CRESSINGTON SPUTTER COATER 108auto

7002, 7002-220

Operation Manual

TED PELLA, INC.

Microscopy Products for Science and Industry

WARNING: If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

DISCLAIMER:

Ignorance of the warnings contained in this operation manual, that is, use of the Cressington Sputter Coater 108auto in a manner that exceeds the given warnings, is an action that falls outside the specifications for the use of the equipment and voids the warranty. In such a case, the distributor and manufacturer disclaim any and all liability (whether based in contract tort, strict liability, or otherwise), for any damages whatsoever, including direct, incidental, consequential, loss of business profits, or special damages, resulting from the misuse of these products, even if the distributor/manufacturer has been advised of the possibility of such damages.

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Section 1: **General**

1.1 Operational Features

The Cressington 108 Auto Sputter Coater is designed primarily for sputtering conducting layers on to non-conductive samples to prevent charging effects in the scanning electron microscope. It uses a planar magnetron sputter target configuration to give efficient high rate sputtering with minimal specimen heating.

The 108 Auto is designed to operate in *automatic* or *manual* modes. Automatic operation gives a complete coating cycle using preset values for pressure, current and time. Manual operation is used for more difficult samples when a wider range of operating conditions is required.

The 108 Auto is fitted with separate argon gas circuits for (1) precision leaking and (2) flushing. This feature is important for the production of high quality stress-free coatings of Au/Pd, Pt/Pd, Au, Pd, and Pt. The system does not sputter Cr, Ni, Ag, Cu, Ir, or Ta.

The Cressington 108 Auto is designed as an integrated vacuum system with its desktop pumping system matched to the coating unit for optimum performance. The production of high quality coatings requires efficient gas handling in the range 0.05mb to 0.1mb.

In the past, sputter coaters for SEM have used the gas pressure control valve to adjust the sputtering current. The Cressington 108 Auto does not operate in this way:- gas control and current control functions have been separated. This allows the user to independently adjust operating pressure *(mb)* and sputter current *(mA)* to obtain the best sample conformity and minimum grain size. The rotary-planetary-tilting sample stage together with variable height sample chamber allows further optimization of the coating.

The Cressington 108 Auto can be factory fitted (or retro-fitted) with the optional MTM-20 thickness monitor system.

1.2: Services Required

The coater requires a single phase a.c. electrical supply from a standard laboratory power point. It is available in 100/120V and 200/240V versions.

The serial number label on the back panel of the control unit is marked with the supply voltage requirement.

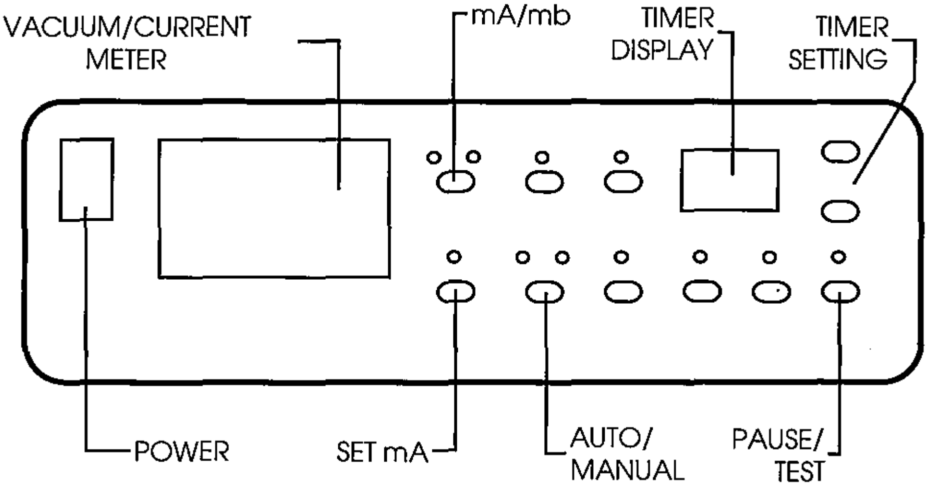
The coater requires a supply of argon gas from a high pressure gas container. The gas purity must be at least *"arc welding grade"* and its supply pressure reduced to 0.3-0.6bar (5-9 psi) by using a pressure reducing regulator valve. Connection requires clear plastic hose (6.0mm internal diameter).

Section 2: **Description**

2.1.1 Control Panel: *General*

The main *POWER* switch turns on the pumping system and the vacuum gauge. The vacuum chamber pressure *(in mb)* is indicated on the lower scale of the meter. At pressures above 0.4mb the sputter supply is disabled and the *mb* light will flash until a pressure below 0.4mb is achieved.

Meter scale changing is automatic and the upper scale will indicate sputter current *(in mA)* when the sputtering supply is energized. A manual over-ride switch *(mA/mb)* is used for momentary change-over between the scales; indicator lights show which scale is selected.



On initial power-up the *AUTO/MANUAL* selector will be in *AUTO*. The switch allows toggling between the two modes.

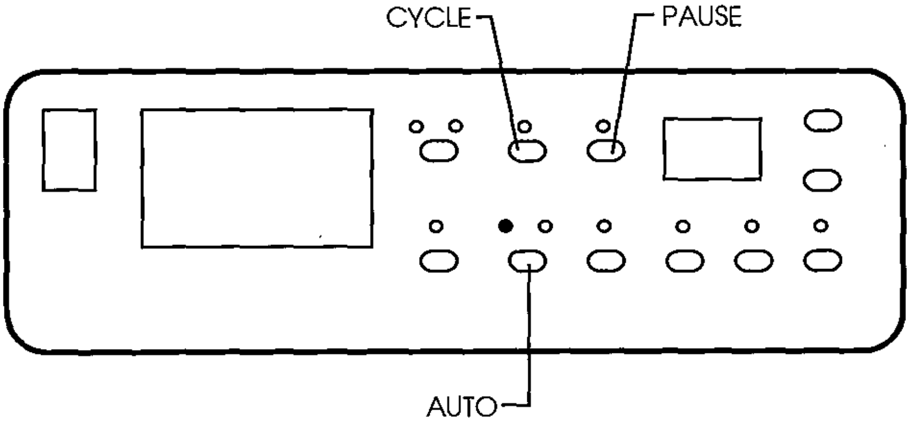
The preset coating time (for automatic and manual modes) is shown on the digital display. It is adjusted by holding down the *PAUSE/TEST* switch and using the setting switches to increase or decrease the displayed value.

The preset power output is checked by pressing *SET mA*. The selected coating current will then be shown on the digital display. It can be adjusted by holding down the *SET mA* switch and toggling between the four available values (10mA, 20mA, 30mA, 40mA) using the timer setting switches.

When a MTM-20 thickness controller is fitted the thickness control mode is selected by simultaneously pressing BOTH the timer setting buttons. The timer display will then show the status of the controller. When both the buttons are pressed again, the coater will revert to timer mode.

2.1.2 Control Panel: *Automatic Operation*

On initial power-up the *AUTO/MANUAL* selector will be in *AUTO*. Pressing *CYCLE* starts a sequence of actions as follows:-

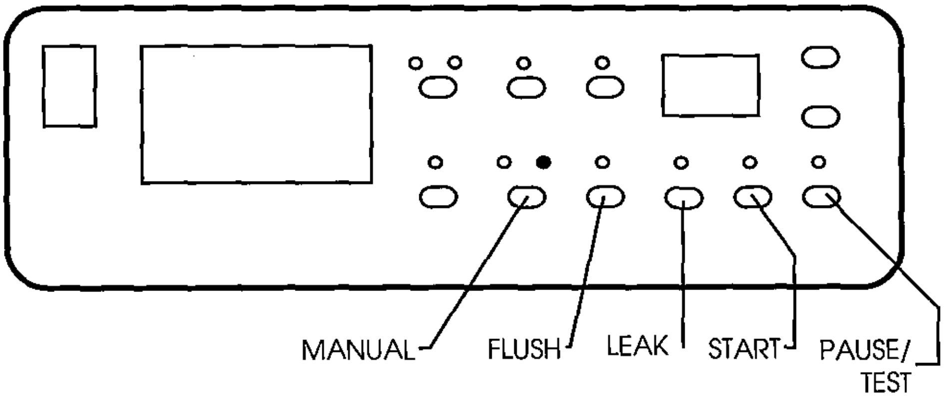


(1) pump to 0.05mb; (2) open argon flush valve for 3 seconds, then close; (3) pump to 0.15mb; (4) open argon flush valve for 3 seconds, then close; (5) open leak valve; (6) pump to 0.15mb and continue pumping for further 20 seconds; (7) switch on sputter supply at preselected current; (8) switch off sputter supply after preselected time.
During the *CYCLE* sequence, the status of the coater is indicated by the LED lights above the manual control switches.

If *PAUSE* is selected during the flushing sequence it will cause a flush to be repeated when *PAUSE* is cancelled. If *PAUSE* is selected while coating, the sputter supply will temporarily be switched off. The maximum pause length is 240 seconds.

2.1.3 Control Panel: *Manual Operation*

In *MANUAL*, the individual functions of *FLUSH*, *LEAK* and *START* can be selected separately. Sputtering is initiated by selecting *LEAK* and then *START*. It can be temporarily halted using *PAUSE/TEST*.



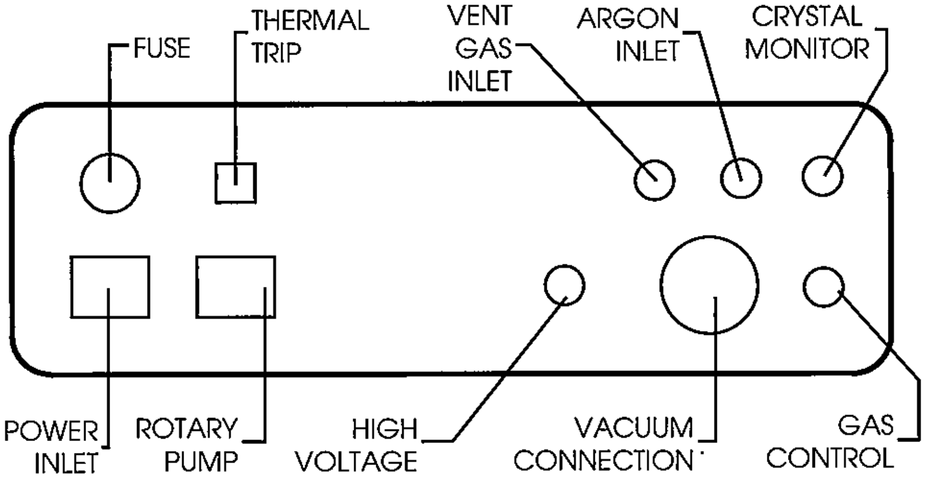
If *PAUSE/TEST* is pressed while *LEAK* is selected the sputter supply will be switched on while the switch is held.

In order to conserve argon, *FLUSH* will time-out after 60 seconds and *LEAK* will time-out after 240 seconds if inadvertently left on.

2.2 Rear Panel

Electrical power to the coater is via the *MAINS POWER INLET* socket. A power cable is provided. The *FUSE* protects the control unit but not the rotary pump (which is separately protected).

The rotary pump is powered from the *ROTARY PUMP POWER* connector port. It is protected by a resettable *THERMAL TRIP.* The pumping port is marked *ROTARY PUMP VACUUM CONNECTION* and has the standard KF16 format to accept the stainless steel flexible hose.



The high voltage power cable from the rear panel *(SPUTTER SOURCE)* to the sputtering head is permanently wired for safety. The output is also controlled by a protection circuit linked to the Pirani vacuum gauge:- the high voltage output cannot be energized until the vacuum is better than 0.4mb. (If the Pirani gauge becomes faulty the supply will not energize and the fault condition will be indicated by the *mb* light flashing and the vacuum meter needle being off-scale in either direction).

The rear panel *ARGON GAS INLET* is connected internally to the gas control valve (marked *"needle valve adjustment, auto models"*) The argon passes from the control valve to the chamber inlet under the baseplate. The *ARGON GAS INLET* should be connected externally (using a 6.0mm internal diameter plastic hose) to a regulated supply of argon (see Section 3.2).

The rear panel *VENT GAS INLET* can be connected to a *separate* supply of dry gas (nitrogen or argon) or to a drying column for air. Care should be exercised when using a gas supply: - the vent valve is the *normally open* type and will allow a permanent flow of gas until the gas supply is turned off.

A rear panel connector position is provided to allow the optional MTM-20 *CRYSTAL MONITOR* to be fitted. (Not shown)

2.3 Vacuum Pumping System

The rotary vacuum pump is sited to the rear of the control unit on a vibration isolated base. It pumps the internal vacuum system via a short stainless steel flexible hose. External vacuum connections are made using the international KF system.

The pump exhaust is filtered to prevent oil mist droplets from diffusing into the laboratory environment.

Inside the control unit, directly underneath the chamber baseplate, is a vacuum manifold with three ports. These ports accept: (1) the Pirani vacuum gauge head, (2) the argon gas supply from the gas control valve and the vent gas supply from the rear panel, (3) the pumping line from the rear panel.

The pumping path from the pump inlet to the sputtering chamber has an unusually high conductance. This gives a rapid pumpdown from atmosphere (less than 2 minutes). It also results in efficient argon gas handling in the pressure range 0.1mb to 0.05mb.

2.4 Chamber and Specimen Table

The cylindrical vacuum chamber (Ø120mm OD x 120mm high x 7.0mm wall) is made from high quality glass. After processing it is annealled to relieve manufacturing stresses. The vacuum seals at each end of the glass cylinder are made by O-rings recessed into the baseplate and top-plate.

The chamber top-plate is attached by a bracket to a support pillar. The bracket is hinged to allow the top-plate to be lifted for access to the chamber. The top-plate incorporates a Planar Magnetron Sputter source for Ø57mm targets. Electrical connection to the deposition source is underneath the painted cover.

A sample table is mounted on a threaded pillar screwed through the centre of the baeplate clamping disc. The table is raised or lowered by loosening the lower locking nut and rotating the table (1 turn equals 1mm).

The baseplate contains a feedthrough port suitable for mounting a thickness monitor feedthrough. If the thickness monitor is not supplied this port is blanked.

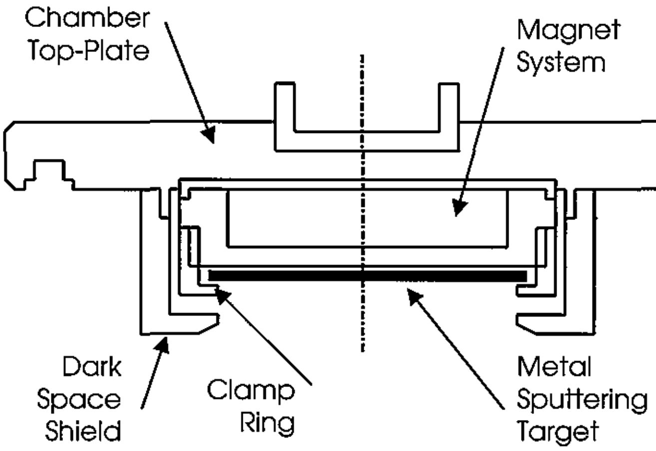
The sample table has a larger hole for mounting the crystal monitor head of the optional thickness monitor system.

2.5 Magnetron Sputter Head

In a diode sputtering system the deposition material is electrically connected to be the cathode of a gas discharge. The discharge current is carried by both gas ions and electrons. Positive gas ions are accelerated in the electric field towards the negative sputter target. When they strike the target the gas ions eject target atoms by momentum transfer. This process is called *sputtering.* The sputtered target atoms then diffuse away from the target and will deposit on any nearby surface.

Sputtering has the specific advantage for coating SEM samples that, if a suitable gas pressure is chosen, the sputtered material arrives at the sample with random direction. This gives the coating good conformity to the sample surface without the use of sample stage movements.

Modern sputtering systems use target arrangements, which include magnetic circuits to enhance the intensity of the discharge and keep the electrons captive in the target area (to reduce heating effects at the sample).
The Cressington planar magnetron sputtering source is shown above. The metal foil target (57mm dia x 0.1mm thick) is held by a threaded clamp ring. The dark space shield must be removed before the target can be changed.



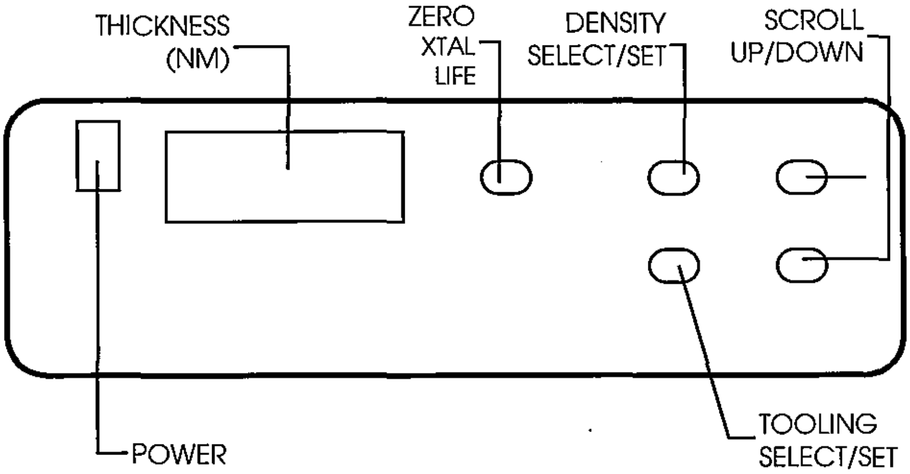
In a magnetron arrangement the sputtering is most intense where the magnetic field is most concentrated. The *effective size* of the present target is about 30mm at center of target.

The most efficient operating pressure for the head (in terms of *amount of material deposited*) is around 0.05mb but this is not the pressure for optimum sample coating by gas scattering. In order to allow a choice of operating conditions the Cressington sputtering supply has been designed to give *constant sputter current independent of gas pressure.*

2.6 Quartz Crystal Thickness Monitor (Optional)

The MTM-10 thickness monitor works on the principle of the *quartz crystal microbalance. When* sputtered material is deposited on an oscillating quartz crystal its frequency is decreased in relation to the mass of material deposited. The frequency change can be used to calculate the film thickness if the density *(gm/cm^3)* of the material is known.

The complete thickness monitor outfit comprises: (1) a crystal head mounted in a special sample table, (2) a vacuum feedthrough mounted in the chamber baseplate, (3) an oscillator unit mounted under the baseplate, (4) connecting cables and adaptors, (5) an MTM-10 control box. The front panel of the control unit is shown below.



The thickness monitor calculates the thickness of deposit every tenth of a second and displays the result in nanometers on the digital *THICKNESS* display. It detects the frequency shift and uses the *DENSITY* and *TOOLING* values keyed into memory. Four sets of values can be stored under *DENSITY* and *TOOLING.*

To display the currently selected value of material density or tooling factor, press the relevant key and the value will appear on the *THICKNESS* display. To select another of the four values scroll up or down to the other values using the scroll keys. To adjust that value, hold down the relevant key for two seconds until the period flashes and scroll up or down using the scroll keys.

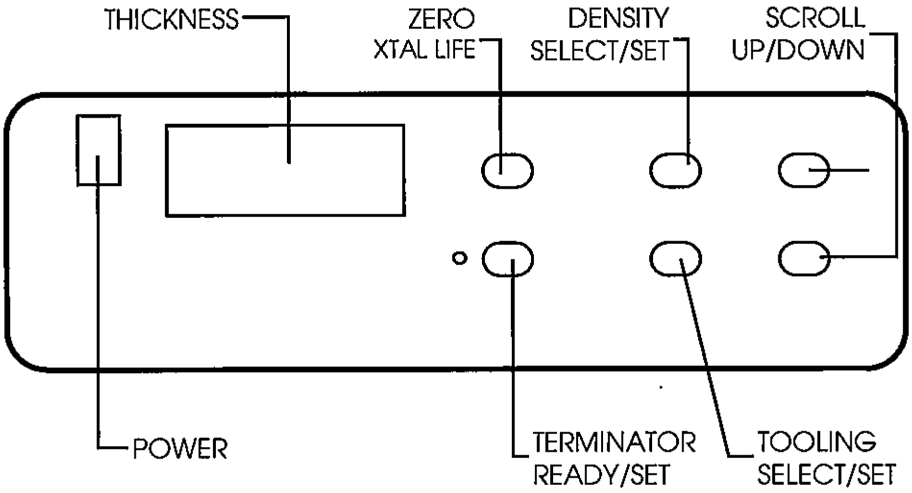
The measuring crystal can only accept a finite amount of sputtered material before becoming overloaded. The amount of offset from its original frequency (6.0MHz) is displayed (in KHz) when *XTAL LIFE* is pressed for two seconds. The expected life of a crystal will depend on the material and the level of stress in the sputtered deposit. When the crystal ceases to oscillate the display shows *"FAIL".* To reset the thickness between runs press *ZERO.*

The thickness of film required to prevent charging is very dependant on the sample and the operating voltage in the SEM. A thickness of between 3nm and 5nm will usually prevent charging. The thickness monitor is a valuable aid for standardizing the conditions for obtaining the optimum coating: Thin enough not to obscure fine detail but thick enough to prevent charging.

2.7 Quartz Crystal Thickness Controller (Optional)

The MTM-20 thickness controller works on the principle of the *quartz crystal microbalance* (see previous page). The complete thickness controller outfit comprises:- (1) a crystal head mounted in a special sample table, (2) a vacuum feedthrough mounted in the chamber baseplate, (3) an oscillator unit mounted under the baseplate, (4) connecting cables and adaptors, (5) an MTM-20 control box. The front panel of the control unit is shown below.

The thickness controller calculates the thickness of deposit using the *DENSITY* and *TOOLING* values keyed into memory and displays the result in nanometers on the digital *THICKNESS* display. The measured *THICKNESS* is compared with the *TERMINATOR* set point and disables the coating process (puts *TERMINATOR READY/SET* status = 0) if *THICKNESS* is equal to or greater than *TERMINATOR* set point.



To display the current film thickness termination press *TERMINATOR* for two seconds. To adjust, hold down the key and scroll up or down using the scroll keys. To disable (0) or enable (1) termination press *TERMINATOR READY/SET.* To display the currently held value of material density or tooling factor held in memory press and release the relevant key. The value will appear on the digital *THICKNESS* display. Four sets of values can be stored under *DENSITY* and four under *TOOLING.* To select another of the four values scroll up or down to the other values using the scroll keys. To adjust a value, hold down the relevant key for two seconds until the period flashes and then scroll up or down using the scroll keys. To reset the display to zero after taking a measurement press *ZERO* and release.

The *TERMINATOR READY/SET* button allows toggling between armed "1" and disarmed "0". When the MTM-20 is wired to the *108auto* the status will be displayed in the timer display (as "t 0" or "t 1") if the controller mode has been selected. To select controller mode press the two timer adjust buttons simultaneously. To reselect timer mode press the toggle buttons again.

The measuring crystal can only accept a finite amount of sputtered material before becoming overloaded. The amount of offset from its original frequency (6.0MHz) is displayed (in KHz) when X*TAL LIFE* is pressed for more than 2 seconds. The expected life of a crystal will depend on the material and the level of stress in the sputtered deposit. When the crystal ceases to oscillate the display shows *"FAIL".*

Target material	Atomic number	Density in gm/cm^3
Cu	29	8.96
Pt/Pd (80/20) (Cressington)	78/46	19.52
Au/Pd (80/20) (Cressington)	79/46	17.84
Au/Pd (60/40)	79/46	16.38
Ni	28	8.90
Pd	46	12.0
Pt	78	21.45
Au	79	19.30

Table 1. Density values for target materials.

The Tooling Factor is used to correct the measured thickness on the crystal head to the actual deposited thickness on the sample. The measured thickness can be different for a variety of reasons:

- the density of thin layers is mostly lower than for solid block material
- there might be a difference in distance to the target for the crystal head and the sample.
- there might be a difference in angle for the crystal head and the target.

If the crystal head is further away from the target, the thickness on the sample is most likely more than measured: use a tooling factor > 1.

Independent calibrated measurements with either step height (AFM) or cross section (SEM) are recommended to determine the correct Tooling Factor for sputtering processes.

3.1 Unpacking

The complete system, (control unit and pumping system) occupies a bench area 420mm wide x 600mm deep (18 x 24") on a standard laboratory bench. The vacuum pump is placed behind the control unit. After clearing the designated area, unpack the control unit and place it on the bench. Remove the packing materials from the sputter chamber baseplate and top-plate.

IMPORTANT
DO NOT ALLOW ANY PACKING MATERIAL TO ENTER THE BASEPLATE PUMPING PORT. TAKE EXTRA CARE WHEN UNPACKING AND HANDLING GLASS CHAMBER COMPONENTS.

Unpack and identify the components of the pumping system:

(1) Rotary pump on anti-vibration base (typical Pfeiffer DUO 2.5);
(2) Stainless steel flexible hose;
(3) KF16 O-ring seals and clamps;
(4) Exhaust mist filter; and
(5) Vacuum pump oil.

3.2 Assembly

3.2.1 Pumping System

Unscrew the hose fitting from the rotary pump exhaust and screw in the exhaust filter.

Fill the rotary pump with oil (according to the manufacturers instructions).

Position the pump behind the control unit with the motor to the left.

Remove the blanking cap from the rear of the control unit. Check that the sealing surfaces and KF16 seals. Connect the rear pumping port of the control unit to the rotary pump inlet using the stainless steel hose (curved into an *"S "* shape) and the two KF16 o-ring carriers and clamps. Ensure that the hose is not under stress when in its final position. Make sure that the power switch on the vacuum pump is in the "ON" pos.

3.2.2 Dual Pumping System

Place the 108 Auto (sputter coater) to the left of the 108 Carbon Auto with the rotary pump placed centrally behind the two coaters. Connect the ball valve to the rotary pump with the lever opposite the pump inlet. Connect the rear pumping ports of the control units to the remaining ports on the ball valve using the stainless steel hoses and the elbow with the 108 Auto (illustrated below).

Section 3: **Installation**

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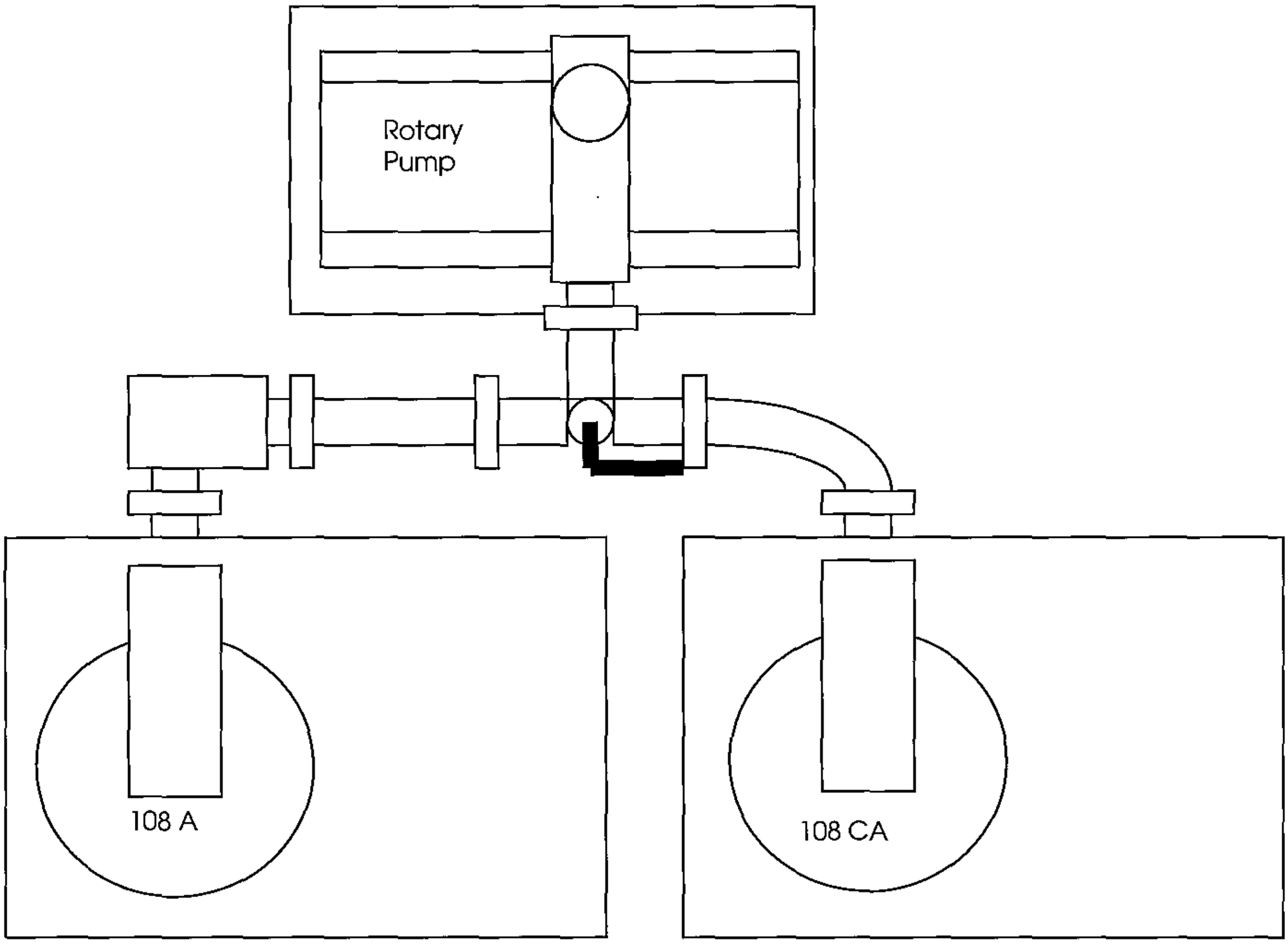
Plug the 108 Auto control unit into the socket on the back of the 108 Carbon Auto control unit. Plug the rotary pump power cord into the socket on the back panel of the 108 Auto control unit. Plug the power cord for the 108 Carbon Auto control unit into a suitable power outlet.



3.2.3 Sample Preparation Chamber

Wipe the insides of the glass cylinder with a lint free tissue (or cloth) that has been moistened with alcohol. The top-plate, which is already fitted with a target, can now be gently lowered. The system is now ready for pumping.

IMPORTANT

BEFORE LOWERING THE TOP-PLATE

CHECK THE POSITION OF THE GLASS CYLINDER. IF THE CHAMBER COMPONENTS ARE NOT CENTRERED THE TOP-PLATE COULD CHIP THE GLASS.

3.2.4 Electrical Connections

Unpack the thickness monitor or control unit (if supplied) together with the electrical cables.

Place the monitor/controller on the main control unit (to the right of the baseplate). It derives its signal(s) from the main unit.

Identify the following loose electrical leads (if supplied):

(1) Power to main control unit (IEC to mains wires);
(2) Power to thickness monitor/controller;
(3) Thickness signal to thickness monitor/controller (BNC connectors); &
(4) Control signal to thickness controller (D connectors).

Plug the rotary pump power cord into the labeled socket on the back panel of the control unit.

Connect the remaining cables as appropriate.

Fit a suitable 3-pin plug to the power cord for the control unit (and the thickness monitor/controller if supplied).

IMPORTANT
THE POWER CORD FOLLOWS THE EUROPEAN CONVENTION. THE PLUG SHOULD BE WIRED AS FOLLOWS: *LIVE* = BROWN; *NEUTRAL* = BLUE; *GROUND* = GREEN / YELLOW.

IMPORTANT
BEFORE SWITCHING ON CHECK THE LABELS ON THE CONTROL UNITS AND ROTARY PUMP TO ENSURE THAT THE SUPPLY VOLTAGE IS SUITABLE.

3.2.5 Argon Gas Supply

IMPORTANT
EXPERT ASSISTANCE SHOULD BE SOUGHT WHEN DEALING WITH HIGH PRESSURE GAS CYLINDERS.

Position a cylinder of pure argon gas close to the 108 Auto sputter coater. The cylinder should be fitted with a pressure reducing regulator valve. The regulator gauge must indicate pressure (not FLOW) and be adjustable in the range up to 1 bar. (It will need to be adjusted to give 0.3/0.6 bar (5-9 psi) during "flushing").

Connect the regulator outlet to *ARGON INLET* on the back panel of the control unit using 6.0mm (internal diameter) clear plastic hose. The hose between the regulator valve and the *ARGON INLET* should be as short as possible. Care should be taken to ensure that the hose has no leaks, which might admit atmospheric air.

IMPORTANT
ALTHOUGH GOLD CAN BE SPUTTERED USING AIR IT IS AN INEFFICIENT PROCESS AND LEADS TO LARGE GRAIN AND SAMPLE HEATING. WHEN SPUTTERING ANY OTHER TARGET MATERIAL ARGON MUST BE USED.

If a dry venting gas is required this should be connected to the *VENT GAS INLET*. Do not use the same argon supply as for *ARGON INLET* as it will be necessary to turn the supply off every time after venting. A more convenient solution is the use of atmospheric air together with a drying column.

Section 4: **Test**

4.1 Vacuum System

IMPORTANT
BEFORE SWITCHING ON CHECK THE LABELS ON THE CONTROL UNITS AND ROTARY PUMP TO ENSURE THAT THE SUPPLY VOLTAGE IS SUITABLE.

Connect the main power cord to the control unit. Check the chamber is correctly closed. Switch on *POWER* on the front panel of the control unit. The vacuum pump will start to pump down the chamber.

The pump has a high rotational speed (3000rpm @ 50Hz or 3600rpm @ 60Hz) and under cold conditions (below 60°F/15°C) the pump may not reach full speed immediately. Confirm that the chamber is pumping down and allow the pump to warm up for 5 minutes before testing the pumping performance.

Switch off *POWER* and the chamber will automatically vent. Lift and replace the top-plate to confirm that the chamber has been fully vented.

Switch on *POWER* and check the pumping time to 0.1mb. This should be about 30sec.

IMPORTANT
THE PRESENCE OF SOLVENTS OR CONTAMINANTS WILL LENGTHEN THE TIME TAKEN TO REACH A GIVEN PRESSURE. IF A LEAK IS SUSPECTED THE GLASS CYLINDER SHOULD BE REMOVED AND THE TWO O-RING SEALS INSPECTED FOR DEFECTS OR SMALL OBJECTS.

Allow to pump for a further 5 minutes and check that the vacuum is around 0.01mb. Switch off the control unit and vent the chamber.

4.2 Argon Gas System

Switch on the coater and pump to better than 0.02mb. Close the gas control valve on the rear panel (clockwise rotation looking from rear).

Open the regulator valve on the argon cylinder until the pressure reads 0.3bar (5psi).

Select *MANUAL* and *FLUSH* and let argon flush through the system for about 10 seconds. Close the FLUSH valve and allow the system to recover to 0.02mb. The flushing system is now primed with argon.

Select *MANUAL* and *LEAK* and wait for the unit to repump. Slowly open the gas control valve until the chamber pressure rises to more than 0.3mb and let argon flush through the control valve circuit for about a minute. The *LEAK* system is now primed with argon. Close *LEAK* and allow the system to recover to 0.02mb.

Fully close the regulator valve on the argon cylinder. Open the *FLUSH* valve. Check that the vacuum recovers to 0.02mb. This verifies that the argon supply circuit is leaktight between the regulator and the coater. Close *FLUSH* and open *LEAK* (the gas control valve should still be wide open). Check that the vacuum recovers to 0.02mb. This verifies that the components of the *LEAK* circuit are free from air leaks.

Note: The flush or leak may time out before vacuum is reached. This may be reason to test the system by plugging the argon line to determine if the regulator is leaking.

Open the regulator valve on the argon cylinder. Partially close the gas control valve and set the pressure to 0.08mb. This will be the operating pressure for initial sputtering tests. Switch off *LEAK* and allow the vacuum to recover. Press *FLUSH* and then *LEAK*; observe the pressure changes. Check that the pressure returns to 0.08mb. Cancel *LEAK*.

4.3 Sputter Supply

Pump the vacuum chamber to a pressure lower than 0.05mb.

Check that the argon regulator valve is open and at the desired pressure (0.3bar). Select *MANUAL*. Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK* (this will cancel *FLUSH*). Wait for the pressure to stabilize at the set value of 0.08mb (adjust if necessary).

Press *SET mA* and hold while adjusting the value on the digital display to 40mA using the timer setting switches.

Press *PAUSE/TEST*.

A glow discharge will be visible in the chamber. The *mb* light will go out and the *mA* light will come on showing that the meter is now reading the sputtering current of 40mA. Release *PAUSE/TEST*.

4.4 Coating Cycle *(Manual and Automatic)*

Switch off. Raise the top-plate and remove the glass cylinder. Place a clean glass microscope slide on top of the stage tables (zero tilt) and replace the cylinder and top-plate. Re-pump to better than 0.05mb.

Select *MANUAL*.. Press *PAUSE/TEST* and adjust the timer display to read 30secs. Press *SET mA* to check the current setting (40mA). Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK*. Wait for the pressure to stabilize. Check the pressure and adjust to 0.08mb if necessary.

If a thickness monitor is fitted, switch *ON* and select *DENSITY* and *TOOLING* following the directions in section 2.6. With the (1) chamber argon pressure, (2) sputter current (3) process time (4) table height and (5) thickness monitor adjusted the system is ready.

Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK*. Wait for the pressure to stabilize. Zero thickness monitor and press *START*.

When the coating cycle is complete the timer will reset to: 30sec. Note the thickness monitor reading. Switch off the power. Lift the top-plate and remove and inspect the microscope slide.

Place another clean glass microscope slide. on the table and re-pump. Select *AUTO* and *CYCLE*. The unit will proceed through its standard cycle of:

(1) Pump to 0.05mb;
(2) Open argon flush valve for 3 seconds, then close;
(3) Pump to 0.15mb;
(4) Open argon flush valve for 3 seconds, then close;
(5) Open leak valve;
(6) Pump to 0.15mb and continue pumping for further 20 seconds;
(7) Switch on sputter supply at preselected current; and
(8) Switch off sputter supply after preselected time.

Each step in the process is shown by the indicator lights over the manual control buttons. If a thickness monitor is fitted the most accurate reading is obtained by zeroing between steps (6) and (7). The final reading should be taken before switching off to vent the chamber.

When the coating cycle is complete the timer will reset to :30sec. Note the thickness monitor reading. Switch off the power. Lift the top-plate and remove and inspect the microscope slide.

IMPORTANT

(A) THE THICKNESS AND DISTRIBUTION OF THE COATING IS A FUNCTION OF

(1) Chamber argon pressure;
(2) Power setting;
(3) Coating time; and
(4) Target to table distance.

(B) THE OPTIMUM VALUE OF THE THICKNESS REQUIRED AND THE TYPE OF DISTRIBUTION REQUIRED WILL DEPEND VERY MUCH ON THE NATURE OF THE SAMPLE AND THE OPERATING CONDITIONS IN THE SEM.

Section 5: **Operating Procedure**

5.1.1 Routine Sample Coating (without thickness monitor)

The following procedure will result in a satisfactory coating in the majority of cases. The procedure may need to be modified if very high resolution is required (see 5.2).

(1) Check that the sample has been mounted on a stub suitable for use on the sample stage tables. Any solvent-based adhesive should have been allowed to dry out thoroughly. The sample should be a suitable shape to allow a conducting path to form during coating.

(2) Check that the valve on the argon gas supply is open and that the regulator gauge is reading about 0.3bar (see 4.2).

NOTE: If the unit is used frequently using the same conditions, steps 3, 4 and 5 can be omitted.

(3) Check the timer value on the digital display. If necessary, press *PAUSE/TEST* and reset the process time.

(4) Check the preset sputter current value by pressing *SET mA* and adjust if necessary. For initial tests 40mA can be used.

(5) Select *MANUAL.*. Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK.* Wait for the pressure to stabilize. Check the pressure and adjust if necessary. For initial tests 0.08mb can be used.

(6) Select *AUTO* and press *CYCLE.*

(7) When the timer resets the coating is complete. Switch off the unit. Lift the top-plate to remove the sample.

(8) If the coating thickness is unsuitable, change the <u>process time</u> for future samples. In the first instance do not adjust power or gas pressure.

5.1.2 Routine Sample Coating (with thickness monitor)

The following procedure will result in a satisfactory coating in the majority of cases. The procedure may need to be modified if very high resolution is required (see 5.2).

(1) Check that the sample has been mounted on a stub suitable for use on the coater sample table. Any solvent-based adhesive should have been allowed to dry out thoroughly. The sample should be a suitable shape to allow a conducting path to form during coating.

(2) Check that the valve on the argon gas supply is open and that the regulator gauge is reading about 0.3bar (see 3.2).

NOTE: If the unit is used frequently using the same conditions, steps 3, 4, 5, and 6 can be omitted.

> (3) Check that the thickness monitor parameters (density, tooling factor) are suitable. Adjust if necessary (see 2.6).
>
> (4) Check the timer value on the digital display. If necessary, press *PAUSE/TEST* and reset the process time.
> **The coating cycle will be terminated manually when the desired thickness is reached. Set a timer value that is longer than the expected coating time, (e.g. 200 seconds).**
>
> (5) Check the preset sputter current value by pressing *SET mA* and adjust if necessary. For initial tests 40mA can be used.
>
> (6) Select *MANUAL*.. Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK*. Wait for the pressure to stabilize. Check the pressure and adjust if necessary. For initial tests 0.08mb can be used.

(7) Select *AUTO* and press *CYCLE*..

(8) Monitor the progress through the cycle and for optimum accuracy; zero the thickness monitor when the *LEAK* pressure is better than 0.15mb (during the stabilization period just before coating commences.

(9) When the thickness monitor reading reaches the desired value press *PAUSE* to stop coating. Check the value and press *CYCLE* to terminate the cycle.

(10) Switch off the unit. Lift the top-plate to remove the sample.

5.1.3 Routine Sample Coating (with thickness controller)

The following procedure will result in a satisfactory coating in the majority of cases. The procedure may need to be modified if very high resolution is required (see 5.2).

(1) Check that the sample has been mounted on a stub suitable for use on the coater sample table. Any solvent-based adhesive should have been allowed to dry out thoroughly. The sample should be a suitable shape to allow a conducting path to form during coating.

(2) Check that the valve on the argon gas supply is open and that the regulator gauge is reading about 0.3bar (see 3.2).

NOTE: If the unit is used frequently using the same conditions, steps 3, 4, 5, and 6 can be omitted.

> (3) Check that the thickness controller parameters (density, tooling factor) are suitable. Adjust if necessary (see 2.7).
>
> (4) Check the thickness *TERMINATOR* set point value. Adjust if necessary,(see 2.7).
>
> (5) Check the preset sputter current value by pressing *SET mA* and adjust if necessary. For initial tests 40mA can be used.
>
> (6) Select *MANUAL*.. Press *FLUSH*, wait for 3 - 5 seconds and press *LEAK*. Wait for the pressure to stabilize. Check the pressure and adjust if necessary. For initial tests 0.08mb can be used.

(7) Change from *timer mode* to *controller mode* by simultaneously pressing the two scroll buttons on the *108auto* front panel. The *108auto* timer display will now show "t 0".

(8) Zero the thickness display and press *TERMINATOR READY/SET* to arm the controller. The *108auto* timer display will show "t 1".

(9) Select *AUTO* and press *CYCLE*.

(10) When the thickness reading reaches the setpoint value the coating cycle will be terminated. Switch off the unit. Lift the top-plate to remove the sample.

5.2 Other Sample Coating Techniques

(If the procedures outlined in 5.1 are not suitable the following techniques can be investigated.)

5.2.1 Tall Specimens

Problem: If the specimen has large vertical dimensions, the coating will be biased towards the tip of the specimen and coating may be too thin towards the base. The problem is caused by the relatively large differences in distance from the target of the various parts of the specimen. The problem can be exaggerated by sputtering at low argon pressures (below 0.05mb) when the sputtered gold is inadequately scattered.

Solution: Position the specimen table close to the baseplate. Sample-to-target distances (top/bottom) are now similar. Also, use a relatively high argon pressure (0.10mb to 0.15mb) to give good scattering and coating from a wider range of angles. If argon pressures over 0.15mb are required, it will be necessary to operate in MANUAL mode.

Note that long distance plus high scattering will give slow coating so the process time will need to be longer.

5.2.2 Porous Specimens

Problem: If a specimen is very porous, pumping the chamber will be slow. Sputtering while the specimen is still degassing may result in poor coating. The specimen surface can "repel" the gold coating by gas scattering. Also, failure to pump to a suitably low pressure for good argon flushing will result in large grain size and stress cracking in the gold coating.

Solution: Longer pumping times and repeated flushings are required. Wait for a very good ultimate pressure before selecting CYCLE. Repeated flushing can be achieved in AUTO by pressing and canceling PAUSE before the second flush is started. If coating into the pores of the specimen is a problem use the techniques in section 5.2.3.

5.2.3 Woven or Tangled Specimens

Problem: Coating at pressures which are "thickness efficient" for magnetron sputter heads (around 0.05mb) can cause inadequate penetration of the coating in this type of sample. The problem is caused by insufficient scattering of the sputtered gold.

Solution: Operate at a relatively high argon pressure (0.10mb-0.15mb) to improve scattering. If argon pressures over 0.15mb are required it will be necessary to operate in MANUAL mode.

If a fine grain coating is required it may be necessary to operate at lower power than usual (10mA - 20mA). Table height will depend on the size of the sample (see 5.2.1).

5.2.4 Specimens with Fine Detail

Problem: Grain structure in the gold coating may obscure some of the fine detail in certain types of specimens.

Solution: The gold coating will need to be as thin and as fine-grained as possible (while still remaining conductive). This implies using relatively low gas pressures (less than 0.05mb) and low powers (10mA-20mA). Unfortunately, the use of low gas pressures can result in poor coverage of the sample.

The alternative is to change the target material from gold to gold/palladium or platinum. These materials give a coating, which has a smaller grain size.

Care is needed when using Au/Pd or Pt targets as they can both form oxides in a plasma. This can result in stress cracking by oxidation of the coating if argon handling is not meticulous. Careful flushing must be used to sweep out any residual oxygen or water vapor from the supply lines. Both FLUSH and LEAK systems should be flushed through before daily use when working with Au/Pd or Pt.

Section 6: **Routine Maintenance**

6.1 Cleaning the Chamber

(1) Switch off the system.
(2) Lift the top-plate and remove the glass cylinder. Take care that the top-plate does not fall forward. The target can be damaged by striking the sample table.
(3) Remove the gold deposit from inside the glass cylinder by wiping with a lint-free tissue or cloth that has been moistened with a vacuum compatible solvent.
A 50:50 mixture of acetone and isopropanol is suitable. Do not use solvents which will attack pump oil. Wear gloves to avoid contact of solvent with skin. Work in a well ventilated area or in a fume hood.
The majority of deposit can be removed by rubbing with a cloth or tissue. Any deposit resisting this treatment may be removed by rubbing with a lint-free cloth and PELCO Bell Jar Kleen™.
(4) Remove any loose deposit from metal surfaces (sample table, baseplate, dark-space shield) by rubbing lightly with a Scotchbrite scouring pad.
Do not use abrasive cleaning pastes on these aluminum alloy surfaces.
(5) Check the surfaces of both chamber o-ring seals for damage and replace if necessary.
(6) Make sure that all surfaces are dry and free from loose material before replacing the glass chamber.
(7) Repump the system and check the pumpdown time as in Section 4.1.

6.2 Target Changing

(1) Switch off the system and vent the chamber.
(2) Lift the top-plate and remove the glass cylinder. Take care that the top-plate does not fall forward. The target can be damaged by striking the sample table.
(3) Remove the dark space shield (see diagram page 6) by loosening the M3 cap screw using a 2.5 hexagon key.
(4) Unscrew the clamp ring with the top-plate close to the horizontal position.
Using this position allows the target to remain in the clamp ring when it is completely removed from the target holder.
(5) Remove the clamp ring and target.
(6) Remove the target from the clamp ring and replace with the new target.
(7) Re-screw the clamp ring to the target holder and replace the dark space shield.
(8) Check the surfaces of both chamber o-ring seals and check that the vacuum chamber surfaces are free from loose material before replacing the glass cylinder. Clean as in 6.1 if necessary.
(9) Repump the system and check the pumpdown time as in Section 4.1.

6.3 Quartz Crystal Changing

When the thickness monitor crystal is either overloaded *or* loaded with stressed material *it will cease to oscillate and the display will read "FAIL". At this stage it will need to be changed.*

(1) Switch off the system and vent the chamber.

(2) Lift the top-plate and remove the glass cylinder.

Take care that the top-plate does not fall forward. The target can be damaged by striking the sample table.

(3) Loosen the 2 small screws in the side of the head. This will allow the crystal holding cap to be separated from the base.

(4) Carefully remove the cap leaving the crystal balanced on the spring. Note the position. Now replace the used crystal with a new crystal. The pattern on the crystal should face outward. Carefully replace the cap.

(5) Push gently to compress the spring. Retighten the small screws.

(6) Check the surfaces of both chamber o-ring seals and check that the vacuum chamber surfaces are free from loose material before replacing the glass cylinder. Clean as in 6.1 if necessary.

(7) Repump the system and check the pumpdown time as in Section 4.1.

6.4 Changing the Oil Mist Filter

The oil mist filter fitted to the exhaust port of the rotary pump should be changed when it shows signs of saturation.

(1) Switch off the system and vent the chamber.

(2) Unscrew the oil mist filter and replace with new filter.

6.5 Changing the Rotary Pump Oil

The rotary pump oil must be changed if its appearance in the sight glass at the end of the pump becomes dark. The maximum period between oil changes should be 6 months if the system is in regular use and should not exceed 12 months.

(1) Drain the oil from the pump and replace according to the manufacturer's instructions as provided.

If the style of pumpbase fitted makes it difficult to remove the drain plug, the pump may be inverted and drained through the filler cap.

Section 7: **Trouble shooting**

7.1 Sputtering

Problem: CYCLE is selected (AUTO mode) but sputtering does not start.
Solution: Argon pressure in specimen chamber too high.
Reduce pressure (select MANUAL and LEAK) below 0.1mb.

Problem: Pressure is higher than 0.5mb after FLUSH and LEAK.
Solution: Argon pressure is above safety trip point.
Reduce Argon pressure with the leak valve below 0.5 mb.

Problem: Meter shows selected sputter current (mA) but no plasma visible.
Solution: Short circuit between target and dark space shield.
Remove dark space shield and clean target clamping ring and target adaptor ring.

7.2 Thickness Monitor MTM-10

Problem: MTM-10 display shows "*FAIL*".
Solution:
1 - Change crystal.
2 - Check SMB cable connections.
3 - Replace Oscillator.

Problem: MTM-10 display shows “or”.
Reason: Thickness has exceeded measurable limits.
Measurement >999.9nm (or less than -99.9nm).

Solution: Press “ZERO”.

7.3 Thickness Controller MTM-20

Problem: Sputtering does not start in MTM mode.
Solution: Check *TERMINATOR* set point value.
Arm MTM-20 by selecting “t 1” with *TERMINATOR READY/SET* switch.

Problem: Sputtering process stops before the selected thickness is reached.
Solution: In MTM mode the maximum sputtering time will be 900 sec.
Change operating parameters to a higher sputter rate or start again in MANUAL mode by selecting LEAK and START.

7.4 Vacuum System

Problem: Rotary pump doesn’t start.

Solution:
1 - Check cable.
2 - Press Thermal Trip on rear panel (viscosity of the rotary pump oil is low due to low temperature.
3 - Make sure ambient temperature is not below 15^{0}C (60 F).

Problem: After 2 minutes of pumping pressure is not better than 0.5 mb.

Solution:
1 - Check O-ring seals.
2 - Check if top-plate seals on glass chamber.
3 - If sample has high porosity reduce sample size or accept long pumping times.
4 - If sample is outgassing bake or accept long pumping times

Section 8: **Spares and Options**

8.1 Consumables

91110	Gold Target (Ø57 x 0.1mm)
8071	Gold Target (Ø57 x 0.2mm)
91114	Platinum Target (Ø57 x 0.1mm)
91112	Gold / Palladium Target (Ø57 x 0.1mm)
91111	Gold / Palladium Target (Ø57 x 0.2mm)
91129	Silver Target (Ø57 x 0.3mm)
8079	Copper Target (Ø57 x 0.3mm)
896	PELCO Bell Jar Kleen™

8.2 Thickness Monitor

93004	MTM-10 Thickness Monitor System (complete kit)
93006	MTM-20 Thickness Controller System (complete kit)
93008	Thickness Monitor Crystals, pkg/3
93009	Thickness Monitor Crystals, pkg/10

8.3 Specimen Chamber

9608	Replacement Glass Chamber (Ø120 x 120mm)
9607	Replacement Chamber O-rings (Ø120mm / Pk 2)

8.4 Vacuum System

7010	Rotary Pump, 115VAC, anti-vibration table, all-metal, integrated coupling system
7030	Dualvacset Conversion Kit (allows connecting two sputter coaters or one sputter coater and one carbon coater to be connected to one vacuum pump)
9609	Oil Mist Filter (for Prod. No. 7010 Rotary Pump)

8.5 Carbon Coater

9602	Automatic Carbon Coater 108carbon/A, 115VAC
9602-220	Automatic Carbon Coater 108carbon/A, 220VAC
9603	Automatic Carbon Coater 108carbon/A-SE, 115V
9603-220	Automatic Carbon Coater 108carbon/A-SE, 220V

Section 9: **Specifications**

9.1 Sputter Coater 108auto control unit

Specimen Chamber	Ø120 mm OD, height 120mm
Sputter target	Ø57 mm diameter x 0.1 mm thick, Gold fitted as standard
Sample Stage	Static table Height adjustable 65mm
Sputter supply	Programmable digital control 10/20/30/40 mA Constant current control Safety interlocked
Sputter head	Low voltage planar magnetron Quick target change Wrap-around dark space shield
Analog Metering	Vacuum: Atmos - 1×10^{-3} mbar Current: 0 – 50mA
Control Method	Automatic/Manual mode selection Automatic operation of gas purge Automatic process sequencing Automatic vent Digital timer 5-300 seconds Full manual control of all functions

9.2 Pumping System

Rotary Pump	High Speed direct drive, 2 stage rotary pump
Pumping Speed	41.6 / 50 ltr/min (50 / 60Hz)
Chamber Pumpdown	30 sec to 0.1mb
Ultimate Pressure	5×10^{-3} mbar
Desktop System	Rotary pump mounted on desktop compatible anti-vibration table

9.3 Thickness Monitor MTM-10

MTM-10	Microprocessor based 4 digit display Push button zero 6Mhz crystal with lifetime check
Memory	Four settings storing Density and Tooling factor
Update rate	10 Hz
Thickness range	0.0 - 999.9nm
Resolution	Better than 0.1nm
Density range	0.50 - 30.00 gm/cm^3
Tooling factor range	0.25 - 8.00
Error Messages	Fail / or

9.4 Thickness Controller MTM-20

MTM-20	Microprocessor based 4 digit display Push button zero 6Mhz crystal with lifetime check
Memory	Four settings storing Density and Tooling factor Termination point
Update rate	10 Hz
Thickness range	0.0 - 999.9nm
Resolution	better than 0.1nm
Density range	0.50 - 30.00 gm/cm^3
Tooling factor range	0.25 - 8.00
Terminating range	0.0 - 999.9nm
Error Messages	Fail / or

9.5 Services Required

Power for 7002	100-120VAC, 50/60Hz, 120W max.
Power for 7006	220-240VAC, 50/60Hz, 120W max.
Gas	Argon; 99.9 min. purity Pressure regulated to 5 - 9 PSI (0.3 - 0.6bar) 6mm ID hose

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