INSTRUCTIONAL MANUAL
CAT. 55220-501B
Twin Jet Electropolisher – Model 501B
1. Introduction

1.1. Description
The Metalthin Mk4 is a twinjet electro-polishing unit comprising thinning cell and control/power supply. It has an automatic foil perforation detection system, which uses a photocell to detect red or infra-red radiation passing through the hole produced in the foil during electro-polishing. When perforation is detected the dc supply and jetting cease automatically, and, if selected, an audio alarm will sound as well. At the termination of electro-polishing a small positive potential can be applied automatically to the foil. There is also the option to continue jetting.

The electro-polishing process can be done in automatic mode to avoid the operator having to make minor adjustments to the thinning current in response to variations in temperature and/or the electro-polishing process.

The standard dc voltage supply is smooth. However, a ripple of about 20% may be introduced. This can be beneficial when thinning multiphase materials.

1.2. Control/Power Supply
The front panel of the Control/Power Supply is shown in Figure 1-1.

1.2.1. Mains voltage
110-120v 50Hz or 220 V, 60 Hz, ac mains supply, as required.

The voltage of the supply is set by the switch in the rear of the Control/Power Supply. Ensure that the voltage set complies with the laboratory mains supply. The Serial Number is on the internal chassis.

The mains cable exiting from the rear of the Power Supply unit requires fitting with a suitable three pin plug wired to the convention:

GREEN/YELLOW – EARTH
BROWN – LIVE
BLUE – NEUTRAL

1.2.2. Output
0-100 V dc, 0-1 A continuously variable and isolated.

1.2.3. Jet pump motor supply
0-10 V dc, 0-500 mA continuously variable and isolated.

1.2.4. External pump motor supply
0-10 V dc, 0-500 mA continuously variable and isolated.

1.2.5. Meters
Voltmeter - single range; 0-100 V dc.
Ammeter - single range; 0-1999 mA dc.

1.2.6. Fuses
Mains input 2 A ac
Thinning output 1 A dc
Pump supply (internal) 1 A dc
Pump supply (external) 1 A dc
1.2.7. **Front Panel - Switches**

**MAINS** - Activate the power supply by inserting the key into the KEY SWITCH and turning it.

The **PUSH SWITCHES** illuminate when their function is operative.

**TRIPPED** (red) - Pressing this switch re-sets the functions cancelled when the perforation detector is activated by light. The sensitivity of the detector is controlled by the knob labelled **SENSITIVITY**.

**RIPPLE** (blue) - Pressing this switch causes the dc supply to the specimen to vary by about ± 20% of the set voltage. Initiating RIPPLE in the latter stages of thinning may assist in retaining particles in multi-phase alloys.

Note: **RIPPLE** will not operate during **CURRENT CONTROL** conditions.

**AUDIO** (blue) - Pressing this switch activates an audible alarm that sounds when perforation is detected.

**REVERSE VOLTAGE** (orange) - Pressing this switch activates a circuit that supplies a small reverse potential to the specimen when perforation is detected to prevent corrosion by the electrolyte. Note: the reverse potential does not show on the voltmeter.

**CONTROL CURRENT** (yellow) - Pressing this switch activates a circuit that automatically adjusts the thinning current to compensate for variations in the temperature and electro polishing process. This avoids the operator having to make small adjustments whilst the specimen is thinning. The range of current conditions over which automatic adjustment will occur is limited. If they are exceeded, then the illumination will change from constant to flashing. When the current control switch is pressed the current drops – increase the dc output to restore it to the required level.

**JETS TRIP CANCEL** (orange) - Pressing this switch cancels the automatic pump switch-off that occurs when perforation is detected and jetting continues

**NOTE:** When the JETS TRIP CANCEL is activated the flow of electrolyte through the jets continues after perforation. Extra care must be taken when removing the specimen holder to avoid splashes of electrolyte from exiting via the slot in the cell lid. To avoid splashes the JETS TRIP CANCEL must be de-activated immediately before removing the specimen holder.

**JETS START** (orange) - Pressing this switch starts the electrolyte pump. Initially the pump operates at full speed to fill the jetting chamber quickly. The speed then reduces automatically to that set by turning the knob labelled JET SPEED. **Do not operate unless immersed in liquid, which is required for lubrication.**

**COOL START** (blue) - Pressing this switch activates an external UNIPUMP, see Section 7.1, that circulates coolant through the cooling coil in the thinning cell. Turning the adjacent knob controls the pumping speed. **Do not operate unless immersed in liquid, which is required for lubrication.**
1. Introduction, continued

1.2.8. Front panel - Lamps
RUNNING (green) - This lamp illuminates when the perforation detector is not receiving sufficient light to activate it.

1.2.9. Front Panel - Control knobs
SENSITIVITY - This knob controls the sensitivity of the detector. The working range is between 7 and 10. Use the maximum sensitivity (i.e. 10) to produce the smallest perforation.

DC OUTPUT - Turning this knob clockwise increases the dc supply to the cell as shown on the meters. The maximum voltage is approximately 100 v. The current produced by any set voltage will vary as a function of electrolyte composition and temperature. After perforation the voltage reading will increase because the current is switched off.

JET SPEED - This knob controls the speed at which electrolyte emerges from the jets after the pump has run at full speed to fill the jetting chamber. The usual working range is 3 to 5. Do not operate unless immersed in liquid.

UNIPUMP SPEED - This knob controls the speed at which the Unipump (see Section 7.1) circulates liquid nitrogen or other coolant through the cooling coil. Do not operate unless immersed in liquid.

1.3. Thinning cell
The cell, see Figure 1-3, Figure 1-4 and Figure 1-5, is constructed from solid PVC and PTFE. The parts are bolted together with stainless steel screws so that it can be completely disassembled if necessary. The main parts are:

a) twin-jetting unit with integral pump and motor
b) electrolyte tank that push-fits into a stand to provide additional stability, and to collect drips of electrolyte
c) electrolyte cooling coil.

Electrolyte in the tank is drawn into the pump chamber and pumped through the internal flow channels. It emerges from the jets into the jetting chamber where it becomes charged with electricity from the platinum wire electrodes coiled around the jets. The electrodes are connected to a terminal post in the lid.

The temperature of the electrolyte may be measured by inserting a suitable thermometer through the hole in the lid and into the corresponding hole in the base of the jetting unit.

1.4. Specimen holder
An exploded view of the specimen holder is shown in Figure 1-6. The specimen holder has a small insert and a large insert. The large insert should not normally be disturbed, except to gain access to the platinum foil. The small insert is used for loading and unloading of the disc specimen that is placed in the shallow recess on the inner face. The foil lies on a circle of platinum wire that connects to the spade fitting on top of the holder.

Two keys are provided; one for screwing in and unscrewing the small insert, the other fits the large insert.

DO NOT UNSCREW THE LARGE INSERT except to replace the platinum foil and then take particular care not to screw it in too tightly, see Section 5.2

1.5. Connecting leads
The connecting leads are all standard wire cables held within a spiral binding that can be unwrapped easily to allow leads to be replaced.
Figure 1-1: Control/Power Supply (front)

Figure 1-2: Control/Power Supply (back)
Figure 1-3: Cross-section of thinning cell (schematic)

Figure 1-4: Plan view of thinning cell
Figure 1-5: Thinning Cell (Schematic)
2. Setting Up

2.1. Safety precautions

The electrolytes used in foil preparation are highly corrosive and all precautions must be taken to avoid contact with skin and eyes.

Suitable gloves, eyes protection and a laboratory coat must be worn. If possible, install and operate the thinning cell in a fume cupboard. DO NOT operate the pump when the specimen holder is removed.

DO NOT operate the Metalthin pump without immersion in a liquid.

2.2. Interconnections

The leads that connect the thinning cell to the power supply are held within a spiral binding. Each of the DIN plug connectors has a unique pin configuration. They can only plug into the sockets on the back of the power supply with a matching configuration.

a) **DETECTOR** lead: Push the DIN plug with 5 pins positioned over 240 degrees into the corresponding socket on the power supply labelled **Detector**, see Figure 1-2. Push the stainless housing into the window on the right hand side of the cell as viewed from the cable clamp end (see Figure 1-4). If required the housing can be securely fastened into the window socket by gently turning the grub screw in the side of the socket using a 1.5mm (Allen) hexagonal key.

b) **LAMP** lead: Push the DIN plug with 3 pins into the corresponding socket on the power supply labelled **Lamp**, see Figure 1-2. Push the stainless housing into the other window socket (see Figure 1-4). If required the housing can be securely fastened into the window socket by gently turning the grub screw in the side of the socket using a 1.5mm (Allen) hexagonal key.

c) **PUMP** cable: push the DIN plug with 5 pins positioned over 180 degrees into the corresponding socket on the power supply labelled **Jets** (see Figure 1-2).

d) **DC CURRENT** lead: the black cable that contains a red and a blue wire is fitted to a DIN plug with a round and flat pin. It goes into the socket labelled **Thinning current** in the rear of the Supply Unit, see Figure 1-2. Push the connector on the end of the **RED** wire onto the spade terminal on the specimen holder (see Figure 1-3 and Figure 1-4). Push the connector on the end of the **BLUE** wire onto the spade terminal on the tank lid (see Figure 1-3 and Figure 1-4).

e) **UNIPUMP** lead: push the DIN plug into the socket on the power supply labelled **Unipump Cool** (see Figure 1-2). Do not operate unless immersed in liquid because it relies on liquid for lubrication and will be damaged without it.

When the leads contained by the spiral wrap have been connected to the power supply they can be secured in a cable clip at either side of the case. Place the cables in the clip and the fasten it. To unfasten the clip insert the tip of a medium size screwdriver where the clip fastens and prise it apart.
2. Setting Up, continued

Loading a specimen

a) Unscrew the small insert from the specimen holder using the small key provided.

b) Place the disc specimen into the central recess in the rear of the small insert. Note: The most consistent results are obtained with discs that are 0.1 to 0.15 mm thick.

c) Screw the small insert back into the holder. Do not use excessive force since this might damage the platinum foil electrode in the holder.

d) Place the specimen holder into the slot in the lid of the thinning cell.

2.3. Charging the thinning cell with an electrolyte

CAUTION:
Make sure that the thinning cell is clean and free from any contamination.
Prepare 220 ml of electrolyte, remove the lid, plus jetting unit, then pour the electrolyte into the tank. Replace the lid ensuring that the jetting unit is properly located in the recesses on either side of the tank.

CAUTION:
DO NOT spill or drip electrolyte onto the leads. If electrolyte is seen on the leads, wash off immediately with water and dry with a paper towel.
Take particular care that electrolyte does not enter the detector and lamp housings.

3. Operation

3.1. Setting the controls

Before switching on:

a) Ensure that there is a specimen holder in the slot in the tank lid and that the JETS READY push switch is OFF.

b) Set the OUTPUT control to the fully counter-clockwise position, the SENSITIVITY to 1 and the JET SPEED control to 3 or 4.

c) Set the AUDIO alarm, REVERSE VOLTAGE, JET TRIP OFF, and RIPPLE switches as required.

3.2. Specimen thinning

a) Obtain the desired electrolyte temperature.

b) Operate the MAINS switch to energize the unit. If the TRIPPED red switch illuminates press it to reset it.

c) When the RUNNING green switch is illuminated turn the SENSITIVITY control to 10 for maximum sensitivity. During testing light through 25 micron hole was detected with the sensitivity set between 8 and 9.

d) Adjust position of the SET OUTPUT knob to obtain the desired voltage and/or current indicated by the meters.

e) Press the JETS READY switch to start the thinning process. The pump will run at full speed initially, to fill the jetting chamber quickly, and then slow down to the pre-set pumping speed.

3.3. Specimen perforation

When perforation is detected the TRIPPED red switch will illuminate, jetting will cease and the thinning current will be switched off. If set, the AUDIO alarm will sound and a REVERSE VOLTAGE will be applied to the specimen. The switches appropriate to these two functions will illuminate.

If the JET TRIP OFF switch has been set jetting will continue.
3. Operation, continued

3.4. Specimen washing and drying
Immediately a perforation is indicated remove the specimen holder from the thinning unit and wash with methanol or ethanol. Then remove the specimen from the holder and, holding it with tweezers, wash again with alcohol. Drop the washed disc onto a laboratory tissue and dry it gently.

Examine the specimen in the TEM as soon as possible.

3.5. Repeat thinning
To thin another specimen under the same conditions, place it in the specimen holder, fit the holder into the slot in the thinning cell and press the TRIPPED red switch.

3.6. Low temperature operation
The Metalthin is ideally suited for operation at low temperatures because the small volume of electrolyte can be cooled easily and quickly.

The power supply has a separate power and control facility for driving the Unipump (available as an optional extra) to send coolant from a modified Dewar flask, via Neoprene tubing, through the cooling coil in the electrolyte tank.

Before using the Metalthin at low temperature make sure that the unit is thoroughly clean and dry. Any residual moisture will become ice crystals and may cause seizure of the pump or blockage of the jets.

Electrolytes containing acetic acid can partially freeze below about -10°C, causing the pump to behave erratically resulting in vibration.

During prolonged low temperature operation condensation/ice may form on the windows inside the jetting unit. This will impair the operation of the perforation detection system. To prevent condensation/ice on the windows: unplug the lamp and detector housing from the cell by holding the end of the PVC socket and pulling it and the stainless steel housing, without turning, directly out from the knurled PVC bush, as shown in the photograph, then direct a fine spray of methanol onto the widows. Note that the stainless steel housings are a sliding fit into the knurled PVC bush, but may have been secured by the stainless steel grub screw in the PVC bush. DO NOT unscrew the PVC bush - it presses the window against the PTFE “O” ring forming a seal that prevents the electrolyte from gaining access to the lamp and to the detector.

4. Troubleshooting
If consistent results are not produced, the cause is often a simple one that can be solved with the aid of the following notes, summarized in Table 1. Please do not hesitate to contact EMS for further advice.

4.1. Electrolyte
a) Contamination: this can occur by various means and is often the cause of poor results. A fresh charge should be made from Analar grade reagents using very clean and dry measuring and storage containers. The thinning cell should be kept thoroughly clean and dry.

b) Temperature: is the cooling being maintained and the temperature being monitored correctly? Is the temperature too low for the electrolyte being used?

c) Volume: Check that the level of electrolyte in the tank is correct. f it is too low the efficacy of jetting will be impaired. Air bubbles may form inside the jets and reduce the sensitivity of the detection system.
4. Troubleshooting, continued

4.2. Specimen Characteristics  
Small changes in specimen composition and heat treatment can affect the thinning process. Is the specimen exactly the same as thinned previously?

4.3. Electrical Interconnections  
Ensure that all leads are connected as described in Section 2.2. It is possible to plug the lamp connector into the jets socket. No damage will result, but the lamp will not operate.

4.4. Electrical Continuity  
An abnormally high electrical resistance in the cell and/or sample holder will result in a lower current for a specified voltage and poor results. NOTE: If the voltage and current readings are not those expected, check the continuity of all electrical connections leading to the cell and sample holder, and those from the terminal on the cell top to the platinum wiring around the jets. Also check the continuity from the spade terminal on the sample holder to the platinum foil, see Section 4.5.

4.5. Specimen Holder  
a) Check that the hole diameter in the small insert is correct for the disc, i.e. for 3 mm discs the hole should be about 2 mm and for 2.3 mm discs about 1.6 mm.

b) Check that the platinum foil has not been damaged and that there are no particles of dirt on the inner surface of the holder and none on the insert.

c) In the top of the holder there is a screw to which the spade terminal is attached, see fig 1.4. Do not move the screw counter clockwise because doing so will reduce electrical contact inside the holder and result in poor quality foils.

4.6. Pump Speed  
a) If the pump speed is too low the electrolyte chamber may not fill sufficiently for jetting to occur beneath the surface of the electrolyte.

b) Avoid using too high a pump speed because turbulence can affect the quality of the polish and cause off-center perforation. Also powerful jets can damage very thin foils.

4.7. Non or impaired Jetting during Pumping  
a) Check that the universal coupling connecting the motor shaft to the pump shaft is securely fastened to both shafts.

NOTE: In the following operation use ONLY WATER in the electrolyte tank WEAR THE PROTECTIONS specified in Section 2.1.

b) Check that the jets have equal velocity. Remove the specimen holder, place a piece of glass over the slot in the lid, operate the pump and observe. If the jet velocities are not equal, inspect for particles of matter in the jets, jet chamber and other channels.

4.8. Deterioration in detection of perforation  
During operation at low temperatures, condensation/frost can form on the windows resulting in impaired light transmission. This results in larger perforations. To resolve the problem, unplug the light source and detector, and spray methanol on the windows.
### 4.9 Troubleshooting Table 1: Summary of Operational Faults and Possible Cause

<table>
<thead>
<tr>
<th>FAULT IDENTIFIED</th>
<th>POSSIBLE CAUSES</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Poor detection of perforation</td>
<td>Opaque electrolyte, Debris in jets, remove widows and look through jets to check, light source extinguished, remove lamp holder from tank to check, air bubble in jet/window area, ice condensation on window, Surface contamination on widow needs removing.</td>
</tr>
<tr>
<td>2 Trip will not reset</td>
<td>Light entering cell, condensation on detector.</td>
</tr>
<tr>
<td>3 Poor or no jetting</td>
<td>Jets blocked by debris/ice, remove widows and look through jets to check, air bubble in pump chamber, internal flow channel(s) blocked, remove screws from T piece, insert 2.5mm rod into flow channels to clear them, electrolyte level too low, pump shaft not turning but motor is, remove motor cover and check if universal joint is turning, if it is then tighten grub screws.</td>
</tr>
<tr>
<td>4 No polish on one side of specimen</td>
<td>The platinum wire to the jet has become disconnected from the terminal underneath the lid or broken somewhere, The flow of electrolyte through the jet is not happening because: a) there is a blockage in the jet or b) there is a blockage in the internal flow channels, c) the platinum foil in the specimen holder has been replaced but no hole was made in it after fitting.</td>
</tr>
<tr>
<td>5 Poor polish on one side of specimen</td>
<td>Electrode foil damaged, specimen not held against foil one jet blocked. Sample holder spade screw has been unscrewed causing poor electrical contact inside the holder. It should be at close to a right angle to the face of the holder as shown in Fig 1.4.</td>
</tr>
<tr>
<td>6 Specimen not held in holder</td>
<td>Small insert needs refacing after being worn (use fine abrasive paper), platinum foil worn/damaged.</td>
</tr>
<tr>
<td>7 Non operation after switching on</td>
<td>Interconnections not as described in Section 2.2, Fuse(s) blown.</td>
</tr>
<tr>
<td>8 Excessive vibration of pump assembly</td>
<td>Insufficient electrolyte in tank, freezing of electrolyte.</td>
</tr>
</tbody>
</table>
5. General Notes

5.1. Electrolyte Removal and Storage
Empty the tank and flush the cell with methanol. This MUST be done when changing electrolytes and at the end of every working day.

Note: the safety procedure in Section 5.5 before flushing with methanol or changing electrolyte.

Prepare and store electrolytes in accordance with safety regulations.

5.2. Replacing the Platinum Foil
Use the larger key to remove the large insert. Remove the old foil and clean the inside of the specimen holder with alcohol. Take care not to dislodge the platinum wire from the circular recess. Cut small pieces off each corner of the new foil so that it just fits into the base of the recess. (It is advisable to start by cutting off very small pieces and then trying the fit because diameter of the base of the recess is slightly larger than the internal diameter of the screw threads.) Place the new foil in position over the platinum wire and replace the large insert. Tighten the large insert CAREFULLY trapping the foil against the small insert. Use a pin to pierce the foil and then finish the hole with a sharp drill (2mm diameter for 3 mm disc samples). Make sure that the edges of the foil are free of slivers of platinum. These can have a deleterious effect on the polishing process.

5.3. Cleaning and Removal of Jets
To gain access for cleaning, remove the lamp and detector housings, unscrew the knurled PVC fittings and remove the windows. The holes (1.6 mm) in the jets may be cleared in situ using a soft wire probe, e.g. solder wire.

To remove a jet, take off the sidepieces, place a piece of plastic rod against the rear face and then give the rod a sharp tap. Removal of a jet must be done CAREFULLY to avoid damaging the platinum wire electrode that is attached to the jet and the underside of the thinning tank lid.

5.4. Foil Thinning from One Side Only
Choose the side of the disc to be thinned and place this side against the platinum foil in the specimen holder. Cover the disc with a thin, circular, light microscope glass slide to protect it from the electro-polishing process. Screw the small insert into the holder carefully, trapping the disc between the glass and platinum foil. Thinning will occur at the large insert side only. Perforation will be detected in the usual way through the glass.

5.5. Use of Solvents for Cleaning the Thinning Cell
DO NOT use acetone. Methanol or ethanol will not affect the material of the cell.

SAFETY WARNING: Some electrolytes are not chemically compatible with methanol or ethanol and explosions can occur if such electrolytes come into contact with ethanol/methanol.

BEFORE introducing ethanol/methanol to the cell for cleaning purposes OR as a constituent of a new electrolyte CHECK that any electrolyte residue in the cell is compatible with ethanol/methanol.

If cleaning or removal of residual moisture from the cell has been done using ethanol/methanol, CHECK that an electrolyte is compatible with ethanol/methanol BEFORE introducing it into the cell.

When you are SURE of the chemical compatibility of the old and new electrolyte with methanol and ethanol, cleaning and removal of residual water may be done using methanol/ethanol. Charge the cell with 220 ml and run the pump for 5-10 minutes.

If you are UNSURE about the chemical compatibility of methanol/ethanol with the electrolyte that is to be use, WAIT 24 hours for the methanol/ethanol to evaporate from the internal passages in the thinning cell BEFORE charging the cell with the electrolyte.
5. General Notes, continued

5.6. Fitting a New Light Source
The light source is polarity sensitive. The longer of the two wires is POSITIVE + and has a RED sleeve for identification. The shorter wire is NEGATIVE - and has a BLUE sleeve.

1. Hold the cap and unscrew the STAINLESS STEEL HOUSING to expose the light source. The light source is plugged into a white socket that is identified for polarity by RED + and BLUE - markers.
2. Remove the light source and keep it as a standard for the length of the leads.
3. MAINTAIN POLARITY while cutting the two leads on the new light source to the same length as those on the old source.
4. Plug in the new light source with the longer lead in the side marked RED.

5.7. Fitting a New Detector
The detector has four pins that plug into the socket that is revealed when the stainless steel housing is removed (refer to Figure 5-1).

1. Hold the cap firmly so that it cannot rotate. Unscrew the stainless steel housing to reveal the old detector.
2. Pull out the old detector.
3. Insert the new detector pins into the socket with the tab on the detector aligned with the paint mark on the side of the socket. Push in firmly, without undue pressure, until the detector is fully in position.
4. Hold the cap and screw on the stainless steel housing.

5.8. Fitting a New Coupling to the Motor Shaft
When the Thinning Cell is assembly is submerged in liquid nitrogen the PVC contracts more than the stainless steel shaft. To allow for this: a) the shaft is a sliding fit in the lower part of the coupling and b) the coupling is positioned so that the top of the shaft does not contact the upper part of the coupling.

1. Remove the motor cover and unscrew the four sockets screws holding the motor to the stainless steel support pillars.
2. Lift the motor off the pillars to gain access to the old coupling.
3. Measure the distance between the face of the motor and the upper face of the coupling.
4. Remove the two grub screws holding the coupling to the motor shaft and slide the old coupling off the shaft.
5. Fit the new coupling in exactly the same position as the old one, tighten the grub screws and replace the pump taking care to see that the top of the shaft fits into the slot in the lower part of the coupling.

NOTE: If you are fitting a new motor the new motor shaft must be the same length as the old one. If it is not then part of it must be cut-off.
5. General Notes, continued

Figure 5-1: Fitting a New detector

Enlarged end view of detector, fitted into the socket
### 6.1 Metalthin Parts List and Order Codes

<table>
<thead>
<tr>
<th>CODE</th>
<th>ITEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>501/001</td>
<td>Specimen holder for 2.3mm specimens</td>
</tr>
<tr>
<td>002</td>
<td>Specimen holder for 3.0mm specimens</td>
</tr>
<tr>
<td>003</td>
<td>Tee piece body</td>
</tr>
<tr>
<td>004</td>
<td>Pump chamber</td>
</tr>
<tr>
<td>005</td>
<td>Pump chamber insert</td>
</tr>
<tr>
<td>006</td>
<td>Impeller rotor (available as an assembly with 012)</td>
</tr>
<tr>
<td>007</td>
<td>Jetting chamber sides Mk3 (pair)</td>
</tr>
<tr>
<td>008</td>
<td>Jets standard 1.5mm (other sizes to special order)</td>
</tr>
<tr>
<td>009</td>
<td>Screwed mountings for detector and lamp plugs (pair)</td>
</tr>
<tr>
<td>010</td>
<td>Windows and PT FE ‘O’ rings (pairs)</td>
</tr>
<tr>
<td>011</td>
<td>Pillars for motor mounting (set four)</td>
</tr>
<tr>
<td>012</td>
<td>Stainless steel shaft (available in other materials)</td>
</tr>
<tr>
<td>013</td>
<td>Motor cover</td>
</tr>
<tr>
<td>014</td>
<td>Lid to assembly</td>
</tr>
<tr>
<td>015</td>
<td>PTFE screwed bush</td>
</tr>
<tr>
<td>016</td>
<td>Electrolyte/washing tank</td>
</tr>
<tr>
<td>017</td>
<td>Platinum wire for specimen holder</td>
</tr>
<tr>
<td>018</td>
<td>Platinum wire for cell electrodes</td>
</tr>
<tr>
<td>019</td>
<td>Terminal screw stainless steel with washer and nut</td>
</tr>
<tr>
<td>020</td>
<td>Stainless screws tee piece-pump chamber (four)</td>
</tr>
<tr>
<td>021</td>
<td>Stainless screws Jetting chamber sides (eight)</td>
</tr>
<tr>
<td>022</td>
<td>Stainless screws motor pillar (eight)</td>
</tr>
<tr>
<td>023</td>
<td>Cable clamp and stainless screws (two)</td>
</tr>
<tr>
<td>024</td>
<td>Stainless steel screws lid-tee piece (four)</td>
</tr>
<tr>
<td>025</td>
<td>Motor cable complete with plug</td>
</tr>
<tr>
<td>026</td>
<td>Motor</td>
</tr>
<tr>
<td>027</td>
<td>Universal coupling</td>
</tr>
<tr>
<td>028</td>
<td>Platinum foil electrode for specimen holder</td>
</tr>
<tr>
<td>029</td>
<td>Motor plate (stainless steel)</td>
</tr>
<tr>
<td>030</td>
<td>Connecting leads PSU-Cell (pair)</td>
</tr>
<tr>
<td>031</td>
<td>Lamp pack</td>
</tr>
<tr>
<td>032</td>
<td>Fuse pack</td>
</tr>
<tr>
<td>033</td>
<td>Specimen holder key small</td>
</tr>
<tr>
<td>034</td>
<td>Specimen holder key large</td>
</tr>
<tr>
<td>035</td>
<td>Detector lead without the detector, PVC sleeve and SS housing</td>
</tr>
<tr>
<td>036</td>
<td>Lamp lead without the lamp, PVC sleeve and SS housing</td>
</tr>
<tr>
<td>037</td>
<td>Extension lead for motor pump (state length required)</td>
</tr>
<tr>
<td>038</td>
<td>Infra-red lamp visible</td>
</tr>
<tr>
<td>039</td>
<td>Stand for electrolyte tank</td>
</tr>
<tr>
<td>040</td>
<td>Photocell detector for Mk4 version only</td>
</tr>
<tr>
<td>041</td>
<td>Photocell sleeve (PVC)</td>
</tr>
<tr>
<td>042</td>
<td>Lamp sleeve (PVC)</td>
</tr>
<tr>
<td>043</td>
<td>Stainless Steel housing for either lamp or detector</td>
</tr>
</tbody>
</table>
7. Metalthin Parts List and Order Codes, continued

7.1. UNIPUMP
The pump power supply and control built into the Metalthin Mk3 is designed to drive the Materials Science UNIPUMP. The Unipump circulates coolant from a modified Dewar Flask through the cooling coil in the thinning cell.

7.2. Precision Shear
The Materials Science PRECISION SHEAR is a miniature shear designed for laboratory use. It cuts strips of metal from sheet without general distortion. It is particularly useful when reducing the width of samples prior to punching out discs for TEM work.

7.3. Disc Punch
The Materials Science UNIDISC is designed especially for the rapid production of disc specimens from strip.

Punches are available for producing discs of 1 to 6mm diameter.

7.4. Graduated Grinding Jig
Discs can be reduced in thickness uniformly, and in a controlled manner, using the Materials Science GRADUATED GRINDING JIG. Jigs are available for discs of 1 to 6mm.

7.5. Laboratory Spark Erosion Machine
The Materials Science SPARK EROSION MACHINE can perform a variety of machining tasks including wire cutting. It is used extensively for producing discs for TEM work. The unique safety features make it suitable for use by unskilled personnel.

7.6. Diamond Saw
The Materials Science DIAMOND SAW is a low cost saw designed for general laboratory use where cutting, without damaging the underlying structure, is required.

7.7. High Speed Slitting Saw
The Materials Science HIGH SPEED SLITTING SAW is a small general purpose saw primarily used for cutting metals. However, it will cut a variety of other materials, e.g. ceramics, plastics, fiberglass.

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>55222-32</td>
<td>UniPump Cryogenic Pump</td>
</tr>
<tr>
<td>55222-40</td>
<td>Dewar Flask (Optional Accessories)</td>
</tr>
<tr>
<td>55222-06</td>
<td>Graduated Grinding Jig</td>
</tr>
<tr>
<td>55230-08</td>
<td>UniDisc 3mm Disc Punch</td>
</tr>
</tbody>
</table>
8. Preparation Of Thin Foils By Electro-Polishing

8.1. Principle of Electro-polishing
The principle of electro-polishing is straightforward. The specimen is made the anode in an electrolytic cell, a potential is applied, and the specimen surface dissolves.

The practice is complex. The composition, the temperature and the flow rate of the electrolyte and the applied potential are major variables.

8.2. Electrolyte Composition
To ensure that dissolution is uniform and the surface is smooth, a thin viscous film must be formed at the surface during electro-polishing. To achieve this the electrolyte has to contain an oxidizing agent, a film former and a solvent for the oxide containing viscous film.

8.3. Electrolyte Temperature
Electro-polishing at sub-zero temperatures (-10°C to -20°C) has two important advantages for foil preparation:

a) The current density at the anode is reduced resulting in a slower, more controllable polishing action.
b) The width of the polishing plateau is greatly increased making it easier to establish and maintain good polishing conditions.

8.4. Value of the Applied Potential
The value of this potential can be established by experiments in which the current (I) and voltage (V) are plotted.

STATIC CONDITIONS: the ideal I-V plot is shown in Figure 8-1. At potentials lower than V1 etching occurs when dissolution occurs without the formation of a stable layer. Above V2 gas bubbles evolved from the surface disrupt the polishing and pitting occurs. The surface may appear to be polished but there are small pinpricks and few, if any, thin areas.

DYNAMIC CONDITIONS: When the electrolyte is stirred or jetted a plateau region is not observed because the viscous layer is not able to stabilise. The type I-V plot obtaining under these conditions is shown in Fig. 8.2.

Figure 8-1: Relationship between Current and Voltage during Electro-polishing under Static Conditions

Figure 8-2: Relationship between Current and Voltage during Electro-polishing under Dynamic Conditions
8. Preparation Of Thin Foils By Electro-Polishing, continued

8.5 Twin-Jet Electro-polishing

When jets of electrolyte are directed, from both sides, at the centre of a 3mm TEM disc specimen dishing occurs rapidly. The disc profile is a function of the current, see Figure 7-3.

![Figure 7-3: Effect of Polishing Current on Disc Profile](image)

The largest thinned areas occur when the profile is flat, Figure 8-3b, but detection of perforation can be irregular. The ideal profile is a combination of Figure 8-3a and Figure 8-3b i.e. central perforation. The conditions for this profile occur at, or just above, the point of inflexion in Figure 8-2. If too high a current is used crescent shaped perforations are produced, see Figure 7-3c.

Central perforation, the ideal condition, is a function of pump speed, electrolyte viscosity and temperature, sample material and current density.

The composition of electrolytes for thinning most metals is now well established. The two remaining variables are the electrical potential and the flow rate of the electrolyte (jet speed). The values given in 'recipes' may not always give the best results. The recommended procedure for varying these parameters to establish the optimum conditions for making good foils is given below:

a) Electrical potential: begin with the potential given in the recipe.
   - If etching occurs, increase the voltage.
   - If pitting occurs, decrease the voltage.

b) Electrolyte flow rate: if the flow rate is too low polishing may not occur in the center of the disc. If the flow rate is too high the viscous film breaks up and polishing is poor or non-existent. At just above the correct flow rate good polishing may occur but the thin areas around the perforation can be deformed or destroyed. The optimum flow rate is determined by trial and error.

When the correct conditions for preparing good foils from a particular sample have been established in an automated twinjet electro polisher they can be reproduced as required.

While central perforation is the ideal condition it is not always achieved. This does not mean that the specimen should be discarded always examine the foil in the electron microscope because usually there will be sufficient thinned material.
8. Preparation Of Thin Foils By Electro-Polishing, continued

8.6. Recipes for Preparing Thin Foils Using the Metalthin

**SAFETY WARNING**

There is a potential explosion hazard when using electrolytes containing perchloric acid (HClO4). The Safety Officer MUST be consulted before using such electrolytes.

The constituents of the electrolytes are harmful to skin and, particularly to eyes. Before preparing and using electrolytes users MUST study the suppliers’ recommendations and safety procedures for mixing of chemicals. Also appropriate safety clothing, gloves and eye protectors MUST be worn.

The Metalthin jets MUST NOT be operated unless there is a specimen holder in the slot in the jetting tank lid. The holder prevents splashes of electrolyte from leaving the tank.

**QUALITY OF ELECTROLYTE CONSTITUENTS**

The quality of the electrolyte constituents can vary according to the source of supply and this can affect the quality of the resultant thin foil. The use of standard grade or Analar grade (AR) is a choice for the user. But to produce high quality foils from materials that are difficult to prepare the use of ANALAR grade materials is essential.

**DISC THINNING CONDITIONS**

Table 2 summaries the conditions for thinning metals that are most frequently encountered by users of the METALTHIN. The optimum results are obtained when the thickness of the disc is 0.1 to 0.15 mm thick. Thicker discs can be reduced to this range by use of the Graduated Grinding Jig

These conditions are GUIDES, not infallible procedures, because the optimum conditions can vary depending upon the heat treatment, degree of cold work and minor variations in composition.

Users are recommended to start with these conditions then to change the current, jet speed and temperature, systematically, noting those conditions that produce large areas of good quality foil.

To provide users with a starting point for metals not covered in Table 2 a list of metals and corresponding electrolytes is given in the following pages, use these in conjunction with the procedures described earlier. Determining the optimum conditions for producing good thin foils is a matter of trial and error.

Further information on electrolytes and foil preparation procedures can be found in the text books listed in Section 8.7.
### Table 2
Summary of the Conditions for Thinning the Metals most Frequently Encountered by Users of the Metalthin

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>ELECTROLYTE (See key below)</th>
<th>TEMPERATURE</th>
<th>CURRENT</th>
<th>PUMP SPEED</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>N1</td>
<td>-15 °C</td>
<td>120 mA</td>
<td>4</td>
</tr>
<tr>
<td>Al - Cu - Co</td>
<td>P3</td>
<td>-15 °C</td>
<td>110 mA</td>
<td>4</td>
</tr>
<tr>
<td>Cobalt</td>
<td>P2</td>
<td>-30 °C</td>
<td>100 mA</td>
<td>4</td>
</tr>
<tr>
<td>Copper</td>
<td>N2</td>
<td>-40 °C</td>
<td>200 mA</td>
<td>4</td>
</tr>
<tr>
<td>Brass</td>
<td>N3</td>
<td>-40 °C</td>
<td>200 mA</td>
<td>4</td>
</tr>
<tr>
<td>Phosphor-Bronze</td>
<td>N3</td>
<td>-40 °C</td>
<td>200 mA</td>
<td>4</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>P1</td>
<td>18 °C</td>
<td>100 mA</td>
<td>2</td>
</tr>
<tr>
<td>Steel - 2% Silicone</td>
<td>P1</td>
<td>-25 °C</td>
<td>60 mA</td>
<td>3</td>
</tr>
<tr>
<td>High Carbon Steel</td>
<td>P2</td>
<td>18 °C</td>
<td>80 mA</td>
<td>3</td>
</tr>
<tr>
<td>Magnesium</td>
<td>N3</td>
<td>-15 °C</td>
<td>80 mA</td>
<td>4</td>
</tr>
<tr>
<td>Nimonic 75</td>
<td>P2</td>
<td>-25 °C</td>
<td>190 mA</td>
<td>5</td>
</tr>
<tr>
<td>Nimonic 115</td>
<td>P2</td>
<td>-25 °C</td>
<td>210 mA</td>
<td>5</td>
</tr>
<tr>
<td>Nickel</td>
<td>P1</td>
<td>-30 °C</td>
<td>200 mA</td>
<td>3</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>S2</td>
<td>-18 °C</td>
<td>150 mA</td>
<td>4</td>
</tr>
<tr>
<td>PE16</td>
<td>P1</td>
<td>-45 °C</td>
<td>70 mA</td>
<td>4</td>
</tr>
<tr>
<td>Tungsten</td>
<td>A1</td>
<td>18 °C</td>
<td>110 mA</td>
<td>4</td>
</tr>
<tr>
<td>Zinc</td>
<td>N2</td>
<td>-15 °C</td>
<td>120 mA</td>
<td>4</td>
</tr>
<tr>
<td>Zirconium</td>
<td>P1</td>
<td>-50 °C</td>
<td>60 mA</td>
<td>4</td>
</tr>
<tr>
<td>Titanium</td>
<td>P1</td>
<td>-40 °C</td>
<td>50 mA</td>
<td>4</td>
</tr>
<tr>
<td>Vanadium</td>
<td>S1</td>
<td>18 °C</td>
<td>100 mA</td>
<td>4</td>
</tr>
</tbody>
</table>

**ELECTROLYTES:**

- **P1**: 5% Perchloric acid in Methanol
- **P2**: 10% Perchloric acid in Methanol
- **P3**: 5% Perchloric acid and 5% Nitric Acid in Methanol
- **N1**: 10% Nitric acid in Methanol
- **N2**: 20% Nitric acid in Methanol
- **N3**: 30% Nitric acid in Methanol
- **A1**: 2% Aqueous K.O.H. (Sodium Hydroxide)
- **S1**: 20% Sulphuric acid in Methanol
2S 10% Sulphuric acid in Methanol

Silver and its Alloys
Ag 9% aqueous potassium cyanide
Ag 67.5 g/l potassium cyanide
15 g/l rochelle salt
19.5 ml/l orthophosphoric acid
2.5 ml/l ammonia
15 g/l potassium ferrocyanide

Ag - 1.94 to 6% aqueous potassium cyanide
20% Zn

Ag - 0.69 to 70% ethyl alcohol
5% Al 20% perchloric acid
10% glycerol

Silver and its Alloys
Ag 9% aqueous potassium cyanide
Ag 67.5 g/l potassium cyanide
15 g/l rochelle salt
19.5 ml/l orthophosphoric acid
2.5 ml/l ammonia
15 g/l potassium ferrocyanide

Aluminium and its Alloys
A1 20% perchloric acid
80% ethyl alcohol

Al - Cu 817 ml orthophosphoric acid
Al - Ag 134 ml sulphuric acid
Al - Zn 156 g chromic oxide
Al - Zn - Mg 40 ml water
Al - Zn - Mg - Cu
Al - Mg - Si

Al - Cu 25 ml methyl alcohol
Al - Cu 25 ml nitric acid
1 ml hydrofluoric acid

Al - Cu 25 ml methyl alcohol
Al - Cu 25 ml nitric acid
1 ml hydrofluoric acid

Al 10% perchloric acid
Al - Mg 10% water
10% glycerol
70% ethyl alcohol

Al - Ag 40 gms of Sodium Thiocyanate (hydrated 33 1/3 gm if Anhydrous and 6 2/3 Cl if distilled water)
220 ml ethyl alcohol
10 ml glycerol
Temp. O° C -- +5° C

Two stages as follows
PROFILE @ 200 mA or at MAX voltage for 3/4 time to perforation.

Pump speed 6.

POLISH @ 70 - 90 mA. Pump speed 4.
If current drops whilst polishing wash specimen vigorously in ethyl alcohol.

For 0.010" thick disc preparation time is: Profile 90 seconds. Polish 60 seconds.

Copper and its Alloys
Cu 33% nitric acid
Cu - Zn ( ) 67% methyl alcohol
Cu - Al
Cu - Ge
Cu - Sn
Cu - Si

Cu 70% orthophosphoric acid
Cu - Te 30% distilled water

Cu 50% orthophosphoric acid
Cu - Zn ( ) 50% distilled water

Cu 250ml phosphoric acid
Cu - Be 250ml ethyl alcohol

Cu - Sn 500 ml distilled water
Cu - Ni 2 ml Vogels economy caustic
0.5 g Golpanol B

Cu - Au 33 ml glacial acetic acid
Cu - Ag 25 g Cr 0 7 ml water

Zone refined and OFHC 20% water+2g NiCl 80% concentrated nitric acid
### Iron and its Alloys
- **Fe**: 10 parts glacial acetic acid
- **Low C steels**: 1 part perchloric acid
- **Fe**: 135 ml glacial acetic acid
- **Plain C steels**: 25 g chromic oxide
- **Uranium steels**: 20 ml water
- **Silicon iron**: 7 ml water

### Low alloy steels
- **Weld metal**: 7 ml water
- **20% Ni steel**: 133 ml glacial acetic acid
- **Fe**: 25 g chromic oxide
- **Low C steel**: 20 ml water

### Stainless steel
- 60% orthophosphoric acid
- 40% sulphuric acid
- 10% perchloric acid
- 90% acetic acid
- 20 g/l chromic oxide
- 10 g/l nickel chloride
- 200 ml glycerol
- 720 ml sulphuric acid
- 70 ml water

### Magnesium and its Alloys
- **Mg**: 33% nitric acid
- 67% methyl alcohol
- 30% hydrofluoric acid
- 70% nitric acid
- 17.5% hydrofluoric acid
- 17.5% nitric acid
- 65% water
- 90% sulphuric acid
- 10% hydrofluoric acid

### Niobium and its Alloys
- **Nb**: 23% perchloric acid
- 77% acetic acid
- 860 ml orthophosphoric acid
- 50 ml sulphuric acid
- 108 g chromium trioxide
- 390 ml sulphuric acid
- 290 ml acetic acid
- 58 ml perchloric acid
- 192 ml acetic acid

### Nickel and its Alloys
- **Ni**: 23% perchloric acid
- 77% acetic acid
- 860 ml orthophosphoric acid
- 50 ml sulphuric acid
- 108 g chromium trioxide
- 390 ml sulphuric acid
- 290 ml acetic acid
- 4% perchloric acid
- 96% acetic acid

### Silicon and its Alloys
- **Si**: 98% sulphuric acid
- 2% water
- 98% sulphuric acid
- 2% water
- 12.5% sulphuric acid
- 87.5% methyl alcohol
- 10% sulphuric acid
- 90% acetic acid
- 12.5% sulphuric acid
- 87.5% methyl alcohol
- 25% sulphuric acid
- 75% ethyl alcohol
- 25% sulphuric acid
- 75% ethyl alcohol
- 12.5% sulphuric acid
- 87.5% methyl alcohol
- 25% sulphuric acid
- 75% ethyl alcohol
- 25% sulphuric acid
- 75% ethyl alcohol
- 25% sulphuric acid
- 75% ethyl alcohol

### Tungsten and its Alloys
- **W**: 2% sodium hydroxide in water
- 2% sodium hydroxide in water
- 10% sodium hydroxide in water
- 2% sodium hydroxide in water

### Zinc and its Alloys
- **Zn**: 20% nitric acid
- 80% methyl alcohol
- 20% perchloric acid
- 80% ethyl alcohol
- 10% perchloric acid
- 90% acetic acid

### Zirconium and its Alloys
- **Zr**: 20% perchloric acid
- 80% ethyl alcohol
- 10% perchloric acid
- 90% acetic acid
8.7 Bibliography


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