E5100 series II

'COOL' SPUTTER COATER

1. SETTING UP EQUIPMENT

The unit is packed in 4 parts :

(i) Main Cabinet

(ii) Vacuum Collar including Specimen Stage

(iii) 'Cool' Sputtering Head

(iv) Accessories including Vacuum Tubing, Gas Tubing and Specimen Holder.

1.1 Unpack the main cabinet and set it on a suitable bench. Figure 1. is a diagram of the left hand side panel (looking from the front) including inset service panel and figure 2. illustrates the right hand side panel. First ensure that the voltage on the label located near to the mains electrical cable is correct for the laboratory supply.

1.2 Check that the baseplate is clean and if necessary dust off. Unpack the vacuum collar and place it carefully on the base-plate so that the blanking plates are on the sides and rear of the collar.

1.3 Next place the cool sputtering head on the glass spacer and secure on the upright pillar at the rear using the locking pin provided.

2. CONNECTION OF UNIT

Refer to figures 1 and 2 for layout of services on the E5100 unit.

1 Pa = 0,01 mbar = 0,1

1 Pa = 1 N/m²

1 bar = 10⁵ Pa

1 psi = 0,1019 1 . 10⁻⁶ kp/mm²

2.1 Unpack accessory kit and remove black Vacuum Tubing. Slide the tube over the outlet fitting on the left hand side panel (LH Panel) and secure the other end over the inlet port of the rotary pump.

N.B. If the rotary vacuum pump is supplied by Polaron the unit is fitted with a 15mm tube inlet. If the pump is obtained otherwise it maybe necessary to obtain the required inlet fitting.

2.2 The accessory pack also contains a 3 pin plug which must be fitted on the electrical power lead of the rotary pump. The pump lead can then be inserted with the socket on the LH Panel of the Sputter Coater.

2.3 A quantity of 6mm tubing supplied is for flushing the Sputter Coater with Argon gas. This tubing connected to a gas cylinder fitted with a 2 stage regulator so that the gas pressure can be adjusted to around 5 psi, the other end is fitted over the gas inlet located on the LH side panel (figure 1).

2.4 There are 2 electrical leads from the vacuum collar, one a 3 pin power lead for the thermoelectric device, the other a flat yellow 2 pin plug for the thermocouple which measures the specimen stage temperature. Insert the power lead into the socket provided on the LH side panel and the thermocouple plug into either of the 2 yellow sockets located near the T/C power socket.

2.5 The HV lead for the Sputtering Lead is then screwed into the socket located on the base of the upright pillar at the rear of the unit.

2.6 On the RH side panel are 2 fuse holders and the electrical mains lead for the Sputter Coater. Check that the 2 fuse holders contain fuses (the HV fuse is 450mA, the power fuse is 10A for 200/240v line voltage and 15A for 100/120v line voltages). Fit the standard power plug into the electrical mains lead (a moulded plug is provided on USA models) and insert plug into laboratory socket.

1 Torr = 1.33 mbar

0,02 mbar = $1.5 \cdot 10^{-2}$ Torr

The unit is now ready for use.

3. OPERATION OF UNIT

3.1 Figure 3 shows a layout of the Front Panel.
Check the following on the Front Panel before using the unit.

(a) 'Leak' valve located on the front panel is closed (check by turning clockwise).

(b) 'Vent' valve located on the front panel is closed (check by turning clockwise).

(c) The Timer is set to the minimum position of 30 seconds.

(d) The Voltage control is set at zero.

(e) The 'Operation Switch' is at the 'off' position.

3.2 Turn the 'Operation' switch on the front panel to the 'Pump' position. The rotary pump should be activated and audible. As the work chamber is pumping down the vacuum gauge (Pirani) will indicate the state of the vacuum. Wait until the gauge $1,5 \cdot 10^{-2}$ Torr shows a ready of 0.1 mbar. The time to achieve this will depend on how long the system has been open to the atmosphere and the pump down will be particularly slow when first using a new unit. Open leak valve by rotating it about 3 complete turns. The vacuum gauge will indicate that Argon is being flushed through the system. Flush for about 30 seconds with the gauge indicating $3,76 \cdot 10^{-1}$ Torr 0.5 mbar. Close the leak valve and allow the system to pump down. It the unit is clean and free from contamination a pressure of about 0.01 mbar will be reached. $2.518 \cdot 10^{-3}$ Torr

3.3 Do not allow system to pump at ultimate pressure for long periods since this will cause backstreaming of oil from the rotary pump with subsequent deposition on the Target.

3.4 If system is not pumping well, open leak valve and allow pressure to rise to between 0.1 - 0.2 mbar. Leave pumping for about 30 minutes with Argon flow and repeat as required.

$7.5 \cdot 10^{-2} - 1.5 \cdot 10^{-1}$

3.5 Close leak valve and pump down to about 0.04 mbar. Turn 'Operation' switch on front panel to 'SET H.T' position and turn voltage control knob to 2.5kV. ThemA meter will indicate 5-10mA or less. Gradually open leak valve until milliameter reads 20mA. A plasma glow will be observed as soon as the voltage is applied and a current of about 5mA is flowing. If a gold target is fitted, the specimen stage becomes covered with a gold film.

3.6 There is always some degradation of the vacuum as soon as the discharge is switched on due to the vaporisation of volatile materials absorbed on the target and other parts. If the amount of volatiles present in the system is excessive the vacuum in the chamber will be rather poor, that is 0.1-0.2 mbar or worse. In this case the sputtering current is high and cannot be reduced to 20mA at the set voltage of 2.5kV. The following procedure should be carried out to clean the system :

$2.5 \times 10^{-2} - 1.5 \times 10^{-1}$ Torr

3.6.1 Turn operation switch to 'SET HT' and set reduced high voltage and leak valve as necessary to attain a discharge current of about 20mA.

3.6.2 Wait for the Pirani Gauge reading to degrade by about 0.05 mbar. 3.75×10^{-2} Torr

3.6.3 Switch off HT and immediately open leak valve fully for 10 seconds.

3.6.4 Repeat steps 3.6.1 to 3.6.3 until the target is clean.

If the vacuum system is kept clean, it should not be necessary to repeat this procedure.

3.7 The function of the electronic timer module can be checked in the following way. Set up the Sputter Coater as described in section 3.5. The timer should be on its minimum position of 30 seconds. Turn the operation switch to 'Control'. The plasma will extinguish but the red LED on the timer module will illuminate. The plasma can be reactivated by depressing the push button on the timer module. Sputtering will then take

place for 30 seconds (set on the timer) and the plasma will extinguish at the end of this period. At the same time a green LED will illuminate on the timer module signalling the end of the Sputtering period. This procedure can be repeated for other Sputtering times. Turn the operation switch to the 'Off' position.

3.8 Turn the vent valve fully open (3-4 rotations) to vent the system to Argon. Note there is a vent valve in the top plate which can be used to admit air to the system after sputtering. Although this is much faster than venting to Argon a longer pump down may subsequently be observed.

3.9 The check the thermoelectric cooling of the specimen stage proceed as follows. On the standard E5100 a 2 way switch is positioned to the left of the operation switch. It is labelled 'STAGE' and controls the thermoelectric cooling of the specimen holder. Select stage switch 'ON' and turn the operation switch to 'PUMP'. In this position the stage cooling is operative as the work chamber pumps down. A double thermocouple socket is located on the LH side panel (see figure 1). One of these sockets is used to monitor the stage temperature. For this purpose a spare plug is provided in the accessory pack. It can be fitted onto a Chromel-Alumel thermocouple indicator such as the Polaron MAT 1200K. The stage temperature is set at $4^{o}C$ and can be checked. This temperature can be adjusted internally if required. Separate instrutions are available for this adjustment.
On turning the operation switch to 'OFF' the thermoelectric cooling is switch off. If is desirable therefore to wait a minute or two before venting the work chamber to air so that the stage temperature is close to ambient. This is particularly true is the stage temperature is set below the dewpoint-
Note If the Sputter Coater is pumped down during the day but not used, the stage cooling should be turned off to prevent heating of the vacuum collar.

4. COATING SPECIMENS

4.1 The specimen holder is in position on the specimen stage and can be removed by hinging back the top plate and unscrewing the socket head screw with the key provided. It is designed to accept the mounts which you have specified.

4.2.1 Place the specimen mounts in the specimen holder and lock the mounts using grub screws. Place specimen holder on the stage and secrue it with the hexgonal headed screw provided. Replace the top plate and pump down the chamber by turning operation switch to 'PUMP' position.

4.2.2 When the pressure in the chamber is below 0.1 mbar, use the Argon Leak Valve to flush the chamber with argon for a period of about 10 seconds, (needle of vacuum meter should read approximately 0.5 mbar during flushing). $3.75 \cdot 10^{-1}$ Torrs

4.2.3 Allow vacuum to recover by closing leak valve and when the pressure is around 0.07 mbar repeat the flushing procedure. $5.2 \cdot 10^{-2}$ Torrs

4.2.4 After the second flushing pump down the specimen chamber to around 0.02 - 0.03 mbar. Allow 4-5 minutes for the specimen chamber to reach thermal equilibrium at around 5°C.
$1.5 \cdot 10^{-2}$ – $2.2 \cdot 10^{-2}$ Torrs

4.2.5 Turn operation switch to 'SET HT' and turn voltage control to 2.5kV. Adjust the leak valve to give a current reading of about 18mA.

4.2.6 When specimens are coated, rotate voltage knob to zero, turn operation switch to 'OFF' and open leak valve fully to vent chamber to Argon gas.
Wherever possible, avoid running the Sputter Coater in the Sputter condition for more than 10 minutes continously.
As mentioned previously the vent in the top plate can be used to admit air to the chamber after sputtering. This reduces the venting period but may result in longer periods of pumping when coating the next set of specimens.

4.2.7 Remove specimen holder, close leak valve and pump down chamber.

5. THICKNESS OF COATING

Experiments using interferometric techniques have shown that the thickness of a Au or Au/Pd coating sputtered in argon gas can be calculated at 2.5kV according to :

$th = 7.5It(\text{Å})$ (V = 2.5kV, target to specimen distance 50mm)

t = time in minutes

I = current in mA

th = thickness in Å

Average coating times will be of the order of 2-3 minutes using V = 2.5kV and I = 20mA.

Platinum targets when fitted will give approximately half the deposition rate.

ADDITIONAL INFORMATION

6. CHOICE OF ROTARY PUMP

Since the Series II 'Cool' Sputter Coater operates at lower pressures than conventional diode sputterers, it is advisable to use a higher capacity rotary pump. We recommend a 100 Lit/min 2 Stage Rotary Pump. Should this not be available, good results can be obtained with a 50 lit/min 2 Stage Pump.

Rotary Pump Suck-back

The modern rotary pumps (eg. Polaron GVD-100, Edwards E2M5) are fitted with anti-suck back devices such that if the Sputter Coater is felt under vacuum, the oil will not creep up the vacuum hose. However, we recommend that the unit be vented overnight.

7. TARAGET REPLACEMENT

The annular target is simply removed by unscrewing the aluminium ring on which it is mounted. (see figure 4).

8. ADJUSTMENT OF THE TARGET HEIGHT

The target assembly can be raised or lowered by loosening the collar on the top plate. The recommended distance between target and specimen is about 4cm (1.5 in).

9. SAFETY CUT-OUT

The electricaly system of the Sputter Coater is fitted with a protection device so that if air is admitted to the vacuum chamber, the High Voltage cuts out. The cut-out is set to operate at a pressure of about 0.1 mbar . It can be altered by adjusting a potentiometer on the Pirani P.C. Board. This potentiometer is the one located furthest from the Relay.

10. HIGH VOLTAGE FUSE

The High Voltage circuit is separately fused to blow if the current between Target and Specimen exceeds 50mA. The fuse holder is located on the Main Cabinet close to the Mains Input lead.

11. SOME COMMON FAULTS AND THEIR DIAGNOSIS

11.1 Fault : Specimens loaded, unit will not pump down.
Check : Top plate gasket is seated correctly on glass spacer.

11.2 Fault : Specimen loaded, unit pumps down very slowly.
Check : (i) Specimens or adhesive is wet and outgassing. Remove specimen and check pump down time.
(ii) Small vacuum leak.

11.3 Fault : On rotating the voltage control knob a small amount, the Plasma current exceeds 50mA.

Check : HV Cap on target assembly not locating properly and HV electrode shorting to earth.

11.4 Fault : On rotating voltage control there is no Plasma current.

Check : (i) HV fuse

(ii) Vacuum is too low (eg about 10^{-3}mbar) or too high (vacuum interlock).

11.5 Fault : Rotary pump is pumping 'hard' but vacuum gauge still indicates around 'ATM'.

Check : Probably a fault on Pirani PCB

11.5 Fault : Specimen stage not cooling.

Check : (i) Out-put transistor of Peltier power supply.

(ii) If voltage across peltier device is about suggest peltier device itself faulty.

USEFUL PARTS LIST

Targets

E5100-314A	Gold Target
E5100-314B	Gold/Palladium Target
E5100-314C	Platinum Target
E5100-314D	Nickel Target
E5100-314E	Silver Target

Specimen Holders

E5100-326A	6	½in.	Cambridge or Philips or Etec
E5100-326B	6	10mm	JEOL
E5100-326C	4	15mm	ISI
E5100-326D	6	9/16 in.	AMR

Specimen Holder fixing screw and Allen Key Set (2)

E5100-1200K Digimat Temperature Meter

E5100-1200KL Lead for Portable Digital Temperature Meter.

HV Fuse (400mA delay) Pkt of 10

Mains Fuse 10A (15A use) Pkt of 10

Gas Tubing per meter

Glass Spacer (specify external diameter which is engraved on collar base internally)

O-Ring Kit

Pirani Gauge Head

Pirani P.C. Board

1 Vacuum Hose (Nitrile Rubber) 1m length.

FIGURE 1. - Left Hand Side Panel

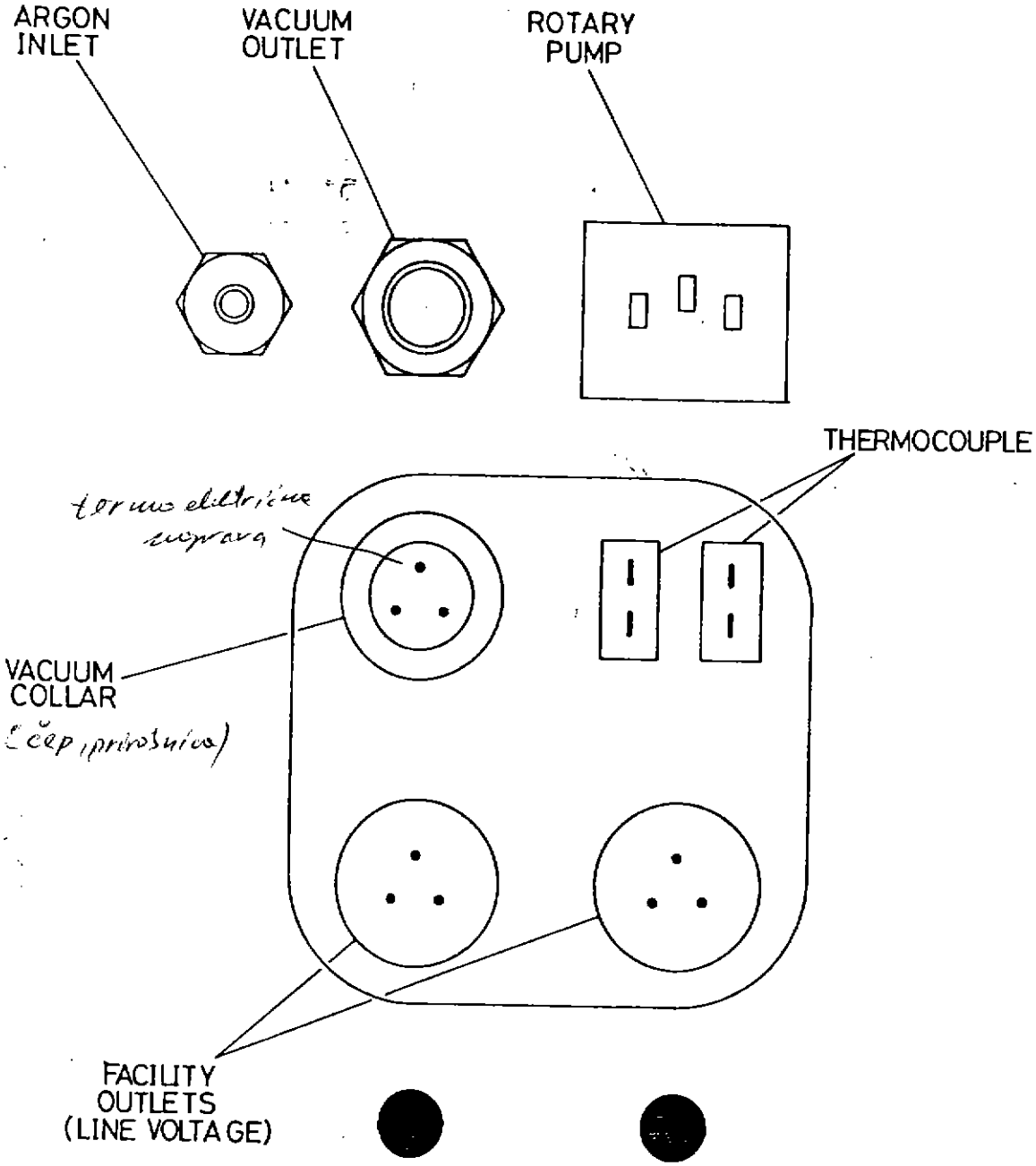
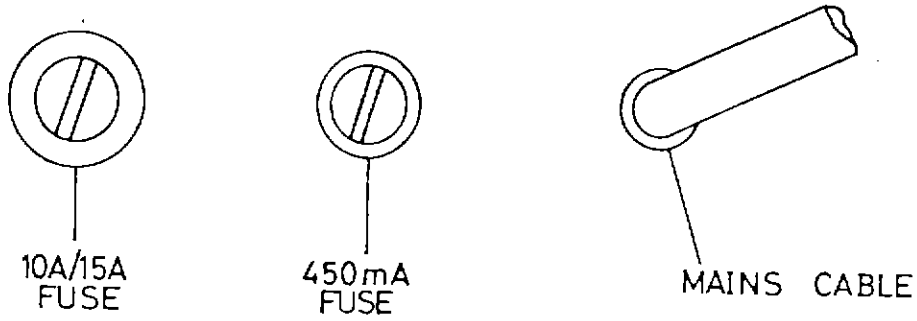


FIGURE 2. - Right Hand Side Panel



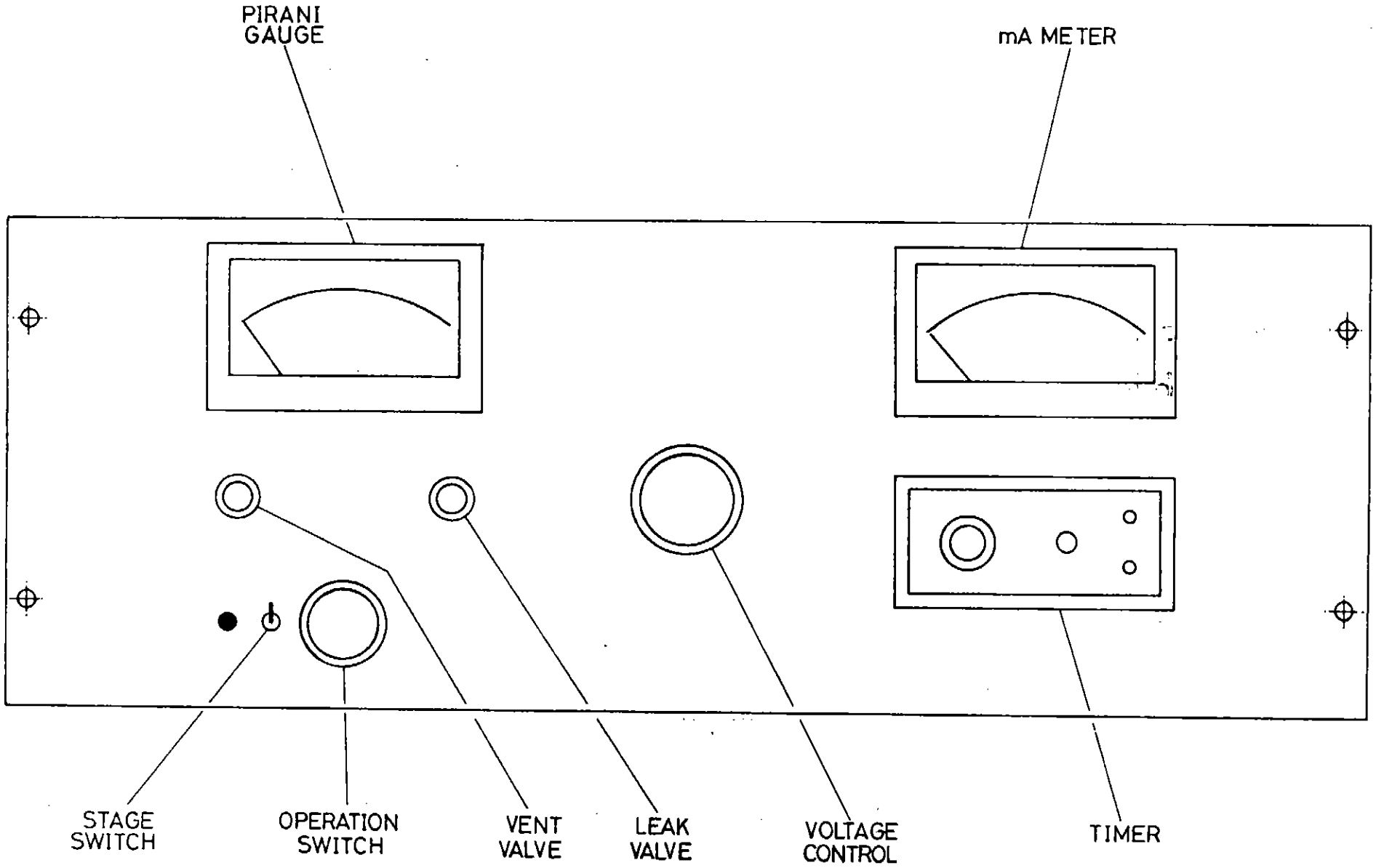
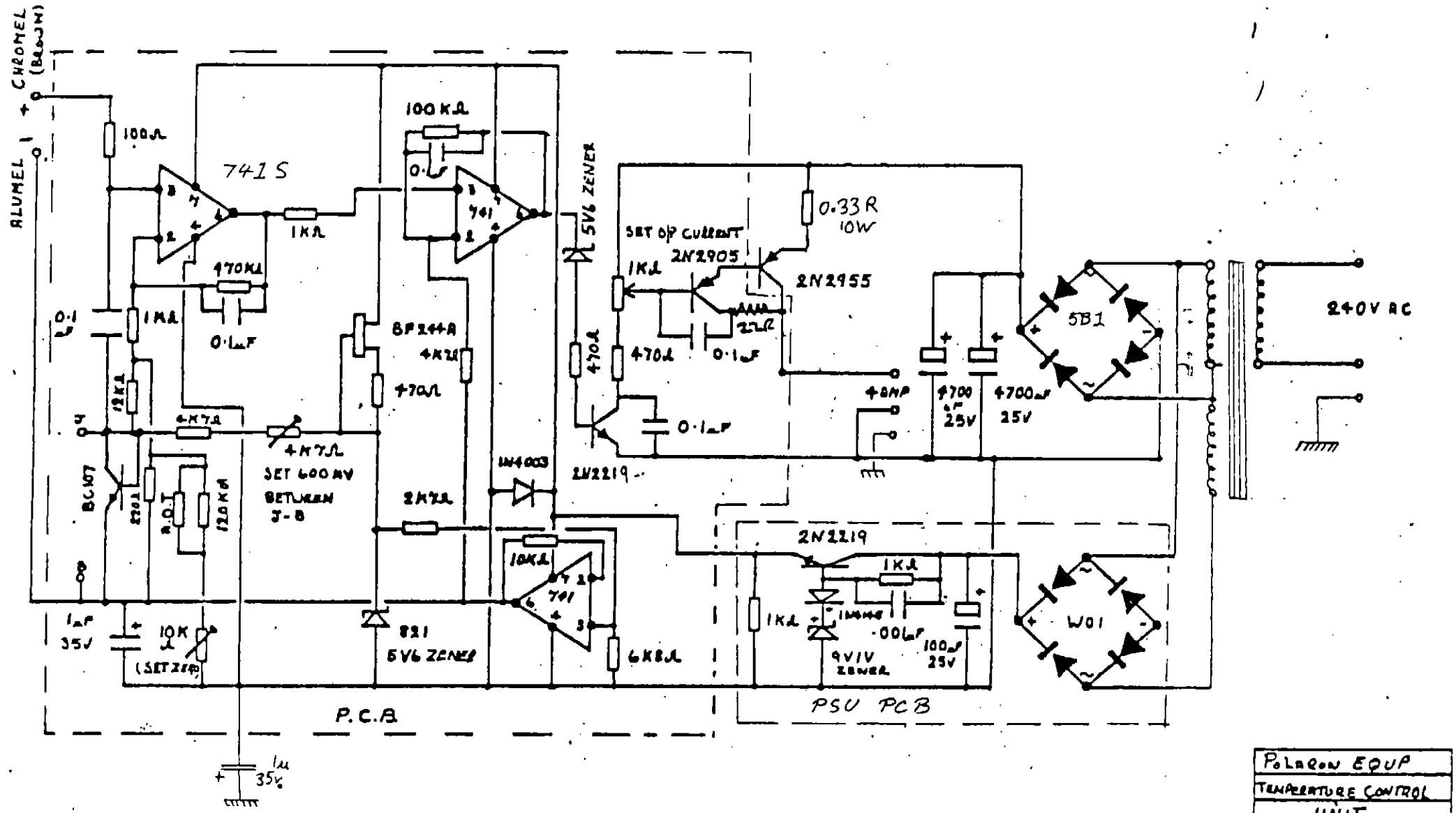
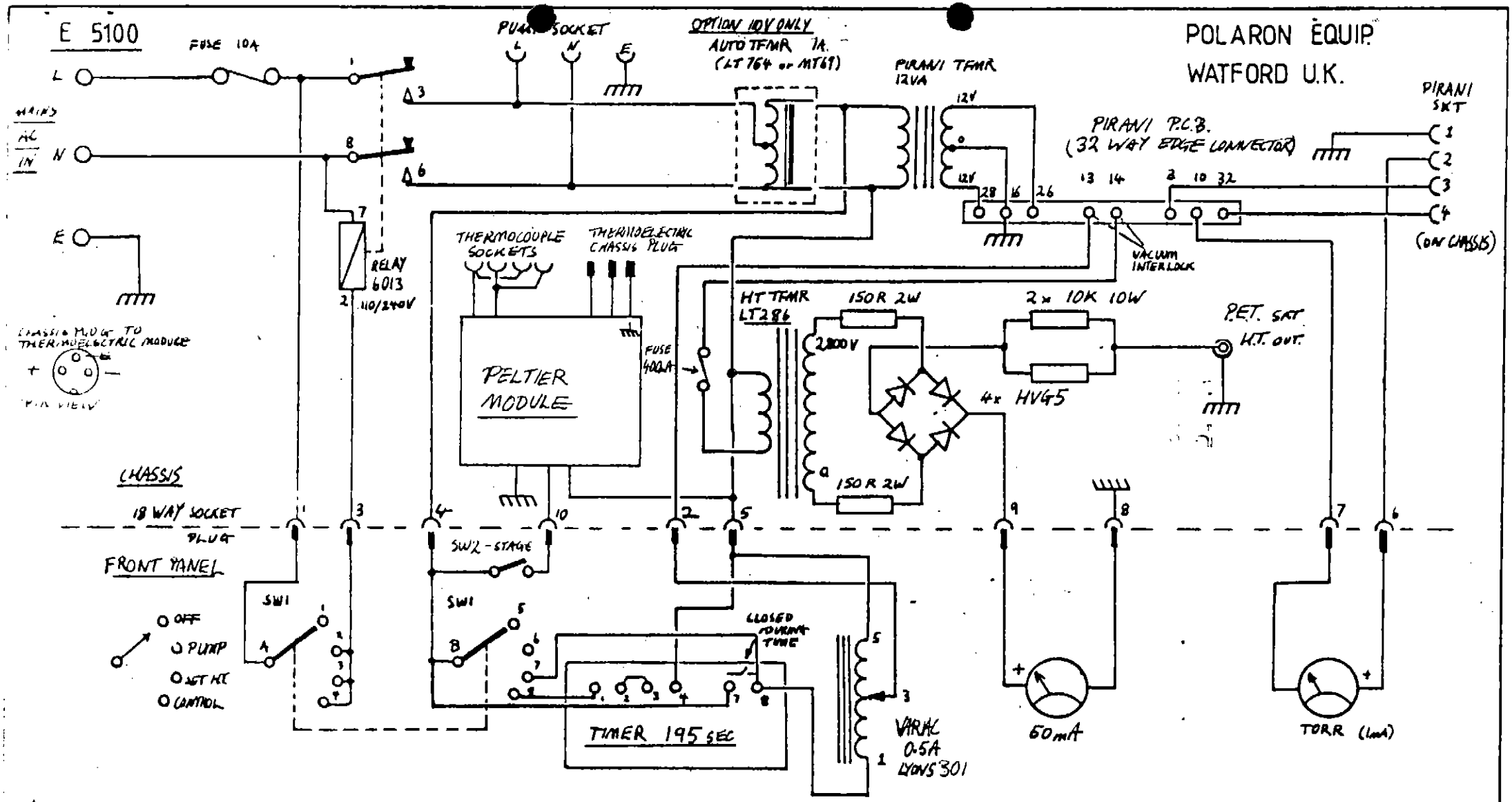


Figure 3 - Front Panel Layout



E 5100
PELTIER
MODULE

POLARON EQUIP
TEMPERATURE CONTROL
UNIT



No.	DESCRIPTION	DATE

REVISION

E 5100 SPUTTER COATER
WIRING DIAG.

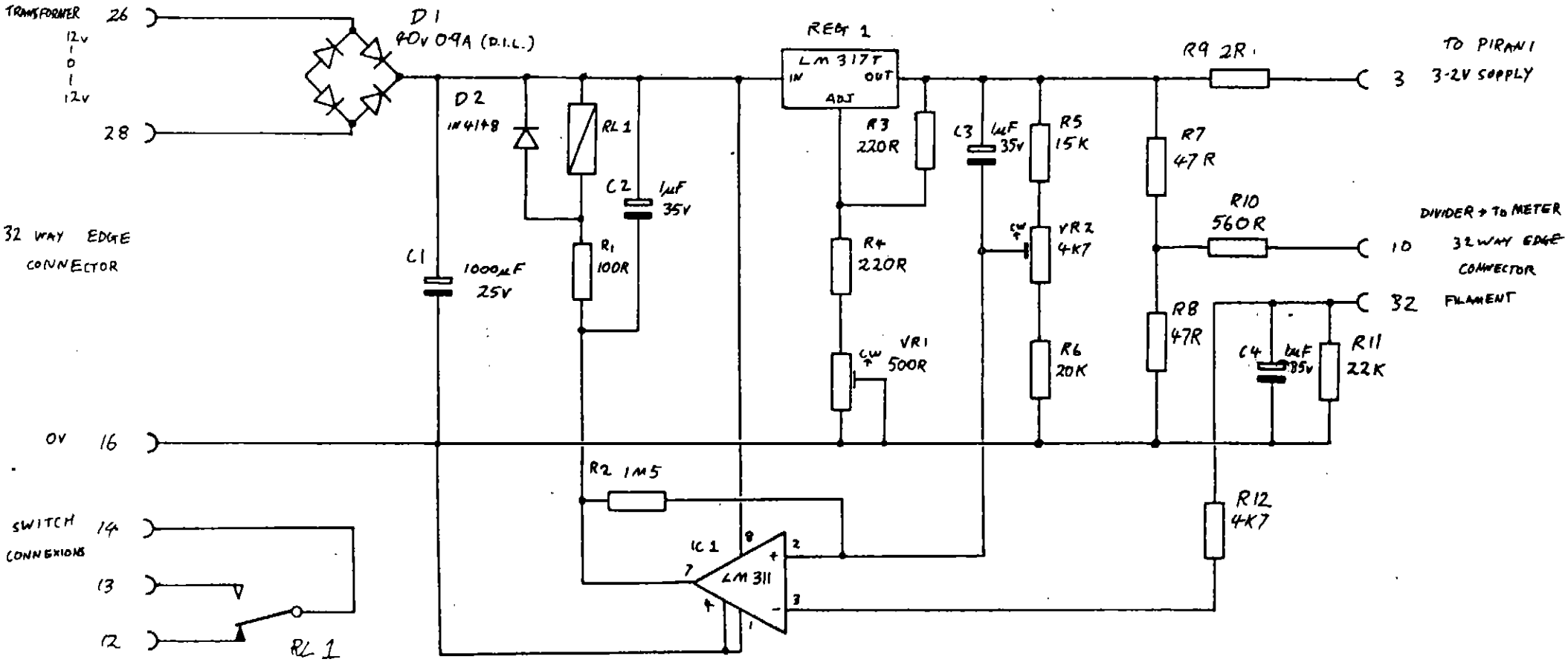
DRAWN	TRACED	CHECKED	APPROVED	DATE	SCALE
V.H.				19 MARCH 84	

DRAWING No.

297 x 210mm A4

PIRANI SWITCH iss. 5

POLARON EQUIP



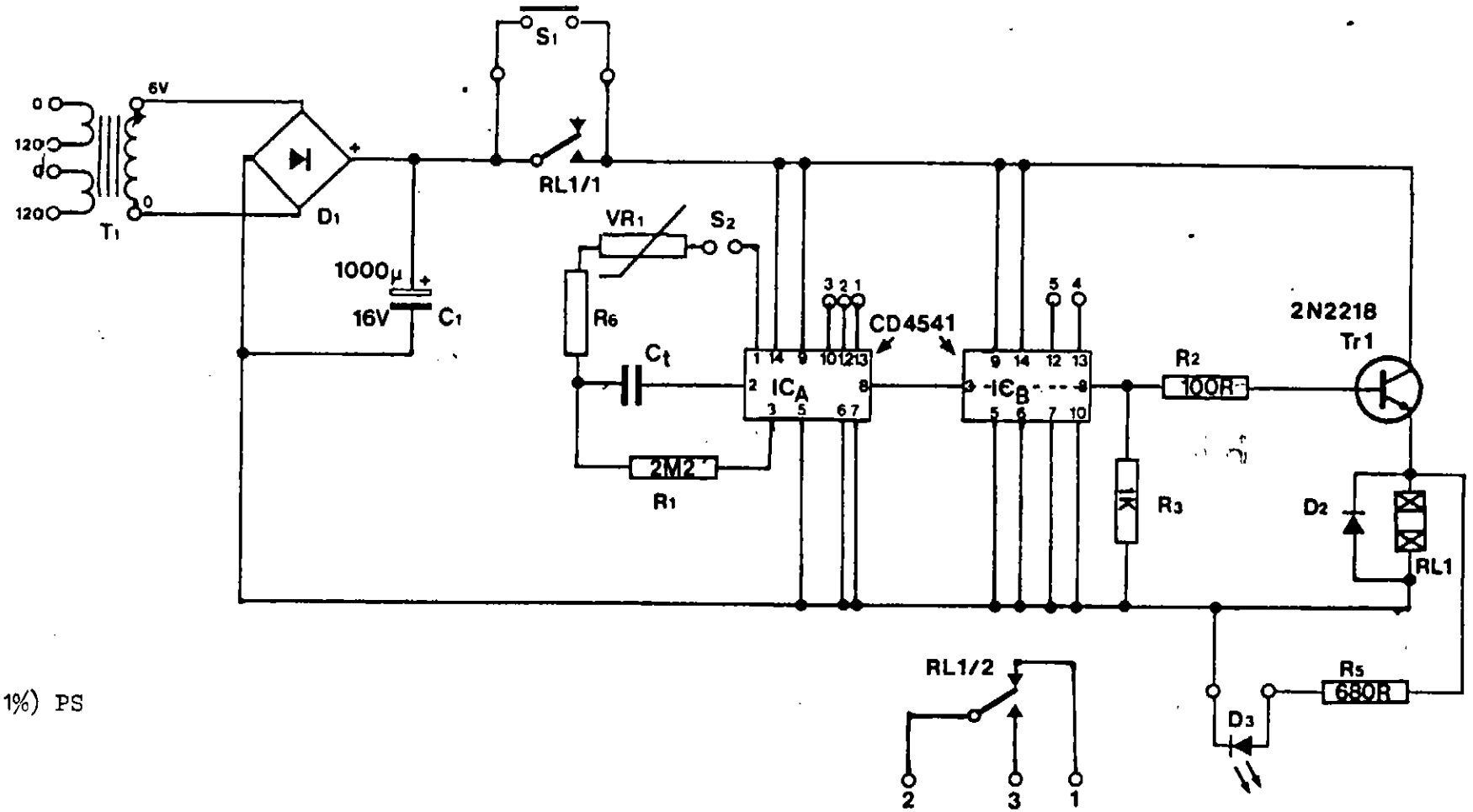
No.	DESCRIPTION	DATE
REVISION		

PIRANI SWITCH CIRCUIT DIAGRAM

DRAWN	TRACED	CHECKED	APPROVED	DATE	SCALE
KH.				13 JUNE 83	

DRAWING No.

A4



SC TIMER

C_t 8200 pF (1%) PS

R_6 24K3

S_2 11 x 24K3

ICB LINKED OUT

T_1 3VA

No.	DESCRIPTION	DATE

REVISIONS

POLARON TIMER 4541

DRAWN	TRACED	CHECKED	APPROVED	DATE	SCALE
				2/82	

DRAWING No. CIRCUIT DIAGRAM POL TIMER 4541

1WC-90 SERIES III TILTING AND ETCHING STAGE

This attachment has been designed to provide a method of etching the surface layers of a specimen prior to sputter coating (with e.g. Gold) for examination in the SEM. The etching is achieved by bombarding the specimens with argon ions accelerated by up to 2.8kV. The process generates heat in the surface layers and it may not therefore be possible to etch very sensitive materials without damaging them. The unit is for use with the E5100 Series II Sputterer. Although it can be physically fitted onto the E5000 Series I Sputter Coater, the1.4kV provided with this unit is not sufficient to give adequate etching.

DESCRIPTION OF UNIT

The unit is based on a vacuum collar having 3 entry ports. One entry port contains a tilting specimen arm with insulated specimen stage. The specimen stage can be tilted between the horizontal (sputter coat) and vertical (etch) position. The specimen block can be water cooled - an inlet and outlet nozzle being provided to which tubing can be fitted. An electrical cable terminating in a high voltage plug leads from the insulated specimen stage and can be inserted into the socket at the bottom of the pillar behind the baseplate.

A second entry port is fitted with a conical anode assembly.

A specimen holder, is available for various types of SEM mounts and screws into the insulated specimen stage, beneath the dark space shield.

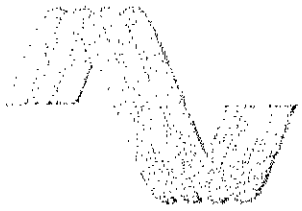
INSTALLATION ON E5100 SERIES II SPUTTER COATER

Remove the thermoelectrically cooled specimen stage from sputter coater and replace it with the tilting and etching stage.

USE

Having installed the 1WC-90 tilting and Etching stage, pump down the unit and check that there are no vacuum leaks. If required, the water hose can be connected to inlet and outlet pipes on the tilting arm.

Cont/d. .....



NORMAL SPUTTERING OF SEM SAMPLES

Tilt the specimen so that the insulated specimen stage is facing the anode, unscrew and remove the dark space shield. A central screw hole is visible in the centre of the specimen block surface now horizontal. A standard specimen holder can then be screwed onto the flat surface. Replace the dark space shield. The HT lead from the top plate is inserted in the socket in the pillar base. Gold sputtering can then be carried out in the usual way. Water cooling can be used if the specimens are extremely delicate.

ETCHING MODE

Tilt the specimen arm so that the insulated specimen stage is horizontal. Load the specimen holder containing the specimens to be etched and tilt the arm so that the specimens are facing the conical assembly.
The High Voltage lead from the insulated specimen stage is inserted into the socket in the pillar base. (The HT lead from the sputtering head is therefore disconnected.)
The unit is then pumped down to approximately 20-30 micron ($2\text{-}3 \times 10^{-2}$mmHG). Argon gas is admitted to the chamber to a pressure of approximately 8×10^{-3}mmHG and the voltage increased to give a current of about 30mA. A discharge results which is most intense between the specimen stage and the conical anode. Typical running conditions are 1.8kV at 30mA.

Higher voltages than 2.0kV are not normally practicable. Also if a current of less that 30mA is used it will be necessary to decrease the voltage. Unfortunately the pirani gauge reading is not a reliable indicator that the correct conditions are being met, so that a visual inspection of the appearance of the discharge must be made. It should always be ensured that two distinctly different regions are present in the space between the specimen stage and the conical anode. These are a dark region adjoining the specimen stage and a bright conical region adjoining the anode which is between 5mm and 15mm wide.

ETCHING RATE

A Gold coating which is sputtered onto a mount using the conditions of 2.6kV 20mA for 2 minutes approximately (150A°) will require 1.8kV at 30mA for at least 3 minutes to etch it completely.

NOTE: A - Do not run the etching process for more than 10 minutes at a time.

B - Check that the correct High Voltage Lead is inserted in the socket in pillar base.

IWC-90 TILTING AND ETCHING STAGE

Vacuum Collar containing:

(a) Tiltable specimen arm

(b) Anode assembly

(c) Blanking Plate

(d) Glass Cylinder

Specimen Holder (specify SEM) including

Allen Keys (2) and screw.

Water Hose

Extension Arm for Pillar