Operating Manual

Cressington 208carbon

High Vacuum Carbon Evaporator

Cressington Scientific Instruments Ltd

Contents

§1. Services Required

1.1 Electrical Power

The 208carbon coater requires a single phase electrical supply from a standard laboratory power point.

The 208carbon is available in two versions for use with 100/115V or 200/230V. A power cable and connector are provided.

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

IMPORTANT **Before connecting the system to a source of electrical power it is essential to check this label to ensure that the supply voltage is suitable**

§2. Installation

2.1 Pumping System

(i) Fitting the Turbo Pump

The Cressington 208carbon high vacuum pumping system comprises a turbo pump (Pfeiffer ATP80) and a two-stage rotary pump (Pfeiffer DUO2.5). The complete coating unit including pumps occupies an area 600mm wide x 600mm deep on a standard laboratory bench.

The turbo pump is packed with the main control unit. Unpack the control unit and remove the turbo pump, vacuum seals, clamps and turbo cable from their box.

Remove the protective material from inside the pumping port (rear panel) of the control unit. Remove the protective cap from the turbo pump inlet and put the high vacuum seal in place.

IMPORTANT Do not allow packing material to enter the pumping ports

Bolt the turbo to the pump port flange using the 4 claw clamps and orientate as shown below. Fit the cable to the turbo pump and fit the other end of the cable in the top receptacle of the "pump supplies" area on the rear panel.

The turbo pump obtains its backing vacuum from a two-stage rotary pump. The vacuum connection from the turbo pump to the rotary pump is a stainless steel flexible (packed in the rotary pump carton). The KF16 x 250mm flexible has a KF16/25 adaptor to suit the KF25 backing port of the turbo pump.

(ii) Fitting the Rotary Pump

Unpack the smaller carton and identify the components of the backing system including:

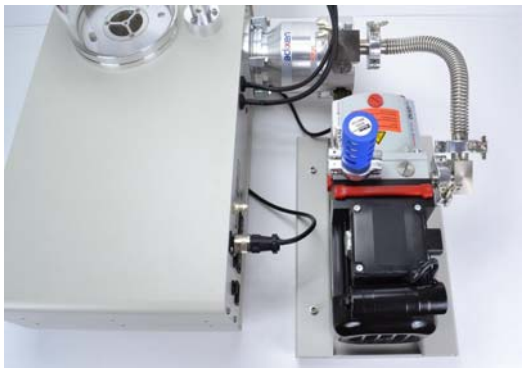
(a) the rotary pump on its anti-vibration base,
(b) stainless steel flexible hose with KF16/25 and KF16/16 elbows,
(c) o-ring seals and clamps,
(d) exhaust mist filter.

Remove the protective caps from the rotary pump ports. The rotary vacuum pump is shipped "dry". Fill the pump with oil and place it behind the 208carbon as shown below.

IMPORTANT Running the pump without oil will cause serious damage.

For details of the filling procedure see the manufacturers instructions.

Connect the backing port (KF25) of the turbo pump to the rotary pump inlet (KF16) using the stainless steel flexible. Check that the sealing surfaces and o-ring seals are clean. Ensure that the hose is not under stress when in its final position as this can cause vibration.



Fit the exhaust filter to the rotary pump outlet port. The exhaust is filtered to prevent oil mist droplets from diffusing into the laboratory environment.

Remove the black plastic tubing from the VENT GAS INLET nozzle.

The rotary pump power cord can be connected to the back panel at this stage but do not connect the main power cord until the vacuum chamber and evaporation source have been prepared. For initial pumpdown see §2.6

2.2 Carbon Evaporation Chamber

The cylindrical vacuum chamber is made from high quality glass. The vacuum seals (o-rings) are recessed into the baseplate and top-plate.

After unpacking. wipe the surfaces of the glass cylinder with a lint free tissue that has been moistened with alcohol. Carefully place the glass cylinder and metal spacer ring on the baseplate.

The chamber top-plate is attached by a bracket to a support pillar. The bracket is hinged to allow the top-plate to be raised and lowered for access to the chamber.

IMPORTANT **Before fully closing the chamber, check that the glass cylinder and top-plate are correctly aligned. If they are not concentric the top-plate could chip the glass.**

If required, the top-plate position can be adjusted by loosening the M5 screw (8mm A/F wrench) which holds the bracket to the pillar.

The chamber support pillar is telescopic and is adjustable to three different height settings using an indexing pin.

The pin is spring loaded and is actuated by a pull ring.

The variable chamber height feature allows the use of different chamber configurations including metal spacer rings and rotary stages (R-P-T stage P/N 87053 and R-T stage P/N 87054) which are retro-fittable to the system

The standard basic chamber configuration includes a metal spacer ring.

2.3 Sample Table

The sample table is mounted on a threaded pillar screwed through the centre of the baseplate clamping disc. The table is raised or lowered by loosening the locking nut (10mm A/F wrench) and rotating the table (1 turn equals 1mm).

The adjustment of the deposition distance is a compromise. Shorter distances give thicker coatings; longer distances give more uniform coverage. Initially adjust the bottom surface of the sample table to a height of about 30mm above the baseplate. This gives a deposition distance of about 85mm. This can be re-adjusted later depending on the requirements of the samples.

The sample table has multiple 3.2mm holes for standard pin stubs. Other designs of sample mount can be placed on the surface of the table.

The 20mm hole at the front of the table (see above) is for mounting a thickness monitor crystal holder.

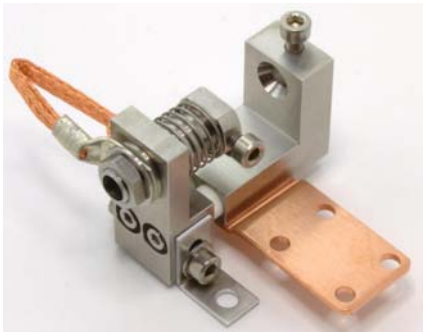
The 208C chamber baseplate has 4 feedthrough holes. The hole to the left and rear is permanently blanked off.

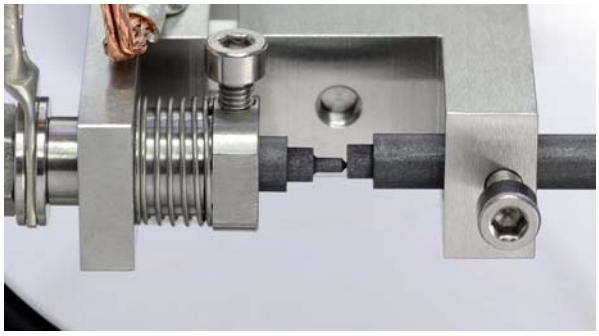
The larger blanked hole on the left is for fitting an RF10 thickness monitor feedthrough. The front hole on the left is the vent gas inlet to the chamber. The blanked hole on the right is for a leak gas inlet which is required if a Glow Discharge Accessory is fitted.

2.4 Evaporation Source (using profiled graphite rods)

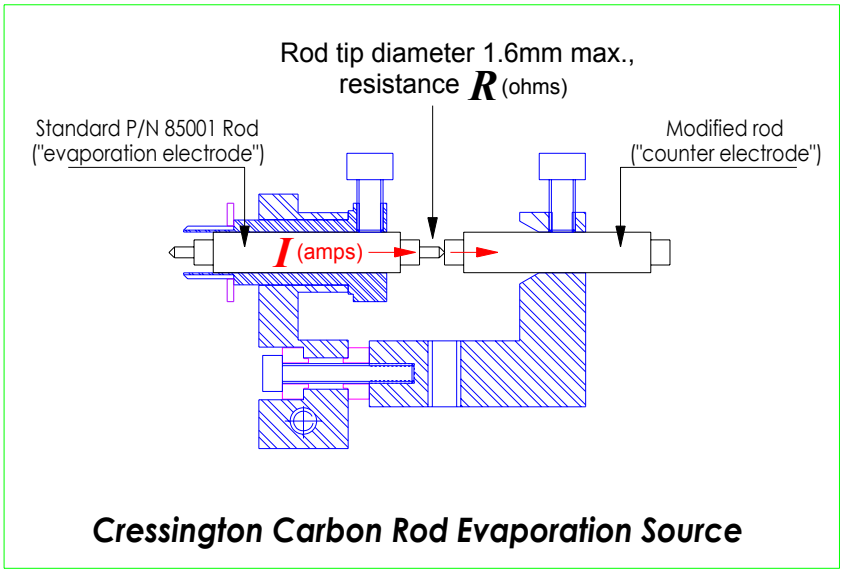
When graphite is heated to around 2800^{0}C in vacuum it vaporises directly from the solid state without melting and condenses as an amorphous carbon film.

Graphite is usually evaporated by resistive electrical heating of rods. The heating effect is localised by profiling the graphite rods to produce a small "hot zone" of high electrical resistance. As material is vaporised from the rod tip, a spring moves the rod forward to maintain electrical contact.





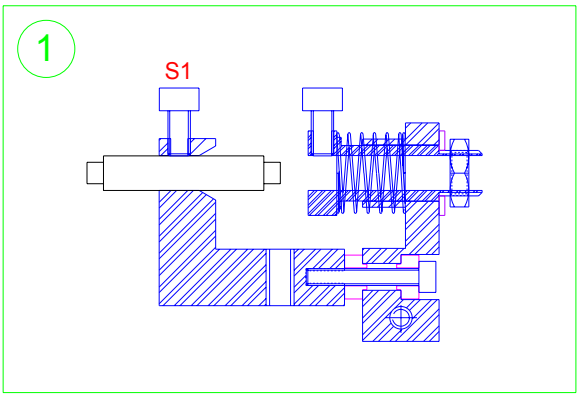
The 208carbon top-plate incorporates a carbon evaporation source based on this principle.



Cressington Carbon Rod Evaporation Source

The source uses graphite rod of 10^{-3}ohm.cm and 6.15mm diameter. For initial tests Cressington precision machined carbon rods (P/N 85001) should be used. After experience has been gained, different profiles can be used.

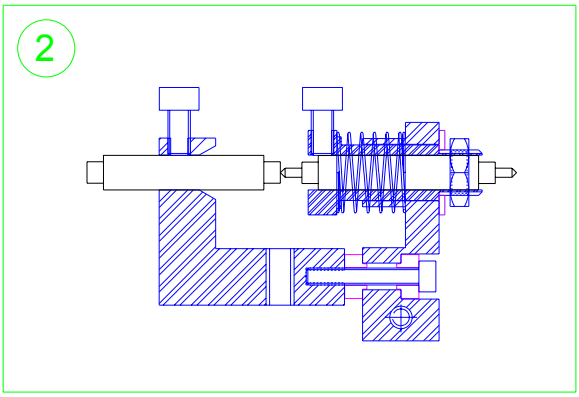
2.5 Loading the Rod Evaporation Source



Modify a standard rod by removing the tips. Smooth and flatten an end face using fine carborundum paper.

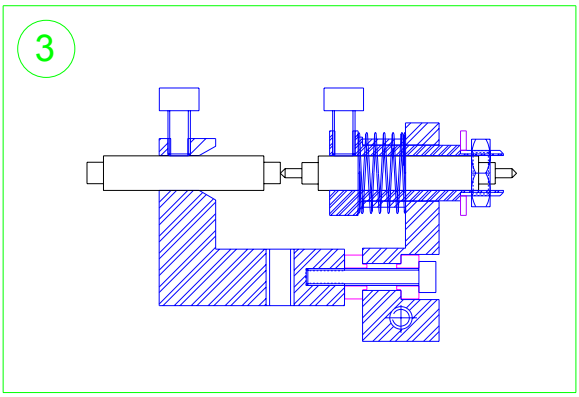
Position the rod in the frame so that the end face is over the screw-hole in the frame. Tighten the fixing screw S1 lightly.

This is the static rod or "counter-electrode" (see previous page).

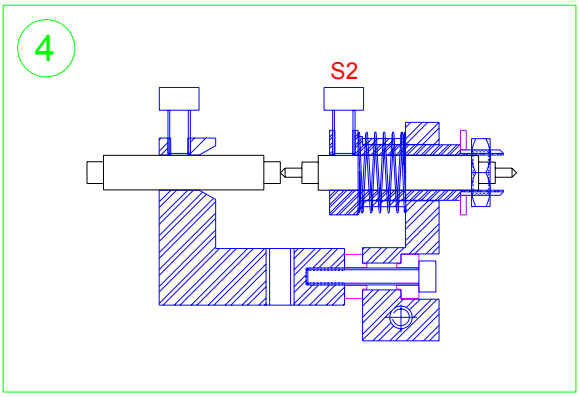


Slide a standard rod through the sprung rod holder until its tip is touching the static rod.

This is the moving rod or "evaporation electrode" (see previous page).



While keeping the two rods touching, slide back the rod holder against spring pressure.



Maintain the spring pressure and tighten the fixing screw S2 lightly.

The source is now ready for use.

2.6 Initial Pumpdown

Before initiating the first vacuum pumpdown, confirm that:

(1) the pumping system has been installed correctly (see §2.1 on pp4/5)
(2) the sample chamber is closed (see §2.2 on p6)
(3) the POWER switch on the 208C front panel is OFF.

IMPORTANT Before connecting any power cords check equipment labels to ensure that the supply voltage is suitable.

Plug the power cord from the rotary vacuum pump into the lower socket of "PUMP SUPPLIES" on the back panel of the 208C (see below).

Plug the mains power cord into the socket "POWER IN".

IMPORTANT Before switching on check the oil level in the rotary vacuum pump. Running without oil will cause serious damage.

Switch on the POWER on the front panel to activate the pumping system.

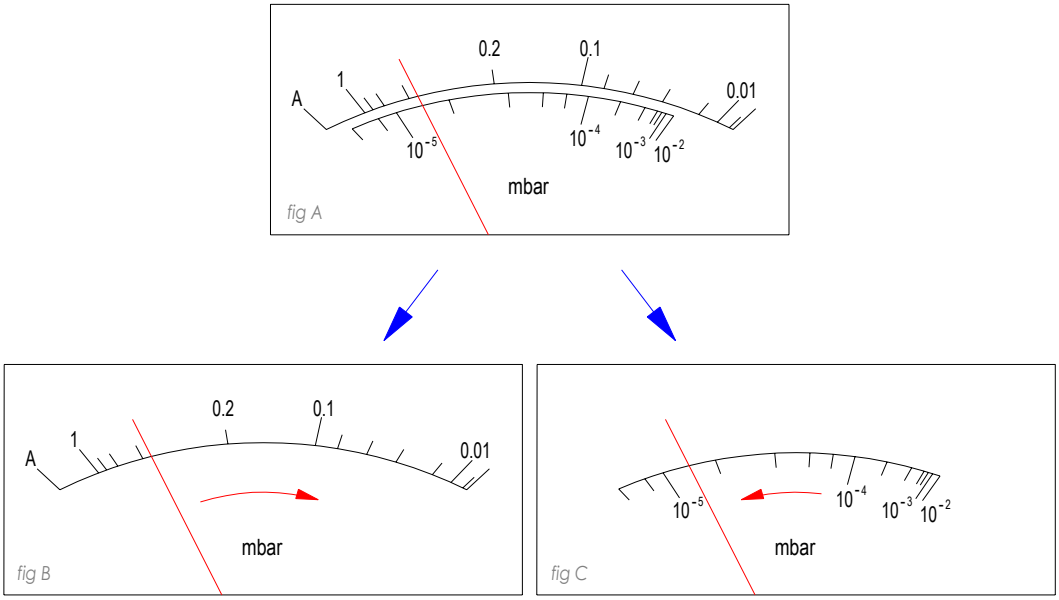
The chamber vacuum is monitored on the front panel meter and the turbo pump status is indicated by lights on the right hand side of the front panel.

2.7 Reading the Vacuum Gauge

The vacuum meter on the front panel of the 208C is a combination of Pirani gauge and Penning gauge. The dual scale of the vacuum meter is shown below in fig A. The dual scale has been separated into fig B and fig C for the purpose of explanation.

The Pirani gauge measures vacuum from atmospheric pressure down to 10^{-3}mb and the reading is displayed on the upper scale. This upper scale is shown separately in fig B. Atmospheric pressure is to the left of the scale and vacuum is improving when the needle moves in the direction of the red arrow.

The scale gives values at major points and sub-divisions between 0.01mb and 1.0mb are given at 2,4,6,8. In fig B the needle is indicating between 0.2 and 0.4mb.



Pirani Vacuum Gauge
(scale from atmosphere down to 10-3mb)

Penning Vacuum Gauge
(scale from 10-2mb down to 6 x 10-6mb)

On initial pumpdown, with the TURBO STATUS orange, the vacuum meter will indicate Pirani gauge pressure.

When the TURBO STATUS goes from orange to green the vacuum meter will switch from Pirani to Penning gauge.

The Penning gauge measures vacuum from 10^{-2}mb down to 6 x 10^{-6}mb and the reading is displayed on the lower scale. This lower scale is shown separately in fig C. 10^{-2}mb is to the right of the scale and vacuum is improving when the needle moves in the direction of the red arrow.

The scale shows the decade points gives sub-divisions at 2,4,6,8. In fig C the needle is indicating between 1 x 10^{-5}mb and 2 x 10^{-5}mb. It could be estimated to be about 1.5 x 10^{-5}mb

2.8 Vacuum Performance

The 208C produces carbon coating of suitable quality for

(1) sample coating for FESEM or

(2) preparation of TEM support films.

For FESEM applications a vacuum of 5 x 10^{-5}mb is usually considered to be adequate before commencing evaporation. The 208C pumps to 5 x 10^{-5}mb in around 8 minutes. The actual time taken can be as short as 4 minutes or as long as 12 minutes and will depend on the condition of the chamber.

IMPORTANT **Pumpdown times and ultimate vacuum will deteriorate if the system is kept at atmospheric pressure for long periods.**

When producing quality TEM carbon support films it is usual to pump for longer. It is generally thought that better vacuum gives better carbon films.

For TEM support films a vacuum of better than 2 x 10^{-5}mb is usually required before commencing degassing and evaporation. If operated with care, the 208C pumps to 2 x 10^{-5}mb in between 10 and 15 minutes depending on the condition of the chamber.

IMPORTANT **For TEM applications the 208C should be prepared for use (at the beginning of the working day) with a pre-pumping cycle to better than 1 x 10^{-5}mb. When in use the system should be kept pumping and vented only for minimum time for sample loading.**

When the system is switched off it automatically vents to atmosphere. The time to vent fully (before the top-plate can be lifted) is about 10 seconds.

2.9 Thickness Monitor

The 208C can be supplied with an MTM-10 thickness monitor. It can not be supplied with an MTM-20 thickness controller because carbon deposition is not suited to thickness control by quartz crystal (see §3.6).

When a thickness monitor (MTM-10) is supplied the following external cables will need to be connected.

(1) mains power cord from the 208C rear panel to the MTM-10 rear panel

(2) bnc signal cable from the MTM-10 rear panel to the 208C rear panel

See p12 of this manual for information on setting up the MTM-10.

For a complete description of the use of this tool see the Operating Instructions for the Cressington MTM-10.

§3. Operating Modes

3.1 Mode Selection

The 208carbon can be operated in AUTOMATIC CONTROL or MANUAL CONTROL modes. The AUTO/MANUAL selector allows switching between the two modes.

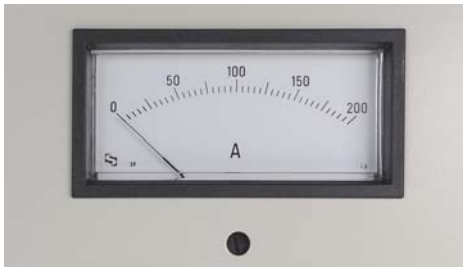
In MANUAL CONTROL the system can be operated in CONTINOUS (Co) or PULSED (Pu) modes. These are selected by pressing the SET switches. The mode is indicated in the digital display by "Co" or "Pu".

3.2 Manual Continuous Mode

Select this mode using the MANUAL and SET switches and, before pressing START/STOP, confirm that:-

(1) the evaporation source is correctly loaded (see §2.5)
(2) the system is pumped below 5 x 10^{-5}mb (see §2.7)
(3) the VOLTS knob is turned fully anti-clockwise
(4) the digital display shows "Co"

Press START/STOP. In continuous "Co" mode the supply is latched and the switch LED will remain illuminated. Rotate VOLTS to apply the heating voltage to the evaporation source. The heating current is displayed on the upper meter.



If VOLTAGE is pressed, the "Co" in the digital display is replaced by the voltage reading.

To confirm that the source is loaded and operating correctly, increase the voltage to 2.0 volts. The current should read between 25A and 30A when using precision machined graphite rods (Cressington P/N 85001).

3.3 Manual Pulsed Mode

Select this mode using the MANUAL and SET switches and, before pressing START/STOP, confirm that:-

(1) the evaporation source is correctly loaded (see §2.5)
(2) the system is pumped below 5 x 10^{-5}mb (see §2.7)
(3) the VOLTS knob is turned fully anti-clockwise
(4) the digital display shows "Pu"

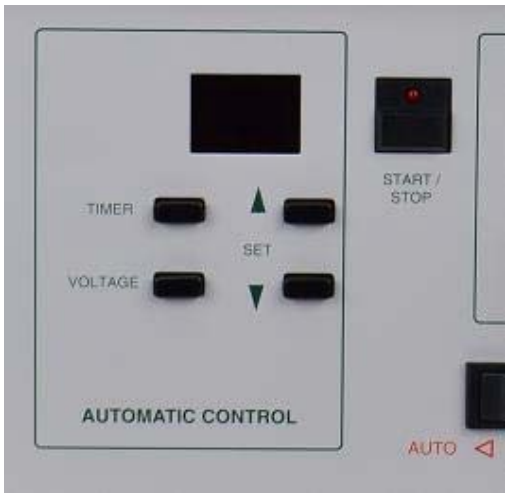
In this mode the START/STOP is "momentary"; the supply is only ON while the switch is pressed.

Before pressing START/STOP, rotate VOLTS and monitor the digital display with VOLTAGE pressed. Set the VOLTS to 2.0 volts. This voltage will not be applied to the source until STASRT/STOP is pressed.

To confirm that the source is loaded and operating correctly, press START/STOP. The current should read between 25A and 30A when using precision machined graphite rods (Cressington P/N 85001). When START/STOP is released the heating current will return to zero amps.

3.4 Automatic Mode

Select AUTOMATIC CONTROL using the AUTO/MANUAL switch. The digital display will now show the setting of the automatic TIMER. This can be altered by pressing TIMER and using the SET switches for up/down adjustment.



When VOLTAGE is pressed the digital display shows the setting of the automatic VOLTAGE control. This is adjusted using the SET switches with VOLTAGE pressed.

For initial tests (with Cressington rods P/N 85001) use the following settings:

VOLTAGE between 3.6V and 4.0V
TIMER between 4 secs and 6 secs

When START/STOP is pressed the preset VOLTAGE will be applied for the preset TIME. The evaporation source current will be displayed on the analogue meter.

This mode is essentially an automated pulse control. Repeat until the desired thickness is obtained.

3.5 Setting the Thickness Monitor MTM-10

The sensing head of the MTM-10 thickness monitor contains an oscillating quartz crystal. When evaporated carbon is deposited on the crystal the frequency of oscillation is changed. The thickness monitor uses this change in frequency to calculate the mass of material.

For the frequency change to be displayed as "film thickness", it is necessary to set the MTM-10 with the coating parameters:-

(a) the density (g.cm-3) of carbon film

(b) the geometrical tooling factor

The density of amorphous carbon (a-C) thin films is not known with any degree of confidence. The density of the original graphite evaporation rods is in the range 1.65 - 1.85 $g.cm^{-3}$ but the structure of a-C is very different from graphite.

When MTM-10 readings are compared with results from the "colour-change" technique (using a polished brass block), good agreement is obtained if a density value of 1.4 $g.cm^{-3}$ is used.

The value of the tooling factor depends on the relative positions of the evaporation source and the sample table (and the height of the sample).

For a full description of the tooling factor calculation see the MTM-10 Operating Instructions.

3.6 The Effect of Radiation on the MTM-10

The quartz crystal increases in mass as material is deposited and responds by reducing its frequency of oscillation. The reduction in frequency is displayed as an increase in thickness.

The quartz crystal is also sensitive to radiation. It responds to an increase in temperature by increasing its frequency of oscillation. The increase in frequency is displayed as a decrease in thickness.

Under normal circumstances these two conflicting effects do not cause a problem because the frequency shift resulting from deposition is much larger than the shift resulting from radiation.

However, in the case of carbon evaporation the situation is unusual. The very high temperature of the source results in a large radiation reaction at the crystal; the low density of carbon (1.4 $g.cm^{-3}$) results in a small frequency change with thickness.

The result is a confusing reading during the evaporation process. It is difficult to follow the coating thickness during deposition and it is not possible to manually terminate the deposition at the desired thickness with any degree of accuracy.

IMPORTANT The MTM-10 does not display accurate thickness during deposition because of transient radiation (temperature) effects.

IMPORTANT The thickness displayed on the MTM immediately after deposition finishes will be changing as the crystal cools down. The true thickness is displayed when the crystal has returned to normal temperature (after approximately 45 seconds).

§4. Operation

4.1 Standard Coating Procedure (MANUAL/Continuous)

(1) Mount the sample

(2) Load the evaporation source

(3) Load the sample (and adjust table height if required)

(4) Switch ON and allow to pump

(5) Select MANUAL and SET "Co"

(6) Wait for desired vacuum reading

(7) Press START/STOP

(8) Increase and control evaporation current until desired thickness has been deposited*

(8) Wait 5 minutes for source to cool

(9) Switch OFF and raise the top-plate to remove the sample

* Thickness of deposition can be estimated by traditional visual means and then measured by MTM-10 after deposition (after waiting for stable reading 30/45 secs)

4.2 Standard Coating Procedure (MANUAL/Pulsed)

(1) Mount the sample

(2) Load the evaporation source

(3) Load the sample (and adjust table height if required)

(4) Switch ON and allow to pump

(5) Select MANUAL and SET "Pu". Press VOLTAGE and set volts

(6) Wait for desired vacuum reading

(7) Press START/STOP. Repeat until desired thickness has been deposited*

(8) Wait 5 minutes for source to cool

(9) Switch OFF and raise the top-plate to remove the sample

* Thickness can be estimated between pulses by traditional visual means. Alternatively thickness can be measured by MTM-10 (after waiting 10/15 secs between pulses)

4.3 Standard Coating Procedure (AUTO)

(1) Mount the sample

(2) Load the evaporation source

(3) Load the sample (and adjust table height if required)

(4) Switch ON and allow to pump

(5) Select AUTO and check the AUTO settings (TIMER/VOLTAGE)

(6) Wait for desired vacuum reading

(7) Press START/STOP and repeat until desired thickness deposited*

(8) Wait 5 minutes for source to cool

(9) Switch OFF and raise the top-plate to remove the sample

* Thickness can be estimated between pulses by traditional visual means. Alternatively thickness can be measured by MTM-10 (after waiting 10/15 secs between pulses)

§5. Routine Maintenance

5.1 Evaporation Source

During evaporation the tip of one of the rods is vaporized and the rod becomes shorter. The rod holder must slide forward under spring pressure to maintain electrical contact between the two rods. To allow sliding, the electrical connection to the rod holder must be flexible.

The function of the copper braid is to carry the large evaporation current without impeding the movement of the rod slider.

fig.2a

fig.2b

The flexible copper braid is made up of a large number of very fine wires. Over time these fine wires become oxidised and increase in resistance.

Each wire in the braid is only 0.15mm in diameter. An oxidation depth of only 25 microns will reduce the effective diameter to 0.10mm and more than double the electrical resistance. This extra resistance reduces the voltage applied to the rods and hence reduces the evaporation current.

IMPORTANT The effects of oxidation can be reduced by waiting a few minutes before venting the system after an evaporation.

IMPORTANT From time to time the copper braid should be replaced. It is advisable to change the spring at the same time.

The spring used in the evaporation source is stainless steel and is not prone to oxidation. However, its strength is reduced by repeated heating cycles. The spring strength should be checked when replacing a copper braid.

The "free length" of the used spring should be compared that of a new spring. If it is significantly shorter it should be replaced.

The oxidised copper braid is removed by undoing the connections
(1) to the frame (M4 socket cap screw, 3mm hex key)
(2) to the carbon rod slider (M8x0.75 nut, 7/16"A/F or 12mm A/F).

When replacing the copper braid (Cressington P/N 85112) it will usually be necessary also to change the stainless steel spring (Cressington P/N 85111).

IMPORTANT When replacing the braid and spring carefully check the alignment of the braid and slider

The braid is shaped in such a way that it offers little resistance to movement when installed correctly. The 'V' section of the braid folds with a scissors action as the holder slides in the frame (*see photographs*).

The braid must not interfere with the smooth sliding action of the rod holder or this will interrupt the evaporation process. When installing a new braid this free sliding movement should be checked.

IMPORTANT When replacing the braid and spring ensure that the electrical contacts are fully tightened. The stabilised voltage applied to the evaporation source rods will be reduced if contact resistances are introduced.

It is important that the copper braid electrical contacts to the carbon rod slider (large nut, M8 x 0.75) and the evaporation source frame (M4 skt cap screw) are carefully tightened. Loose contacts will introduce electrical resistance and reduce the efficiency of the evaporation source.

5.2 Cleaning the Chamber

If the system is in regular use, the chamber will eventually become heavily coated with carbon.

The carbon deposit can be removed from the glass chamber as follows:

(1) Switch off the system and disconnect from electrical power

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

(3) Remove any loose or flaking carbon from inside the glass cylinder by wiping lightly with a lint-free tissue or cloth which has been moistened with a vacuum compatible solvent (alcohol; isopropanol)

(4) Remove well-adhered carbon from inside the glass cylinder by rubbing firmly. Any deposit resisting this treatment may be removed by rubbing lightly with a dry Scotchbrite scouring pad

(5) Remove any loose carbon from metal surfaces (sample table, baseplate, evaporation source) by wiping/rubbing as in (3/4) above.
Do not use abrasive pastes on aluminium alloy surfaces

(6) Check the surfaces of both chamber o-ring seals for damage and replace if necessary

(7) Make sure that all surfaces are dry and free from loose material before replacing the glass chamber

5.3 Replacing the Crystal in the Thickness Monitor

The measuring crystal changes frequency as carbon accumulates on its surface. The amount of offset from its original frequency (6.0MHz) is displayed (in KHz) when *ZERO CHECK* is pressed for more than 2 seconds.

The crystal oscillator can only accept a finite offset and will eventually become too heavy to oscillate. At this point the display of the monitor reads "FAIL".

IMPORTANT **The crystal can also "FAIL" if carbon becomes loosely adhered to its surface. Carbon will begin to lift in a humid atmosphere.**

IMPORTANT **Loose carbon flakes from the chamber wall or top-plate will also cause "FAIL" if they land on the surface of the crystal.**

When "FAIL" is indicated on the MTM-10 display the crystal will need to be changed. The procedure for changing the crystal is described in §7, p10 of the Operating Instructions for the MTM-10/20.

5.4 Replacing 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

5.5 Changing the Rotary Pump Oil

The rotary pump oil, as seen through the sight glass at the end of the pump, should appear colourless. It must be changed when it begins to have a darkened appearance. The maximum period between oil changes should be 12 months if the system is in regular use.

The oil should be drained from the pump and replace according to the manufacturers' instructions (7.2 Changing the Operating Fluid, page 49). If the shape of the pump base makes it difficult to remove the drain plug, the pump may be inverted and drained through the filler cap.

§6. Troubleshooting

6.1 Carbon Evaporation

PROBLEM No evaporation current when STASRT/STOP is selected

SOLUTION Poor contact between carbon rods. Reload evaporation source or change spring.

PROBLEM Large evaporation current but no carbon evaporation

SOLUTION Carbon rod tip exhausted. Replace rods in source.

PROBLEM Large evaporation current but no carbon evaporation

SOLUTION Carbon rod tip wrongly shaped. Reshape rod with smaller diameter tip.

6.2 Vacuum

PROBLEM Rotary pump does not start

SOLUTION Press Thermal Trip on rear panel. Temperature of the rotary pump oil is low due to ambient temperature below 15^0C.

PROBLEM Pumping time too slow

SOLUTION Small vacuum leak. Check top and bottom surfaces of glass chamber. Check for surface contamination or damage on O-ring seals.

PROBLEM Pumping time too slow

SOLUTION Porous sample. Allow longer pumping time.

§7. Spares and Consumables

7.1 Carbon Source

30816 Carbon Rod Shaper

85001 Precision Machined Carbon Rods for 50/60Hz (Pk 10)
85103 Carbon Rods (∅6.15 x 305mm) for 50/60Hz (Pk 10)

85110 Carbon Rod Slider
85111 Carbon Rod Slider Springs (Pk 5)
85112 Copper Braids (Pk 5)
85113 Ceramic Insulators (Pk 8)

85114 Feedthrough Insulator Set (2 x PTFE washers with O-ring)

85116 Static Rod Holder
85117 Moving Rod Frame
85118 Slider Nut (with washers)

7.2 Vacuum System

87010 Replacement Glass Chamber (∅150 x 150mm)
87015 Replacement Chamber O-rings (∅150mm / Pk 2)

21300 Thickness Monitor Crystals (Pk 10)

80156 Rotary Pump Exhaust Filters (Pk 5)
81121 P3 Rotary Pump Oil (0.5 litre)

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