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MODEL 600 DUOMILL INSTRUCTION MANUAL

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About this Guide

This *DuoMill User's Guide* is written to provide procedure for the installation of the unit, instruction on basic operations of the unit, and procedures for routine maintenance and servicing.

The Guide provides the following comments:

Warning: Used when failure to observe the instruction could result in per-

sonal injury.

Caution: Used when failure to comply with the instruction could damage

the equipment.

Note: Used to highlight advice directed at getting the best perfor-

mance from the equipment.

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1. GENERAL DESCRIPTION

The Models 600DDM/600CDDM DuoMill™ are instruments that can simultaneously ion mill two independent specimens for transmission electron microscopy (TEM). The DuoMills are protected by US patent 4272682. This Guide pertains to both DuoMill models.

Each DuoMill contains four subassemblies:

- Main Work Chamber
- · Gas control system
- Vacuum system
- Electrical system

1.1 Main Work Chamber

The Work Chamber contains all the facilities for independently ion milling two specimens. A molecular drag pump (MDP), backed by a four-stage diaphragm pump, is suspended below the center of the Chamber and pumps both the right and left milling stations (see Figure 1.1). Each station is equipped with a pair of ion guns (Octoguns[™]) mounted on a high precision, rotatable stage. The stage centers the ion beams accurately on the specimen over the full ±40° range of sputtering angles and also provides the necessary ductwork for feeding the ionizing gas independently to the two Octoguns. A four-position, high-voltage switch enables the operator to energize either one or both Octoguns.

A Whisperlok™ assembly in each milling station provides specimen rotation about the vertical axis and a pneumatically driven Airlock system for fast specimen exchange without affecting the milling conditions at the twin milling station. The Whisperlok also provides transmission illumination for specimen viewing and a window for close specimen viewing. A bellows-activated safety switch is mounted at the back of each milling station to sense the vacuum in the Airlock. This switch prevents the Whisperlok piston from being lowered if the Airlock has not been evacuated. A port is provided at the center of the Chamber for a liquid nitrogen trap (optional). A cold-cathode gauge tube, mounted at the center rear of the Chamber monitors the vacuum at the top of the pump.

1.2 Gas Control System

Two parallel gas circuits are fed from an inlet Tee located at the rear of the cabinet. The inlet gas pressure must be regulated to 8 psig (0.55 bar) to operate the two pneumatic Whisperlok mechanisms; this pressure is then further reduced to 3 psig (0.2 bar) by two miniature regulators positioned at the entrance to each pair of Octoguns. These regulators prevent interaction between the two ion-milling stations when their gas supplies are turned on and off. Four precision needle valves and four 2-way solenoid valves are provided to regulate the gas flow to each Octogun. The gas supply for the two Whisperloks is controlled by two 3-way solenoid valves that pressurize the Whisperlok pistons in the energized state and vent them to atmosphere when de-energized (see Figure 1.2).

1.3 Vacuum System

Vacuum is maintained in the DuoMill by a 260 l/s MDP, backed by a 33.3 l/min roughing pump (see Figure 1.3). The ultimate pressure in the main Chamber with no guns operating is typically better than 1 x 10⁻⁶ torr. The diaphragm pump evacuates the Airlock through manually-operated, spring-loaded evacuation valves in series with flow-restricting orifices. Each Airlock is connected to a bellows-type vacuum sensing switch that prevents the Whisperlok piston from being lowered into the main vacuum system if the Airlock is not evacuated. Each DuoMill model is provided with an automatic vent valve to vent the chamber during shutdown. In the event of a power failure, all 2-way solenoid valves controlling gas flow to the ion guns will close down. However, the 3-way solenoid valves for the Whisperlok will open to allow the specimen-holder pistons to rise automatically into the Airlock. In the Model 600DDM, a power failure will also cause the MDP vent valve to open and vent both the MDP and the Work Chamber. A manual vent valve is also provided for the Work Chamber and is located on the right rear wall of the main chamber under the cabinet lid.

1.4 Electrical System

The DuoMill is designed to run on 115 V 60 Hz, 230 V 50 Hz, or 220 V 60 Hz. The DuoMill can be adapted to operate at other voltages, e.g., 100 V or 200 V, by installing an appropriate transformer in the cabinet base. The electrical system consists of the two basic circuits shown in Figures 1.4a and 1.4b. The first circuit controls all the vacuum components and is operated by Switch A (right vertical panel). The second circuit controls the ion-beam production and is protected by two vacuum-sensing switches and a specimen height-sensing switch. This second circuit is duplicated in parallel so that each of the two ion-milling stations can be operated independently. The power input to both ion-beam circuits is controlled by the output of the vacuum control circuit so that ion-beam production will stop if there is a vacuum malfunction. Figure 1.4c shows the complete system wiring diagram.

Each Whisperlok contains a specimen height-sensing switch that automatically shuts off power to the high-voltage supply and gas to the ion guns if the specimen is raised into the Airlock. The high-voltage supply to each pair of ion guns is controlled by two potentiometers mounted on the sloping front panels. An orange MDP Ready lamp is provided on the right vertical panel and is lit when the MDP is rotating at the correct speed. The high-voltage supplies will not turn on if this lamp is not illuminated.

The ion-beam circuits can be placed under the control of a Dual Laser AutoTerminator that senses a perforation in the specimen and automatically shuts off the ion beam to prevent further milling. The AutoTerminator socket is shown in Figure 1.4b. Briefly, Terminals 3 and 4 are connected to a light-activated switch that opens when a hole forms in the specimen, thus cutting power to the high-voltage supply. Terminals 1 and 2 are connected to the light-sensing photoresistor mounted at the base of the Whisperlok. See Figure 6.1a and Section 6.1 for details.

WARNING: Always disconnect the power cord to the DuoMill and its accessories before opening any of the covers protecting electrical components. The ion guns and laser power supplies generate high voltages that are particularly hazardous.

2. INTRODUCTION TO ION MILLING

2.1 Factors Influencing Sputtering Rates

The primary factors influencing the sputtering rate are:

- The flux and velocity of the incident particles, i.e., ions and neutrals.
- The angle of incidence of the sputtering beam.
- The relative masses of the specimen atoms and incident particles.
- The binding energy of the specimen atoms.

Secondary factors include the specimen temperature, the concentration and chemical nature of any background gases, the crystallinity of the specimen, any chemical interactions occurring between the incident particles and the specimen, and specimen contamination.

Incident Flux

Within the range of flux levels normally used, the sputtering rate is directly proportional to the incident flux of particles of a given energy striking the target. The rate also increases almost linearly with increasing incident energy up to ~ 10 kV. Above this energy, the rate levels off and then actually decreases above ~100 kV as implantation occurs, i.e., most of the energy of the incident particles is deposited too far below the specimen surface to contribute to the sputtering process. It can be seen from Figure 2.1a that the sputtering rate of the Octoguns actually increases greater than linearly with increasing kV because increasing the kV not only increases the energy of the particles but also focuses the beam to a higher particle density on the specimen.

Angle of Incidence

In conventional ion milling, the sputtering rate is roughly inversely proportional to the mean free path of an incident atom in the target and varies widely from one material to another. Incident particles undergo elastic collisions that develop into cascades and sputtering occurs when the energy of an atom in a cascade reaching the specimen surface is greater than the surface binding energy. The number of energetic cascades reaching the surface increases as the angle of incidence between the specimen surface and the incident beam decreases. However, the momentum transferred to the specimen correspondingly decreases and hence there is an optimum angle for maximum sputtering, which typically is in the range 20-25° (see Figure 2.1b).

The angular dependence of milling rates has the undesirable effect of producing surface structuring that can be reduced (but not entirely eliminated) by rotating the specimen during ion milling. In the DuoMill, the specimen rotation axis is fixed in the vertical orientation and tilting the ion guns controls the angle of the incident beam. This design also enables milling to be performed at very shallow angles, which is important when it is necessary to minimize the depth of radiation damage.

Mass Ratio

The momentum transferred to the target (and hence also the sputtering rate) varies with the mass ratio of the incident and target atoms. The sputtering rate is at maximum when the masses of the incident and target atoms are equal and decreases as their differences in mass increases. In practice, some increase in milling rate can be achieved for certain specimens by using gases other than argon in the ion guns. In general, however, the gains obtained do not usually justify the inconvenience and cost of stocking the special gases.

Binding Energy

A background pressure of electronegative gases, such as oxygen, generally increases the yield of sputtered ions from clean surfaces. However, except for reactive materials, the background gas has very little influence on the total sputtering rate of ions plus neutrals. Most atoms are sputtered as neutrals, not ions, and, for a clean metallic surface, the ejected neutrals outnumber the ejected ions by a factor of 10⁴. In some cases (for example aluminum), a reactive gas can significantly reduce the sputtering rate by forming chemisorbed layers that increase the binding energy of surface atoms. It is important in such cases to minimize the amount of oxygen and water vapor introduced into the vacuum system during specimen exchange by using airlocks or by backfilling the Work Chamber with dry nitrogen. A liquid-nitrogen cold trap should also be used to reduce the partial pressure of water vapor and minimize hydrocarbon contamination.

2.2 Ion Current and Specimen Thinning Rate

The sputtering performance of commercially available ion mills is often related to the so-called ion current picked up either by the specimen or by a removable current sensor (see Figure 2.2a). This practice is unfortunate because it not only neglects to take into consideration the sputtering power of the energetic neutrals, but it also fails to recognize that the current actually measured is not the true ion current.

Particles striking a target from an ion gun eject electrons and secondary ions and, hence, produce a secondary current that must be separated from the total probe current if the true ion current is to be measured. This measurement can be performed by surrounding the target with a conductor (Faraday cage) that collects the secondary current and returns it to the target as illustrated in Figure 2.2b. With this arrangement, the net current measured is the true ion current. The inclusion of a Faraday cage around a specimen in an ion mill is very restrictive and, for this reason, the continuous monitoring arrangement shown in the last example of Figure 2.2a has been adopted in the DuoMill.

The current measured by this arrangement is termed the 'specimen current' and it serves mainly to give a quick confirmation that the ion beams are on and are striking the target. The monitor also gives some idea of the degree of contamination of the vacuum system and ion guns with water vapor, etc., since such contamination will produce an unusually high specimen current for a given gun voltage and gun current. A graph showing typical specimen current values for various beam angles in the DuoMill is shown in Figure 2.1b.

The proportion of ions to neutrals produced by the Octoguns in the DuoMill has been measured using deflector plates to separate the ions from the neutrals before they strike the current sensor. It was found that at 5 kV almost 99% of the Octoguns output is in the form of energetic neutrals. Even at 10 kV, only about 10% ions are present. The DuoMill could therefore be called a fast atom beam mill (FAB Mill) rather than an ion mill if it is considered important to emphasize the nature of the particles at the target rather than at the source.

Irrespective of whether the particles are ions or neutrals, it is the milling rate that is of primary importance in rating the performance of an ion-milling machine. In order to check that the DuoMill is meeting its milling rate specification, the time necessary to perforate a 50- μ thick sample of 99.99% pure copper is measured at 6 kV and 1 mA total gun current. This time must be 2.5 hr or less for the ion gun pair to be accepted for shipment. A total gun current of 1 mA (0.5 mA per gun) is shown in Figure 2.1a. The ion guns used for the tests had been operated for approximately 50 hr and consequently the cathode holes were enlarged. New cathode holes produce more sharply focused beams and the thinning rates would be about 50% higher than that shown in Figure 2.1a. Electron microscope examinations indicate that specimens prepared at lower sputtering rates and better vacuum conditions exhibited less surface structuring and tended to contain larger thin areas. Better specimens are also obtained if the milling time is minimized by mechanically thinning the specimen as much as possible prior to milling.

3. INSTALLATION PROCEDURE

Gatan recommends that you read the following sections carefully and organize User Instruction, Servicing, and a Logbook before releasing the DuoMill for general use.

3.1 Site Preparation

The Model 600DDM(CDDM) requires a well lit, cool, clean room, with electrical power source of approximately 1 kW. It is helpful to locate the instrument close to a workbench where spares and accessories can be stored and servicing can be done efficiently. The DuoMill can be moved easily to the installation site on its large-diameter, swiveling, rubber wheels. The unit weighs approximately 400 lb (180 kg). Units shipped to sites in USA, UK, France, Germany, Sweden, Norway, Netherlands, and Austria are equipped with suitable power plugs (see Figure 3.1a). In other countries, a power plug has to be supplied by the customer and should be connected according to one of the following color codes:

Brown = Live Black = Live

Blue = Neutral or White = Neutral

Green/Yellow = Ground Green = Ground

A cylinder of argon (Grade 4.8) with a regulator will be required. The outlet should be regulated to a pressure of 8 psig (0.55 bar) and be coupled with a 1/8" internal diameter (3 mm) plastic hose to the tapered hose fitting at the rear of the DuoMill. Allow approximately 3 ft (1 m) excess length of hose so that the instrument can be pulled away from the wall for easy cleaning and servicing.

3.2 Unpacking Instructions

- a. Lift off the accessory box from the top of the cabinet and remove accessories.
- b. Cut and remove the vertical banding holding down the DuoMill.
- c. Lift off the corrugated packing from the top of the DuoMill and at each end of the vacuum chamber.
- d. Unbolt and remove the wooden hold-down bar extending across the front of the base.
- e. Remove the wooden lifting lever and the plywood spacers located on either side of the base.
- f. Use the lifting lever to pry up each side of the DuoMill enough to slide one spacer under each pair of wheels.
- g. Remove the corrugated pad that supports the underside of the cabinet.
- h. The DuoMill can now be rolled off the wooden base onto the floor of the installation site.
- Open the DuoMill doors and remove the upper portion of the corrugated block beneath the MDP.
- j. Remove the corrugated spacer from the left side.
- k. Unlock the cabinet lid and remove the protective packing from around the thermocouple gauge (right side). The key to the lid is in an envelope attached to the front of the DuoMill vacuum chamber.

3.3 Installation

- a. The DuoMill is shipped under vacuum.
- b. Remove the pipe plug from the automatic vent valve located at the back of the MDP and install the small vent filter provided. This plug is used for shipping purposes only and can be discarded.
- c. Connect the argon cylinder fitted with a regulator to the gas input port located at the center of the cabinet back. See Figure 3.1b.
- d. Turn on the main valve of the gas cylinder adjusting the outlet pressure to 8.0 psig (0.55 bar).
- e. Check for argon leaks by closing the main valve of the gas cylinder and observing the high-pressure regulator gauge. No decrease in the pressure reading should be visible during a period of 2 min after closing the main valve.
- f. Plug the DuoMill into a grounded AC power outlet having the voltage designated on the Gatan label located on the right side of the cabinet. In units shipped without a power plug, it is important to wire the power input according to one of the color codes given in Section 3.1.
- g. Proceed to the detailed operating instructions, Section 5.

4. OPERATING INSTRUCTIONS (short form)

4.1 Pre-Start-Up Check List

- a. Switch A should be off, i.e., depress lower face of switch.
- b. Raise the main circuit breaker (right side of cabinet).
- c. Check that the argon pressure is set at 8 psig (0.55 bar).
- d. Set the red pointer of the thermocouple gauge to the far right position.
- e. Check that both Airlock capsules are in place.
- f. Depress the lower face of both High Voltage switches, i.e., to Off position.
- g. Rotate both High Voltage Control knobs fully counterclockwise.
- h. Rotate both Current Limit knobs fully counterclockwise.
- i. Rotate both High Voltage Selector knobs on the chamber to their neutral positions (unmarked position on selector).

4.2 Start-Up Procedure

- a. Depress the upper part of Switch A (diaphragm and molecular drag pumps will start).
- b. Observe MDP Ready light come on and Penning gauge (left side) start to read.
- c. Allow chamber pressure to drop to 10⁻⁵ torr before proceeding.

4.3 Specimen Loading

- a. Depress Raise on the Airlock rocker switch.
- b. Depress the Vent button to vent the Airlock. Remove the Airlock capsule.
- c. Unscrew the specimen holder.
- d. Load a specimen and screw the holder back onto the Whisperlok piston.
- e. Place the sputter shield directly on top of the specimen platform, being careful to orient the shield as shown in Figure 5.3. Replace the Airlock capsule.
- f. Depress the Vac button intermittently to evacuate the Airlock.
- g. Depress Lower on the Airlock rocker switch.

4.4 Switching on the Ion Beams - Current Limit Mode (not recommended for low-energy guns)

- a. Remove the gun mount rotation tool from its holder on the inside of the left-hand door of the cabinet.
- b. Insert the tool into its socket on the circumference of the gun mount and rotate the gun mount to the appropriate sputtering angle (usually 15°).
- c. Check that the chamber pressure is better than 10⁻⁵ torr before depressing the upper face of the High Voltage switch (light comes on).
- d. Set the Process Timer to the desired number of milling hours and minutes. Depress Start/Stop button. The Timer panel light must be blinking for the HV output to be enabled.
- e. Turn High Voltage Selector knob on milling chamber to White position and turn on Gas Control switch for White gun.
- f. Turn High Voltage knob fully clockwise (4 kV maximum if using low-energy guns).
- g. Turn Current Limit knob clockwise to desired current (usually 0.5 mA).
- h. Adjust the Gas Flow knob (needle valve) for the White gun to obtain the desired operating voltage (the needle valve controls the gas pressure in the ion gun and the gas pressure controls the voltage in the ionization chamber).
- i. Repeat Steps e through h for Red gun.
- j. Turn High Voltage Selector knob on milling chamber to White/Red position.
- k. Increase Current Limit knob clockwise for each gun to give total gun current of 1.0 mA (0.5 mA per gun).

4.5 Switching on the Ion Beams - Voltage Regulated Mode

- Select desired sputtering angle (see Steps 4.4a and b) and check that the chamber pressure is better than 10⁻⁵ torr
- b. Depress upper face of High Voltage switch (light comes on) and set the Process Timer. Depress Start/Stop button.
- c. Turn High Voltage Selector knob on milling chamber to White position.
- d. Turn on Gas Control switch to White gun.

- e. Turn Current Limit knob fully clockwise.
- f. Turn High Voltage knob clockwise to desired voltage (not above 4 kV for low-energy guns).
- g. Adjust Gas Flow knob (needle valve) to give desired current (usually 0.5 mA).
- h. Turn High Voltage Selector knob to Red position.
- i. Turn on Gas Control switch for Red gun.
- j. Adjust Gas Flow knob (needle valve) to desired current (0.5 mA).
- k. Turn High Voltage Selector to White/ Red position.

4.6 Switching on the Ion Beams - Voltage and Current Regulated Mode

This mode of operation is only possible using the Model 600.21000 Electronic Gas-flow Control (EGC) option. For detailed operating instructions see Section 6.7.

4.7 Switching off the Ion Beams

- a. Bring specimen into Airlock by depressing Raise on the Airlock switch or,
- b. Switch high voltage off, or,
- c. Press Process Timer Start/Stop button.

CAUTION: Do not use the Gas Flow knobs (needle valves) to shut off the gas flow to the guns. Use the Gas Control switches.

4.8 Specimen Viewing

When bringing a specimen into the Airlock for close viewing, it is not necessary to interfere with any of the beam controls. Simply depress Raise on the Airlock switch and following inspection (under vacuum) depress Lower on the switch. The ion beams will turn off and come on automatically, retaining their original settings.

NOTE: During specimen viewing, the Process Timer continues to count down the time.

A microscope with illuminator is available as an optional accessory for detailed inspection of the specimens. Holes in specimens are best examined using the transmission illuminators, which are operated by the red push-buttons located at the front left and right corner of the DuoMill cabinet. These illuminators are not available on the liquid nitrogen cooled Whisperloks.

4.9 Cleaning Specimen Viewing Windows

Each time the specimen is exchanged, the window in the capsule should be popped out of its O-ring retainer, turned over and pressed back in place. The sputter-coated surface of the glass window is now outside the vacuum and can be cleaned at any time. Use a non-abrasive household cleaner to remove deposits. Make it a habit to clean the window as frequently as possible. A long-neglected window is difficult to clean.

5. DETAILED OPERATING INSTRUCTIONS

5.1 Pre-Start-Up Check List

- a. Both High Voltage switches (sloping front panels) should be off. (Depress bottom part of rocker switches).
- b. Both Voltage Control knobs (sloping front panels) should be turned fully counterclockwise.
- c. Rotate both High Voltage Selector knobs (front of the main chamber) to their neutral positions (unmarked position on knob).
- d. Both Airlock capsules should be in place on top of the Work Chamber.
- e. The red pointer of the thermocouple gauge should be set to the far right position.
- f. The argon pressure should be set at 8 psig (0.55 bar).
- g. Switch A (right vertical panel) should be off.
- h. The circuit breaker (right side of cabinet) should be on (raise breaker arm).

5.2 Start-Up Procedure

- a. Depress the upper part of Switch A (diaphragm and turbo pumps will start).
- b. Observe MDP Ready light come on and Penning gauge start to read.
- c. Allow chamber pressure to drop to 10⁻⁵ torr before proceeding.

5.3 Specimen Loading and Sputter Shield

- a. Depress Raise on the Airlock switch to raise the specimen platform into the Airlock.
- b. Depress the Vent button to bring the Airlock to atmospheric pressure. Any increase in foreline pressure when the Vent button is depressed indicates a leak between the Airlock and the main Work Chamber. Investigate this fault before proceeding further.

CAUTION: Never depress the Vent button when the Airlock piston is lowered.

- c. Remove the Airlock capsule.
- d. Unscrew the specimen platform.

CAUTION: Never push down on the Airlock piston as this may vent the main Work Chamber to atmosphere.

- e. Insert a specimen into the recessed hole of the specimen platform. The gentle operation of the Whisperlok mechanism eliminates the need for clamping the specimen discs. However, unusual specimen shapes, such as wires or ribbons, should be fixed to the specimen holder using two small (0.2-mm diameter) beads of silver paint. Specimen clamping plates are also available for curled specimens that need to be held flat during ion milling. It is recommended that specimen loading into the holder and specimen examinations are done with a 20X laboratory stereomicroscope.
- f. Replace the specimen platform onto the Airlock piston. Be sure that the specimen holder is completely screwed down, otherwise the specimen will not be centered with respect to the ion beams and the holder may hit the specimen-viewing window when the Airlock capsule is replaced.
- g. Place the sputter shield directly on top of the specimen platform, being careful to orient the shield as shown in Figure 5.3. Replace the Airlock capsule.
- h. Depress the Vac button intermittently to evacuate the Airlock.
- i. Depress Lower on the Airlock switch to lower the specimen to its normal working position. The piston should descend relatively slowly under the 8 psig regulated pressure. If the pressure is too high, the piston will descend too quickly and the specimen may be disturbed in its mount. Excessive pressure may also squeeze grease from the Airlock piston O-ring, which may result in sticking on the raise cycle.
 - The sputter shield will come to a stop at the Airlock piston O-ring. If you have oriented the shield correctly you will be able to visually check the operation of the Octoguns by looking through the two off-center holes.

NOTE: The sputter shield is designed to reduce the build-up of sputtered material onto the Airlock capsule window and the Airlock O-ring seal.

The advantages of the sputter shield are as follows:

- Improves the accuracy of the laser AutoTerminator by minimizing the change of transmitted light intensity during specimen thinning.
- Eliminates sticking and leaking at the Airlock piston seal caused by sputter deposits.
- Reduces considerably the frequency of maintenance of the capsule window and Airlock piston seal.

5.4 Milling Modes

The DuoMill high-voltage supplies can be operated in any of three modes:

- Current limiting
- Voltage regulated
- Combined voltage and current regulated

All cold-cathode type ion guns require periodic adjustment of the inlet gas pressure to maintain a constant ion current due to temperature changes, out-gassing, and erosion of the cathode aperture. Since these adjustments can be bothersome, Gatan has developed an Electronic Gas-flow Control (EGC) that constantly monitors the gun current and automatically adjusts the gas supply to obtain a constant, pre-selected specimen current. Thus, the DuoMill now becomes both current and voltage stabilized, ensuring accurate reproducible milling conditions without constant attention.

If the DuoMill is not equipped with an EGC, the current-limiting mode of operation is generally preferred. In this mode, the voltage is automatically adjusted by a feedback circuit in the high-voltage power pack to maintain the desired gun current. An alternative voltage-regulated mode is used for specimens that are subject to ion damage and can only be successfully thinned below a particular voltage.

Current-Limiting Mode (not recommended for low-energy guns)

To use this mode, proceed as follows:

- a. Remove the gun mount rotation tool from its holder on the inside of the left-hand door of the cabinet.
- b. Insert the tool into its socket on the circumference of the gun mount and rotate the gun mount to the appropriate sputtering angle (usually 15°).
- c. Check that the chamber pressure is better than 10⁻⁵ torr before depressing the HV switch (light comes on).
- d. Set the Process Timer by pressing its Start/Stop button and depress the Up Arrow button to set the desired thinning time.
- e. Press the Process Timer Start/Stop button again; the blinking panel light indicates that the HV output is enabled and the Process Timer is counting down.

NOTE: The Process Timer panel light must be blinking for the HV output to be enabled.

To change the desired process time, press the Start/Stop button and depress the Up Arrow button to set the desired thinning time; press the Start/Stop button to reinitiate the milling and the timing. It may be useful to note that the Process Timer remembers its previous setting.

- f. Turn the High Voltage Selector knob on the milling chamber to the White position and switch on the White Gas Control valve.
- g. Turn Current Limit knob clockwise half a turn.
- h. Turn High Voltage knob clockwise and set to 1.0 kV above desired operating voltage (not above 4 kV for low energy guns)
- i. Adjust the White Gas Flow valve to obtain the desired operating voltage (this needle valve controls the gas pressure in the ion gun and the gas pressure controls the voltage in the ionization chamber).
- j. Repeat steps f through i for Red gun.
- k. Turn High Voltage Selector knob on milling chamber to White/Red position.
- Turn Current Limit knob clockwise to obtain the total gun current desired (usually 1.0mA, which means 0.5 mA per gun).

Voltage-Regulated Mode

To use this mode, proceed as follows:

- a. Select desired sputtering angle (usually 15°) and check that the chamber pressure is better than 10⁻⁵ torr.
- b. Depress HV switch (light on) and set the Process Timer.
- c. Turn High Voltage Selector switch on milling chamber to White position.
- d. Turn on Gas Control switch to White gun.
- e. Turn Current Limit knob fully clockwise.

- f. Turn High Voltage knob clockwise to desired voltage (not above 4 kV if using low-energy guns).
- g. Adjust Gas Flow knob to give desired current (usually 0.5 mA).
- h. Turn High Voltage Selector knob to Red position.
- i. Turn on Gas Control switch to Red gun.
- j. Adjust Gas Flow knob to obtain desired current (0.5 mA).
- k. Turn High Voltage Selector switch to White/Red position.

In this mode, the guns will require periodic slight adjustments to maintain the desired current. The procedure is to (1) turn High Voltage Selector knob to White and adjust gas flow to bring current back to original (usually 0.5 mA); (2) repeat with Red gun. (3) Turn High Voltage Selector to White/Red position.

Voltage- and Current-Regulated Mode

This mode of operation is only possible using the EGC option. For detailed operating instructions see Section 6.7.

5.5 Changing Cathode Hole Positions in the Octoguns

- a. Depress the High Voltage switch to the Off position.
- b. Rotate the Octoguns knob to align the marker precisely with the next higher number. To avoid confusion, always change the cathode holes on both guns simultaneously.

NOTE: If the marker is not precisely aligned, the beam will not hit the center of the specimen and the thinning rate will be reduced.

c. The reading on the total timer should be noted to monitor the life of the new cathode hole (typically 100 hr).

NOTE: To get the maximum life from the Octoguns, keep an accurate record of the life of each cathode hole (see attached sample chart, Figure 5.5, which can be copied for your use). Always change cathode holes in sequence (1 through 8) so that it is clear which ones are worn out.

5.6 Specimen Examination

- a. Depress Raise on the Airlock switch to raise the specimen into the Airlock. The high voltage will automatically switch off as the specimen rises.
- b. The specimen can now be examined closely through the viewing microscope.
- c. After viewing, the specimen can be returned to its normal working position. Depress Lower on the Airlock switch. The ion beams will switch on automatically when the specimen reaches its working position. (The guns take approximately 30 sec to attain their original operating conditions; 2 min with EGC.).

NOTE: While the specimen is raised, the Process Timer continues counting down.

A specimen-viewing light is available to observe the specimen in transmission mode in both the lowered and raised positions. The viewing lamp is located in the bottom of the Whisperlok assembly and operated by red push buttons located at the front left and right corner of the DuoMill chamber.

NOTE: The specimen-viewing lamp is not available on the cold style Whisperlok.

5.7 Specimen Removal

- a. Depress Raise on the Airlock switch to raise the specimen into the Airlock.
- b. Vent the Airlock by pressing the Vent button.

CAUTION: Do not vent the Airlock unless the Airlock piston has sealed the opening to the main Work Chamber.

c. Invert the window in the Airlock capsule so that the sputter-coated surface is outside the vacuum system for easy cleaning.

5.8 Shut Down and Venting Procedure

- a. Turn both Voltage Control knobs fully counterclockwise.
- b. Turn off both high-voltage supplies.

- c. Raise both specimen holders into their Airlocks.
- d. Rotate both High Voltage selector knobs to their neutral positions.
- e. Turn off Switch A if the DuoMill is not going to be used for more than a few days. Both the diaphragm pump and MDP will stop and the MDP will automatically vent.

6. OPERATION OF ACCESSORIES

6.1 Dual AutoTerminators

WARNING: Do not stare directly into the laser assembly. Always point the laser away from the eyes and close the shutter when the laser is not in use. The laser uses high voltages that can be lethal. Disconnect power before servicing the laser power supply. The laser tube must be electrically connected before turning on the Term switch.

6.1.1 Principle of Operation

The laser AutoTerminator consists of a control unit, a laser source, and a detector. The control unit is used to turn off the DuoMill high-voltage power supplies and the ionizing gas supplies to either the left or right milling station upon specimen perforation. The laser source is accurately aligned on its mounting plate over the Airlock so that the beam passes down through the center of the specimen and through a small aperture at the top of the Airlock piston. The beam then passes through a window and onto a photoresistor(detector) mounted at the base of the Whisperlok. Light received by the photoresistor produces a voltage reading (mV) that is displayed on the front panel of the control unit. When this voltage exceeds a set value (200 mV), the main relay in the control unit trips and switches off the ion-beam control (see Figure 1.4b). The actual light level corresponding to a 200 mV reading 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. An optical filter can be used to extend the sensitivity range of the AutoTerminator (see following section). A circuit diagram of the dual AutoTerminator control unit is shown in Figure 6.1a. The laser head assembly is shown in Figure 6.1b.

6.1.2 Installation and Laser Alignment Check

NOTE: The DuoMill must be under vacuum and the MDP switched on to operate the AutoTerminator.

WARNING: As a safety precaution, be sure that the laser shutter is closed (see Figure 6.1b).

General Setup

- a. Connect the laser assembly to one of the coaxial sockets at the rear of the laser control unit and plug the control unit into the appropriate socket (left or right) at the rear of the DuoMill cabinet.
- Switch on the control unit by depressing the top of the appropriate Term switch mounted on the left or right front vertical panels.
- c. Remove the specimen holder and if necessary, clean the specimen-viewing window.
- d. Lower the piston into the Work Chamber. This will actuate the Airlock micro-switch that feeds power to the laser control unit.
- e. Specimen Rotate switch to Off.

Standard Whisperlok

- a. Place the laser assembly over the appropriate Airlock port and open the shutter.
- b. Adjust the sensitivity control to 6.0 on the dial and observe the reading on the digital meter increase to 1400 ±100. An alarm sounds and the red Trip light is illuminated. The alarm is controlled by a timer and sounds for 60 seconds.
- c. If the meter reading does not show an increase or the 1400 level is not achieved, check that the laser assembly is properly seated on the Airlock port.
- d. If this does not correct the problem, remove the laser assembly from the Airlock port and point the assembly toward a nearby surface to verify that the beam is present.

Warning: Do Not Stare Directly Into The Laser Assembly To View The Beam.

If the beam is on, then either the Whisperlok aperture assembly in the piston rod is blocked (see Section 7.3) or the laser tube has been knocked out of alignment (see Section 6.1.5).

- e. Turn the sensitivity control fully counterclockwise (essentially turning the sensitivity control off). The digital reading on the meter should decrease to zero. Depress the Reset switch, which will turn on the green Operate light and turn off the audible alarm.
- f. Turn the sensitivity control slowly clockwise and verify that the Trip relay operates when the reading on the digital meter reaches 200 (this should correspond to about one revolution of the control knob).

- g. Adjust the sensitivity control to produce a reading of 150 on the digital meter. If this reading varies by more than 10% during one revolution of the specimen rotate motor, the laser requires alignment (see Section 6.1.5).
- h. Close the shutter of the laser assembly. Switch off the TERM switch, remove the assembly from the Airlock capsule and place it on top of the laser control unit.

Cold-Style Whisperlok

NOTE: The laser beam passes the detector only once per revolution in the Cold-Style Whisperlok (see Figure 6.2b).

- a. Place the laser assembly over the appropriate Airlock port and open the shutter.
- b. Turn On the Process Timer, the HV switch, and the Specimen Rotate switch.
- c. Adjust the sensitivity control to 6.0 on the dial. The reading on the digital meter slowly increases and decreases as the piston rotates. As the reading increases and passes through the preset trip point, an alarm sounds, the red Trip light is illuminated, and the piston rotation stops.
- d. Hold down the Reset button to permit the rotation to continue for several revolutions to verify that the readings are consistent.
- e. Release the Reset button when the highest reading is observed on the digital meter (1400 ±100) and switch Off the Rotate switch.
- f. If the meter reading does not show an increase or the 1400 level is not achieved, check that the laser assembly is properly seated on the Airlock port.
- g. If this does not correct the problem, remove the laser assembly from the Airlock port and point the assembly toward a nearby surface to verify that the beam is present.

Warning: Do Not Stare Directly Into The Laser Assembly To View The Beam.

If the beam is on, then the laser tube is out of alignment (see Section 6.1.5). If the digital meter reads 1400, proceed.

- h. Turn the sensitivity control fully counterclockwise (essentially turning the sensitivity control off). The digital reading on the meter should decrease to zero. Depress the Reset switch, which will turn on the green Operate light and turn off the audible alarm.
- i. Turn the sensitivity control slowly clockwise and verify that the Trip relay operates when the reading on the digital meter reaches 200 this should correspond to about one revolution of the control knob.
- j. Close the shutter of the laser assembly. Switch off the TERM switch, remove the assembly from the Airlock capsule and place it on top of the laser control unit.

6.1.3 Operating Procedure

The operating procedure used depends on whether the specimen is initially opaque or transparent/translucent. The specimen must be in the Work Chamber for the AutoTerminator to function.

Opaque Specimens

- a. Turn the sensitivity control fully clockwise and turn the appropriate Term switch on.
- b. Load the opaque specimen and lower it into the Work Chamber.
- c. Set the Process Timer to the maximum thinning time required.
- d. Switch on the ion guns and start thinning the specimen at the desired current, voltage, and beam angle.
- e. Place the laser assembly over the Airlock capsule (the reading on the digital meter should not change from zero).
- f. Open the laser shutter (see Figure 6.1b).
- g. When a hole forms and begins to grow in the specimen, the meter reading will gradually increase until termination occurs at a reading of about 200 mV.
- h. Before leaving the AutoTerminator for an extended run, check the following: (a) the green Operate light is on, (b) the laser shutter is open, (c) the Process Timer is set for a sufficient number of hours.

NOTE: If the red Trip light is illuminated, it can be reset to Operate simply by depressing the Reset button.

Transparent/Translucent Specimens

- a. Place the optical filter on the specimen capsule-viewing window (see Figure 6.1b).
- b. Determine the setting of the sensitivity control that just causes the Trip light to come on when there is no specimen in the specimen holder. This position marks the maximum allowable counterclockwise setting of the sensitivity control.
- c. Place the transparent/translucent specimen in the specimen holder and readjust the sensitivity control until the lamp trips. This marks the maximum clockwise setting of the sensitivity control.

NOTE: If the intensity oscillates during specimen rotation, then set the sensitivity to the maximum value.

- d. Set the sensitivity control halfway between the maximum clockwise and counterclockwise positions.
- e. Proceed as described in Steps c through h under Opaque Specimens above.

NOTE: If the AutoTerminator trips prematurely, adjust the sensitivity control further counterclockwise to bring the reading below 200.

6.1.4 Removing the Laser AutoTerminator

- a. Close the shutter on the laser assembly.
- b. Switch off the Term switch.
- c. Unplug the control unit from the rear of the cabinet.
- d. Remove the control unit and laser assembly from the DuoMill.
- e. Store all components in a clean area where they are unlikely to be accidentally damaged.

6.1.5 Laser Beam Alignment

The laser is accurately aligned before shipment and the following procedure should only be carried out if either the laser tube is being replaced or it is determined the tube has gone out of alignment.

- a. Properly set up the laser assembly (see Section 6.1.2) but tilt the laser tube carefully by hand (left to right and front to back) until the maximum intensity is observed on the digital meter.
- b. Note the direction of deflection required to maximize the intensity since this will quickly indicate which screws must Loosen the four locking screws, after removing the covers.
- c. Adjust the alignment screws to obtain maximum intensity. This adjustment should be done on the Room Temp Whisperlok with the motor rotating and on the Cold Style Whisperlok with the rotation stopped at the peak reading.
- d. Retighten the locking screws. Check that all four alignment screws are tightly pressed against the surface of the support base. Replace the four screw covers.

6.1.6 Replacement of the Laser Tube

- a. Remove the mounting nut, shown in Figure 6.1b.
- b. Lift off the bad laser tube.
- c. Place the new tube onto the support base and attach the mounting nut.
- d. Align the laser beam as described in the previous section.

6.1.7 Maintenance

- a. Periodically check that the laser is aligned properly (see Section 6.1.5).
- b. Clean the Whisperlok aperture window about every 100 hr of ion mill operation (see Section 7.3).

6.2 Liquid Nitrogen Cold Stage

Certain specimens, e.g., plastics, many minerals, and some electronic materials will melt, decompose, or become damaged in other ways during ion milling. The extent of damage in these beam-sensitive materials can often be reduced or even eliminated by cooling the specimen. In the DuoMill, specimen cooling is done by extending the vertically oriented specimen rotation drive rod down into a dewar of liquid nitrogen mounted below the Whisperlok system. The specimen drive rod and extension braid are made of high-purity aluminum and copper for good thermal conduction and the rod is insulated from the Whisperlok piston by a thin-walled stainless-steel tubing. The rotating specimen holder will reach a temperature of approximately -130°C in about 60 min after immersing the copper braid in liquid nitrogen. However, during ion

milling, the heating effect of the beams at maximum intensity will raise the specimen temperature to as much as -90°C. Beam heating can be minimized by (a) using a low beam voltage and current, (b) ensuring good thermal contact between the specimen holder, and (c) ensuring good thermal contact between the specimen holder and the Whisperlok piston.

6.2.1 Installation

- a. Remove the Standard Whisperlok assembly as described in Section 7.2 (a to h and shown in Figure 6.2c).
- b. Replace with the Cold Style Whisperlok shown in Figure 6.2a.
- c. Start up the DuoMill as described in Section 5.2. Note that the Whisperlok piston is hollowed out to provide vacuum thermal insulation for the aluminum conductor and consequently the Airlock pump-down time during specimen exchange will be longer than normal, (approximately 2 min).
- d. Screw the copper braid onto the lower end of the Whisperlok piston.

CAUTION: the copper braid fitting has a left hand thread. Do not remove the thermal joint compound from the threads otherwise the specimen temperature will not reach the guaranteed factory test temperature of -150°C at the specimen holder.

e. The 4-liter capacity liquid nitrogen dewar can be stored in the cabinet base until ready for use.

6.2.2 Operation

- a. Load a specimen onto the special copper specimen holder. Use a thin film of vacuum grease in the specimen-locating groove to maintain efficient thermal contact when the specimen is under vacuum. Silver paint, mounting wax, or any other low vapor-pressure surface-contact material can be used as an alternative to vacuum grease. However, if a mounting material cannot be used because of specimen contamination, etc., then a mechanical clamping plate can be used. In this case, the efficiency of thermal contact will depend on the magnitude of the clamping force applied but in general it will not be as great as when a mounting material is used.
- Screw the specimen holder onto the Whisperlok piston, once again using vacuum grease on the threads to provide good thermal contact.
- c. Replace the Airlock capsule and evacuate the Airlock. The pump-down time will be longer than usual, as explained above.
- d. Fill the dewar with liquid nitrogen and install it under the Whisperlok centering it relative to the end of the piston rod. Place the Anti-Icing collar over the neck of the dewar, pass the copper braid through the collar and immerse it in the liquid nitrogen. The Anti-Icing collar eliminates icing of the piston rod around the neck of the dewar.
- e. Lower the Whisperlok piston.

Wait 50 min before switching on the ion beams (a) to allow time for the specimen to cool and (b) to allow the aluminum conductor rod to contract to its correct height.

NOTE: If you wish to mill a specimen on the cold stage at room temperature, a special short specimen holder is available. It can be recognized by its brass (not copper or stainless steel) threaded base ring.

Leaks

If there is a leak in the cold stage or the DuoMill, the specimen temperature will increase. Also, the specimen will rise above its correct position because the aluminum rod will not contract as much. Symptoms of a leak are: (1) poor vacuum reading, (2) the frost level on the thin walled stainless steel tube rises to the laser window, (3) the specimen thinning rate is unusually low (because the specimen is not at the beam focus level).

The most probable location of a leak in the cold stage is a hairline crack in the laser window. The window can be removed for inspection by sliding it off the stainless-steel tube after first unscrewing the lower nut. **Do not use a solvent to clean the laser window as this may subsequently cause stress cracks to develop. Clean with soap and water only.** Before sliding the window back into place: (a) check that the laser mirror is clean and properly aligned with the opening in the stainless steel tube, (b) make sure the tube is clean and free from scratches, (c) lubricate the tube with a thin film of vacuum grease. When the window is back in place reform the low-temperature vacuum seal by screwing on the nut until the reference marks on the aluminum conductor and the nut are aligned (see Figure 6.2b).

6.2.3 Operation of the AutoTerminator with the Cold Stage

In the Cold Stage Whisperlok, the laser beam is reflected by a mirror out of the hollow aluminum cooling rod onto the photoresistor. The location and design of this mirror are shown in detail in Figure 6.2b. The cold stage is supplied with a special January 1999, Rev. 1

vertically oriented photoresistor mounted in the path of the reflected beam. The signal detected by the photoresistor will fluctuate as the specimen rod and mirror rotate. This makes no difference to the AutoTerminator operation when thinning opaque specimens. However, with transparent specimens, the trip point must be set carefully to the maximum value of the oscillating signal.

6.3 Liquid Nitrogen Cold Trap

The cold trap considerably reduces the level of hydrocarbons and water vapor in the Work Chamber and is useful when specimen contamination by hydrocarbons must be avoided. The cold trap fits into the top central port of the Work Chamber. The trap is installed by shutting down and venting the DuoMill as described in Section 5.8. Remove the blanking cover to the central port and replace it with the cold trap; see Figure 6.3.

6.4 Specimen Viewing Microscope

This microscope with illuminator (see Figure 6.4) is positioned onto the Airlock capsule and top plate. The lamp is switched on automatically when the microscope is in position and switches off when removed. The microscope can be rotated 360° to optimize observation of structural detail from different angles and can be illuminated for viewing in transmission mode.

To view the specimen, bring it up into the Airlock. There is no need to turn off the ions guns; they will automatically stop and resume with the original settings when the specimen is lowered. Focus the microscope on the specimen to observe structural detail. Depress the illuminator button located at the front left and right corner of the DuoMill chamber for viewing in transmission mode. These illuminators are not available on the liquid nitrogen cooled Whisperloks. To continue ion milling after examining the specimen, lower the specimen into the Work Chamber. The original settings will be reached in a few minutes when the ion guns have warmed to their equilibrium temperatures.

6.5 Electronic Gas Flow Control (EGC)

6.5.1 Installation

- a. Turn off the DuoMill, close the main gas valve on the argon cylinder, unplug the DuoMill from the main supply voltage, and vent chamber.
- b. Remove existing manual valve assembly after pulling off the three argon hoses and the blue 4-pin plug.
- c. Connect the 4-pin plug to the EGC and attach the gas lines to the hose connectors (the middle connector is the gas Inlet. See Figure 6.5b).

6.5.2 Operating Procedure

- a. Set guns for the desired sputtering angle (usually 15°) and check that the chamber pressure is better than 10⁻⁵ torr.
- b. Connect the leads, one end to each gun.
- c. Turn on both the Red and White gas control switches.
- d. Turn High Voltage Selector knob on milling chamber to the White/Red position.
- e. Depress High Voltage Switch (light on) and set Process Timer.
- f. Turn Current Limit knob fully clockwise.
- g. Turn High Voltage knob clockwise to 1.0 kV above desired voltage. (Not above 4.0 kV if using low-energy guns).
- h. Allow sufficient time for valves to open (approximately 2 min). The pressure reading on the Penning gauge will be observed to increase. Also, the gun current should begin to rise.
- i. Rotate the Current Limit knob counterclockwise until the current is set to 1.0 mA (0.5 mA if using only one gun.)
- j. Adjust High Voltage Set knob on EGC panel to obtain desired voltage. Guns will now maintain the above voltage and current without need for any additional adjustment.

NOTE: When the gas controls are initially operated or after being shut down for extended periods, the valve may stick in the Off position. If this occurs, rotate the High Voltage Set control fully counterclockwise until the valve begins to open, then proceed to Step h.

6.5.3 EGC Balance Procedure

After following the Operating Procedure above, if one gun appears to be more brilliant than the other or if only one of the guns appears to be operating, proceed as follows:

a. Turn off High Voltage switch on the sloping panel and both the Red and White switches on the EGC panel.

- b. Lift the EGC from its compartment and rotate it slightly allowing it to rest on top of the cabinet platform.
- c. Connect a digital voltmeter (DVM) (range 50 VDC) between TP1 and the ground pin (see Figure 6.5a).
- d. Turn on the White switch and follow Steps d through j of Section 6.5.2 above on the WHITE gun only. Record the voltage measured.
- e. Turn off the White switch, connect the DVM between TP2 and ground, turn on the Red gun, and again record the voltage.

NOTE: Should only one gun work or if a voltage differential greater than 7.0 VDC is measured, the valve body is possibly defective. You need to proceed to the EGC functional setup procedure.

6.5.4 EGC Functional Setup Procedure

Have the instrument running and pumping to the best vacuum it can achieve with *no* argon on, such that there is *no* load on the system. Vacuum should be approximately 3×10^{-6} torr.

Look at the EGC valve body (this is the circular part attached to the bottom of the electronic module). You will see knobs on either side of the valve—one with a red button, one with a white button. Rotating the knobs (CCW) in very small increments (1/8 to 1/4 turn) will reduce the spring load on the crystal and lower the required operating voltage.

CAUTION: Rotating the knobs in full-turn increments will cause the valve to bleed large volumes of argon into the Chamber.

- a. Rotate one of the knobs CCW until you see the vacuum changing (e.g., from 3×10^{-6} to 6×10^{-6} torr). This means the valve is beginning to leak.
- b. Rotate the knob CW, very slowly and in very small increments, until the leak is just eliminated. The pressure might now reach 4×10^{-6} torr instead of the original 3×10^{-6} torr. This is OK.
- c. Repeat this procedure for the other knob.
- d. Now turn on the valve and check TP-1 and TP-2.
- e. Adjust the knobs until you can get the voltages to within 7.0 VDC of one another.

6.5.5 Switching off the Ion Beams

- a. Bring the specimen into the Airlock by depressing Raise on the Airlock switch, or
- b. Switch High Voltage off, or
- c. Press the Start/Stop button on the digital Process Timer.

CAUTION: Do not use the switches on the EGC panel to shut off the gas flow to the guns unless only a single gun is to be used.

6.6 Chemical Milling

Background

Chemical milling is widely practiced in the semiconductor industry, with the most popular chemicals used being the halogens and halogen compounds. In the electron-microscopy field, chemical milling is used for preparing specimens that are difficult to make by conventional inert gas milling techniques, e.g., indium containing type III-V compound semiconductors and metals like tungsten. The chemical-milling capability offered with the DuoMill is the Chemically Assisted Ion Beam Etching (CAIBE™) option. This option may only be used with Model 600 CDDM, a model specially adapted for chemical milling.

CAIBE option

The CAIBE option permits an intense, slow, molecular beam of a reactive species to be directed at a specimen while it is being simultaneously milled with a conventional, fast, inert gas atom Octogun source. Chemical species are not passed through the Octoguns, therefore avoiding corrosion of the gun components (see Figure 6.6a). The CAIBE uses small quantities of a reactive gas to produce beneficial chemical effects, minimizing pump maintenance and pollution problems. Since complexities associated with ionization and decomposition inside the gun are avoided, a wide range of chemicals can be used.

WARNING: An external exhaust system must be used if the DuoMill is to be operated with toxic gases.

CAUTION: The Liquid Nitrogen Cold Trap must not be used to trap the reactive gases during operation of the CAIBE option since trapped gases may be released more quickly than can be handled by the chemical pumping system when the Cold Trap is allowed to warm to room temperature.

6.6.1 Installation of the CAIBE System

- a. Shut down the DuoMill according to Section 5.8.
- b. Vent the chamber and remove the Airlock capsule and the tan colored top plate.
- c. Press the Whisperlok piston to its full down position.
- d. Remove the gun mount rotation tool from its holder on the inside of the left-hand door of the cabinet.
- e. Insert the tool into its socket on the circumference of the gun mount and rock the mount back and forth while at the same time pulling outward. Do not remove the Teflon connectors. The HV leads will pull out with the gun mount.
- f. Insert the CAIBE assembly into the bearing at the end of the chamber.
- g. Reaching through the top port, plug the two HV leads into the Teflon connectors. Make sure they are fully inserted.
- h. Replace the top plate and the Airlock capsule

6.6.2 Loading the Chemical Source into the CAIBE System

WARNING: It is recommended that loading or unloading of chemical sources be done in a well-ventilated hood or glove box. To minimize corrosion, do not vent the DuoMill until you are ready to load the chemical source.

- a. Shut down and vent the DuoMill according to the procedure described in Section 5.8.
- b. Pull the CAIBE assembly out of the gun mount.
- vent the chemical capsule in the CAIBE assembly by turning the valve stem fully counterclockwise (see Figure 6.6a).

Note: The capsule is held in by an O-ring and is not threaded to the knob.

- d. Remove the capsule screen and add the solid chemical source until the capsule is half full (if iodine is used as the chemical source, use approximately 10 g, enough for about 50 hr of continuous operation).
- e. Replace the screen and clean off any particles of the chemical source stuck to the outside of the capsule.
- f. Apply a very small amount of vacuum grease to the 032 size O-ring and insert the capsule back into the gun knob. Repeat Steps b through g for the other gun.
- g. Open both gas control valves as described in Step c above.
- h. Start-up the DuoMill according to Section 4.2.
- i. Once the chamber pressure reaches the 10⁻⁴ torr region, close the Gas Flow Control valves (holding the gun knob, rotate the valve knob CW against its stop).
- j. Wait until the pressure stabilizes around 10⁻⁶ torr before operating the CAIBE system.

6.6.3 Operation of the CAIBE System

The CAIBE system consists of a modified standard gun mount having a central port that accepts the CAIBE jet assembly. After installing the system, the DuoMill should be evacuated to the 10⁻⁶ torr level.

- a. Retract the CAIBE assembly and open the valve to evacuate the capsule before loading a specimen (see Figure 6.6a).
- b. Before loading the specimen, the valve should be opened fully for about 5 min to obtain a steady state flow. It is then adjusted using the Penning gauge. The optimum setting is very material dependent, i.e., for InP the optimum flow results in an increase of 1 to 1.5 x 10⁻⁶ torr from the ultimate pressure and for InSb it is about 1 to 1.5 x 10⁻⁷ torr.
- c. To determine the optimum amount flow for a given material, it is necessary to examine the prepared specimen in the TEM. If the specimen is heavily etched, it means the flow is excessive and must be reduced. However, if the specimen is covered with argon ion-milling artifacts, e.g., indium islands in the case of indium containing semiconductors, then the flow should be increased (it is interesting to note that CAIBE milling will quickly clean indium islands off specimens that have been previously argon ion milled). When the correct valve setting is determined, the same setting may be used for subsequent specimens of the same material.
- d. Load the specimen by placing it on the special specimen support post provided (see Figure 6.6b). This support post has three advantages over the conventional specimen platform. It is positioned close to the ion mill axis such

that the chemical jet will be directed onto the specimen. Also, specimens are kept cleaner because contamination by secondary sputtering of reactants from the specimen mount is avoided. Finally, specimens can be thinned at much lower angles, thereby reducing radiation damage and creating larger thin areas. The disadvantage of the support post is that it does not allow two-sided milling. However, the above three advantages are usually considered to outweigh this disadvantage.

- e. Begin milling using the optimal valve setting.
- f. When thinning is half complete, the specimen must be inverted to complete thinning from the non-dimpled side.
- g. When specimen perforation occurs, the high voltage should be switched off and the CAIBE assembly should immediately be locked in the retracted position with the valve closed. The specimen can then be removed in the normal way, as described in Section 5.7.

NOTE: The CAIBE assembly should always be latched in the out position when not in use. In this position, inert gas ion milling can be performed in the conventional manner.

CAUTION: To minimize corrosion and pump maintenance, always turn off the CAIBE jet when not in use.

6.7 Digital Process Timer

The Process Timer is a countdown interval timer with a digital countdown display, an LED indicator, and an audible alarm to indicate the end of the timing cycle. Two arrow buttons on the front of the timer are used to set the time. Timing intervals from 1 min to 99 hr 59 min can be set in one minute intervals. Hold down the Up Arrow button to increase the time or the Down Arrow button to decrease the time. The longer the button is depressed, the faster the time display will change.

The Start/Stop button performs three functions:

- Pressing the button when the timer is not active will cause the timer to begin counting down the time displayed.
- Pressing the button when the timer is active will stop the timer and turn off the flashing LED (the timer will display
 the remaining time in the cycle).
- Pressing the button a second time will cause the timer to continue timing from the point at which it was stopped.

Depressing the Start/Stop button for more than 2 sec while in the Stop mode will reset the timer to the initial preset time interval. Should power fail during a timing cycle, the internal memory will backup the time to the last five seconds. Upon restoration of power, the timer will display the time remaining in the previous timing cycle.

Setting the Process Timer

- Depress the Up Arrow button to set the desired thinning time. If you overshoot the desired time, depress the Down Arrow to decrease the time.
- b. Depress the Start/Stop button to start the process timer. A flashing dot indicates the timer is counting down. The digital readout will count the time down in 1-min intervals until it reaches 0 at which time the audible alarm will sound.
- c. Should it be necessary to change the preset time during the thinning process, depress the Start/Stop button to stop the count down, depress the Up or Down Arrow button to set the new time. Depress the Start/Stop bar to reinitiate

6.8 Sector Speed Control (SSC)

Introduction

Ion-milled, cross-sectional TEM specimens are used extensively to study the microstructure of multi-layered materials. Unfortunately, it is difficult to get all the layers in a cross section equally electron transparent because the various components usually mill at different rates. Milling at low angles can minimize differential thinning so that the slow milling layers shadow the fast milling layers from the ion beam. However, even at low angles, uneven milling still occurs when the ion beam travels along the multi-layer interface (glue line). Although this problem can be overcome by using a shield to cut off the ion beam in these directions, the shield has the disadvantages that it slows the overall thinning rate, it is cumbersome to change the shielded angle, and the shield can contaminate the specimen. A more efficient solution is to rotate the specimen rapidly as the beam passes along the glue line and reduce the rotation speed in other areas. The Sector Speed Control (SSC) has been designed to do just this (see Figure 6.8a).

SSC is available in both room temperature or liquid nitrogen versions. The two versions are similar except the latter has a lower port for the liquid-nitrogen-cooled conductor and a right-angled sensor to detect the light from the laser AutoTermina-

tor. Both versions incorporate a DC motor to rotate the specimen at two different angular speeds: 2 rpm in the selected sector (Φ) and 10 rpm outside it. Additionally, continuous rotation at either of the two speeds is available (see Figure 6.8b for circuit diagram).

Single or double sectors (diametrically opposed) may be selected with angles of 40°, 60°, or 80°. The precise alignment of the sectors is accomplished through the use of an optical encoder. This encoder is set so that a reference mark at the top of the piston is directly in line with the centerline of the rear ion beam when the specimen rotation is in the middle of the slow speed sector. The optical encoder has the advantage that the reference zero is maintained even in the event of a power failure or a power fluctuation.

6.8.1 Operating Procedure

The double-sector mode of thinning is generally preferred and is used when a fast thinning (soft) layer is protected on both sides by slow thinning (hard) layers.

The single-sector mode of thinning is used when a soft layer is protected on only one side by a hard layer. In this case, only the rear, upper ion gun should be used and the hard layer should be oriented towards the ion beams during the slow speed sector. With this set-up the hard layer will protect the soft layer so that ideally both layers will become electron transparent simultaneously.

The selection of the sector angle is generally dependent on the nature of the specimen. If the soft layer(s) are uniformly thin and the hard layer(s) are continuous along the glue line, it may be better to use a larger sector angle to obtain a more uniform thin area. On the other hand, if the soft layers are wide and the hard layers are discontinuous along the glue line then smaller sector angles should be chosen to maximize the shadowing effect.

6.8.2 Specimen Loading

A reference mark must be drawn on the base of the specimen holder in line with the reference mark at the top of the Whisperlok piston (Figure 6.8a). The mark should be placed when the holder is screwed fully down onto the Whisperlok piston. This mark indicates the location of the rear ion beam when the specimen is in the middle of the single, slow-speed sector. Cross-sectioned specimens should therefore be mounted so that the glue line is perpendicular to the reference mark. If single-sector milling is required the "hard" side of the specimen should be on the same side as the reference mark.

6.8.3 Switching on the SSC

The SSC will operate automatically so that the symmetry axis of the slow-speed sectors is parallel to the ion beams (i.e. perpendicular to the ion mill chamber axis). The various operating modes are set by opening the sloping panel and rotating the knob on the SSC front panel to one of the following eight positions (going clockwise):

- 2 rpm Continuous rotation at two revolutions per minute.
- 40° Rotation at 2 rpm in a single sector of 40° and 10 rpm for the remaining 320°.
- Rotation at 2 rpm in a single sector of 60° and 10 rpm for the remaining 300°.
- 80° Rotation at 2 rpm in a single sector of 80° and 10 rpm for the remaining 280°.
- 80° Rotation at 2 rpm in two sectors of 80° each (180° apart) and 10 rpm for the remaining 200°.
- 60° Rotation at 2 rpm in two sectors of 60° each (180° apart) and 10 rpm for the remaining 240°.
- 40° Rotation at 2 rpm in two sectors of 40° each (180° apart) and 10 rpm for the remaining 280°.
- 10 rpm Continuous rotation at ten revolutions per minute.

The operating mode can be changed at any time during the milling process by rotating the knob to a new position. This change does not alter the alignment of Φ , its centerline always remains fixed. Generally it takes two complete revolutions for the SSC to update the new switch position.

The piston can be raised at anytime during the milling process and upon lowering, the sector will remain properly aligned but again it may take two revolutions to update. After following the normal Airlock evacuation procedure, the piston can be lowered and the ion beams turned on according to Section 5.4.

7. ROUTINE MAINTENANCE AND SERVICING

7.1 Cleaning the Airlock Capsule Viewing Window

- a. After ion milling, raise the specimen into the Airlock and vent.
- b. Remove the capsule and push out the viewing window. Check capsule O-rings and if necessary clean or replace.
- c. Flip and replace the window so that the dirty side is exposed to atmosphere.
- d. When the next specimen is being ion milled, place a few drops of a non-abrasive kitchen cleaner on the dirty window and rub the cleaner over the surface using a soft tissue. Alternatively, a 4-6 μ diamond polishing compound may be used.
- e. Continue rubbing until the window is clean.

NOTE: To prevent the formation of difficult to remove sputtered deposits, it is strongly recommended that the viewing windows be cleaned after every specimen exchange.

7.2 Servicing the Vacuum Seals in the Whisperlok Mechanism

- a. Shut down the DuoMill as described in Section 5.8.
- b. Remove the specimen Airlock capsule.
- c. Remove the specimen holder.
- d. Press the Airlock piston down into the Work Chamber and remove the tan-colored Airlock top plate.
- Remove the three O-rings from the top plate and clean the underside of the top plate and the O-ring grooves with a laboratory grade solvent.
- f. Clean and lubricate the O-rings and replace them in the top plate.
- g. Disconnect the electrical plug from the Whisperlok base assembly.
- h. Withdraw the complete Whisperlok assembly by rotating it clockwise to the end stop and pulling downward (see Figure 6.2c). If only the upper Flouroseal is to be replaced go to Step p.
- i. Remove the four screws that hold the cover to the base assembly.
- j. Remove the four screws, the four shoulder washers, and the contact ring that hold the cover to the cylinder body. Be careful to retain the limit switch plunger that activates the limit switch in the base.
- k. Remove the piston by pulling on the large nylon bevel gear (see Figure 6.8a). Now thoroughly clean all parts.
- I. Lubricate the piston plate O-ring and the inside wall of the cylinder body with a silicone lubricant.
- m. The flanged ball seal may be removed after unscrewing the piston plate. Be careful not to damage the seal during removal. The ball seal is made of Teflon and can be cleaned in most solvents. It does not require lubrication.
- n. Lightly lubricate the piston rod and place the assembly back into the cylinder body.
- o. Assemble the cover assembly such that the limit switch plunger is in line with the limit switch.
- p. Remove the piston rod shield from the piston rod.
- q. Unscrew the cone shaped cap from the top of the cylinder body.
- r. Remove the old Fluoroseal and carefully clean the groove.
- s. Lightly lubricate both the OD and ID surfaces of the seal with silicone vacuum grease and slip it over the piston rod; now lightly press down on the seal until it just begins to enter the groove.
 Replace the cone-shaped cap by slowly screwing it onto the cylinder body. This will properly seat the seal in the groove.

NOTE: Lightly lubricate the threads in the cap with vacuum grease before replacing.

- t. Replace the piston rod shield.
- u. Replace the Whisperlok assembly and reconnect the electrical plug.

7.3 Cleaning the Whisperlok Aperture Assembly

NOTE: This operation can be performed without venting the main Work Chamber.

- a. Raise the Whisperlok and Vent the Airlock capsule. Remove the capsule and specimen holder.
- b. Using a coin or similar device unscrew the aperture assembly.

CAUTION: Do not push the Airlock piston down as this may vent the main Work Chamber.

c. Remove the aperture assembly and unscrew the window-retaining cap at the bottom of the tube (see Figure 7.3).

- d. Remove and clean the small window.
- e. Remove any dirt from the window O-ring seal and the aperture tube.
- f. Remove and clean the upper O-ring
- g. Lubricate both O-rings, assemble them along with the window and screw the retaining cap on lightly; then screw the assembly back into the Airlock piston.

NOTE: Steps a to g should be done every 100 hr of ion-mill operation.

7.4 Changing Cathodes and Insulators

- a. Shut down the DuoMill according to Section 5.8.
- b. Withdraw the gun requiring servicing by pulling outward on the engraved knob (see Figure 7.4a).
- c. Pull out the insulator with its anode and anode contact. Disassemble further by gripping the tip of the anode contact between the fingernails of the thumb and second finger and pulling the contact straight out (see Figure 7.4b).

CAUTION: Never attempt to rotate the anode contact in the insulator. If the anode contact becomes jammed, immersing the insulator in methanol for about 5 min can usually loosen it.

- d. The anode can now be popped out of the insulator using the anode contact as a piston.
- e. Unscrew the cathode retaining screw and pull off the cathode.
- f. Attach the new cathode using the retaining screw.

CAUTION: Check that the screw does not protrude above the surface of the cathode to be sure it will not scratch the inside surface of the gun mounting plate.

- g. Clean the anode and insulator using 600-grit abrasive paper and then ultrasonically clean all parts in a clean degreasing type solvent. Any glaze on the face of the insulator around the anode hole must be filed off, otherwise the gun will tend to short out. Replace the insulator O-ring and assemble the parts in the new cathode. Clean and lubricate O-rings with minimal amount of silicone grease. Be careful not to let any grease get onto the anode or the face of the insulator as this will create shorting problems during subsequent gun operation.
- h. Assemble the insulator into the new cathode.
- Replace the gun in the mounting plate in such a manner that the slot at the end of the insulator faces the direction of the ion beam. The gun fits in only one orientation - do not use excessive force.

CAUTION: Special attention must be paid to the cleanliness of the insulator, the anode, and the anode contact. The primary cause of ion-gun arcing is contamination from vacuum grease or grease from finger prints, which decomposes and provides a current-leakage path. If arcing is left to continue the ceramic insulator will become glazed and a permanent leakage path will develop. If the glaze cannot be completely removed the insulator will have to be replaced.

7.5 Using the Airlock O-Ring Lubrication Tool, see Figure 7.5

After a period of time, the Airlock O-ring will become contaminated and may prevent the specimen piston from ascending fully into the Airlock. The lubrication tool remedies this problem without having to shut down the ion mill or vent the chamber.

- a. Depress Raise on the Airlock switch to bring the specimen piston up into the Airlock.
- b. Remove the Airlock capsule and specimen holder.
- c. Replace with the capsule containing the sliding lubrication tool. The grooved surface of the tool should be liberally coated with vacuum grease.
- d. Evacuate the Airlock and push down on the back of the lubrication tool allowing the piston to push the knob back up; repeat this operation four or five times. This will remove deposited material from the O-ring as well as lubricate it (see Figure 7.5).
- e. Vent the Airlock and remove the tool.

7.6 Cleaning the Cold Cathode Gauge Tube

Contamination of the measuring chamber within the tube will affect the pressure reading and generally produces an indication of poor vacuum. If the contamination becomes severe, instability occurs due to sputtered layers peeling off producing a short that shifts the needle to the right end of the scale. If this occurs, the gauge tube must be dismantled and cleaned.

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

NOTE: The anode, igniter, and O-ring are consumable items and must eventually be replaced.

Disassembly

- a. Unplug the connector from the gauge tube by first unscrewing the retaining screw at the center of the connector.
- b. Remove the gauge tube by pulling it straight out from its chamber.
- c. Use the 1.5 mm Allen wrench to loosen the setscrew on the side of the electronics module and remove the module (see Figure 7.6).
- d. Use the 7.0 mm wrench to remove the hex-head screw from the magnet and slide off the magnet.
- e. Remove the locking ring and the pole insert from the front of the measuring chamber.
- f. Use the 3.0 mm Allen wrench to remove the two socket-head screws at the back of the tube and remove the retainer.
- g. Carefully remove the anode, support ring, and Viton O-ring. These parts can be individually cleaned or replaced if necessary.

Cleaning

- a. Clean the inside of the tube and the front pole insert using an abrasive pad (such as Scotchbrite) or polishing cloth (500 grain).
- b. Rinse both parts using methanol; dry with compressed air or nitrogen gas.
- c. Carefully clean the anode and igniter using a polishing cloth; the igniter can be moved on the anode by sliding it up or down. Rinse the anode and ceramic with methanol and dry nitrogen.

CAUTION: The igniter 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 since these help form the vacuum seal.

Assembly

- a. The igniter must be positioned 20 mm from the end of the anode pin.
- b. Insert the O-ring and support ring into the tube. The sealing surface, O-ring, and ceramic part must be clean.
- Carefully insert the anode with igniter into the tube; replace the retainer and tighten the screws uniformly until the stop position is reached.
- d. Place the mounting tool (if available) over the anode pin to accurately position the igniter on the pin. Remove the tool and dust out the tube to remove any loose particles.
- e. Slide the pole insert into the front of the tube and mount the locking ring against the pole insert.

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

- f. Mount the magnet onto the tube and lock it with the screw and clamp.
- g. Carefully push on the electronics module until it stops, position the connector rotated 180° from the magnet retaining screw. Lock the module in place with the socket set screw.

CAUTION: Do not tighten down on the setscrew, it need only be snug.

- h. Replace the gauge tube into the chamber and position the magnet retaining screw.
- i. Plug the connector into the gauge tube and tighten the retaining screw.

CAUTION: *Do not* allow the DuoMill to run for more than 1 hr with the Penning gauge at pressures above 10⁻³ torr, since a glow discharge will occur in the tube causing it to become contaminated.

7.7 Argon Gas Leaks

Typically a cylinder of argon gas will last about six months to a year. If one runs out in a much shorter time, there is probably a leak in the gas distribution assembly. To verify a leak, close off the main valve of the argon cylinder and observe the high pressure gauge. If the pressure falls over a period of a few minutes, a significant leak is indicated.

The gas-distribution assembly consists of a pressure regulator with gauge and a normally closed three-way solenoid valve, SV2 or SV3. As can be seen from Figure 1.2, when SV2 or SV3 is energized, argon pressurizes the Whisperlok assembly lowering the piston to the down position. When SV2 or SV3 is de-energized the argon supply is shut off and the Whisperlok

assembly is vented to atmosphere through the valves, allowing the piston to rise into the Airlock. Should the valve seats become dirty or defective, SV2 or SV3 may not seal properly when de-energized and cause argon to leak from the tank.

Leak Detection

- a. Locate the gas-distribution assemblies behind the cabinet doors in the upper left and right hand corners.
- b. The valve SV2 is mounted on the left side with its vent port directed toward the floor of the cabinet.
- c. Attach a short length of plastic tubing to this port and place the opposite end in a beaker of water.
- d. Any bubbling indicates a leaking seal that must be cleaned or replaced.

Replacing Seals

- a. Shut off the argon supply at the tank and raise the piston up into the Airlock.
- b. Disconnect the two electrical leads going to the solenoid coil by disconnecting the spade lugs.
- c. Insert a flat blade screwdriver into the end of the coil-retaining nut and unscrew the valve stem.
- d. Remove the plunger from the valve stem, inspect and clean the valve, seat the pads at either end of the plunger.
- e. Reassemble in reverse order and repeat for SV3.

7.8 Vacuum System Maintenance

7.8.1 Pfeiffer Molecular Drag Pump

The Pfeiffer molecular drag pump is shipped already charged with lubricating oil and only requires changes after approximately 5000 operating hours. However, if the bearings begin to sound louder than normal, the oil cartridge should be changed. The lube kit consists of a stack of felt discs saturated with oil and is replaced as a cartridge. Changing the cartridge requires the Work Chamber be vented to atmosphere. This is also an opportunity to service the guns, clean the Airlock assembly, and possibly the Whisperlok.

Oil Cartridge Replacement:

- a. Unplug the cables from the rear of the controller.
- b. Separate the controller from the MDP by removing the thumb screws between the two.
- c. Using a wide blade screwdriver, unscrew the cover at the bottom of the pump.
- d. Using a pair of tweezers, remove the existing cartridge and properly dispose it.
- e. Clean the cover and the inside of the pump from where the cartridge was removed.
- f. Insert the new cartridge (already saturated with oil) and reinstall the cover.
- g. Remount the controller and connect all the cables.

CAUTION: Pfeiffer recommends the pump be rebuilt after 20,000 hr to prevent severe damage as well as costly repairs.

7.8.2 Blazers Diaphragm Pump

The Balzers diaphragm pump requires no maintenance under normal operating conditions. The valves and diaphragms will suffer from the effects of wear and tear after a time. If a serious drop in foreline pressure is noted, the working chamber, the diaphragms, and the valves should be cleaned and checked for cracks.

7.8.3 Chemical Filter

Model 600CDDM is shipped with a chemical filter containing activated carbon installed ahead of the diaphragm pump to prevent premature failure caused by high levels of corrosive materials entering the pump (see Fig. 7.8.3). These high levels are present particularly if the CAIBE jet is accidentally left open over a period of several days. The output of the filter couples to a viewing tube containing a chemical indicator strip that gauges the level of contamination of the activated carbon within the canister. As the activated carbon reaches its saturation level, low amounts of reactive materials will begin passing directly through the filter and over the indicator strip. A change in color and finish of the strip from a bright silver to a dull dark yellow color indicates the activated carbon is contaminated and must be replaced as soon as possible.

Recharging the Chemical Filter

- a. Shut down the DuoMill and vent the system.
- b. Remove the filter by uncoupling the KF flanges, one at its output to the flexible hose and the other at the input to the viewing tube.
- c. In a well-ventilated area, empty the contaminated activated carbon into a Zip-Loc storage bag and refill the canister with a new charge.
- d. Place the contaminated material in the empty container from the new charge and dispose of it as Hazardous Waste Material.
- e. Clean the indicator strip with a polishing compound to remove the tarnish.
- f. Replace the strip into the viewing tube, making sure to orient it so that the wide surface is facing the window and it is on an angle across the tube diameter (see Figure 7.8.3).
- g. Install the filter and start the DuoMill.

7.9 Routine Maintenance Schedule

Section	Maintenance Operation	Frequency	Fault Condition
7.1	Clean viewing window	Every specimen exchange	Specimen viewing becomes difficult
7.3	Clean aperture assembly	Every 100 operating hours	AutoTerminator reading fluctuates
7.3	Clean Airlock window	As required	AutoTerminator sensitivity decreases
5.5	Rotate Octogun cathodes	Every 100 operating hours	High chamber pressure, low thinning rate
7.1	Clean Airlock capsule O-ring	As required	Poor vacuum when Airlock piston lowered
7.5	Clean piston Airlock seal	As required	Poor vacuum when Airlock piston lowered
7.4	Complete ion gun service	Every 800 hr	Persistent gun arcing
7.8.1	Change Pfeiffer MDP cartridge	See instructions	Poor vacuum, bearing noise excessive
7.8.3	Change filter charcoal	See instructions	Indicator strip changes color to dull dark yellow
7.6	Clean cold-cathode gauge	As required	Erratic reading, meter bounces to right
7.2	Service Whisperlok assembly	As required	Poor vacuum during specimen rotation

Complete overhaul of DuoMill every 8000 hr.

8. SPARE PARTS LIST

8.1 Specimen Holders

Standard specimen holder	600.380A2 (2.3mm) 600.380A3 (3mm)
Cold style specimen holder	600.380B2 (2.3mm)
	600.380B3 (3mm)
Room temp holder for cold stage(1)	600.380C2 (2.3mm)
	600.380C3 (3mm)
Specimen support plates Molybdenum (Set of 5)	600.38211 (3mm)
	600.38212 (2.3mm)
Specimen clamping plates Molybdenum (Set of 5)	600.38221 (3mm)
	600.38222 (2.3mm)
Platform retaining screws 00-90 x 1/8 (Set of 12)	600.38200
Single sided specimen shroud for top surface milling (Standard)(2)	600.38071.0
Single sided specimen shroud (Cold style)	600.38072.0
Single sided specimen shroud (Short base)	600.38073.0
Low-angle specimen mount (standard)	600.5115B.0
Low-angle specimen mount (cold stage, short base)	600.5115C.0
Specimen post 3 mm	600.51161.0
Specimen post 2.3 mm	600.51162.0
DuoPost, glue type, 3 mm	691.08600
DuoPost, clamping type, 3 mm	691.08601

- (1) The specimen stage descends.050" (1.27 mm) when cooled from room temperature to -150°C. A special short specimen holder is required when the specimen stage is operated at room temperature.
- (2) Sputtered material can deposit on the lower surface of specimens during single-sided milling. The shroud prevents this. Please specify type of specimen holder.

8.2 Octoguns [Models 600.01000 (standard) and 600.37000 (low energy)]

Octogun O-ring kit (4 guns)	600.15002
Ceramic insulator	600.01120.0
Anode	600.01130.0
Anode contact	600.01140.0
Eight aperture cathode	600.01153.0
Low energy anode	600.37010.0
Low energy eight aperture cathode - Stainless Steel	600.37020.0
High voltage end cap assembly	600.01070
Screw-in Teflon connector with ball plunger	600.05310

8.3 Sector Speed Control (Model 600.48000)

Whisperlok O-ring kit (2 assemblies)	600.15004
Whisperlok aperture window (qty 2) (Figure 7.3)	06226
Piston rod vacuum seal (Fluoroseal)	04516

8.4 Airlock (Model 600.05000)

Airlock O-ring kit (2 assemblies)	600.15006
Airlock capsule window	06225

8.5 Auto-terminator (Model 600.32000)

Replacement laser tube 05486
Optical filter 600.32060.0

8.6 Cold-Cathode Gauge (Model 600.22000)

Gauge tube repair kit 600.22160

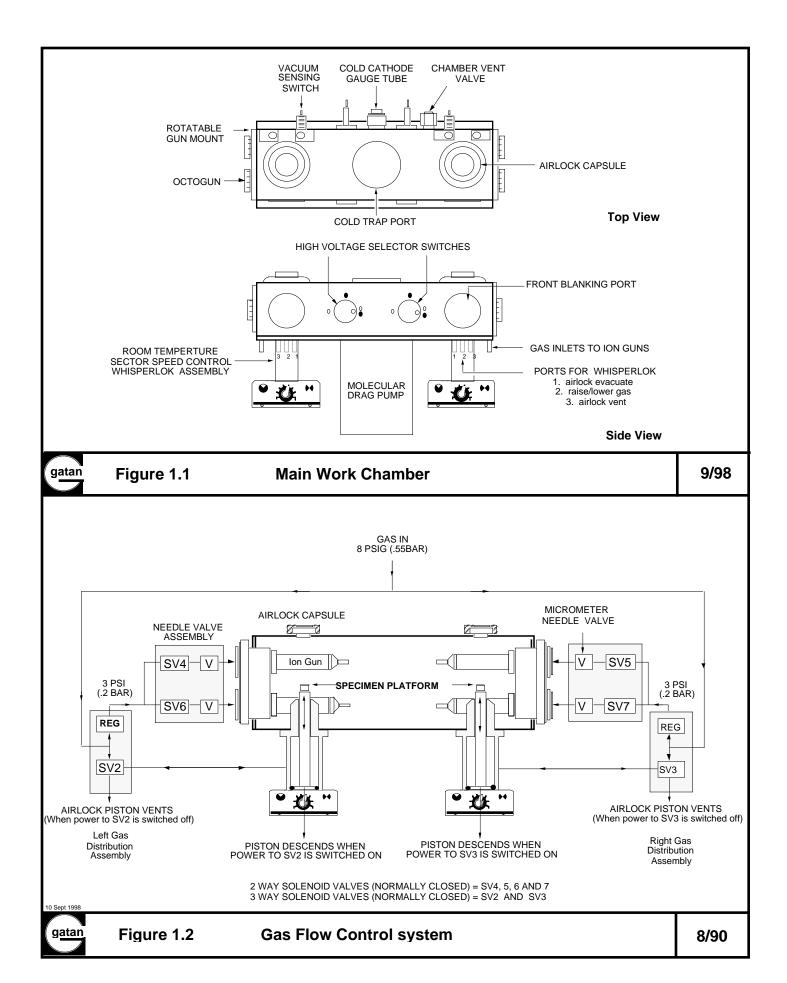
NOTE: Please have your serial # available when ordering.

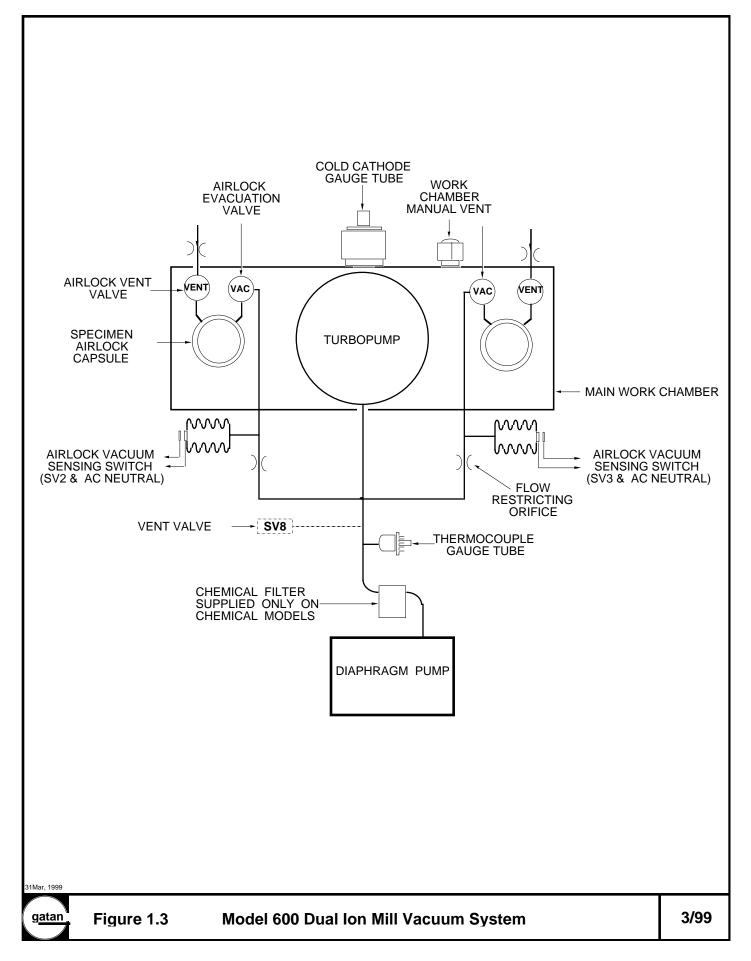
8.7 Gas Distribution Assembly (Model 600.07000)

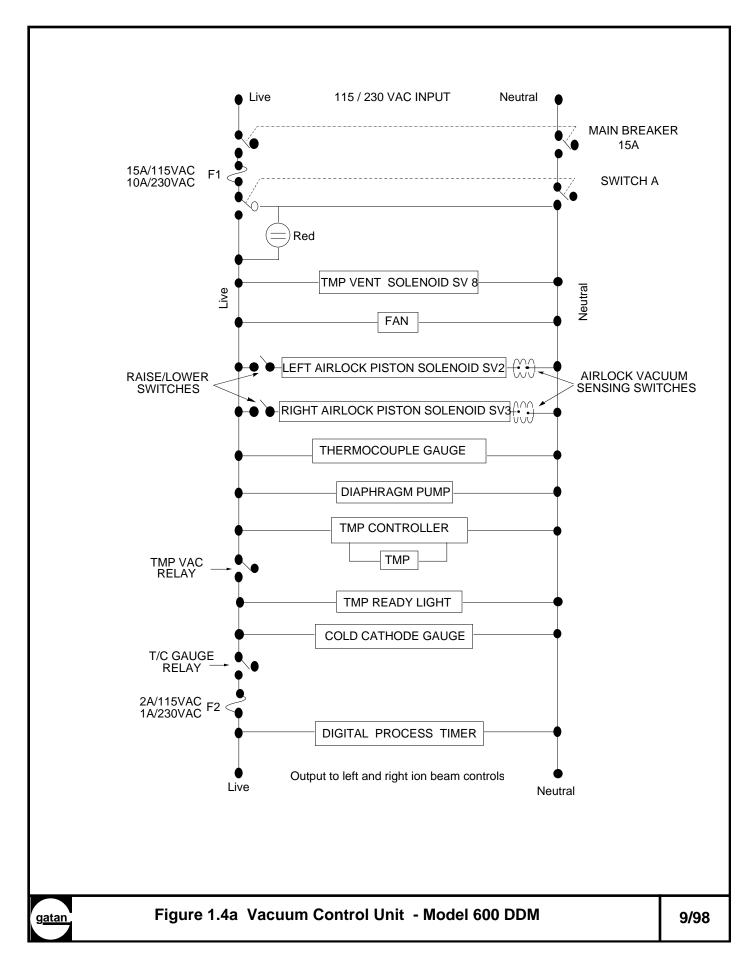
3-way solenoid valve	03564
Solenoid valve coil 115 V	03501
Solenoid valve coil 230 V	03502

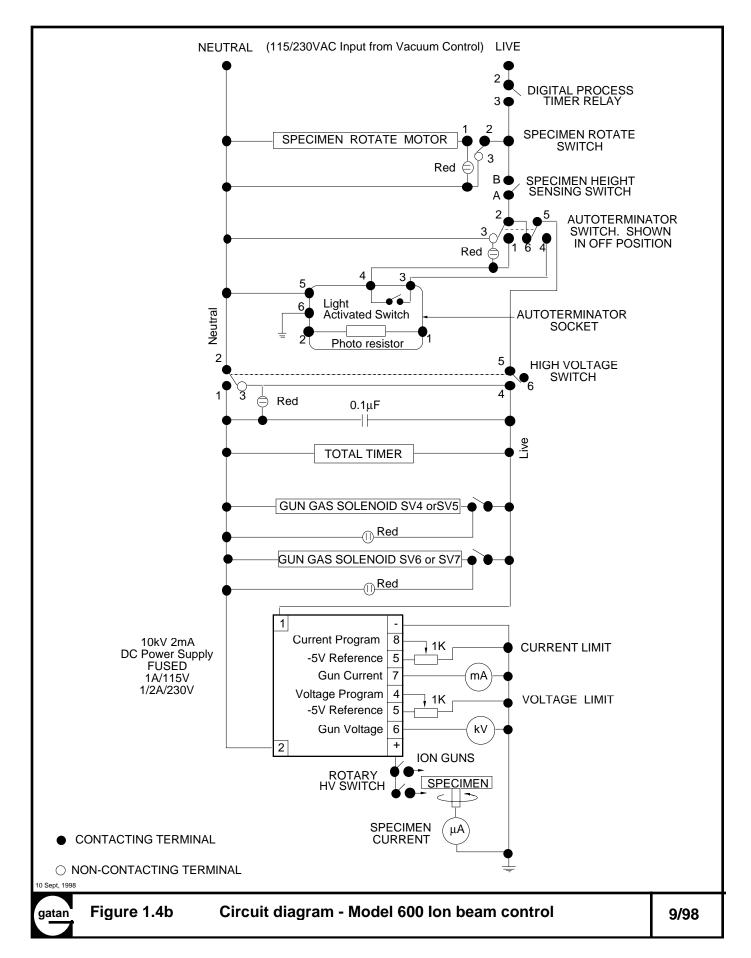
8.8 O-Ring Kits

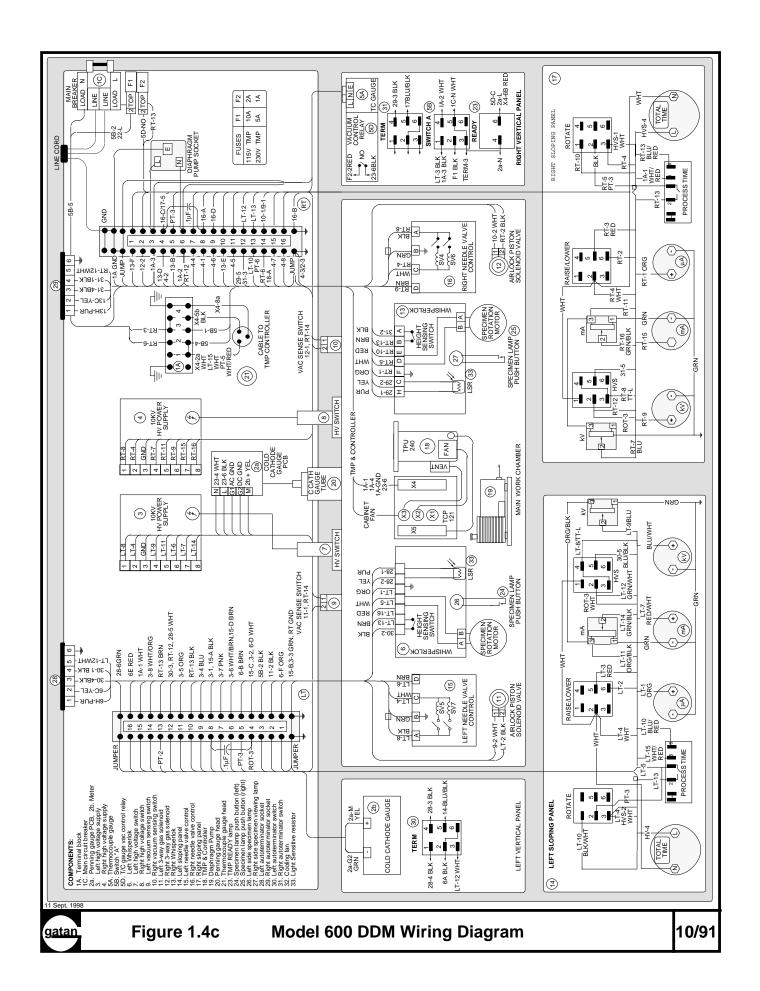
Octogun O-ring kit (4 guns)	600.15002
Whisperlok O-ring kit (2 assemblies)	600.15004
Airlock O-ring kit (2 assemblies)	600.15006
Main working chamber O-ring kit	600.15011
Complete O-ring kit	600.15800

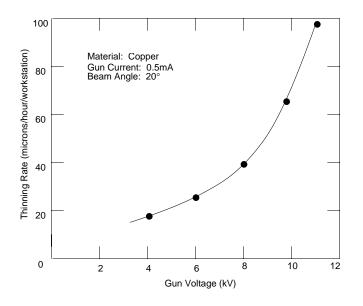








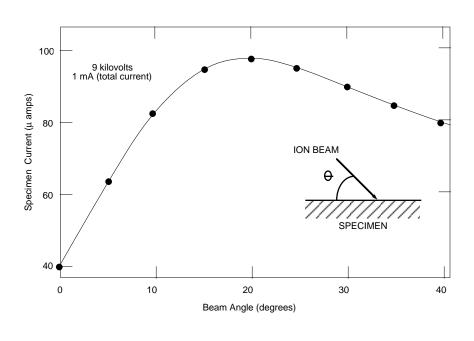




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Figure 2.1a Typical Thinning Rate for Copper in Range 4-11kV

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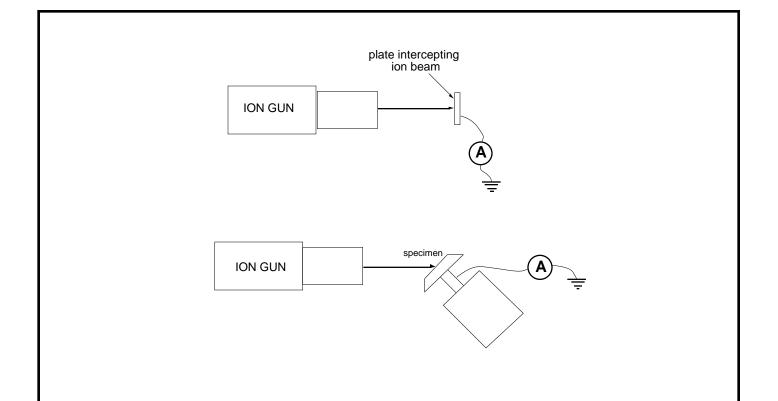


14Aug,1990

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Figure 2.1b Specimen Current versus Beam Angle for Copper

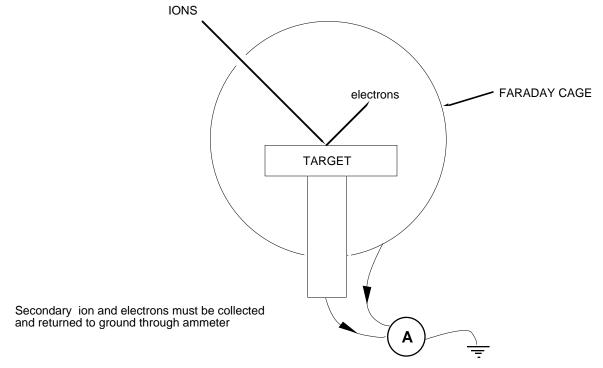
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Figure 2.2a Ammeters in these Circuits do not measure Ion Current

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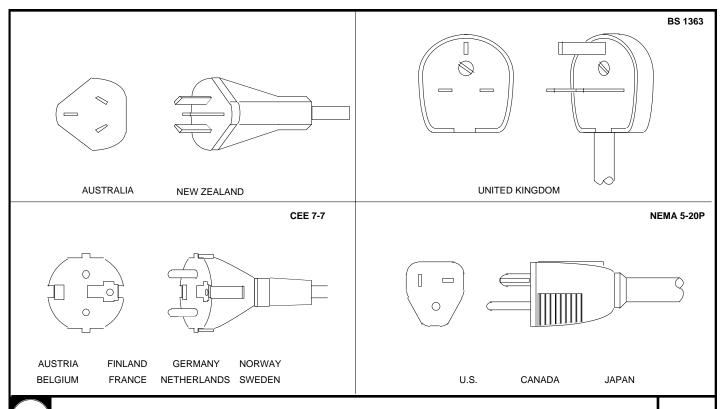


1Jan, 1989

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Figure 2.2b To measure Ion Current a Faraday Cage must be used

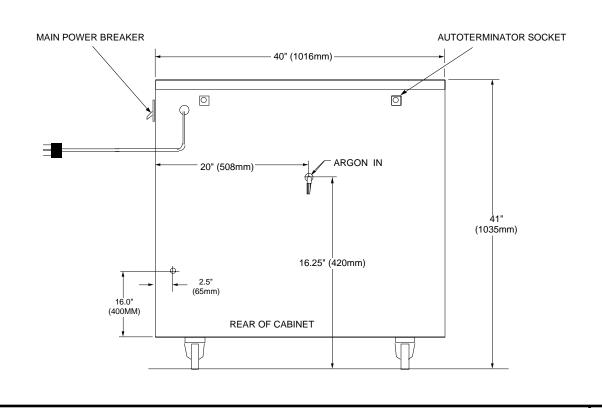
1/86



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FIG 3.1a Plugs supplied with the Model 600 DuoMill

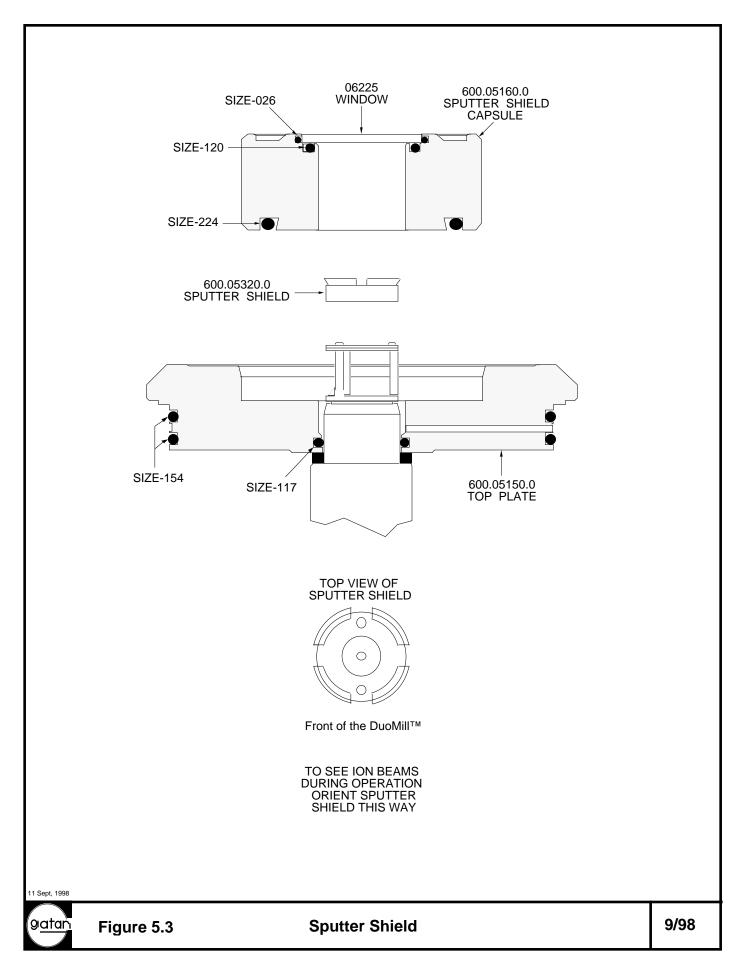
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Fig 3.1b Locations of power and gas fittings

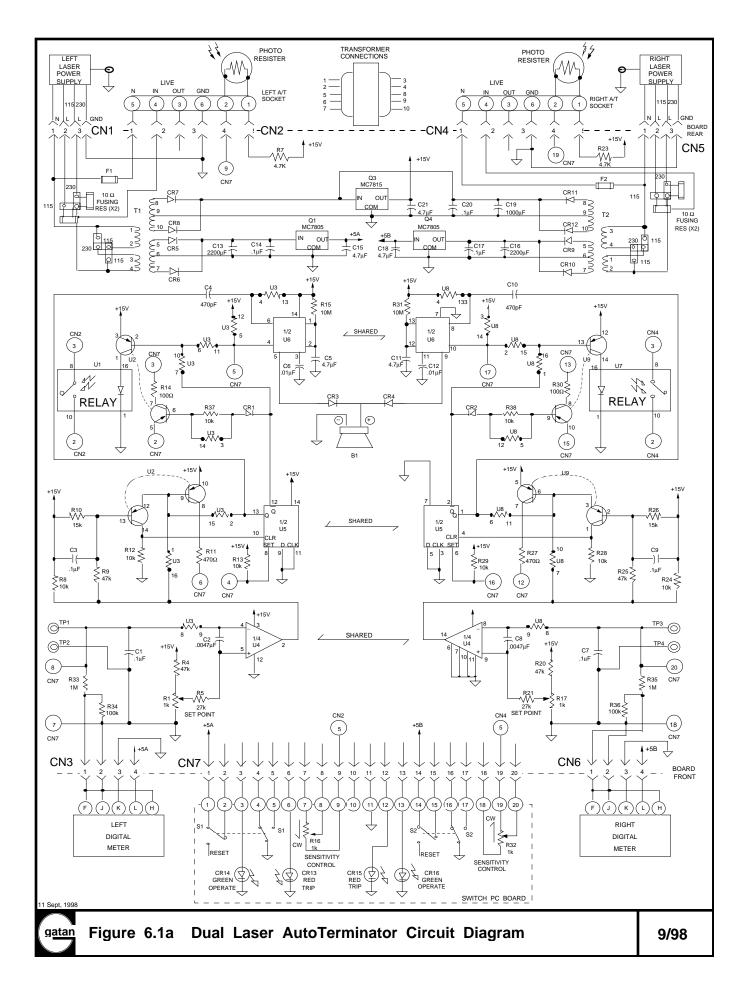
8/90

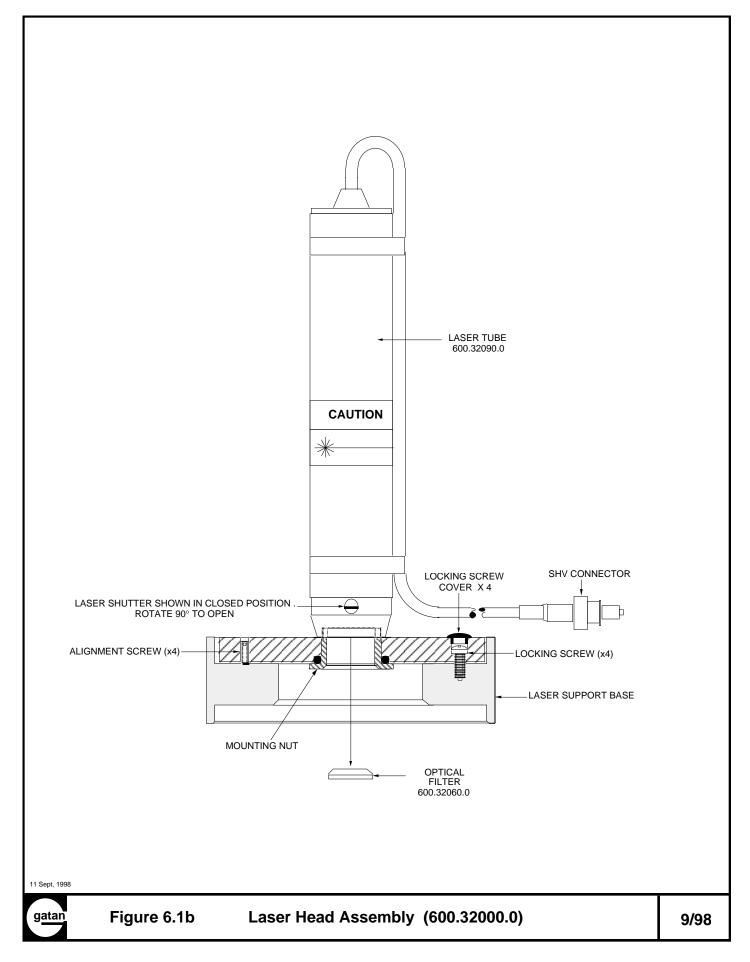


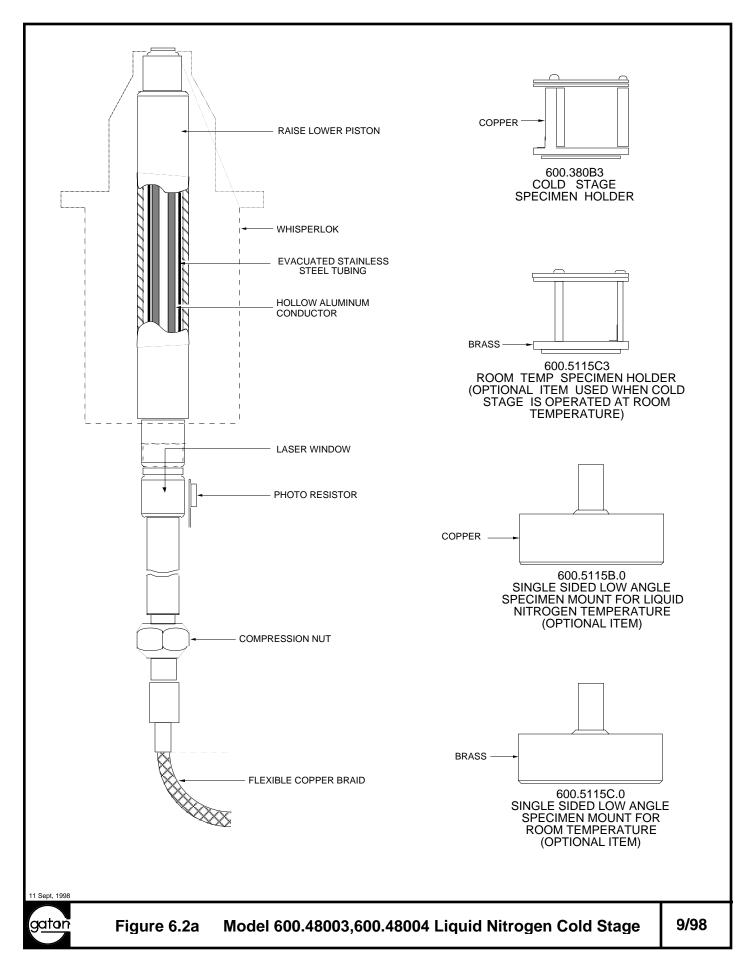
LEFT GUNS						
No	Start	Finish	Hrs	Remarks	Name	Date
1						
2						
3						
4						
5						
6						
7						
8						

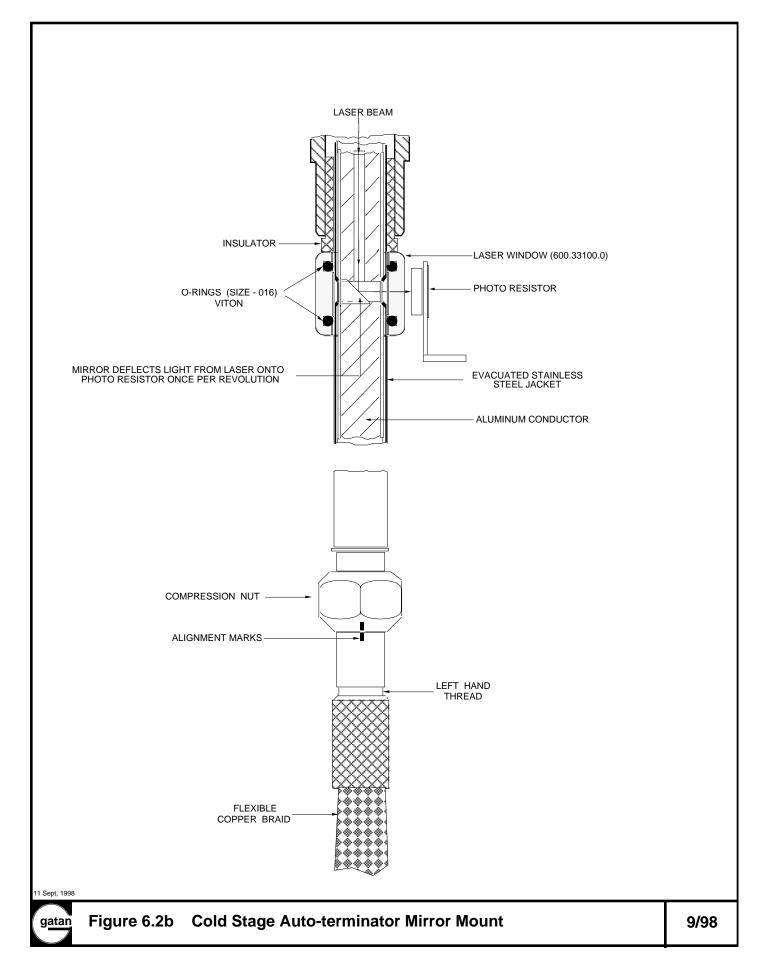
RIGHT GUNS						
No	Start	Finish	Hrs	Remarks	Name	Date
1						
2						
3						
4						
5						
6						
7						
8						

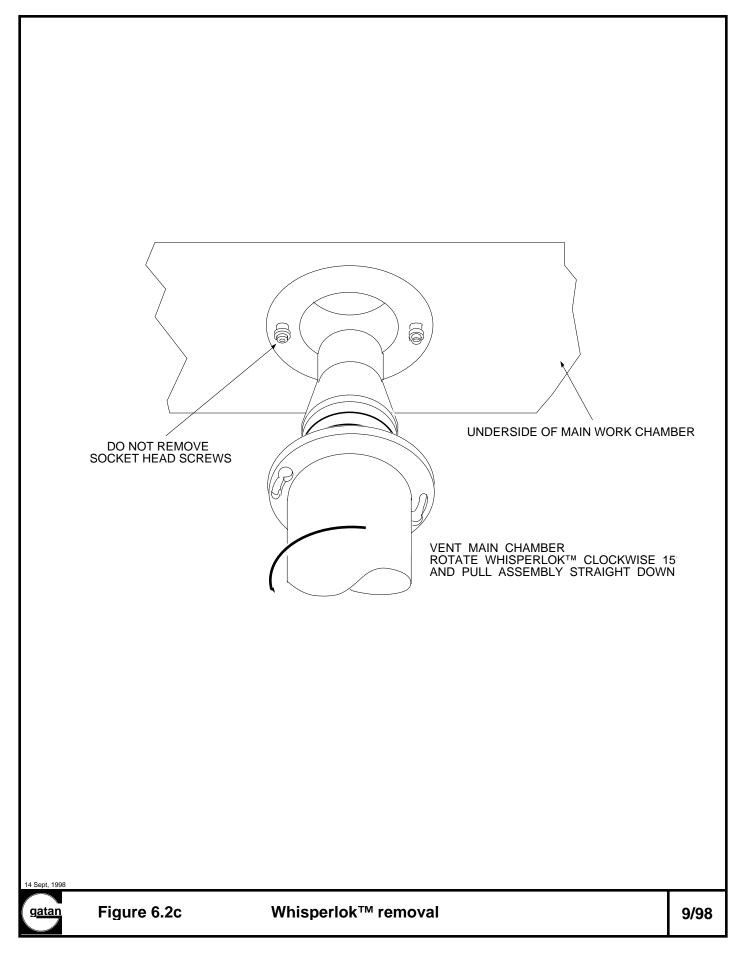
11Jan, 1989		
gatan Figure 5.5	Record of Octogun Use	1/86

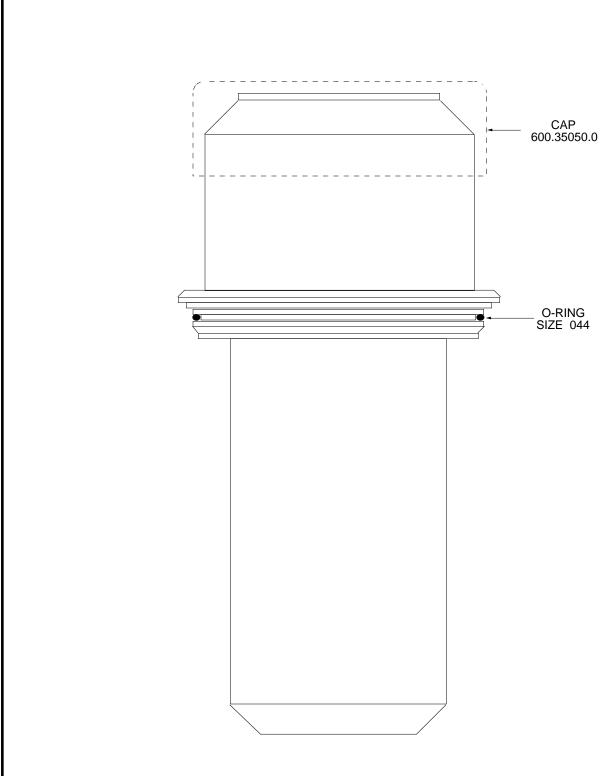












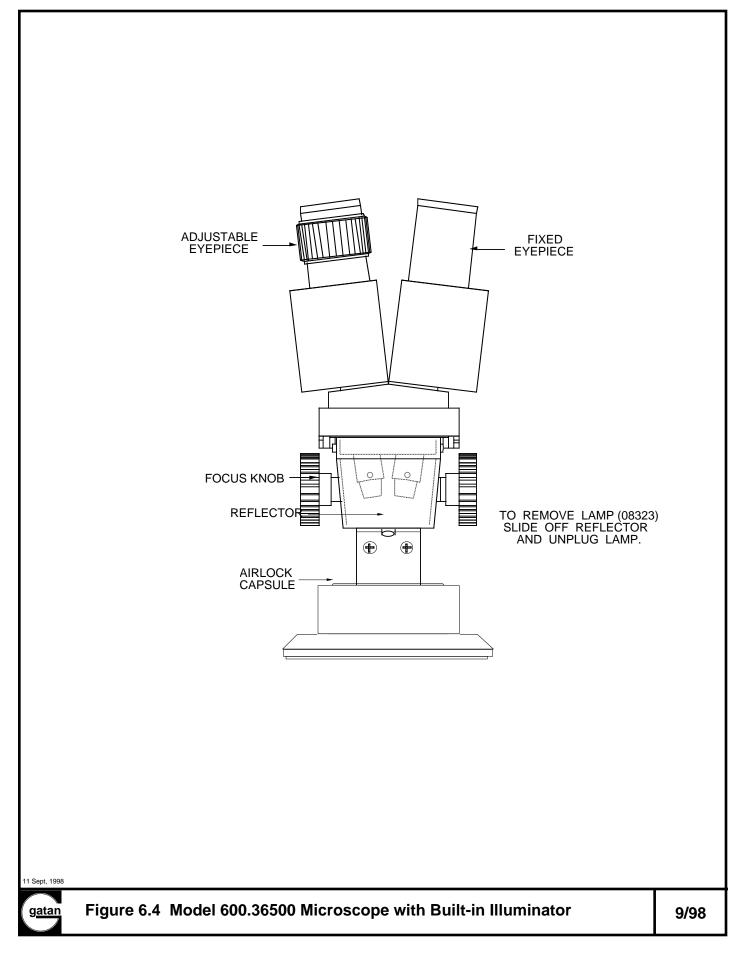
The cold trap reduces considerably the level of hydrocarbons and water vapor in the work chamber and is useful when specimen contamination by hydrocarbons must be avoided. The cold trap is installed in the top central port of the work chamber. First, close down and vent the ion mill as described in Section 5.8 and then pry up the cover to the central port and replace it with the cold trap.

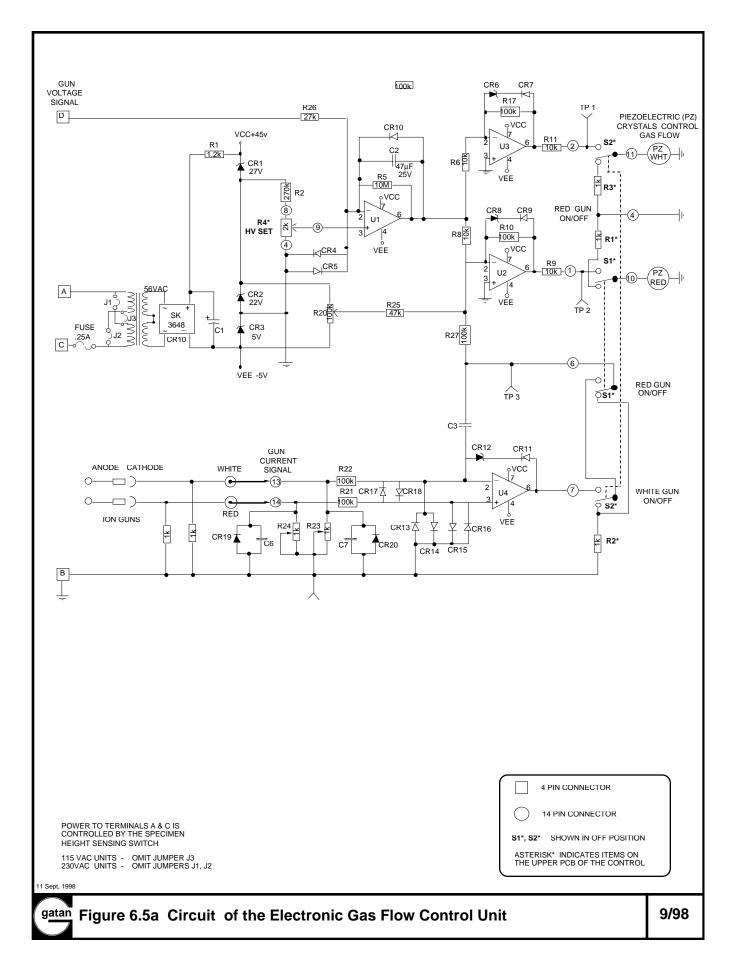
11 Sept, 1998

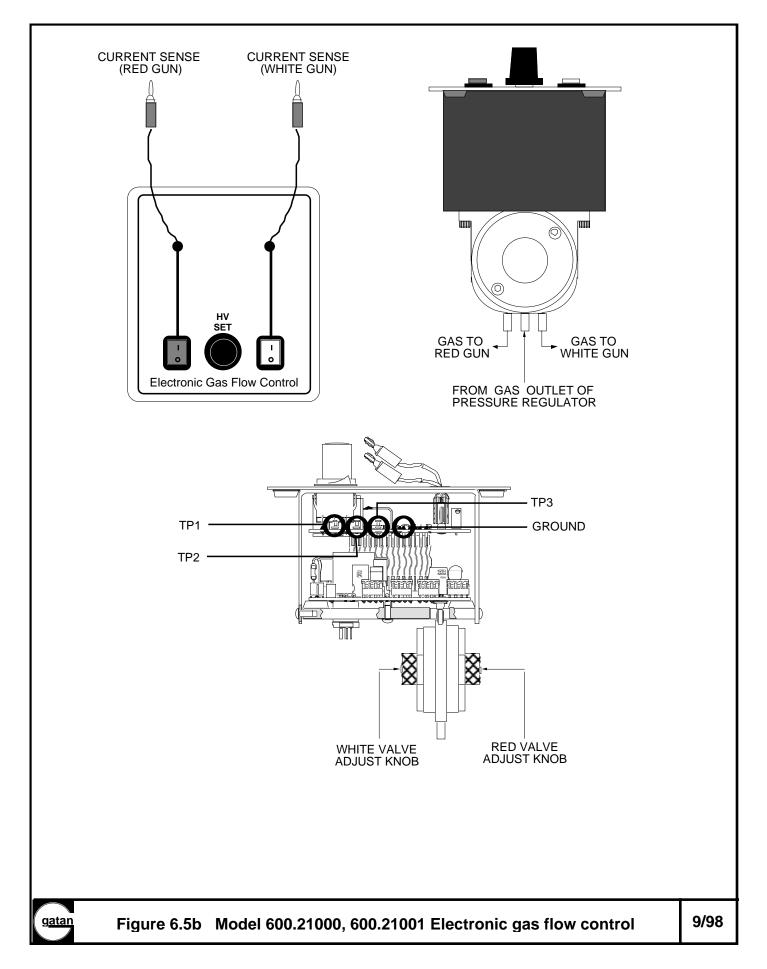
<u>gatan</u>

Figure 6.3 Model 600.35000 Liquid Nitrogen Cold Trap

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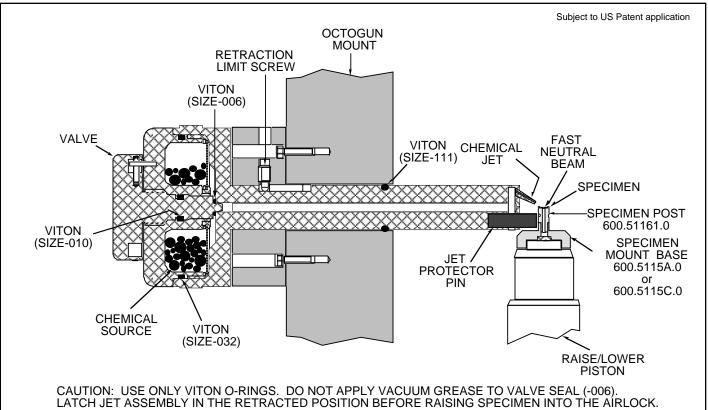


Figure 6.6a Model 600.51000 CAIBE Jet Assembly 3/99

SPECIMEN SETS
IN RECESS

SPECIMEN POST FITS
INTO SPECIMEN MOUNT
TO THIS SHOULDER

600.51161.0
SPECIMEN POST

STANDARD WHISPERLOK™ AND
600.5115B.0 — COLD STAGE AT LN₂ TEMPERATURE
600.5115C.0 — COLD STAGE RT

50 January 1999, Rev. 1

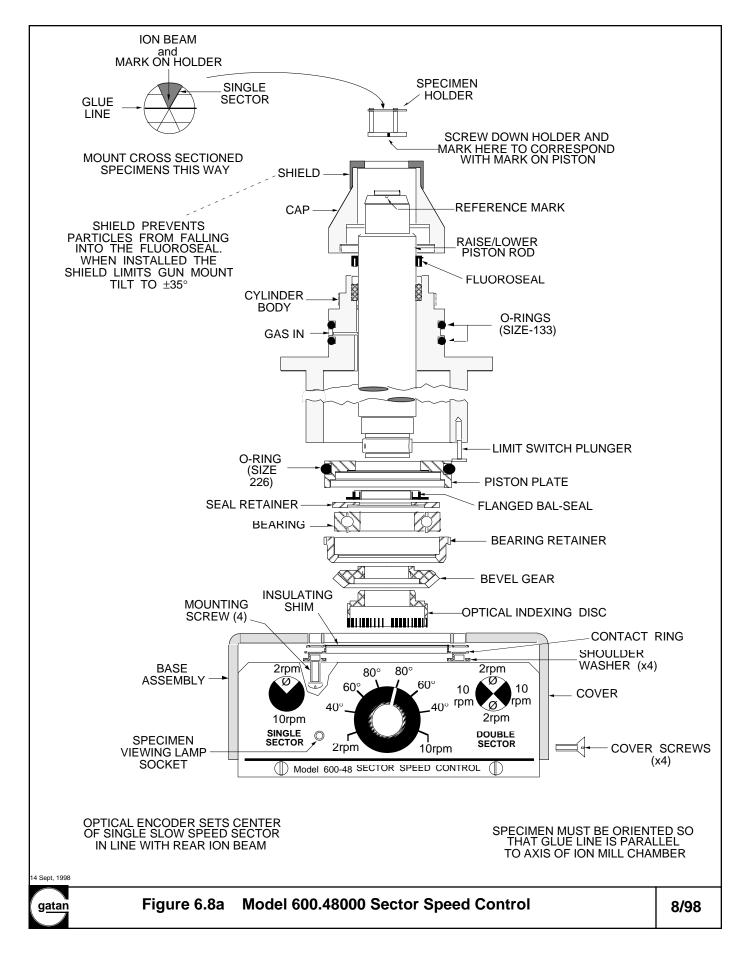
3/99

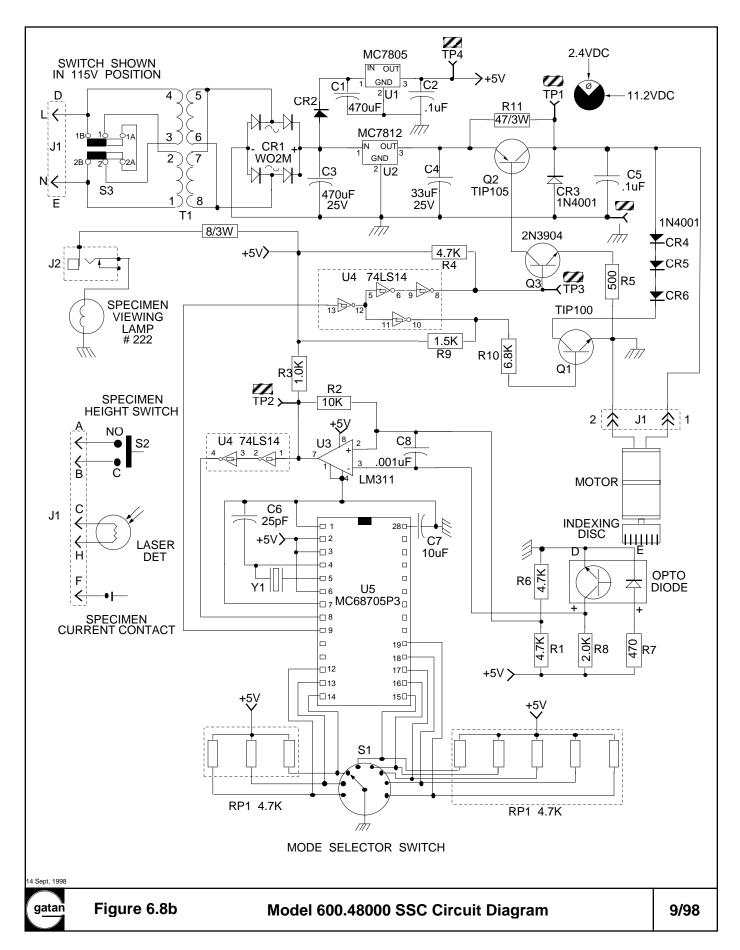
Single Sided Low Angle Specimen Mounts

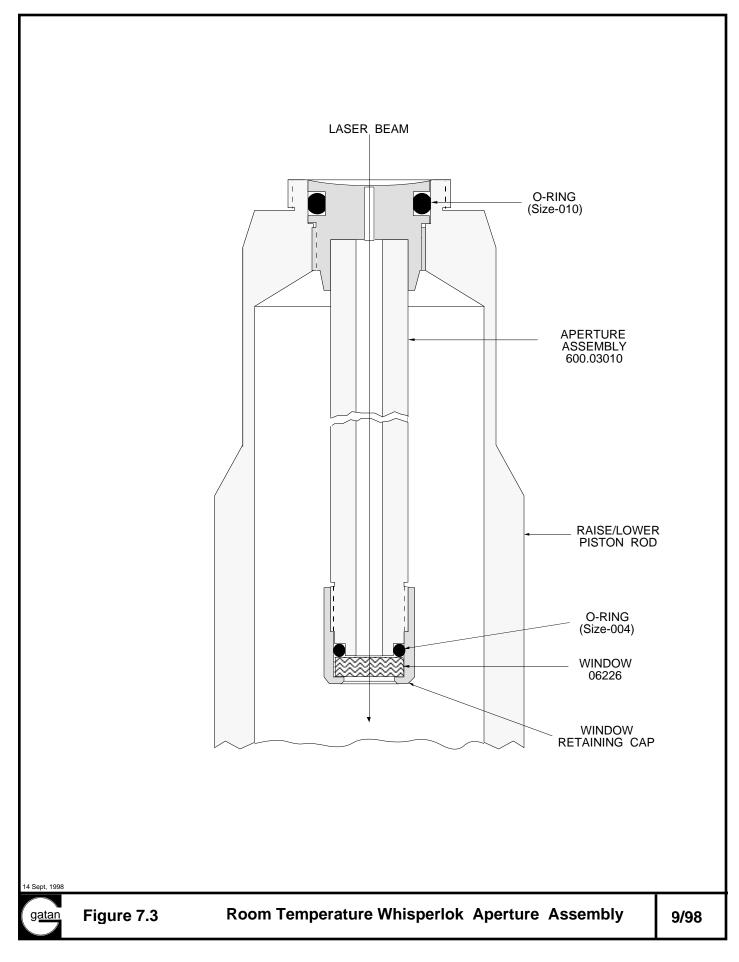
3/31/1999

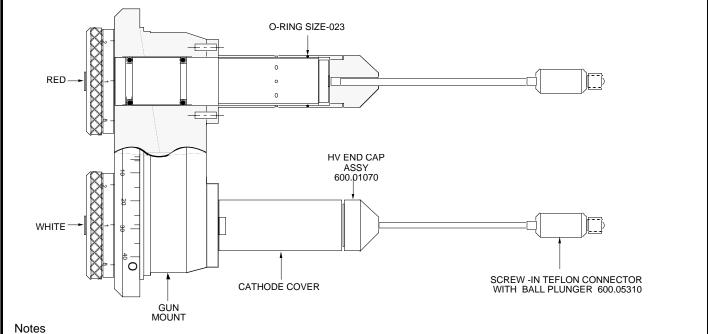
gatañ

Figure 6.6b









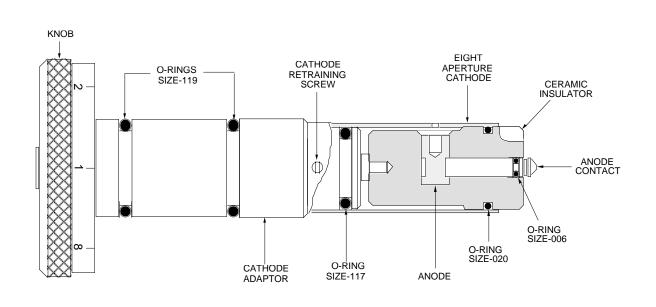
- **1.** To get the highest thinning rate it is important to align the engraved line on the Octogun precisely with the mark on the gun mounting platform.
- 2. The beam is on axis when the HV end cap assy is screwed in as far as possible and then backed off 3/4 to 1 1/4 turn where the reference marks will coincide.

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Figure 7.4a

Model 600.01002 Ion Gun Mount

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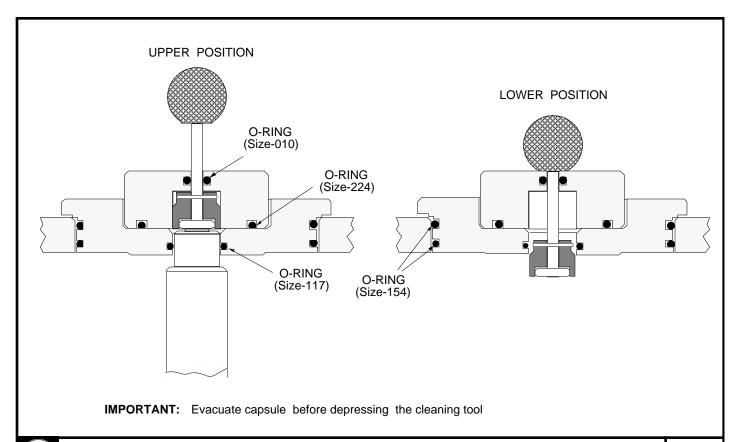
14 Sept, 1998

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Figure 7.4b

Model 600.01090 Octogun Assembly

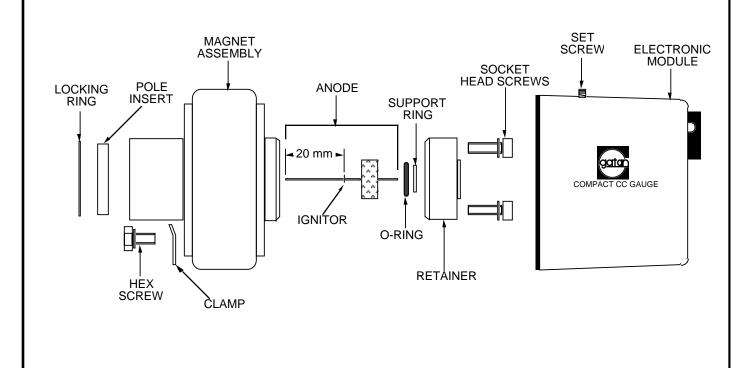
9/98



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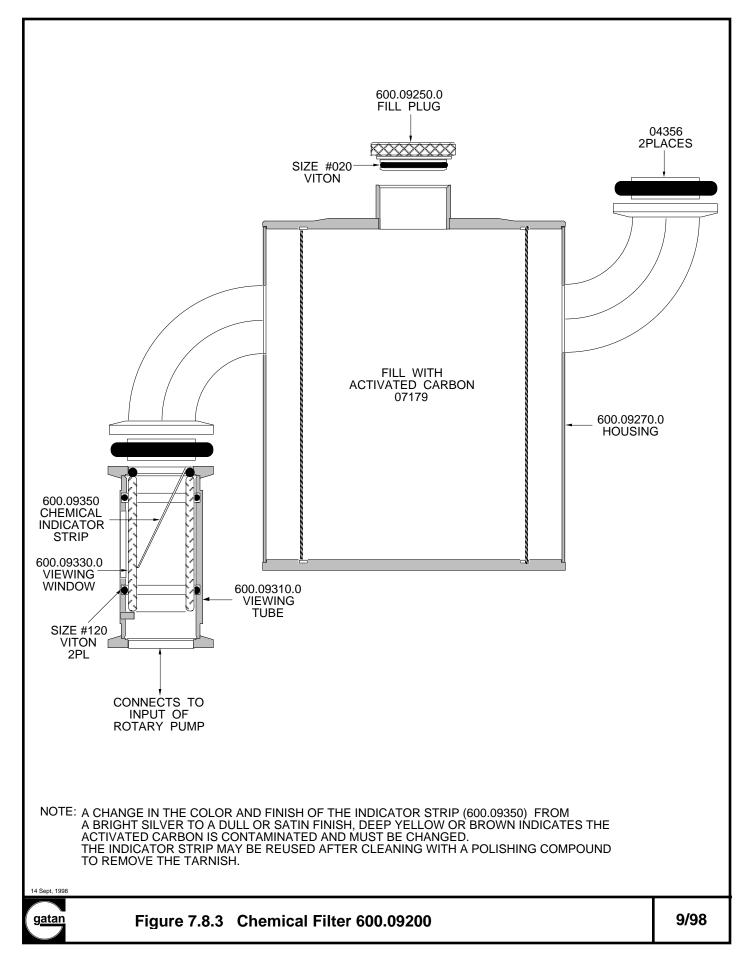
Figure 7.5 Airlock O-ring cleaning and lubrication tool

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gatan Figure 7.6 Cold cathode gauge tube

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Gatan Product Warranty

Gatan warrants that products manufactured by Gatan shall be free of defects in materials and workmanship for the warranty period, which commences at date of shipment. Gatan tests the performance of a unit as part of its final test procedure, prior to shipment from its factory. Gatan warrants that the unit meets Gatan's published specifications at time of shipment from its factory. All product warranties provide, for a period of one year after shipment to customer, parts (excluding all normal consumable, wear, and maintenance items) and labor. For Specimen Preparation Equipment and Specimen Holders, Gatan will correct any defects in the instrument either by repair in our facility or replacing the defective part, with the shipping party responsible for shipping costs. For products which attach to the column (Cameras, DigiScan, GIF, and PEELS), travel of up to 100 miles from a Gatan authorized repair center (Pleasanton, CA; Warrendale, PA; Munich, GmbH; and Corby, UK) is included. Travel expenses for service beyond 100 miles will be charged for.

Instruments, parts, and accessories not manufactured by Gatan will be warranted by Gatan for the specific items and periods in accordance with and provided by the warranty received by Gatan from the Original Equipment Manufacturer. All such accessory warranties extended by Gatan are limited in accordance with all the terms, conditions, and other provisions stated in this Original Equipment Manufacturer warranty. Gatan makes no warranty whatsoever concerning products or accessories not of its manufacture, except as noted above.

Customer Responsibilities

The customer bears the following responsibilities with regard to maintaining the warranty. The customer shall:

- Perform the routine maintenance and cleaning procedures at the required intervals as specified in Gatan's operating manuals. Failure to perform specified
 maintenance will automatically void warranty.
- 9. Use Gatan replacement parts. Failure to use the specified replacement parts will automatically void warranty.
- 10. Use Gatan or Gatan-approved consumables.
- 11. Provide Gatan authorized service representatives access to the products during normal Gatan working hours during the coverage periods to perform service.
- 12. Provide adequate and safe working space around the products for servicing by Gatan authorized service representatives.
- 13. Provide access to, and use of, all information and facilities determined necessary by Gatan to service and/or maintain the products. (Insofar as these items may contain proprietary or classified information, the customer shall assume full responsibility for safe-guarding and protecting them from wrongful use.)

Repairs and Replacements

Gatan will, at its option, either repair or replace defective instruments or components with conforming goods. Repair or replacement of products or parts under warranty does not extend the original warranty period. With the exception of consumable and maintenance items, the replacement parts or products used on instruments out of warranty are themselves warranted to be free of defects in materials and workmanship for 90 days.

Any products, part, or assembly returned to Gatan for examination or repair shall have Gatan's prior approval, with the customer requesting 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. If the item is not under warranty, to obtain an RGA, the customer must provide a Purchase Order (PO) for the repair. 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 be required. A credit against this PO will be issued by Gatan upon receipt of the item as 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 item is under warranty, return transportation will be prepaid by Gatan. If the returned item is not under warranty, return transportation will be charged to the customer.

Warranty Limitations

The warranty does not cover:

- 1. Parts and accessories which are expendable or consumable in the normal operation of the instrument.
- 2. Any loss, damage, and/or instrument malfunction resulting from shipping or storage, accident (fire, flood, or similar catastrophes normally covered by insurance), abuse, alteration, misuse, neglect, or breakage or abuse of parts by User.
- Operation other than in accordance with correct operational procedures and environmental and electrical specifications.
- 4. Performance to specifications or safety of use (including X-ray emissions) if the unit is physically installed on, used in conjunction with, or used as part of a third party's equipment and is not installed by a Gatan service engineer.
- 5. Performance to specifications or safety of use (including X-ray emissions) as a result of the use of Gatan's equipment with that of a third party due to the third party's product design.
- 6. Modification of, or tampering with, the system.
- 7. Improper or inadequate care, maintenance, adjustment, or calibration by User.
- 8. User-induced contamination or leaks.
- 9. Any loss, damage, and/or instruments malfunction resulting from use of User-supplied software, hardware, interfaces, or consumables other than those specified by Gatan.

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 90-day warranty unless otherwise specified.

Post Warranty Period Support and Product Obsolescence

After the expiration of the warranty period described above, 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 repair. Gatan will continue to provide billable service support for the products for a period of three years after discontinuance or design obsolescence by Gatan. After this three year period, service support will be offered at the sole discretion of Gatan.

Liability Limitations

This warranty is in lieu of and excludes all other expressed or implied warranties, including (but not limited to) warranties of merchantability of fitness for a particular purpose. Under no circumstances will Gatan Inc. or Gatan International be liable for any direct, indirect, special, incidental or consequential damages (including lost profit) or loss of any kind, whether based on warranty, contract, tort, or any other legal theory. The limits of Gatan liability in any dispute shall be the price received from the purchaser for the specific equipment at issue. The laws of the state of Pennsylvania apply to all aspects of this warranty.

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DECLARATION OF CONFORMITY

(According to ISO/IEC GUIDE 22 and EN 45014)

Manufacturer's Name: Gatan Inc.

Manufacturer's Address: 5933 Coronado Lane

Pleasanton, CA 94588 U.S.A.

DECLARES THAT THE PRODUCT

Product Name: DuoMill

Model Number: 600

Serial Numbers:

CONFORMS TO THE FOLLOWING EUROPEAN DIRECTIVES

Low Voltage Directive 73/23/EEC EMC Directive 81/336/EEC As Modified by Directive 93/68/EEC

Supplementary Information:

Safety: EN 61010-1:1991/A2:1995

EMC: EN 55022:Class A/Class B

EN 50082-1:1992 EN 50081-1:1992

I, the undersigned, hereby declare that the equipment specified above conforms to the above Directives and Standards.

Place: Pleasanton, CA Signature:

<u>Date:</u> November 1998 <u>Full Name:</u> Robert Buchanan

Position: President and CEO

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