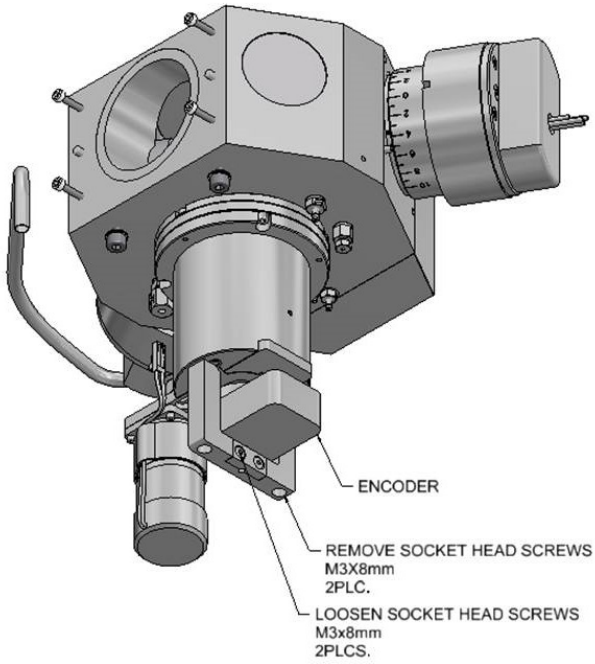

Figure 5-15 Replacing the encoder.

5.14. Replacing the Sample Mount

The Sample Mount is located under at the top of the rotate shaft. It does not need to be replaced unless it fails or is damaged, which typically does not happen during the lifetime of the instrument.

1. Raise the Stage and vent the airlock chamber. This allows the airlock cover to be removed after system power is off.

2. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. Lift off the Viewing Port. Press the Airlock piston down into the Work Chamber if it hasn't already lowered itself.

4. Remove top cover plate. Using the pin end of the Specimen Mount Removal Tool, insert the pin into one of the holes in the top cover plate, push gently and tilt the plate up and out for removal.

5. Remove the Specimen Mount. While holding the rotate shaft, rotate the Specimen Mount counter-clockwise until it is completely free of the rotate shaft.

NOTE: If the specimen mount only rotates but does not unscrew, remove the cover from the PIPS II cabinet and manually restrain the timing pulley at the bottom of the Whisperlok to prevent it from rotating while the specimen mount is being unscrewed.

6. Install the new Specimen mount. If necessary, install the XY stage parts.

7. Replace the Top Cover plate and Viewing Port.

8. Close the Vent valve and turn on the power.

9. Verify that the stage home position is correct. If the stage home position is not correct, calibrate it as described below.

a) Lower the stage and set the home position. Alignment > Home.
b) Observe the actual rotational position of the stage. It will not be at the home position. Note how many degrees in rotation it is from the proper home position.
c) Adjust the home calibration setting to compensate. Maintenance > Calibration > Stage. The calibration setting is in 10ths of a degree, so that if the position is 10 degrees away from the proper position adjust the calibration setting by 100.
d) Repeat this process until the home position is correct.

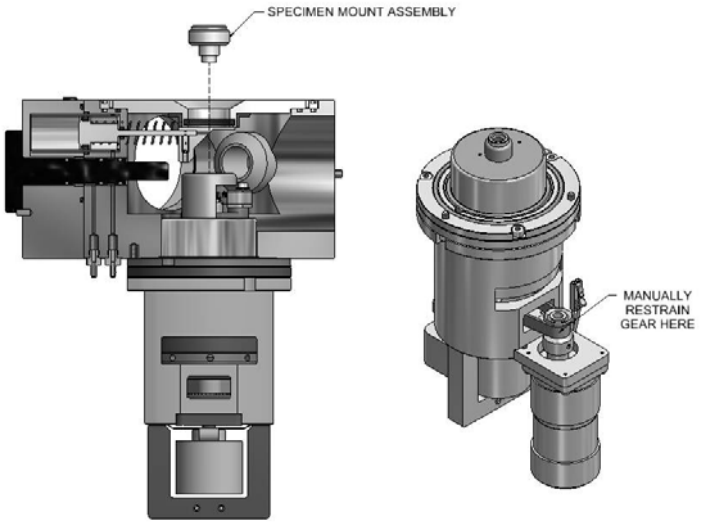

Figure 5-16 Sample mount removal.

5.15. Replacing the Bellows Assembly

The Bellows Assembly is part of the Whisperlok™ assembly. It does not need to be replaced unless it fails.

5.15.1. *To Remove the Whisperlok™ Assembly*

1. Raise the Stage and vent the airlock chamber. This allows the airlock cover to be removed after system power is off.

2. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. Remove the system cover.

4. Lift off the Viewing Port. Press the Airlock piston down into the Work Chamber if it hasn't already lowered itself.

5. Remove top cover plate. Using the pin end of the Specimen Mount Removal Tool, insert the pin into one of the holes in the top cover plate, push gently and tilt the plate up and out for removal.

6. Disconnect the cold stage. If the system has a cold stage, remove the two screws and disconnect the cold stage heater from the hinged conductor assembly.

Caution: The fiber cable must be installed at exactly the same position after removal. Because the stage is raised when the fiber cable is installed, it is possible to install it too deep within the piston. This would result in damage to the fiber cable and perhaps the piston.

7. Remove the fiber cable.

a) Mark the fiber cable with a piece of tape so you can reinstall it at the exact same place.
b) Loosen the 2 set screws that connect the encoder to the drive shaft.
c) Loosen the 2 screws on the fiber cable clamp.
d) Gently pull the fiber cable downward and remove it.

8. Remove the Whisperlok™ assembly.

a) Unplug the motor drive cable.
b) Remove the 3 M3 screws that secure the Whisperlok™ assembly to the work chamber.

c) Gently lower the Whisperlok^{TM} assembly and remove it, being careful not to disturb the vacuum and pneumatic tubing.

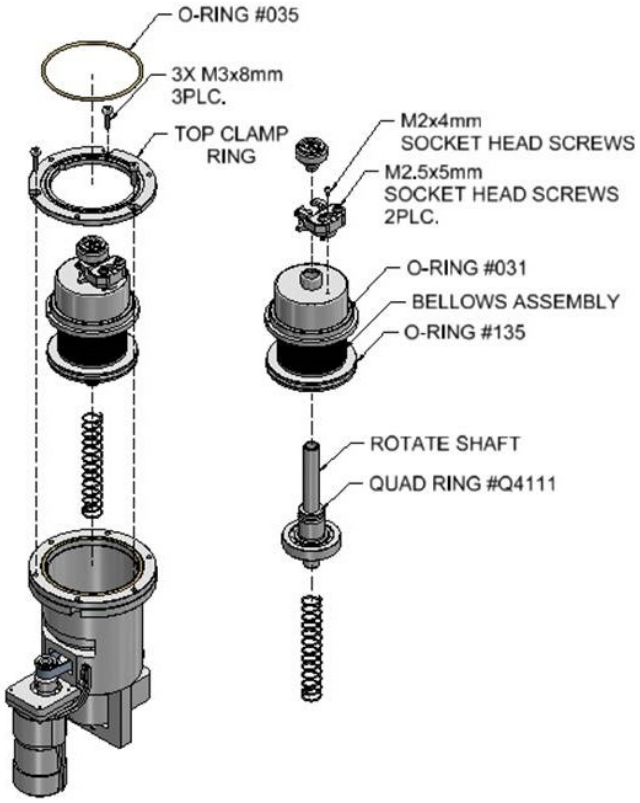

Figure 5-17 Whisperlok assembly.

5.15.2. *To Dis-assemble the Whisperlok^{TM} Assembly*

1. Remove the 3 screws in the top of the assembly.

2. Remove the top clamp ring.

3. Remove the bellows assembly from the cylinder body. Check the inside of the housing where the o-ring at the bottom of the bellows assembly slides up and down. If necessary clean and lubricate with Krytox GPL-206.

4. If the system has a cold stage, remove the brush arms by first removing the spring that clamps them to the hinged conductor assembly.

5. Remove the Specimen Mount. While holding the rotate shaft, rotate the Specimen Mount counter-clockwise until it is completely free of the rotate shaft.

6. Remove the window shield from the rotate shaft, if it did not remain with the specimen Mount.

7. Remove the 4 screws and the clamping plate at the bottom of the Whisperlok™ assembly.

8. Remove the rotate shaft from the bellows assembly. This can be accomplished by pulling on the large bearing at the bottom of the assembly, or by pushing slightly on the top of the rotate shaft.

9. Clean and lubricate the rotate shaft quad-seal. This seal should always be cleaned and lubricated when the Whisperlok is disassembled.

10. Remove the 2 o-rings from the bellows assembly.

5.15.3. *To Assemble the Whisperkok™ Assembly*

Assemble the Whisperlok™ assembly with the new bellows assembly.

1. Install the 2 o-rings on the new bellows assembly.

2. Clean and lubricate the o-ring at the bottom of the bellows assembly with Krytox GPL-206.

3. Clean and lubricate the quad-seal on the rotate shaft with Krytox GPL-206.

4. Clean the o-rings in the cylinder body and clamp ring.

5. Lubricate the inside of the bellows assembly where the quad-seal contacts with Krytox GPL-206.

6. Install the piston assembly into the bellows assembly.

7. Install the Specimen Mount onto the piston assembly.

8. If applicable, install the brush arms and the clamping spring.

9. Lubricate the inside of the cylinder body with Krytox GPL-206.

10. Install the bellows assembly into the cylinder body. The center notch in the outside top flange of the bellows assembly must be oriented toward the front of the system. The motor must be on the right hand side of the Whisperlok™ assembly when viewed from the front of the system. Compress the bellows fully.

11. Install the clamp ring. The 2 pins in the bottom of the clamp ring align to the 2 outside notches in the top flange of the bellows assembly. Install the 3 screws.

12. Install the Whisperlok™ assembly onto the work chamber. The motor assembly must be to the right when viewed from the front of the system.

13. Replace the Top Cover plate and Viewing Port.

14. Close the Vent valve and turn on the power.

15. Verify that the stage home position is correct. If the stage home position is not correct, calibrate it as described below.

a) Lower the stage and set the home position. Alignment > Home.
b) Observe the actual rotational position of the stage. It will not be at the home position. Note how many degrees in rotation it is from the proper home position.
c) Adjust the home calibration setting to compensate. Maintenance > Calibration > Stage. The calibration setting is in 10ths of a degree, so that if the position is 10 degrees away from the proper position adjust the calibration setting by 100.
d) Repeat this process until the home position is correct.

16. Purge the guns at least 4-5 hours before milling samples.

5.16. Cleaning the Rotate-Shaft Quad-seal

A quad-ring separates the chamber from atmosphere and allows for rotation of the piston. If this seal begins to leak, then it should be cleaned and lubricated or replaced. When this seal leaks, a pressure burst is typically observed periodically with each rotation. If this is observed, stop rotation and see if the periodic pressure bursts stop. If you see periodic pressure bursts only when the piston is rotating, then this seal is probably leaking. In this case, follow procedure 5.14 to lubricate or replace this seal.

5.17. Checking the Specimen Height

The specimen height is pre-set at the factory, and should not need adjustment. The height can be checked by aligning the beams to the center of the beam alignment screen, then changing the gun tilt from 10 to 5 degrees. It is helpful to increase the gas flow so as to decrease the length of the ellipse illuminated on the screen. As the gun tilt is changed, the beams will increase or decrease in length and rotate about the center of the screen.

If the Whisperlok™ bellows or the specimen mount is replaced, it may be necessary to re-set the specimen height.

If the specimen is not held at the eucentric height of the guns, the ellipses will move toward or away from the guns as the tilt is adjusted. This will result in a lower milling rate from below. In extreme cases, there will be redeposition on the bottom surface of the sample. If the center of the ellipse moves by more than about 1 mm, then you should consider adjusting the specimen height as described below. As an alternative, you may designate one gun to be used from the top always and the other gun to be used from the bottom. Align the top gun using the alignment screen as usual, and align the bottom gun using a glass disk mounted to a glue type duo-post. In each case, choose the most common average tilt angle for alignment; for example 6 degrees top and 4 degrees bottom.

Note that a slight adjustment change in gun alignment can make it seem as though the height is wrong. This can be caused by insufficient lubrication of the o-ring in the Ion Source assembly, if so then lubricate per Section 5.6.1. Before changing the stage height, be sure that both guns exhibit behavior as described above. Try adjusting the direction perpendicular to the long axis of the ellipse slightly, then re-testing.

1. Check the specimen height during milling. The specimen height must be set so that when the tilt of a gun is rotated from top to bottom the beam always strikes the center of the sample. The beam is typically aligned to the center of the sample when striking the sample from above by using the Beam Alignment Screen provided with the PIPS II.

a) If the height is set at the correct height, the beam will strike the center of the sample from both the top and the bottom.
b) If the height is set too high, the top beam will be centered and the bottom beam will strike the sample past the center.
c) If the height is set too low, the top beam will be centered and the bottom beam will strike the sample before the center.
d) If the height is set either too high or too low the sample will not mill evenly on the top and bottom.

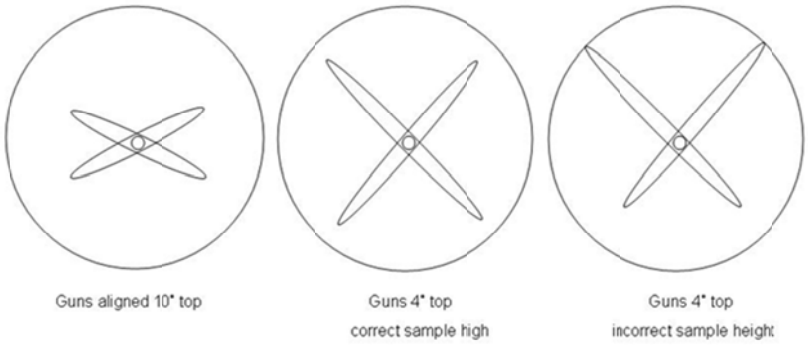

Figure 5-18 checking the specimen height.

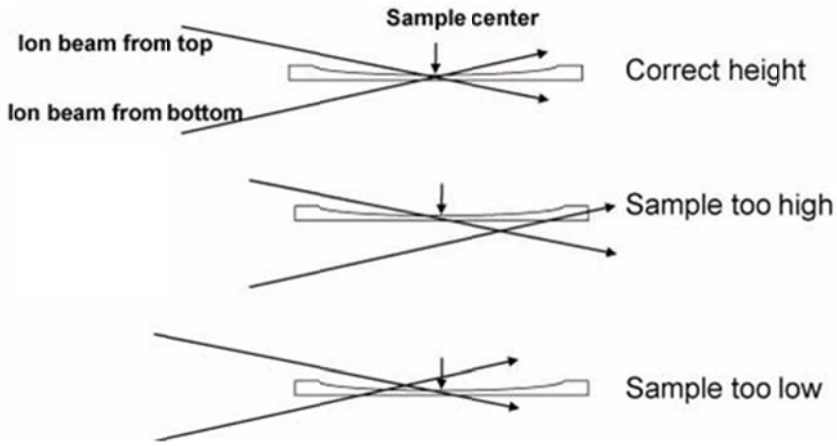

Figure 5-19 Correct beam angle setting.

2. Mount a 3 mm diameter glass screen onto a glue-type Duo- Post. Be sure the screen is flush against the recessed area of the post. A rectangular piece of glass can also be used, and if oriented properly will provide a larger view of the beams.

3. Purge the ion guns.

4. Insert the alignment screen into the PIPS II. Center the XY stage so the hole in the alignment screen does not wobble as the stage rotates.

5. Turn on both guns at 10° top, 6.0 keV, no modulation.

6. Adjust the tilt of each gun to align both beams to the center of the alignment screen. Adjust the gas flow so each beam forms an ellipse on the screen that is about 2-3 mm long. This will require more gas flow than is typically used.

7. Remove the alignment screen and insert the DuoPost with the glass screen.

8. Turn on both guns at 10° bottom, 6.0 keV, dual-beam modulation.

9. If the height is set properly, the beams will be centered on the glass screen.

10. Adjust the height of the sample by inserting an allen wrench in the holes of the height adjuster and moving the hole to the right or left. The height adjuster is threaded and moving the holes left or right results in lowering or raising the stage height. The stage must be raised in order to make an adjustment. Moving the holes to the right will result in raising the stage height. Moving the holes to the left will result in lowering the stage height.

Adjust the height until the centers of the beams are halfway between their starting position and the center of the glass screen.

Caution: We recommend that you turn off the power to the system and unplug the power cord from the back of the cabinet before opening the cabinet. Close the cabinet before turning the system back on.

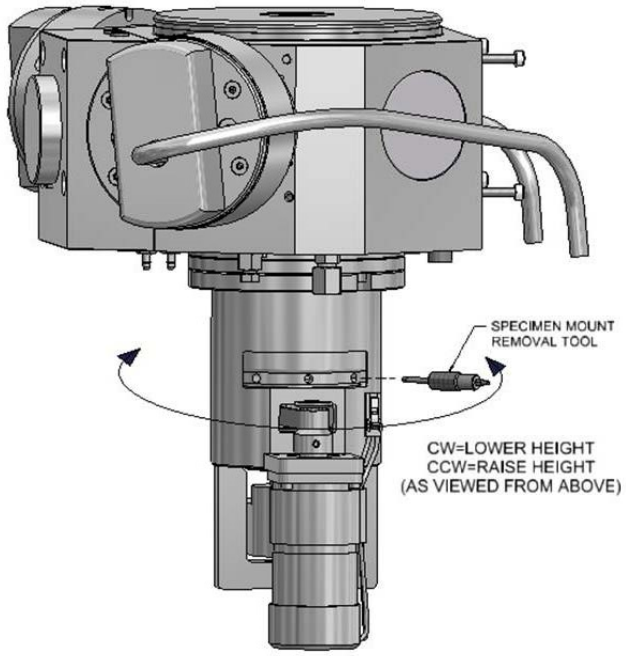

Figure 5-20 Sample height adjustment.

11. Repeat the above procedure until the center of the beams moves less than ~1 mm between 10° top (alignment screen - adjust tilt of guns) and 10° bottom (glass screen - adjust height of stage). Note that both guns may not be aligned identically. In this case, find the best compromise height for both guns.

5.18. Replacing the Gas Manifold

The gas manifold is made of acrylic and must be replaced if a crack develops which creates a leak. A crack can be created if a fitting or screw in the gas manifold is over-tightened. A field replacement gas manifold assembly includes the gas manifold, valves, fittings, and tubing. It does not include the mass flow controllers.

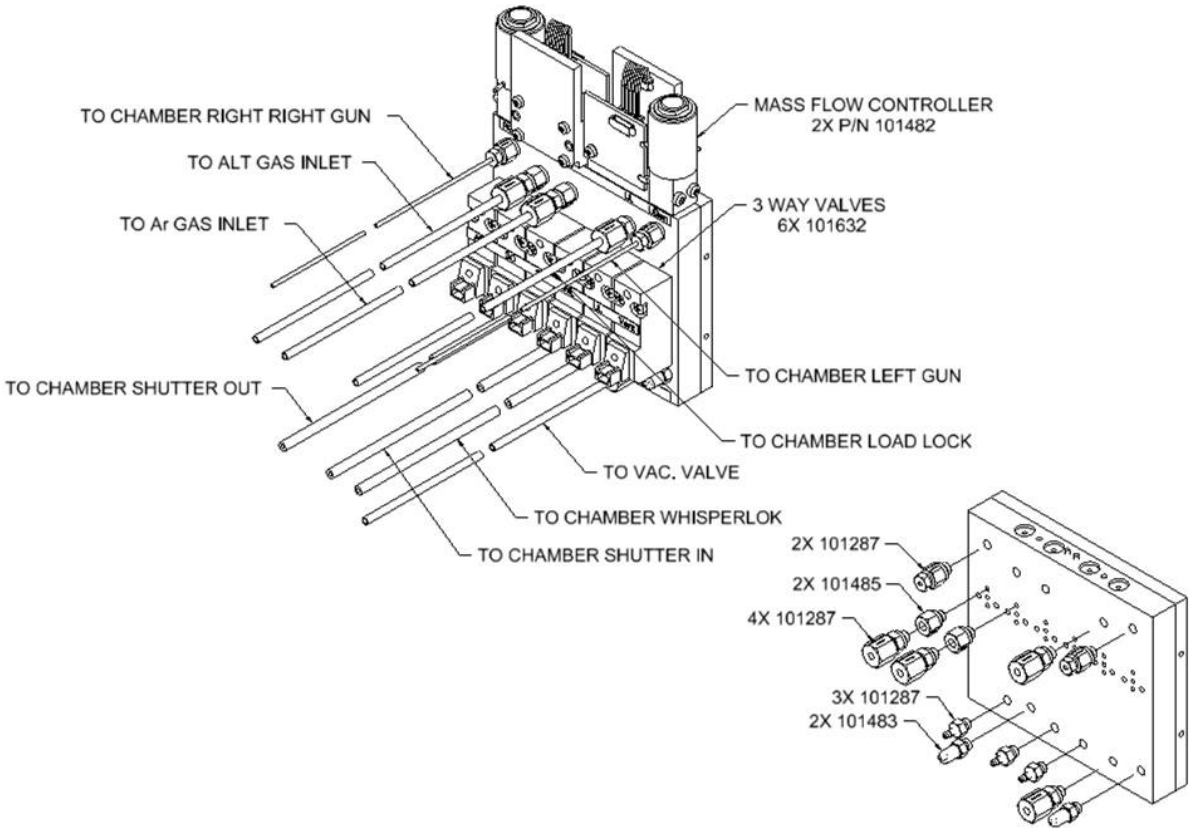

Figure 5-21 Gas manifold assembly.

5.18.1. *Removing the Gas Manifold*

1. Raise the Stage and vent the airlock chamber. This allows the airlock cover to be removed after system power is off.

2. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. Remove the system cover.

4. Turn off the Ar pressure at the regulator which supplies Ar to the system.

5. Unplug the electrical connectors for each of the 6 valves.

6. Unplug the electrical connectors for each of the 2 mass flow controllers.

7. Disconnect the tubing from all of the connectors on the bottom of the work chamber. Remember where each tube was connected.

8. Disconnect the tubing between the gas manifold and the Ar and Alt inputs. This should be done at the cabinet feed through side.

9. Disconnect the tubing between the gas manifold and Vac-valve assembly. This should be done at the Vac-valve assembly side.

10. Remove the gas manifold. Unscrew the 4 screws that secure the gas manifold to the sheet metal bracket. Gently remove the gas manifold. Inspect the gas manifold assembly. The o-ring seals may be observed from the back side through the Acrylic manifold with an optical microscope. Cracks in the Acrylic or other issues may be observed.

11. Remove the Mass Flow Controllers. Remove the screws attaching the MFCs to the gas manifold. Lift the MFCs off the gas manifold. Remove the o-rings from the gas manifold and save for re-use.

5.18.2. *Installing the Gas Manifold*

The new gas manifold field replacement kit includes a full set of tubing, which should be connected to the gas manifold before installing the manifold in the system.

1. Attach the Ar and Alt tubing to the gas manifold. This is 1/8 inch (~3.2 mm) nylon tubing with a Swagelock fitting at one end and a Beswick fitting at the other end. Connect the Beswick fitting to the Ar and Alt fitting of the gas manifold.

Caution: be sure to use 2 wrenches when installing compression fittings. One wrench is to insure that the fitting is not tightened with respect to the Acrylic manifold, which could create a crack in the manifold. Tighten the compression fittings finger tight plus 1/4 turn.

2. Attach the gun tubing to the LG and RG fittings of the gas manifold. This is 1/16 inch (~1.6 mm) green PEEK tubing with a Beswick compression fitting at each end.

3. Attach the LL and Vac tubing to the gas manifold. This is 1/8” nylon tubing with a Beswick compression fitting at both ends. The shorter tubing is connected to the LL fitting.

4. Attach the tubing to the SO, SI, and WL fittings. This is polyurethane tubing with no fittings attached. Press it on to the barbed fittings on the gas manifold. The Vac tubing is the longer of the three.

5. Install the MFCs. Place the o-rings in the o-ring depressions in the top of the gas manifold. Place the MFCs on the gas manifold, with the pins aligned

to the appropriate holes. Install and tighten one screw per MFC until the MFC is flush with the face of the gas manifold. Do not overtighten.

6. Install the Gas Manifold. Using the 4 screws, secure the gas manifold in the sheet metal bracket.

7. Attach the tubing. Attach the tubing to the work chamber, gas inlet fittings, and Vac-valve assembly. Use 2 wrenches to insure the fittings are not over-tightened.

8. Close the Vent valve and turn on the power. The MDP speed and backing pressure may be monitored in the Maintenance > Vacuum screen.

9. Turn on the Ar pressure at the regulator.

10. Purge the guns for 4-5 hours before milling samples.

5.19. Replacing a Mass Flow Controller (MFC)

This is a difficult operation due to space limitations. Gatan recommends that the MFC be replaced without removing the Gas Manifold, because this method is less likely to cause a problem with any of the tubing or fittings. If this cannot be accomplished, then remove the Gas Manifold prior to replacing the MFC.

1. Raise the Stage and vent the airlock chamber. This allows the airlock cover to be removed after system power is off.

2. Shut down the power to the PIPS II. Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. Remove the system cover.

4. Install a ground strap on your wrist. The MFC contains static sensitive components.

5. Turn off the Ar pressure at the regulator which supplies Ar to the system.

6. Unplug the power/signal cable from the MFC.

7. Loosen the M3x20 screw which secures the MFC to the Gas Manifold. Note that you will need a hex tool that is shorter than the tool provided with the instrument. It should be between 38 mm long and about 70 mm long. Note the orientation (the two MFCs have opposite orientation). Carefully remove the MFC.

8. Clean and inspect the 2 O-rings. Replace the O-rings in the pockets in the Gas Manifold.

9. Install the replacement MFC. Note that there is a pin in the top of the Gas Manifold that mates to a hole in the MFC.

10. Carefully plug the power/signal cable into the MFC. The connector and wires are delicate.

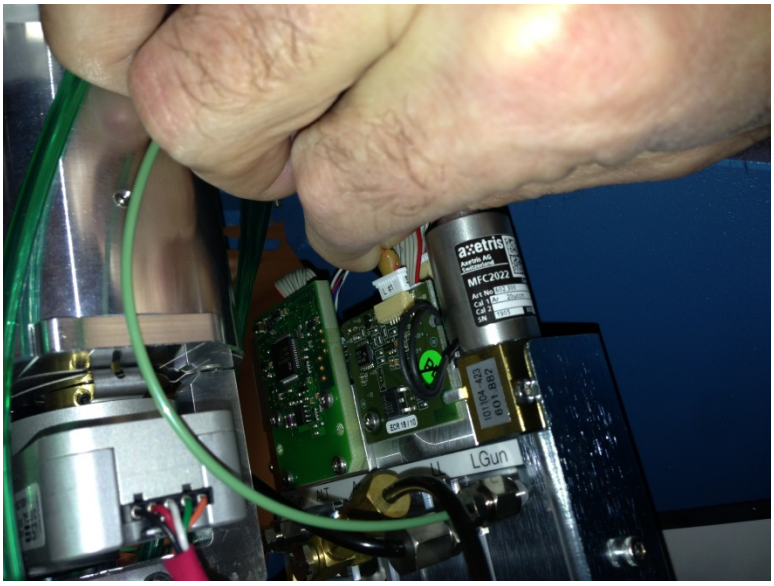

Figure 5-22 MFC removal. Unplugging the power/signal cable from the MFC.

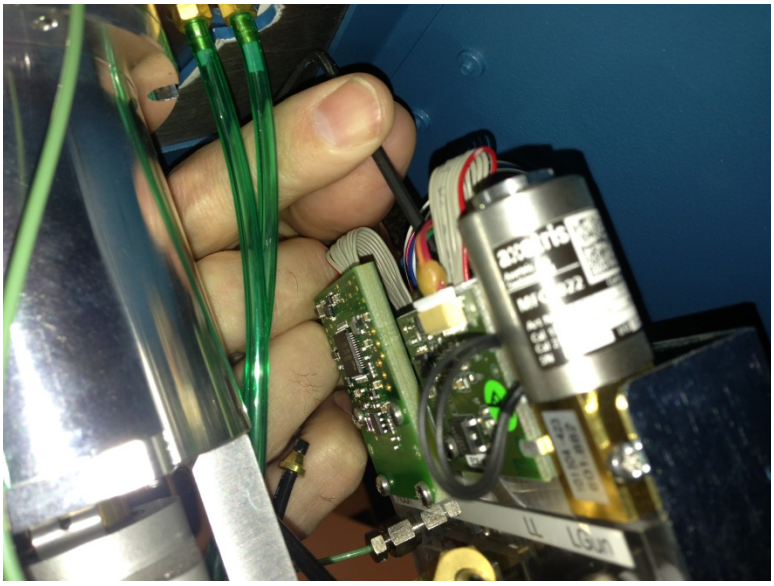

Figure 5-23 MFC removal. Loosening the M3 screw that secures the MFC.

5.20. Replacing the Touchscreen

1. **Shut down the power to the PIPS II.** Unplug the power cable from the back of the system.

2. **Remove the system cover.**

3. **Unplug the touchscreen flex cable from the touchscreen adapter PCA.** Using a small screwdriver, lift the back of the black hinged clamp up and toward the front of the system. Gently remove the flex cable.

4. **Unplug the coax flex cable from the touchscreen adapter PCA.**

5. **Remove the 4 M2x20 screws that secure the touch display to the touchscreen adapter plate.**

6. **Remove the touch display with the coax flex cable attached.** The coax flex cable is glued to the touchscreen.

7. **Install the new touch display and coax flex cable.** Install the 4 screws.

8. **Connect the coax flex cable and touchscreen flex cable to the touchscreen adapter PCA.**

9. **Install the cover.**

10. **Power on the PIPS II.** First plug the power cable into the power entry module.

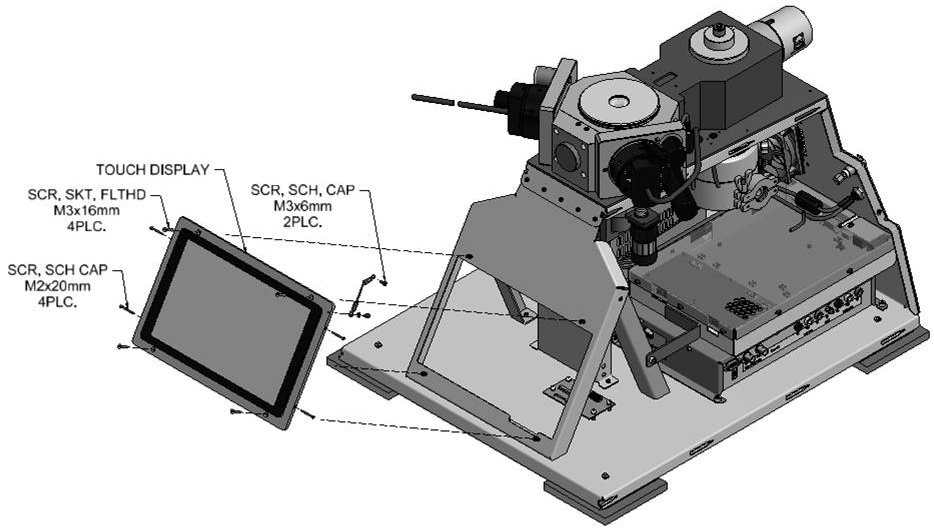

Figure 5-24 Touch display assembly.

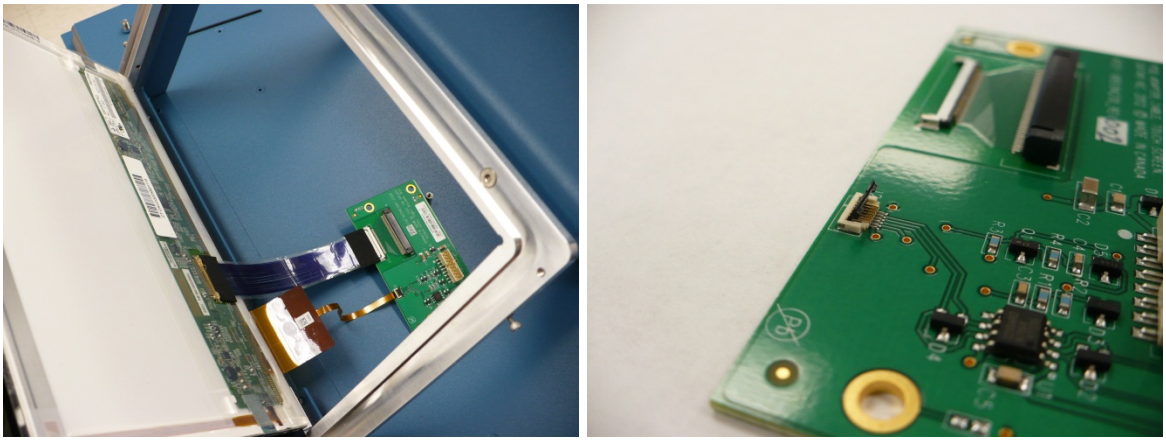

Figure 5-25 Connections for touch display.

Left image shows cable connections made prior to attaching the touch display. Right image shows the clamp for the touchscreen cable in the open position. After the touchscreen cable is inserted into this connector, press down on this clamp. Note that this is a delicate connection.

5.21. Replacing the High Voltage Power Supply

1. **Shut down the power to the PIPS II.** Unplug the power cable from the back of the system.

2. **Remove the system cover.**

3. **Unplug the gun high voltage cables from the high voltage power supply.** Remove the ground wires from the ground lug in-between the two sets of high voltage connectors.

4. Unplug the power and control cables from the high voltage power supply.

5. **Remove the three easily accessible screws that attach the HVPS to the frame.** Loosen the fourth screw but do not remove it, the HVPS is slotted in this location.

6. Remove the HVPS from the cabinet.

7. Install the replacement HVPS. Install screws.

8. Reconnect all the cables to the replacement HVPS.

9. Replace the cover.

10. Power on the system. First replace the power cable.

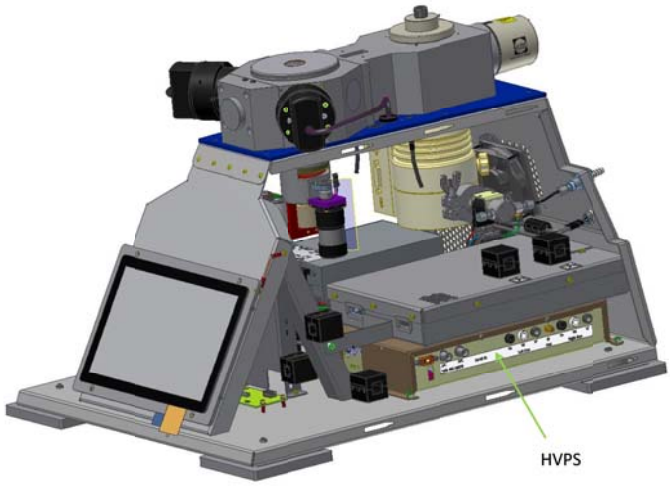

Figure 5-26 HVPS location.

5.22. Replacing the Control PCAs (CPU, I/O)

1. **Shut down the power to the PIPS II.** Unplug the power cable from the back of the system.

2. **Remove the system cover.**

3. **Remove the cover of the electronics enclosure.**

4. **Unplug the gun high voltage cables, if necessary, from the high voltage power supply.** This may be helpful to allow access.

5. Unplug the CC Gauge cable, Dewar cable, Ethernet cable, and camera trigger cable from the back of the system.

6. **Clamp the tubing between the vacuum valve assembly and the backing sensor on the I/O PCA.** A tubing clamp or forceps may be used.

7. Remove the tubing from the backing sensor on the I/O Printed Circuit Assembly (PCA). Be sure the tubing remains clamped and holds vacuum.

8. Clamp the tubing between the Gas Manifold and the Ar pressure sensor. Remove the tubing from the Ar pressure sensor on the I/O PCA.

9. Remove the fiber from the below sample illuminator (BSI) socket.

10. Disconnect the cable connectors from the PCAs. Insert tweezers between the clamping arms and the connector, to release the connector from the clamping arms. Pull the connector out of the socket. Be very careful not to pull on the wires, or they may break.

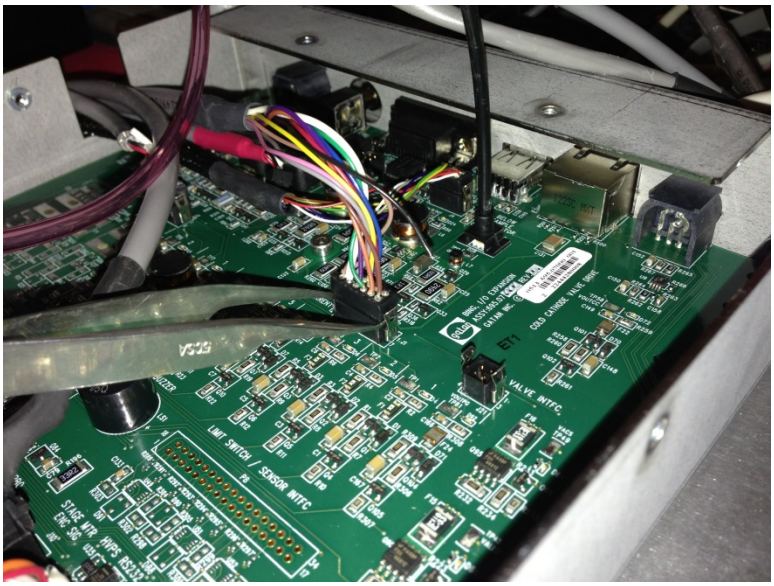

Figure 5-27 Removing cables from the I/O PCA.

11. Remove the 4 screws that secure the I/O PCA.

12. Remove the nuts from the CC Gauge and Dewar connectors on the back of the system.

13. Remove the jack screws from the RS-232 connector on the back of the system.

14. Remove the PCAs from the PCA enclosure.

15. Install the new PCAs. Replace the 4 screws, the 2 nuts, and the 2 jack screws.

16. Connect the cables to the same sockets they were removed from. The captive connectors have an orientation defined by a chamfer on two corners. The image below shows the location of the sockets, and each cable should be labeled with the connector. In addition, the List of Cables shows the associated connector for each cable.

17. Replace the electronics enclosure cover.

18. Replace the system cover.

19. Power on the system. First replace the power cable.

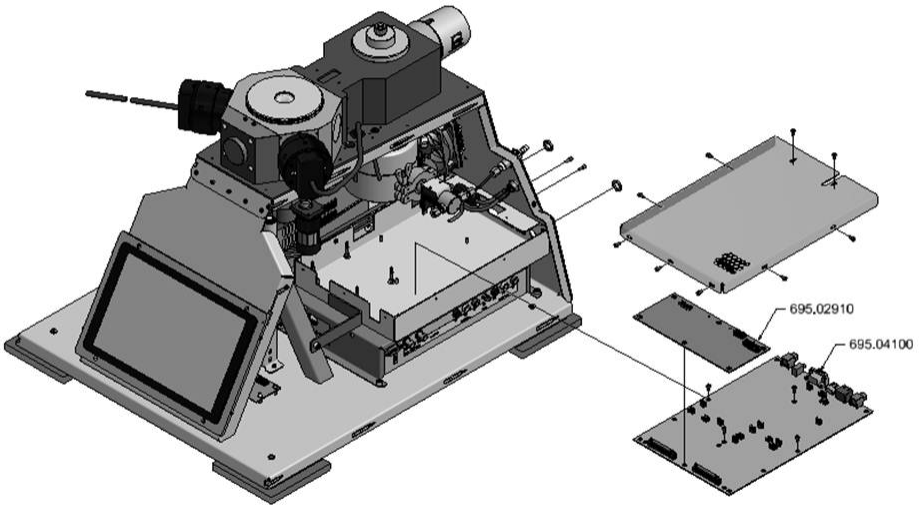

Figure 5-28 PCA assembly.

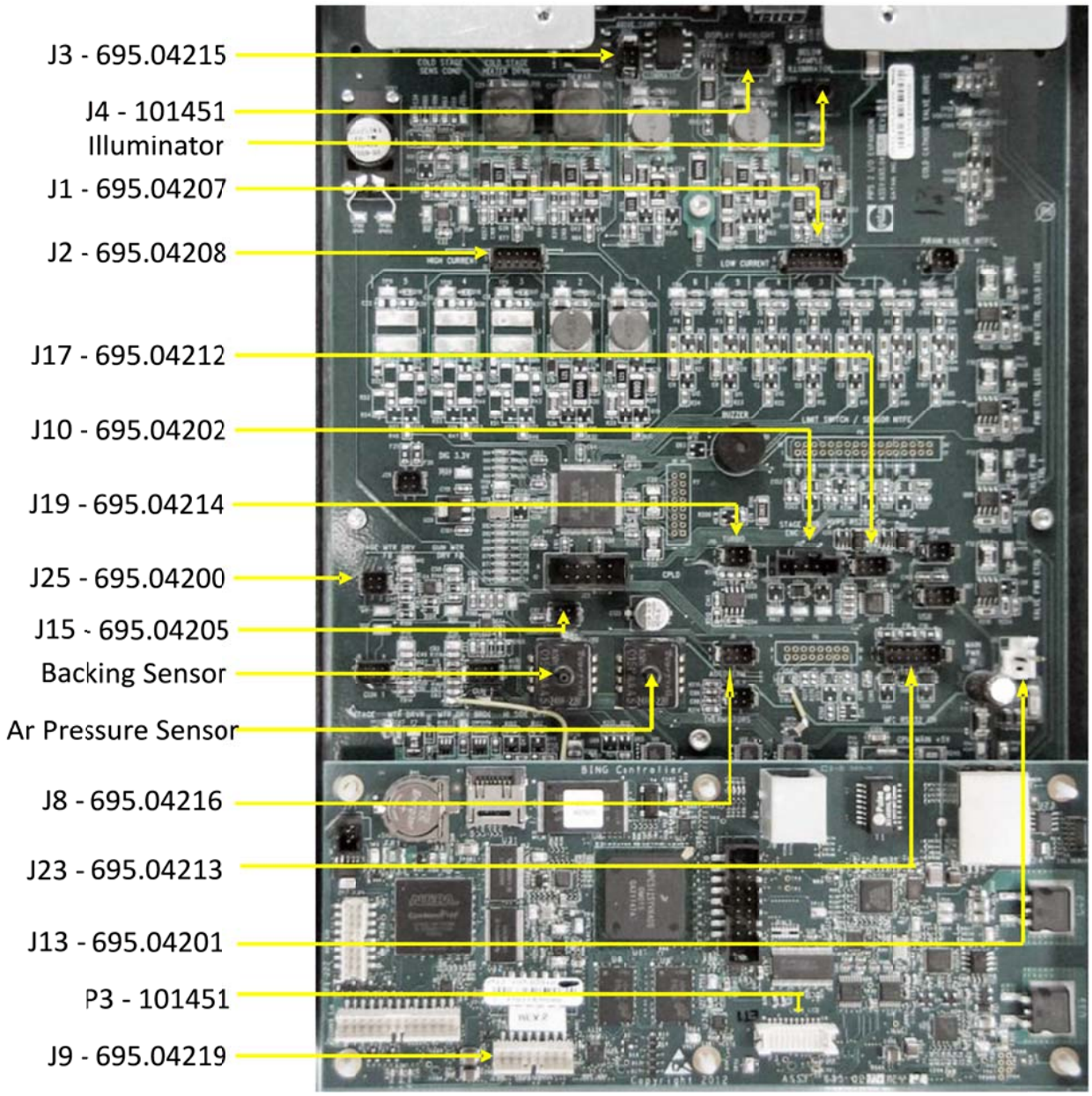

Figure 5-29 PCA connector locations and associated cable part numbers.

5.23. List of O-Rings

Description	Size	Quantity	Location
Diaphragm Pump Assembly	012	1	DP fitting
	M4.5x1.4mm	1	DP fitting
Chamber Assembly	022	1	Airlock window
	019	1	Airlock window
	044	2	Airlock/chamber top
	042	1	Chamber top
	Q4116	1	airlock
	005	1	Shutter piston
	111	1	Shutter piston
	017	1	Front plate
	030	1	Front plate
	012	3	Shutter guide
Beam Stop	022	2	Beam stop
Whisperlok Assembly	004 GLV	1	Sample mount
	035	2	Chamber
	031	1	Chamber
	Q4111	1	Rotate shaft
	135	1	Bellows
Ion Gun Assembly	003	1	Focus electrode
	010	2	Focus electrode
	020	1	Housing
	030	2	Chamber
Manifold Assembly	141	1	Manifold-chamber
	123	1	CC gauge
	039	1	Dewar port
Vent Valve	012	1	Vent valve
	010	1	Vent valve
Cold Stage Dewar	039	1	Dewar port
	014	1	Electrical feedthru
	112	1	Vent valve
Gas Manifold	008	4	MFC

5.24. List of Cables

Description	Part #	Qty	Connector #
Whisperlok motor	695.04200	1	J25
Power	695.04201	1	J13
Whisperlok encoder	695.04202	1	J10
Cold Cathode gauge	695.04203	1	JP2
Cold Stage dewar	695.04204	1	JP1
DP, internal	695.04205	1	J15
DP, external	695.04206	1	Back panel
Valves, manifold	695.04207	1	J1
Vacuum valve	695.04208	1	J2
Gun motor, internal	695.04210	2	J6(left), J7(right)
HVPS, communications	695.04212	1	J17
MFCs	695.04213	1	J23
MDP	695.04214	1	J19
Illuminator, above	695.04215	1	J3
Autoterminator, internal	695.04216	1	J8
Gun motor, external	695.04217	2	Top panel
Camera trigger	695.04218	1	J20
Touchscreen	695.04219	1	J9 (CPU)
Video/backlight	101451	1	J4 (I/O),P3(CPU)
Video, coax flex	101462	1	J2(TS Adapter)

6. Trouble Shooting

Symptom	Problem cause	Solution
Chamber pressure display 0 on Milling screen.	MDP not up to speed. Manual vent valve open. Vacuum leak. MDP failure. DP failure.	Turn off gas flow. Verify MDP speed (Maintenance > Vacuum).
Piston will not lower. Backing pressure does not return within 5 Torr when pumping airlock.	Viewing port or window not seated properly. VE valve failure.	Clean viewport o-rings. Press down on viewport and window while pressing Vac button. (Maintenance > Vacuum) Check VE valve cable/control.
Piston cannot be lowered into the chamber. Backing pressure does return within 5 Torr when pumping airlock.	Argon supply interrupted. WL or LL valve failure.	Check argon pressure 25 psi (1.72 bar) or main valve closed. Check WL and LL valve cable/control, be sure WL valve LED illuminated and LL valve LED not illuminated.
Specimen difficult to see in working position	Sputtered material obscuring viewing window.	Clean or replace viewing window. See Section 5.1.
Specimen will not rise fully into the airlock	Dry and coated airlock vacuum seal.	Service vacuum seal. See Section 5.2.
Poor vacuum when specimen mount rotation is operated, typically observe periodic pressure burst during each rotation.	Dirty or dry quad-ring in Whisperlok piston.	Clean and lubricate piston quad-ring. See Section 5.14.
Shutter will not close or closes only part way	Argon supply interrupted or HV timer may be off. SI or SO valve failure. Shutter piston may be clogged or have failed.	Check argon pressure 25 psi (1.72 bar) or main valve closed. Check SI valve LED is on and SO valve LED is off when shutter is in. Check SI valve LED is off and SO valve LED is on when shutter is out. Check SI and SO valve cable/control. Clean and inspect the shutter piston.
Excessive argon use	Argon leak	Check for leaks. See Section 5.9
Cold-cathode gauge reading fluctuates or reads excessively high.	Gauge tube contaminated.	Service/clean gauge tube. See Section 5.4.
Ion gun has no output. Current=0, voltage on, gas on.	Gun shorted (Anode cup to magnet)	Clean guns. See section 5.6.
Gun output is extremely erratic.	Guns are not purged sufficiently	Purge guns. See Section 3.2.
Chamber pressure very high when stage is lowered.	Leak in bellows.	Replace bellows assembly.
Piston is in raised position and sample mount is rotating.	Argon supply interrupted.	Check argon pressure 25 psi (1.72 bar) or main valve closed.

7. PIPS II Options

7.1. Cold Stage Option

Certain PIPS II models include a cold stage. This option must be installed at the factory on a new PIPS II.

The PIPS II Cold Stage upgrade components replace existing PIPS II components as follows:

- The dewar assembly replaces the existing PIPS II vent-valve assembly or liquid nitrogen trap.
- The cold stage Whisperlok assembly replaces the existing Whisperlok assembly.
- The PIPS II I/O PCA connects to the dewar via a cable, and provides a readout of the cold conductor temperature as well as control of two heaters.

The first heater controls the temperature of the cold conductor, and the second is used to boil-off the liquid nitrogen in the dewar. For instance, if a sample has a phase-transition temperature at -100 °C that you would like to avoid, the conductor temperature can be set to -50 °C prior to inserting the sample. In addition, if the stage is cold and you would like to mill at room temperature, you can set the conductor temperature to 23 °C.

When the dewar is filled with liquid nitrogen, it cools a copper plate that extends into the specimen chamber. Copper braids connect that plate to a cold conductor that sits next to the cold stage spindle. Brushes thermally connect the cold conductor to the cold stage spindle. When the Whisperlok is lowered into the milling position, it makes thermal contact with the cold conductor and the sample is cooled. When the Whisperlok is raised into the air lock, it no longer makes thermal contact with the cold conductor. The Whisperlok then makes thermal contact with the o-ring in the air lock and comes into thermal equilibrium with the chamber walls.

A thin glass rod (specimen mount window) is installed in the bottom of the specimen mount in order to shield the vacuum window from sputtered material. This rod can be removed and polished clean when deposited material begins to block the light from the below sample illuminator. The vacuum window cannot be easily cleaned, so it is important that the glass rod is always installed. The chamber must be vented in order to clean this rod (see Cleaning the Specimen Mount).

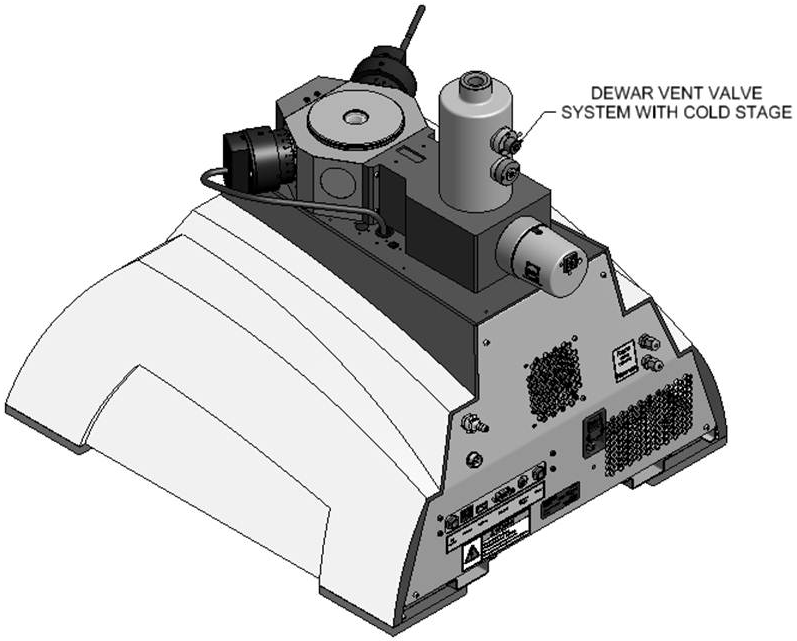

Figure 7-1 System with Cold Stage installed.

7.1.1. *Operation*

Fill the dewar with liquid nitrogen prior to loading a sample into the PIPS II. Once the stage is cold, you may insert and remove samples from the airlock as required.

It is important when removing a cold sample that you allow enough time (at least 20 minutes) for the sample to warm up after raising the stage and before venting the Air- lock. This will prevent water from condensing on a cold sample. In some regions with low dew point, it may be necessary to wait longer before venting in order to prevent condensation.

NOTE: Do not overfill the dewar; the starting level should be just below the bottom of the dewar neck. After about 10 minutes, boiling in the dewar will cease and more liquid nitrogen may be added. The dewar will typically last about 6-8 hours between refills. After the liquid nitrogen has boiled off, it typically takes about 4 hours for the cold conductor to warm up to room temperature.

Filling the Dewar

1. **Raise the stage into the airlock.**

2. **Fill the dewar with liquid nitrogen.**

Do not overfill; the starting level should be just below the bottom of the dewar neck.

3. **The liquid nitrogen will boil off in a few minutes.**

Continue refilling the dewar for about ten minutes to replenish the liquid nitrogen.

NOTE: It may take more than one "top off" to initially cool down the dewar.

4. **After about ten minutes the boil-off rate will have slowed dramatically.**

Top off the dewar.

5. Place the supplied lid on the dewar.

6. **The system is ready for a sample to be installed**.

The liquid nitrogen in the dewar should last 6-8 hours if the heater is not being used.

Loading a Sample

1. Raise the stage into the airlock (if not already raised).

2. Wait at least 20 minutes.

NOTE: It is not necessary to wait if the stage has been in the raised position for at least 20 minutes previously.

3. Vent the airlock and remove the airlock cover.

4. **Insert sample (specimen post).** Be sure the specimen post is properly seated in its lowest position.

5. Replace the airlock cover.

6. **Lower the sample.**

7. **Go to Settings > Heaters page.** Set the cold stage heater temperature to 23° and enable it.

8. Find the center of rotation.

9. Bring the sample to home position and note the position of the center of rotation with respect to a known feature on the sample.

10. Raise the stage and vent.

11. Use the hex key and move the point of interest to the center of rotation.

12. Repeat 1-5, for fine adjustments.

13. After point of interest is centered, go back to Settings - Heaters page and disable the cold stage heater.

14. Wait fifteen minutes for the sample to cool down.

If your sample has poor thermal conductivity you may want to wait longer.

15. Begin milling.

Removing a Sample, method 1

1. **Set cold stage heater to 23 C.** See below for heater instructions.
2. **Turn on cold stage heater.**
3. **Wait 20–25 minutes for sample to reach room temperature.**
4. **Raise the stage into the airlock.**
5. **Turn off cold stage heater.**
6. **Vent the airlock, then remove the airlock cover.**
7. **Remove the specimen post.**

Note: if you experience condensation on the sample, try increasing the cold stage heater to 30 C or waiting longer during step 3.

Removing a Sample, method 2

1. **Raise the stage into the airlock.**
2. **Wait 20-25 minutes.**
3. **Vent the airlock, then remove the airlock cover.**
4. **Remove specimen post.**

Temperature Control

The cold stage is controlled by the GUI (Settings > Heaters).

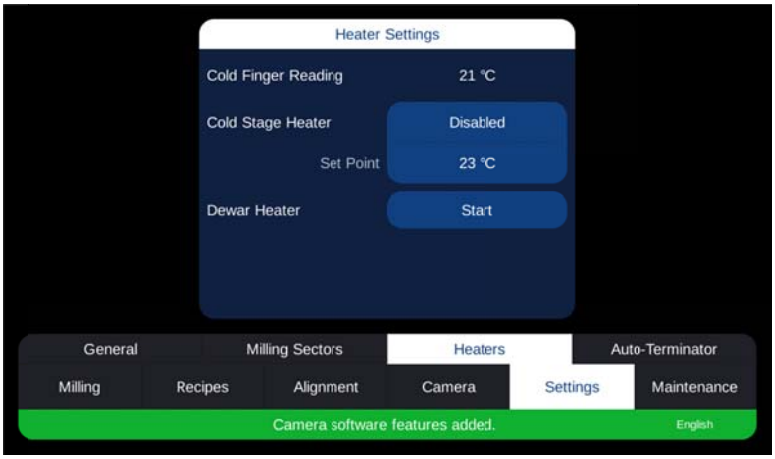

Figure 7-2 Settings Page.

Cold Finger reading: Displays the temperature of the cold finger

Cold stage heater: Turns the heater on and off. If the heater is on and the temperature drops below the heater set point, the heater is turned on until the temperature is 1 deg above the set point

Dewar heater: Activates the dewar heater. This heater stays on continuously until the Cold Finger Temperature reaches 25° C, then it shuts off.

Raising the LN Dewar Temperature

1. **Remove the sample.**

2. **On GUI, Start the Dewar Heater.** This heater stays on continuously until the Cold Finger Temp reaches 25° C, then it shuts off. This typically takes less than one hour.

Setting a Sample Temperature

1. **Raise the stage into the airlock.**

2. **Fill LN dewar (if not already filled).**

3. **On the Settings > Heaters Page, set the Cold Stage Heater Set Point to the desired temperature.**

4. Enable the cold Stage Heater. The temperature will rise (Cold Finger Reading) until it reaches the set point, then will switch off. When the temperature falls below the set point, the heater will turn on again. The controller will cycle this way as long as it is the Cold Stage Heater is Enabled. The sample will not experience temperature swings because there is a long time lag between the conductor temperature and the sample temperature.

5. Lower the stage.

6. Wait twenty minutes for the sample temperature to stabilize.

NOTE: The range of the set point for the Conductor Heater is -200 °C to 50 °C. Setting a set-point temperature lower than the minimum temperature of the stage (~-150 °C) will not result in a lower temperature. The controller can only raise the conductor temperature.

Caution: The Conductor heater will stay active until it is disabled. Do not forget to disable it when it is no longer needed.

7.1.2. *Recommendations*

For best results, consider these additional factors:

- Crystal bond can fail at low temperatures, in addition to being thermally insulating. If you use a glue-type post or a Cu post, consider using silver paint instead of crystal bond.
- Samples that are thicker at the outside rim will generally have better thermal conductivity. Note that this can limit the range of milling angles that can be used.
- Lower ion beam density results in lower sample temperature. Using the gas flow to defocus the beam will result in lower sample temperatures. Likewise, milling at lower beam energy results in lower sample temperature. If you are using beam modulation, increasing the rotation speed will result in a lower sample temperature.
- Gatan recommends method 1 for removing samples. Method 2 does not result in active pumping on the airlock after the stage is raised, and may result in some condensation on the sample if the pressure in the airlock increases during the wait time.

7.1.3. *Performance*

A sample post will reach a temperature of approximately -120 °C +/- 25 °C. The sample post will typically cool to nearly -100 °C in fifteen minutes. It will reach its lowest temperature in 30-40 minutes. The sample will typically reach the same temperature as the post; how long this takes depends upon the thermal conductivity of the sample and its thickness.

With the ion guns on at 5 kV, 5 degrees, and dual beam modulation a bulk sample (e.g., Ta) mounted in a clamp-type DuoPost will increase in temperature by about 25-50 °C. The temperature of a thin specimen will increase more, depending on the thermal conductivity of the sample, the thickness of the sample, and the power density of the ion beam. In general, lower energy and a more defocused (broader) beam will result in a smaller temperature rise during milling.

The temperature measured at the cold conductor is typically about 50-75 °C cooler than the temperature at the sample. In addition, there is a time delay of about fifteen minutes between a change in cold conductor temperature and the corresponding change in sample temperature. For instance, if the stage is lowered and you fill the dewar with liquid nitrogen, the conductor will reach -100 °C about fifteen minutes before the sample.

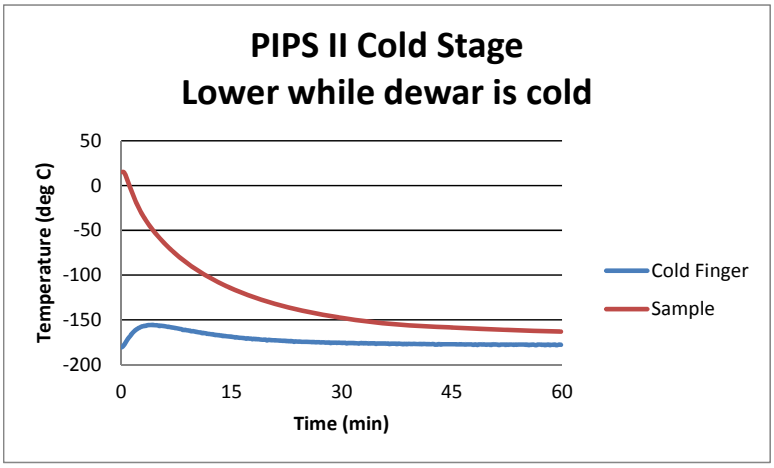

Figure 7-3 Sample and cold conductor temperature over time.

Stage was lowered at zero minutes. Dewar was filled and cold prior to this test.

NOTE: When the stage is raised into the airlock, the temperature of the sample post will typically increase to nearly room temperature in fifteen minutes.

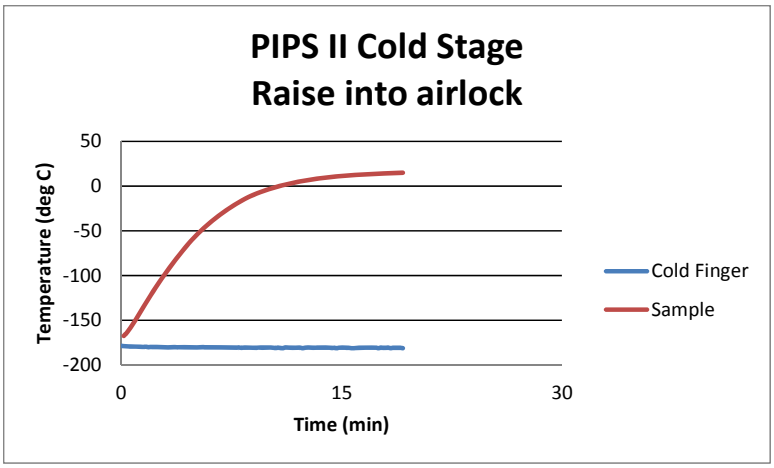

Figure 7-4 Sample and cold conductor temperature over time.

Stage was raised at zero minutes.

7.1.4. *Maintenance*

Checking the Cold Conductor Brush Wear

The brushes that make contact between the cold conductor and the spindle exhibit wear. They are expected to last several years, but should be inspected every six months. The Cold Conductor Assembly brushes can be replaced only with Gatan provided parts, which are designed to meet specific requirements of thermal conductivity, electrical conductivity, and lubricity in vacuum.

1. **Raise the stage and vent the airlock by pressing the Vent button.**

2. **Turn off the power to the system.**

3. **Wait ten minutes for the MDP to spin down, then slowly vent the system.**

4. **Remove the viewing port.**

5. **Remove the cover using the specimen mount removal tool.**

6. **Remove the shutter using a small hex tool.** See section 5.5 for instructions.

7. **Visually inspect the brushes for wear.**

a. **The thinnest part of the brushes are 0.050" (1.27 mm) thick when they are new. When the brushes reach a thickness of about 0.015" (.38 mm), they should be replaced.**

b. Note that there is a mechanical stop which prevents the brushes from wearing too thin.

c. If the two sides of the mechanical stop are in contact with each other, then the cold conductor assembly must be replaced.

8. **Vacuum out any powder or flakes of brush material that has fallen to the region below the brushes.**

This material is a normal part of the wear process of the brushes. If an excessive amount of material is built up on the brushes, you may want to remove the cold conductor assembly and clean the material from the brushes with a dry applicator or similar soft material.

Caution: Do not use any liquid or solvent on the brushes, this will destroy them.

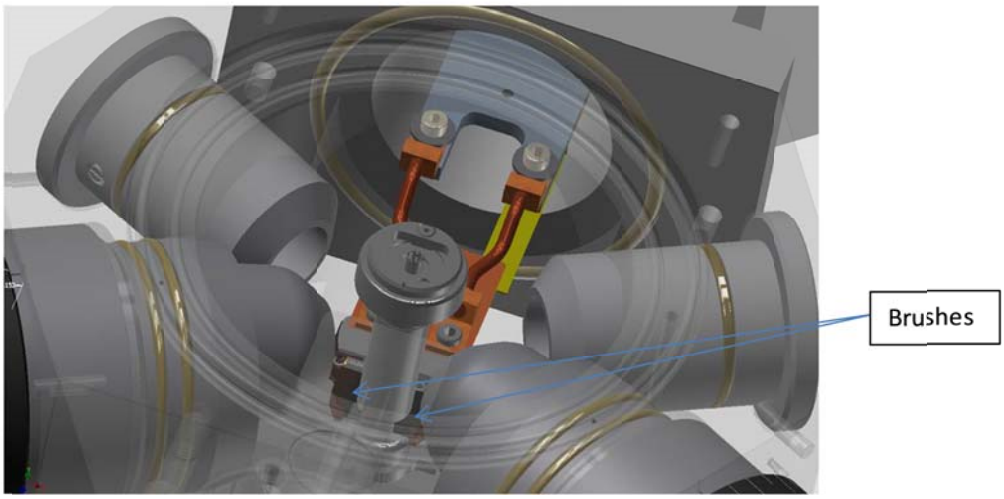

Figure 7-5 Interior chamber showing the cold conductor with new brushes.

Replacing the Hinged Conductor Assembly

1. Remove the shutter (section 5.5).

2. Remove the Sample Mount (section 5.3).

3. Remove the two M2.5 screws and washers on the top of the cold conductor assembly.

4. Move the heater out of the way.

5. Loosen the M2 screw that holds the cold conductor assembly to the bellows assembly.

6. **Carefully lift the cold conductor assembly out of the chamber, making sure not to damage the brushes.**

7. **Insert the new brush arms into the new cold conductor base.** Do not install the spring yet. Note that in addition to the spring that clamps the brush arms together, there is a spring threaded into a bottom hole of the cold conductor base. This spring makes electrical contact between the sample stub and the chassis, and is needed for proper operation.

Caution: Do not use any liquid or solvent on the brushes, this will destroy them.

8. **Install the cold conductor base into the system. Be careful not to damage or drop the brushes.** Be sure the spring between the cold conductor base and the top of the bellows assembly is perpendicular to the bellows assembly (not kinked).

9. Tighten the M2 screw.

10. Install the spring onto the two posts on the brush arms.

i. Place one side over the first post, then use tweezers to stretch the spring over the second post. There is an indent in the posts to capture the spring.

ii. Make sure the brushes are aligned to the spindle (i.e. there is not a gap between them).

11. Attach the Cu braids and heater to the cold conductor assembly with the M2.5 screws and washers. The heater must be installed below the Cu braids.

12. Install the Sample Mount.

13. Install the shutter.

14. Replace the cover and viewing port.

15. Close the vent valve.

16. Turn on the power and wait for the ion gauge to turn on.

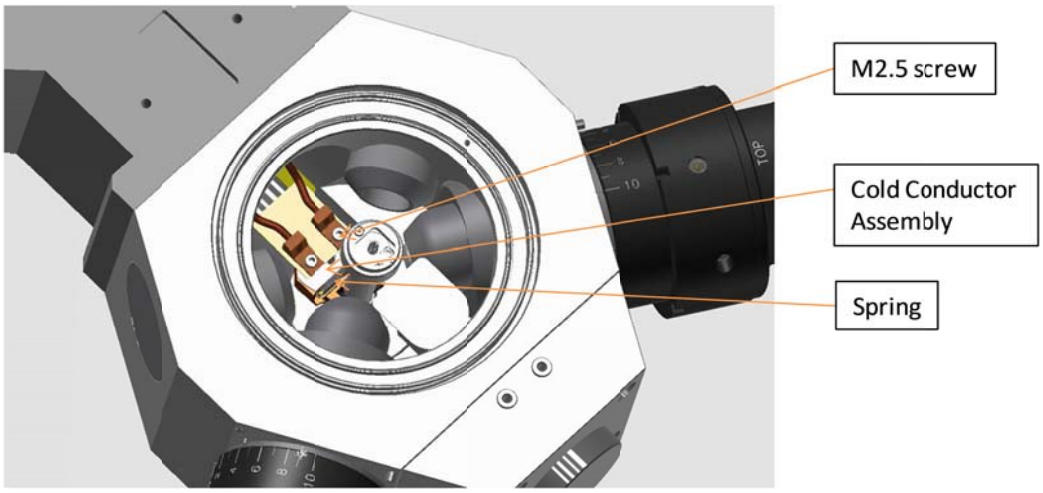

Figure 7-6 Open chamber showing access to cold stage.

Replacing the Dewar Assembly

To Remove the Dewar Assembly

1. **Raise the Stage and vent the airlock chamber.** This allows the airlock cover to be removed after system power is off.

2. **Shut down the power to the PIPS II.** Wait at least 10 min to allow the MDP to come to a complete stop. Then vent the work chamber by opening the Vent valve. Unplug the power cable from the back of the system.

3. **Lift off the Viewing Port.** Press the Airlock piston down into the Work Chamber if it hasn't already lowered itself.

4. **Remove top cover plate.** Using the pin end of the Specimen Mount Removal Tool, insert the pin into one of the holes in the top cover plate, push gently and tilt the plate up and out for removal.

5. **Disconnect the cold stage.** Remove the two M2.5 screws and washers and disconnect the cold stage heater from the hinged conductor assembly. See Figure 7-6.

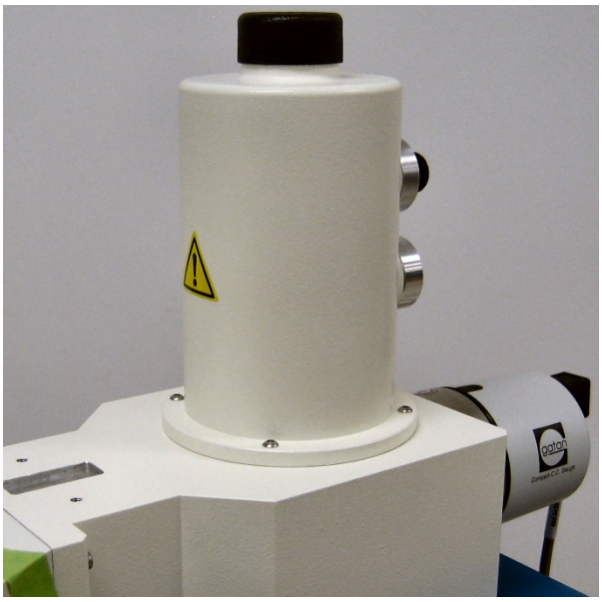

Figure 7-7. Dewar Assembly installed in manifold.

6. Remove the Dewar assembly.

a) Unplug the cable from the back of the dewar.
b) Remove the 4 M3 screws that secure the dewar to the manifold. See Figure 7-7.
c) Lift the dewar assembly upward until it is free from the manifold. Then tilt the top of the dewar forward and move the dewar backward so that the Copper bar clears the manifold. It may be necessary to tilt the dewar sideways to clear a microscope assembly, or even to remove the microscope prior to this work.

7. Install the new Dewar assembly.

a) Insert the dewar assembly into the manifold. Press down firmly to seat the o-ring in the manifold.
b) Install the 4 M3 screws that secure the dewar to the manifold. See Figure 7-7.
c) Connect the cold stage to the hinged conductor assembly. Install the two M2.5 screws and washers that connect the cold stage heater to the hinged conductor assembly. See Figure 7-6.

8. Install the top cover plate.

9. Install the Viewing Port.

10. Turn on power to the system.

7.2. End-point Detection

The PIPS II may be equipped with a Reflection Illuminator, a stereo light microscope or a digital camera, and a Transmission Illuminator. Employing the latter two, one can use the interference-fringes technique to monitor specimen thickness of semiconductors and insulators to aid in end-point detection. With silicon, for example, each colored fringe represents a thickness change of a few hundred Angstroms. When a specimen ceases to produce new fringes, perforation is imminent.

7.2.1. *Autoterminator*

There is usually no warning prior to perforation in the case of opaque materials so the Autoterminator can be used to detect perforation. However, the Timer should be used during the early stages of thinning to prevent accidental overruns.

Perforation detection

Light from the Transmission Illuminator enters a condensing lens in the Autoterminator and is directed onto a sensor to produce a digital readout. When the light intensity is high and the readout exceeds 99, milling is switched off.

NOTE: Contamination of the Viewing Port can be reduced by delaying the use of the Autoterminator until the last stages of the thinning process.

Sensitivity

Operating at maximum sensitivity, the smallest hole the Autoterminator can detect in an opaque specimen is approximately 35 µm in diameter. This diameter can be reduced by manually stopping the milling process before the Autoterminator display reaches 99 and visually checking the specimen to determine how much more thinning is required.

The actual light level corresponding to a reading of 99 can be adjusted within certain limits using the Sensitivity control. This control enables the operator to select varying termination hole sizes in the specimen and also to compensate for varying degrees of specimen transparency. The hole should not be allowed to become too large because this may allow the underside of the specimen to become contaminated.

Perforations in partially transparent samples may be detected by a combination of lowering the intensity of the bottom illuminator and reducing the sensitivity of the Autoterminator. Experimentation is required to find useful conditions for different types of samples.

NOTE: Maximum sensitivity setting and highest light intensity should be used for metals. Lower sensitivities and lower light levels may be preferred for semiconductors and ceramics. For best results, make sure the specimen Viewing Port is clean before using the Autoterminator.

The Autoterminator is mounted on the Airlock cover and plugs into a socket located to the right of the Pumping Manifold. The Autoterminator incorporates a special shutter-control feature to minimize the amount of sputtered material accumulating on the Viewing Port. When the Autoterminator is in place and the guns are operating, the Shutter will open for about 1 sec every specific seconds/rotations (defined by the user) for sampling of the transmitted light intensity. This sampling rate is maintained until the Autoterminator shows 10 on the digital display whereupon the sampling rate is automatically increased to every 16 sec, with this rate maintained until termination.

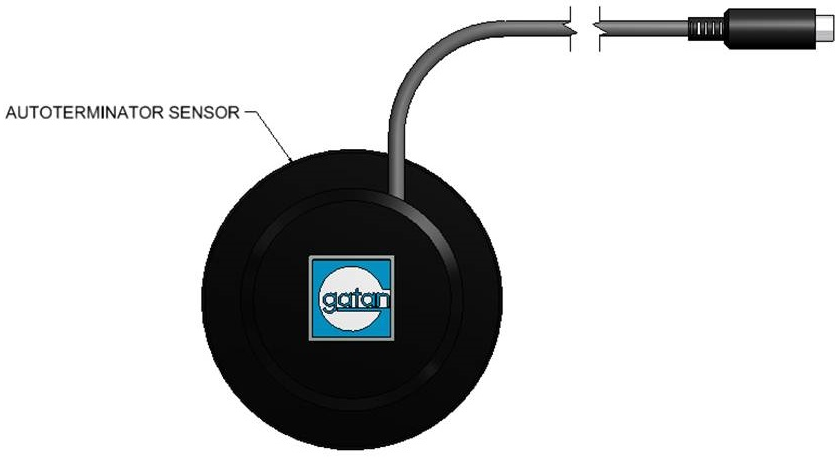

Figure 7-8 Autoterminator sensor top view.

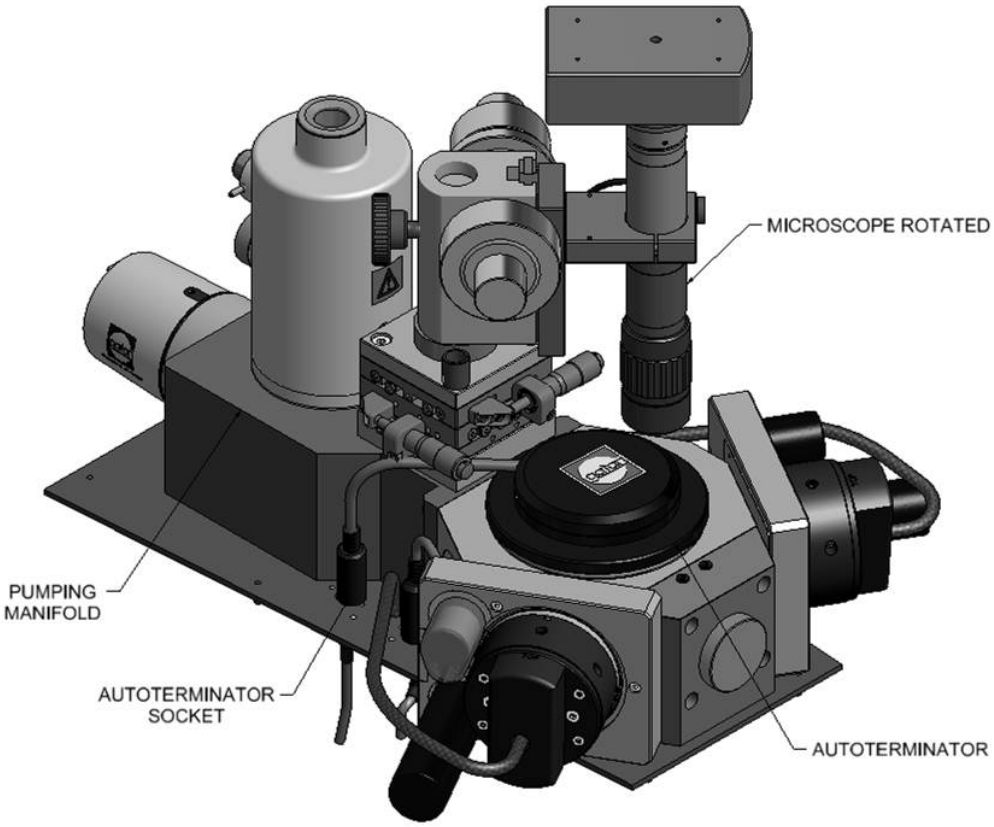

Figure 7-9 Autoterminator shown in working position.

7.2.2. *Installation and Checkout of the Autoterminator*

1. The PIPS II must be powered on, under vacuum, and with a specimen post (but no specimen) in place.

2. **Set up the Autoterminator.** Place the Autoterminator onto the Airlock cover. Plug its cable into the socket to the left of the Pumping Manifold. Turn off the transmission illumination on the Camera page.

3. Set the Sensitivity control to 50%.

4. Check the Autoterminator reading. Go to Settings > Autoterminator on the GUI. Verify that the reading is less than 10%.

5. Turn on the transmission illumination. Go to Camera > Bottom Illuminator. Set the intensity to maximum.

6. Check the Autoterminator reading. To go Settings > Autoterminator on the GUI. Verify that the reading is 100%.

7. Checkout is complete.

7.2.3. *Operation*

The Autoterminator can be configured using from the Settings page on the GUI.

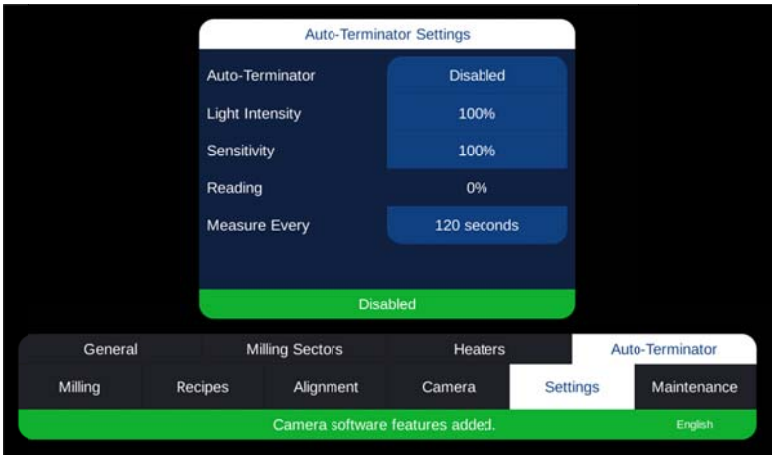

Figure 7-10 Autoterminator page.

Reading: Displays the last signal read by the autoterminator.

Sensitivity: This control enables the operator to select varying termination hole sizes in the specimen and also to compensate for varying degrees of specimen transparency.

Light intensity: Sets the intensity of the bottom illuminator while a reading is being made.

Measure every: Tells the system how often to take a measurement. It can be every X rotation or every X seconds. Once the reading reaches 15% of the trip point, readings will be taken every 16 seconds, instead of the interval set above.

7.3. Digital Zoom Microscope Option

Certain PIPS II models include a digital zoom microscope option. This option must be installed at the factory on a new PIPS II.

The PIPS II digital zoom microscope option consists of the following components:

- XY stage mount holds the microscope assembly and allows the microscope to be positioned in X and Y.
- Microscope assembly.

- Digital camera, with USB cable to the imaging PC and trigger cable to the PIPS II.
- Imaging PC. This PC has an Ethernet cable connected to the PIPS II, and a USB cable connected to the camera.
- DigitalMicrograph™ software installed on the imaging PC which controls the camera and certain functions in PIPS II.

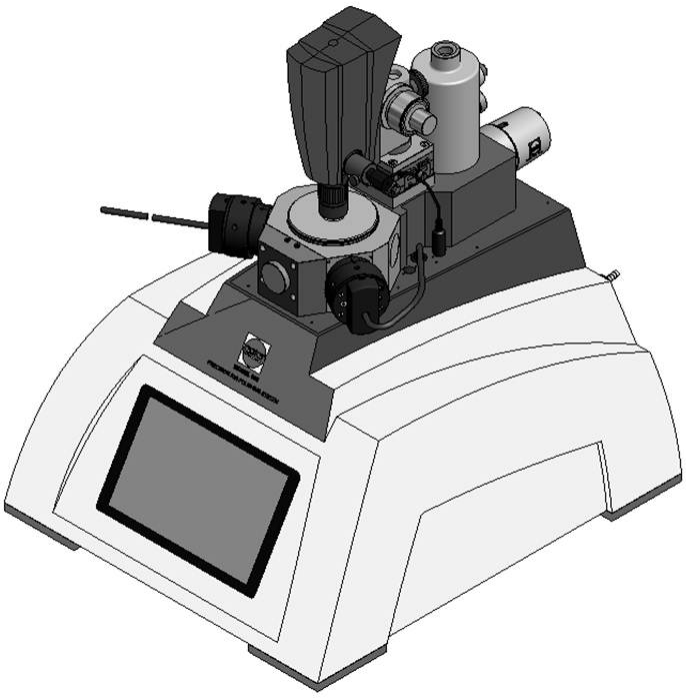

Figure 7-11 System with digital zoom microscope.

7.3.1. *Camera Software Operation (DigitalMicrograph™)*

DigitalMicrograph™ is an application used for acquiring, visualizing, analyzing, and processing digital image data. DigitalMicrograph supports all of the top industry standards for storing files. You can open and store TIFF, GIF, PICT, BMP, and other formats using DigitalMicrograph.

7.3.2. *Basic Concepts*

DigitalMicrograph presents all of its information through the use of windows. Each window contains a set of related information.

Image document windows contain a visible representation of a page of paper. Images can be placed on this page. Other objects such as lines, boxes, and text can also be placed on this page. You can open, save, and print image document windows.

Many aspects of images and objects placed on pages can be controlled through the use of palettes. Palettes "float" above image document and text

document windows. You cannot open, save, or print palettes. Palettes can be recognized by their small title bar.

Text document windows contain text. Text document windows do not hold any other graphical objects. You can open, save, and print text document windows.

DigitalMicrograph can be extended to support acquisition devices through the use of plug-ins. Plug-ins are placed in a folder named "PlugIns".

DigitalMicrograph can also run simple programs (called scripts) which carry out automated tasks.

7.3.3. *The DigitalMicrograph Environment*

Before you can use DigitalMicrograph, you must install it on your computer according to the instructions contained in the Installing DigitalMicrograph manual.

To start DigitalMicrograph

You launch DigitalMicrograph as you would any other application; select DigitalMicrograph from the Start menu, or double click the DigitalMicrograph icon on the desktop.

By choosing commands from the menu bar, you can now create a new image window, open an existing one, or acquire one from an acquisition device.

When opening DigitalMicrograph (DM) you will see the following window

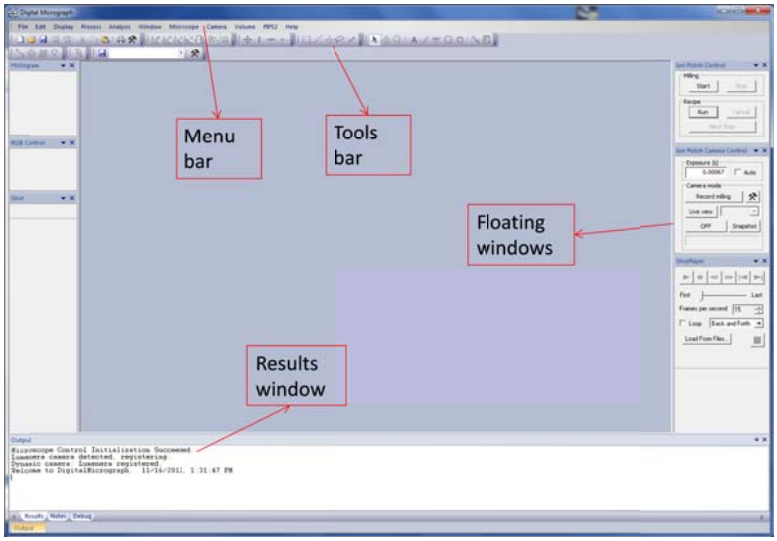

Figure 7-12 DM environment.

This screenshot shows DigitalMicrograph with only its basic plug-ins displayed.

Key areas

Menu bar

At the top is the menu bar containing the File, Edit, Display, Process, Analysis, Window, Microscope and Help menus. In these menus are all the controls for operating the application.

Tool bar

Under the menu bar is a toolbar.

Floating Windows

On the left hand side several Floating Windows are displayed. Floating Windows can also appear on the right hand side of the screen.

Result Window

At the bottom is the Results Window. This window is used to report results and updates of operations performed by DigitalMicrograph. This window may be hidden to increase the area available for image windows.

Image Windows

All images are displayed in Image Windows. They can be displayed anywhere in the application, and many images can be open at the same time.

To Exit DigitalMicrograph

You can exit DigitalMicrograph when you're finished with it. Choose Exit from the File menu, or hold down the Alt key and touch F4 to exit.

If any modified documents are open and haven't been saved, DigitalMicrograph asks whether you want to save the documents.

You can exit without saving any of the files by holding down Control and Alt keys and touch F4 to exit.

Opening an Image

You can either open a single frame image or a series of images (3-dimensional image or a stack).

Single image file: Go to File: Open… and brows to the location the file is saved, then select the file and touch open.

Series of images (stack): Go to File: Open Series… and brows to the location the files are saved, then select the first file in the series, and touch open. The dialog box shown below will appear. Here you can define the number of slices you want to open (Output images, Size). This option is very useful for

viewing all the images taken during a Record Milling session. The images may be conveniently viewed in series with the slice player.

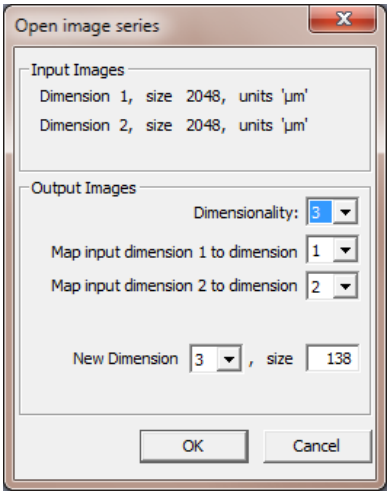

Figure 7-13 DM open image series.

Using Image Windows

DigitalMicrograph provides several ways to customize an image window. Among other things, you can magnify your view of the document, change the page size, and move the image and page around within the window.

To resize the window

Resize the window as you would resize any window on your operating system. To resize manually, click and drag an edge of the window.

If the image document is currently in image mode, its image will change from one integer multiple of its true size to another as you drag the window border. If you hold down the Alt key as you drag the window border then the image will resize to the largest size that fits in the window. If you hold down the Control key as you drag the window border then the image will not resize.

If the image document is currently being viewed in page mode, the page will always be sized to fit as large as possible within the window.

To resize around either the image or the page, click in the maximize box.

Re-sizing around an image or page will size the window so that the image or page fits exactly within the window at its current resolution.

To change printed page size or orientation

You can change the size or orientation of the page. Select Page Setup from the File menu. Enter the desired size and orientation in the dialog that is presented.

To change the magnification of the image document

You can change the size at which DigitalMicrograph displays the image or the page within the window. Click in the image window with the Zoom tool from the Standard Tools. The Zoom tool will display a "+" inside the magnifying glass to indicate you will be magnifying around the point at which you click. Hold down the Alt key to demagnify. The Magnify Page tool will display a "-" inside the magnifying glass to indicate you will be demagnifying.

Figure 7-14 DM standard tools.

You can also use the mouse wheel to zoom in and out around the location of the image where the mouse is located.

To move the image around within the window (page mode only)

You can move the image within the window. Click and drag the image within the image document with the Pointer tool.

To move the page around within the window

You can move the page within the window. Click and drag the page within the image document with the Move Page tool

Saving an Image Document

As you work, save early and often; don't wait until you finish working or until "later." This will prevent you from losing images due to power failures and other unexpected circumstances.

The File menu contains 4 items related to saving images: Save, Save As..., Save Numbered, and Save Display As... With the Save and the Save As items you can save the image data; with the Save Display As item you can save the screen rendering of your image document, and with Save Numbered you can do either depending on the preferences you have supplied. The same functions can also be accessed from the FileTools toolbar.

Figure 7-15 DM main menu.

The different image formats supported in DigitalMicrograph have different capabilities. This means that at some times you may be presented with more format choices than at other times. And sometimes the system has to ask for clarification on how to deal with limitations of a particular format.

The Gatan file format is the only format that can save all information properly at all times, and is the only choice when you are saving an image document that is displayed in page mode.

To save an image document in the Gatan file format

Choose Save from the File menu to save current image or click the Save button in the File Tools.

If this is the first time you've saved the file, DigitalMicrograph displays the Save As dialog box. Type in name for the file, choose the desired directory, choose "Gatan Format (*.dm4)", and click Save.

If the image document has already been saved once or was loaded from a file, DigitalMicrograph saves it to the same file, overwriting the previous version.

Choose Save As from the File menu to save to a new file. DigitalMicrograph displays the Save As dialog box. Type a name for the file, choose the desired directory, and click Save.

To save an image in TIFF format

If your image is not displayed in page mode you can save your image in other formats than Gatan Format. The most important of those formats is TIFF. However TIFF has certain limitations, and other applications implement different levels of TIFF format. For example Adobe PhotoShop does not cope well with negative values stored in images stored as signed 2-byte integer format - it assumes that they are large and positive. This is the format generated by Gatan's MSC cameras. When trying to save an MSC image with scale marker to TIFF the following will happen:

- Choose Save from the File menu to save current image or click the Save button in the File Tools.
- Choose TIFF from the "Save as type" drop list. At this time a warning dialog will appear. This dialog appears because your image contains an annotation (the scale marker) and TIFF cannot handle annotations

as separate objects. So you are given a choice to burn the annotation into the image data, or to ignore the annotation. Touch the OK button.
- Now DigitalMicrograph gives you a choice to convert to 16 bit unsigned so that you can more readily interpret the data in Adobe PhotoShop. Converting to 16 bit unsigned is done by adding 32768 to all image values. Touch the OK button.

The image will now be saved using the preferences you supplied. Note that you can lock in your choices on the warning dialogs by checking the appropriate check boxes, so that you do not have to go through this whole procedure each time.

To maintain compatibility with the largest number of other applications use the Save Display As function in DigitalMicrograph.

If you have problems with other applications, note that you can always load the TIFF image back into DigitalMicrograph and you will get all image data and meta data back. Then try to save in some other format to make the data appear properly in the other application

To save an image in TIFF format using Save Display As...

- Select the image you want to save and select Save Display As... from the file menu or click the Save Display button in the File Tools. The standard Save As dialog will be displayed and you can now choose from another list of file formats, including GIF and JPEG. In this example choose TIFF and pick a name and location for the image.
- Touch the Save button.
- Here you choose whether to save the image in the size displayed on the screen, or in its full resolution. And you can choose to include the annotations. Once again you can lock in on your choices so that you do not have to see this dialog each time you save an image.
- Touch the OK button.

The image will now be saved.

When you open this image in Adobe PhotoShop, you will see exactly the same thing as in DigitalMicrograph.

To save a series of images

DigitalMicrograph can save image documents in a series of files so that each time you save, the image document gets a new filename. Choose Save Numbered from the File menu or click the Save Numbered button in the File Tools.

You can set the directory in which to save the image documents, the name of the series, and the number in the series that you want to begin with. For

example, the first time you do this the image document will be saved with the name "Image Series.1." The next time you do it, the image will be saved with the name "Image Series.2."

Batch Convert

Using the Batch Convert... menu item images saved in Gatan Format can be converted to different data formats. This procedure will always save the data or display at the resolution of the source data, and it will include the annotations in the result. If the Gatan file contained an image document with more than one image, only one of the images is exported and a message to this effect is printed to the results window.

This feature can be useful for saving disk space by converting images to jpg.

- Choose Batch Convert from the File menu.
- A dialog with appear where you can enter the folder name by either typing it or using the Browse... button. If you want to convert all files in sub-folders of the selected folder as well, then check the Convert sub-folders button.
- Next choose to either save the image data in "Data Only" or MRC format, or save the image display in BMP, JPEG or TIFF format. MRC format does not support all data formats and if an image is encountered that cannot be converted to MRC a message will be printed to the results window.
- Touch the OK button. The procedure will now start converting all files in the selected folder and the following progress window is shown.
- Touch the Cancel button to abort the procedure.

Closing Image Documents

When you're finished using an image document, you can close it to remove the image from your computer's memory. When you're finished using DigitalMicrograph, you can exit it to end the current session. When you close image documents or exit DigitalMicrograph, you will be asked if you want to save any of the changes.

To close an image document

You can close image documents when you're finished with them to save on memory.

Choose Close from the File menu or click in the Close box.

Hold down the Alt key while closing the window to tell DigitalMicrograph not to present the dialog asking whether to save the file or not.

Hold down the Shift and Alt keys to close all windows and avoid being prompted to save each one.

Using Floating Windows

Floating windows are used to display information about and directly manipulate images and other objects within image documents.

You can arrange floating windows in a configuration that most suits your requirements. You can group sets of the floating windows together and you can "roll-up" a particular floating window in order to reduce the space it takes on the screen.

Some of the older DigitalMicrograph acquisition plug-ins will present a floating window that cannot be grouped with other floating windows.

DigitalMicrograph will remember the positions and groupings of all of your floating windows from session to session. If you exit DigitalMicrograph and launch it again later, the floating windows and groups will return to the same configuration.

To open a new floating window

DigitalMicrograph lists all of the floating windows in the Floating Windows menu. Select the desired floating window from the Floating Windows submenu under the Window menu.

DigitalMicrograph will add the new floating window to the group at the top-left of the main screen. If no group exists there, DigitalMicrograph will create a new group.

To move floating windows

Floating windows can be moved in the following ways:

Move an entire group of floating windows. Grab the group title bar and drag it to a new location.

Move a floating window above another within a group. Grab the title bar of a floating window and drag and drop it on the title bar of another to place it above the existing window.

Move a floating window below another within a group. Grab the title bar of a floating window and drag and drop it on the contents of another to place it below the existing window.

Move a floating window to another group. Grab the title bar of a floating window and drag it to the new group.

Move a floating window to a new group. Grab the title bar of a floating window and drop it somewhere where there is no other floating window.

To roll up or roll down a floating window

DigitalMicrograph allows you to roll up and roll down floating windows to save screen space and get unused controls out of your way. Click on the Twist Down control to roll up or roll down a floating window.

To close a floating window

You can close floating palettes completely.

Close an entire group of floating windows by clicking in the Close box of the group palette.

Close a specific floating window by dragging the floating window to a new group and close the new group.

Floating Windows Layout Manager

In many cases there are too many floating windows that you want to display simultaneously, and you are forced to open and close the relevant ones. To alleviate this problem there is a "Floating Windows Layout Manager". With this feature, you can easily save and retrieve different configurations of Floating Windows.

For example you can set up a layout called "Acquisition" that includes all panels you need during acquisition, and a layout called "Analysis" that includes post-processing and analysis related panels. All this functionality is located in the Windows menu under the "Layout Manager" sub-menu.

The "Save Layout As…" item allows you to save your current layout and give it a name.

When choosing "Manage Layouts…" a dialog is displayed that allows you to rename and delete existing layouts.

The items under the separator are the actual layouts you have saved. Choosing one of those items forces all floating windows to be redrawn as defined by that layout.

Image Displays

In order to display an image using Raster or Surface Plot display types, DigitalMicrograph must first map the image's data to the values 0 through 255. To display in gray-scale, the values 0 to 255 are then associated with different gray-scale values, e.g. 0 corresponds to black and 255 corresponds to white. To display in color, each value from 0 to 255 is associated with a color. In the rest of this section, gray-scale values are considered to be just a specific case of a color transformation. The section below describes these transformations.

DigitalMicrograph maps an image's original data values to a color or gray-scale value through a sequence of steps.

1. Determine the contrast limits of the image's data

DigitalMicrograph uses two parameters, the low- and high-contrast limits, to map the image's original data into a range suitable for display of the image. Pixels in the original image below the low-contrast limit are treated as if they were at the low-contrast limit and those above the high-contrast limit are treated as it they were at the high-contrast limit.

DigitalMicrograph can determine these contrast limits "automatically" by surveying the image, or "manually" using values entered by the user.

2. Transform each mapped data value into a color index.

The mapped image values are then transformed into a color index that indicates which color in the color table to use for displaying a particular pixel. The Contrast Transform lines in the Histogram depict how this transformation is performed. DigitalMicrograph supplies a number of standard contrast transform methods and allows you to build a custom one if you desire.

3. Display the pixel using the color table of the image.

DigitalMicrograph uses the color index to correlate each color in the color table with pixels with specific intensities in the image. DigitalMicrograph supplies a number of standard color tables, such as the gray-scale table, for use in images and allows you to build a custom one if you desire.

Histograms

DigitalMicrograph will automatically calculate the histogram of an image displayed using Raster or Surface Plot display types, and display it in the Histogram palette. The Histogram palette also displays the Contrast Transform lines and the color table in the Color Bar on the left of the palette.

The horizontal axis of the histogram represents data values with the left-most side corresponding to the low contrast limit and the right-most side corresponding to the high contrast limit. The vertical axis of the histogram represents the number of pixels with a particular data value.

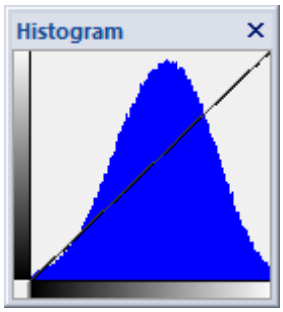

Figure 7-16 DM Histogram Window.

Using Image Regions of Interest

Many times, in order to process or analyze an image, you will need to select a region of interest (ROI) on an image. The region of interest indicates the part of the image you are interested in processing or analyzing.

Figure 7-17 DM ROI menu.

Methods of selecting regions of interest are specific to the type of image display the image is displayed as. The ROI Tools provides a set of tools for indicating regions of interest.

Rectangular ROI

You can make a rectangular region of interest on an image displayed with the Raster or RGB image display type. Use the Rectangle ROI tool to make a region of interest.

Making a region of interest will erase all previous regions of interest. To extend an existing set of regions of interest, hold down the Shift key while making the new region of interest.

Hold down the Shift key while making a rectangular region of interest to restrict it to be a square.

Hold down the Alt key while making a rectangular region of interest to restrict it to be a rectangle with a side that is a power of two (useful when performing FFTs).

The region of interest will appear as a red-dashed rectangle.

Line of interest

You can make a line of interest on an image displayed with the Raster or RGB image display type using the Line ROI tool to make a line of interest.

Making a line of interest will erase all previous regions of interests. To extend an existing set of regions of interest, hold down the Shift key while making the new region of interest.

Hold down the Shift key while drawing a line of interest to restrict it to 45° or 90°.

The region of interest will appear as a red-dashed line.

To specify a point of interest on an image with a Raster or RGB display

Point of interest

You can specify a point of interest on an image displayed with the Raster or RGB image display type using the Point ROI tool to make a point of interest.

Making a point of interest will erase all previous regions of interest. To extend an existing set of regions of interest, hold down the Shift key while making the new region of interest.

The region of interest will appear as a red cross-hair.

To specify a closed-loop region of interest on image with a Raster or RGB display

Closed-loop ROI

You can specify a closed-loop region of interest on an image displayed with the Raster or RGB image display type using the Closed-Loop tool to make a closed-loop region of interest.

Making a closed-loop region of interest will erase all previous regions of interest. To extend an existing set of regions of interest, hold down the Shift key while making the new region of interest.

The region of interest will appear as a red-dashed region.

Open-line ROI

To specify an open-line region of interest on an image with a Raster or RGB display use the Open-Line tool to make an open-line region of interest.

Making an open-line region of interest will erase all previous regions of interest. To extend an existing set of regions of interest, hold down the Shift key while making the new region of interest.

The region of interest will appear as a red-dashed line.

To adjust a region of interest on an image with a Raster or RGB display

Regions of interest are just additional objects attached to images. You can move them around as desired. You can also select, deselect, copy, drag, and delete them.

Rectangular and line regions of interest

Edit rectangle and line regions of interest by dragging their handles.

Hold down the Shift key while changing a rectangular region of interest to restrict it to be a square.

Hold down the Alt key while changing a rectangular region of interest to restrict it to be a rectangle with a side that is a power of two (useful when performing FFTs).

Hold down the Shift key while changing a line of interest to restrict it to 45° or 90°.

Using Line Profiles

You can use a line profile to sample an image along a line and display the sampled data in a line plot. The line plot will represent the data in the source image even if the source data changes or the line-profile position changes in the source image.

You can only create line profiles on images with a Raster display.

Use the Line Profile tool to create a line profile. A new Line Plot window will be created that represents data sampled from the source image beneath the line profile.

Adjusting the endpoints of a line profile

Adjust the endpoints by dragging the handles on the line profile or by double-clicking on the line profile, The Change Profile Info dialog will appear. Enter the desired coordinates in this dialog. The coordinates should be specified in uncalibrated units (i.e. pixels).

Adjusting the integration width of a line profile

You can adjust the integration width of a line profile by two methods: by selecting the line profile and pressing the '+' and '-' keys or by double-clicking on the line profile. The Change Profile Info dialog will appear. Enter the desired integration width in this dialog. The line profile will change to reflect the integration width.

Using the Slice Tool

Some applications, require the use of a three-dimensional image, rather than the standard two-dimensional image. DigitalMicrograph gives you a control to choose which layer (slice) of the three-dimensional dataset to display as the image.

- Select Floating Windows:Slice under the Window menu. This will open the Slice floating window.
- Select the three-dimensional image for which you want to change the slice. The Slice window will be disabled if the data is not three dimensional.
- Drag the top slider to adjust the slices displayed.
- Drag the bottom slider to adjust the number of slices to be integrated and displayed simultaneously.
- Check the Display Center check box to show all coordinates with respect to the center.

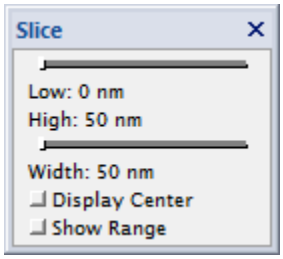

Figure 7-18 DM Slice tool.

Using the Slice Player

Use the slice player to automatically go through (first to last or back and forth) a 3-dimensional image (a stack) or a set of images saved by the Record Milling mode. To view images saved by Record Milling, they must first be opened using the Open Series option.

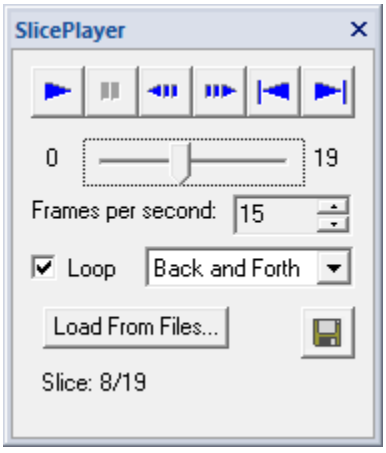

Figure 7-19 DM Slice player.

It is also possible to save a .avi file, by depressing the Disk button on this window. Brows to the location you like to save this file and touch Save, a dialog box will show up as shown below. Select Full frame (uncompressed) and touch ok.

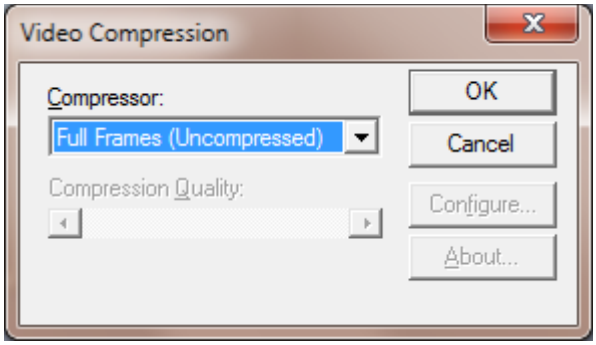

Figure 7-20 DM video compression.

PIPS II Milling Control

Milling can be started/paused/stopped at any time using this option on DM. This is specifically useful in cases where the PIPS computer is remotely accessed and the user is watching the milling process from outside the lab.

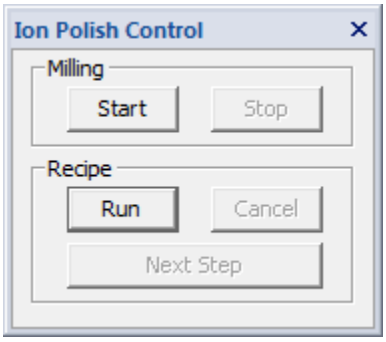

Figure 7-21 DM Ion Polish Control window.

To stop milling, select Stop. This is equivalent to selecting Stop on the PIPS II Milling page.

To start milling, select Start. This is equivalent to selecting Start on the PIPS II Milling page.

PIPS II Camera Control

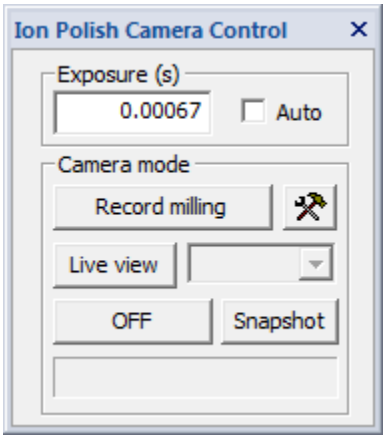

Figure 7-22 DM Ion Polish Camera Control window.

Exposure

Exposure time can be changed in two ways, either type in the exposure time in seconds and touch return or click in the exposure box and change the time by clicking on up and down arrow keys on the keyboard.

Alternatively, the Auto exposure box may be checked and DM will determine the exposure level automatically.

NOTE: Auto exposure mode will cause the live view to be somewhat not smooth. It is recommended to turn off Auto exposure once the exposure level has been found.

Camera mode

This part of the window is used to view the sample in live mode, take a snapshot or record images as the sample is being milled.

Record milling

Use this option for capturing a series of images during the milling process. When selected and the system starts polishing, the software automatically acquires images once every rotation. These images will be retained either in memory or on disk for examination or further processing. The shutter will be opened and the illuminators turned on just prior to image capture.

Gatan recommends using this mode during milling, and using Live View for setting exposure levels.

The frequency at which you want these images to be saved can be set using the toolbox menu. Clicking the toolbox brings up the PIPS Record Options window:

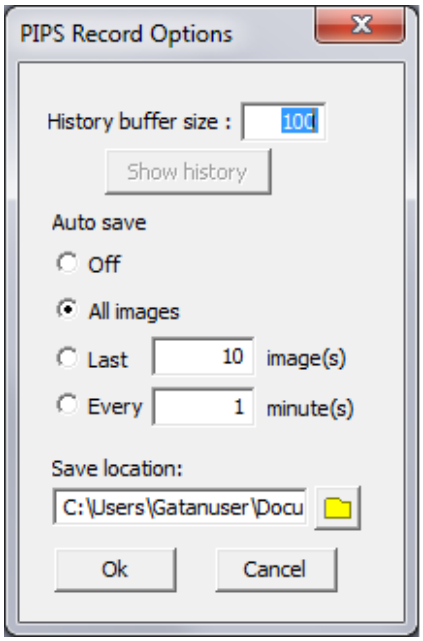

Figure 7-23 DM PIPS II record options window.

History buffer size: Defines the size of the stack that is displayed. This is limited by the amount of available memory on the computer. It is recommended that this be set to 19 or less images. Note that if the memory used by images is larger than the available memory, DM will stop acquiring images.

Auto save: Is used for saving the images that are acquired by the camera on the disk. The user has the option to i. turn the auto save **Off**, ii. to save **All images**, iii. to save the **Last X-images**, or iv. to save the images **Every X-minutes**.

Save location: Use this option to define where the images are saved. Files will automatically be named with a sample number and an image number embedded in the file name. If the stage is raised into the airlock, then a new sample number will be used the next time that the Record Milling mode is used. If milling is interrupted by using the Pause or Stop selection on the Milling page of the PIPS II, then the sample number remains the same when milling is restarted.

NOTE: The exposure time can be adjusted before the milling process is started and/or anytime during the process. In record milling mode it can take up to a full stage rotation before a change is observed, therefore, it may be preferable to switch to Live View, change the exposure time, then switch back to Record Milling mode.

NOTE: Recording can be stopped at any time by selecting Off.

Live View

This is used to watch the milling process live. The shutter is opened and the illuminators are turned on when this mode is selected. Viewing can be stopped at any time by selecting Off. Live View images are always acquired at VGA size (640x480).

Exposure time: can be changed in two ways, either type in the exposure time in seconds and touch return or click in the exposure box and change the time by clicking on up and down arrow keys on the keyboard.

Zoom: three zoom levels are available in the preview mode:

- Zoom 1x: shows the full camera frame, binned by 4
- Zoom 2x: shows the ½ center camera frame, binned by 2
- Zoom 3x: shows the ¼ center camera frame, binned by 1

Snap Shot

This is used to acquire a single full-frame image. Set the exposure time and touch Snapshot.

NOTE: Images in Record and Snap Shot mode will always be in Full frame mode, binned by 1.

NOTE: As shown in figure below, these options are also available on the PIPS GUI Camera Page.

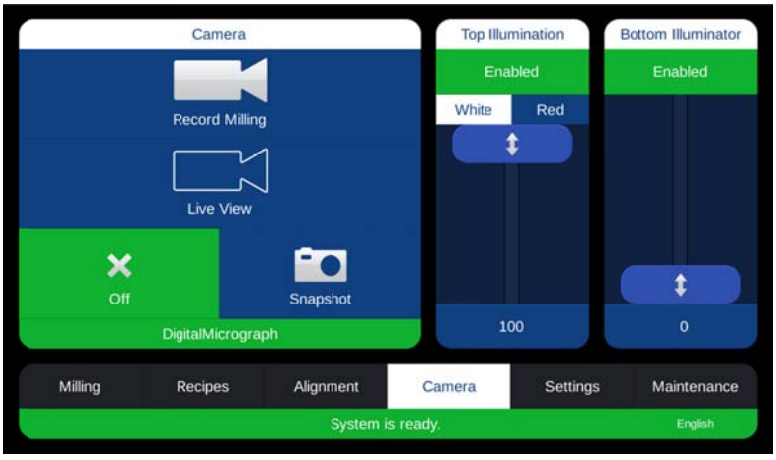

Figure 7-24 Camera page.

7.4. Motorized Gun Tilt

Certain PIPS II models include motorized guns. In these models, the gun tilt angles are set by the GUI or by a recipe. This option must be installed at the factory on a new PIPS II.

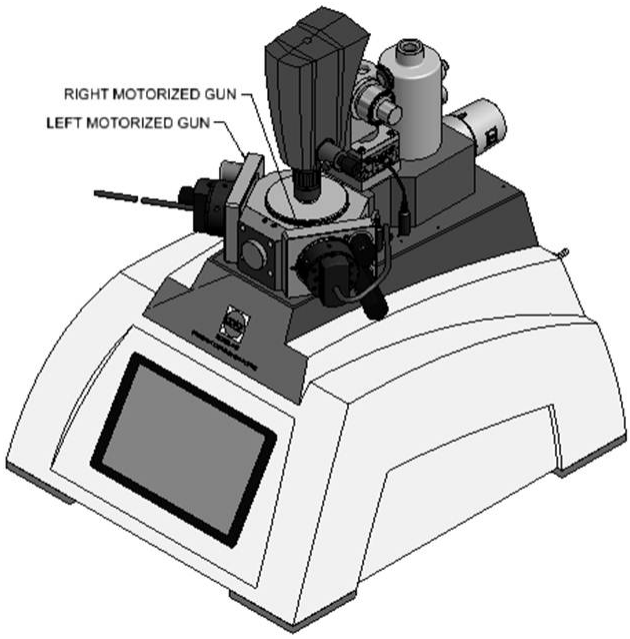

Figure 7-25 PIPS II with motorized gun tilt.

7.4.1. *Operation*

The gun angles may be set on the Milling page at any time by selecting the left and right tilt angle selections just above the chamber temperature readout. Positive tilt angles correspond to milling the top side of the sample, while negative tilt angles correspond to milling the bottom side of the sample. Milling angles may also be set by recipe.

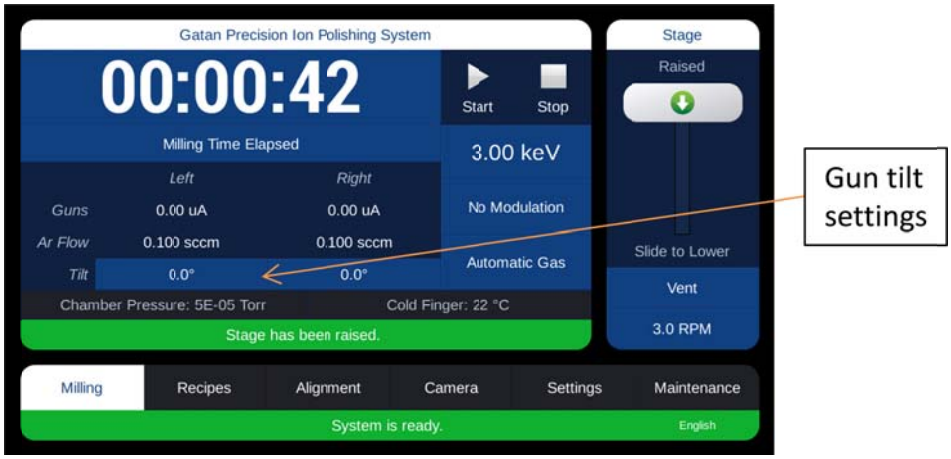

Figure 7-26 Gun tilt settings.

7.4.2. *Maintenance*

Each motorized gun assembly includes the following: large gear connected to the gun knob by 3 pins, motor connected to a small gear, potentiometer connected to a small gear, cable assembly. The motorized gun assemblies may be replaced if they fail. The left and right motorized gun assemblies are different, and must be replaced with the proper assembly.

Replacing the Motorized Gun Assemblies

1. **Shut down power to the PIPS II.** Unplug the power cable from the back of the system.

2. **Unplug the motorized gun assembly cable from the PIPS II.** This is a mini-din connector on the top of the system just behind the chamber.

3. **Remove the gun knob assembly.** Rotate the gun knob to the 10° Top position. Use a 3.0mm hex wrench to release the two screws from the gun knob and pull the knob from the gun housing.

4. **Remove the 3 screws from the front of the motorized gun assembly.**

5. **Carefully remove the motorized gun assembly from the chamber.** The motorized gun assembly should clear the gun housing assembly without need to vent the chamber and remove the gun housings. The backing plate may be removed and replaced, or simply reused. To replace, remove the 2 screws securing the backing plate to the chamber, remove the backing plate.

6. **Install the new motorized gun assembly.**

7. **Plug the cable into the connector on the chamber.**

8. **Turn on power to the system.** First replace the power cable.

9. **Calibrate the motorized gun assembly.**

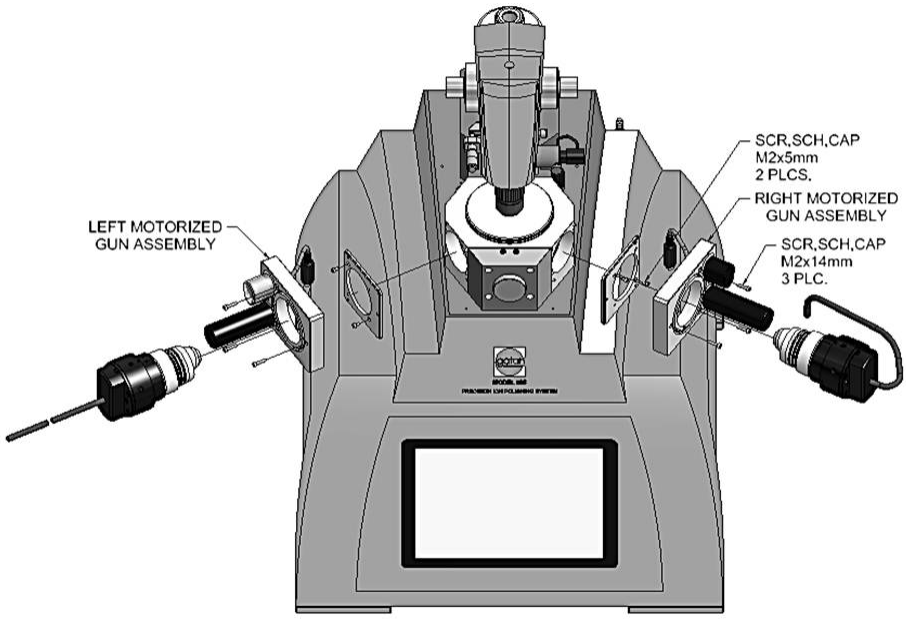

Figure 7-27 Motorized gun assemblies.

Calibrating the Motorized Gun Assembly

Motorized gun assemblies are calibrated at the factory and normally do not require re-calibration. In the event that a motorized gun assembly is replaced, it will need to be calibrated.

1. **Unplug the cable of the motorized gun assembly to be calibrated.**
2. **Manually rotate the knob to 10 deg top.**
3. **Plug the cable back in.**
4. **Touch Maintenance > Guns > Gun Tilt**
5. **Write down the dac reading displayed for that gun.**
6. **Unplug the cable, manually set the gun to -10 deg, plug in the cable.**
7. **Write down the dac reading displayed.**
8. **Touch Maintenance > Calibrations > Guns**

9. **Enter the dac readings for the appropriate settings.**

10. **Touch Maintenance > Guns > Gun Tilt**

11. **Verify that both guns can be set within the full range of -10 to +10 deg.**

Figure 7-28 Gun tilt maintenance screen.

Figure 7-29 Gun tilt calibration screen.

Gatan Hardware Product Warranty

1. WARRANTY. Gatan, Inc. ("Gatan") warrants to the purchaser ("Customer") that products and components manufactured by Gatan (collectively, "Products") shall be free of defects in materials and workmanship for one (1) year ("Warranty") commencing on the date of shipment from Gatan's factory ("Warranty Period"). Gatan warrants that the Products meet Gatan's published specifications at the time of shipment from its factory.

2. REPAIR OR REPLACEMENT.

2.1 During the Warranty Period, Gatan will, at its option, either repair or replace defective Products with conforming goods. Gatan will provide the parts (excluding all consumables, wear, and maintenance parts) and labor necessary to effectuate such repair or replacement of the defective Products. For imaging and analytical Products under warranty, travel of up to 100 miles from a Gatan authorized repair center (Pleasanton, CA; Warrendale, PA; Munich, Germany; Corby, UK; and Tokyo, Japan) will be free of charge. Travel expenses for warranty service beyond 100 miles will be charged for. Warranty repair of specimen holder and specimen preparation products will be done on a return to factory basis, with the shipping party responsible for its shipping costs. Gatan's liability under this Warranty shall be limited to repair or replacement of the defective Products. In no event shall Gatan be liable for the cost of procuring substitute goods.

2.2 Repair or replacement of Products or parts under this Warranty does not extend the original Warranty Period.

2.3 Items not manufactured by Gatan will be warranted by Gatan in accordance with the terms and conditions of the warranty received by Gatan from the original equipment manufacturer ("OEM"). Gatan makes no other warranty whatsoever concerning products or accessories manufactured by an OEM.

3. RETURNED GOODS AUTHORIZATION. The return of any Product, part, or assembly to Gatan for examination or repair shall have Gatan's prior approval, with the Customer requesting from Gatan a returned goods authorization ("RGA") approval. This RGA and the associated RGA number may be obtained through Gatan service or directly from Gatan's Warrendale facility at 724-776-5260 or by Fax at 724-776-3360. (1) If the Product is not under Warranty, to obtain an RGA, the Customer must provide a purchase order ("PO") agreeing to cover all charges associated with the repair. (2) If the item is under Warranty and the Customer is requesting an expedited exchange, as may be the case for a printed circuit board, a PO will also be required. A credit against this PO will be issued by Gatan upon receipt of the Product returned in accordance with the RGA instructions. The returned item should be shipped prepaid by the Customer with the RGA number clearly marked on the exterior of the shipping container and on the enclosed shipping documents. If the returned Product is under Warranty, the return transportation will be prepaid by Gatan. If the returned item is not under Warranty, return transportation will be charged to the Customer.

4. CUSTOMER RESPONSIBILITIES. The Customer bears the following responsibilities with regard to maintaining the Warranty. The Customer shall:

4.1 Perform the routine maintenance and cleaning procedures at the required intervals as specified in Gatan's operating manuals.

4.2 Use only Gatan replacement parts.

4.3 Use Gatan or Gatan-approved consumables.

4.4 Provide Gatan's authorized service representatives with access to the Products during normal Gatan working hours during the Warranty Period to perform service.

4.5 Provide adequate and safe working space around the Products for servicing by Gatan's authorized service representatives.

4.6 Provide access to, and use of, all information and facilities determined necessary by Gatan to service and/or maintain the Products. (Insofar as the information required for Gatan to service and/or maintain the Product may contain confidential or proprietary information, the Customer shall assume full responsibility for safe-guarding and protecting such information from wrongful use.)

4.7 Failure to comply with any of these Customer responsibilities will automatically void the Warranty provided herein.

5. WARRANTY LIMITATIONS. This Warranty does not cover:

5.1 Parts and accessories which are expendable or consumable in the normal operation of the Product.

5.2 Any loss, damage, and/or malfunction resulting from shipping, storage, accident (fire, flood, or similar catastrophes normally covered by insurance), abuse, alteration, misuse, neglect,

breakage, or abuse by Customer or Customer's employees or representatives.

5.3 Operation other than in accordance with correct operational procedures and environmental and electrical specifications.

5.4 Performance to specifications or safety of use (including X-ray emissions) if the Product is physically installed on, used in conjunction with, or used as part of a third party's equipment.

5.5 Performance to specifications or safety of use (including X-ray emissions) due to the design, operation, or fault of the third party's equipment in those special cases where Gatan specifically authorizes in writing the installation and/or use of Products with a third party's equipment.

5.6 Performance to specifications or safety of use (including X-ray emissions) if the Gatan Product is not installed by a Gatan service engineer or Gatan authorized service representative.

5.7 Modification of, or tampering with the Products or components.

5.8 Improper or inadequate care, maintenance, adjustment, or calibration of Products by the Customer or Customer's employees or representatives.

5.9 Contamination or leaks induced by actions of Customer or Customer's employees or representatives.

5.10 Any loss, damage, and/or malfunction resulting from use of software, hardware, or interfaces supplied by Customer or Customer's employees or representatives or consumables other than those specified by Gatan.

6. WARRANTY EXCLUSIONS. In the course of normal use and maintenance, certain parts have finite lifetimes. For this reason, the consumables, wear, and maintenance parts as specified in Gatan's operating manuals carry a ninety (90) day Warranty unless otherwise specified.

7. POST-WARRANTY PERIOD SUPPORT AND PRODUCT OBSOLESCENCE. Upon expiration of the Warranty Period, Gatan will provide service support for Gatan manufactured Products at Gatan's service labor rates and parts pricing in effect at the time of the service support. Gatan will continue to provide billable service support for a period of three (3) years after discontinuance of a Product by Gatan. After this three (3) year period, service support will be offered at the sole discretion of Gatan. Gatan warrants, for a period of ninety (90) days, that the replacement parts or Products used by Gatan during such post-warranty services will be free of defects in materials and workmanship.

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8. LIMITED MEDIA WARRANTY. Gatan warrants the media upon which this Software is recorded to be free from defects in material and workmanship under normal use for a period of one (1) year from the date of shipment. In the event that any media proves to be defective during the Warranty Period, your remedy shall be to return the physical media to Gatan for replacement. Should Gatan be unable to replace the media within a reasonable

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