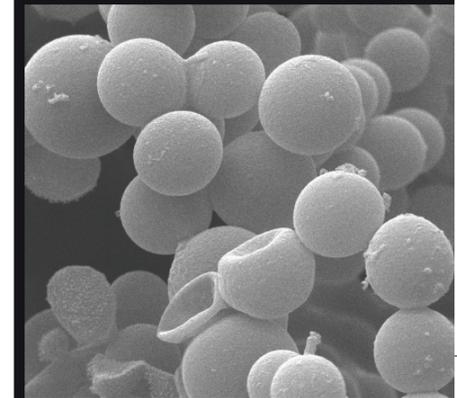




# Equipment Applications and Techniques

2012 EDITION

Cooling  
Sputter Coating  
Carbon Coating  
Vacuum Evaporation  
Plasma Etching  
& Ashing  
Critical Point Drying  
Freeze Drying  
Cryo-SEM Preparation



**Electron  
Microscopy  
Sciences**

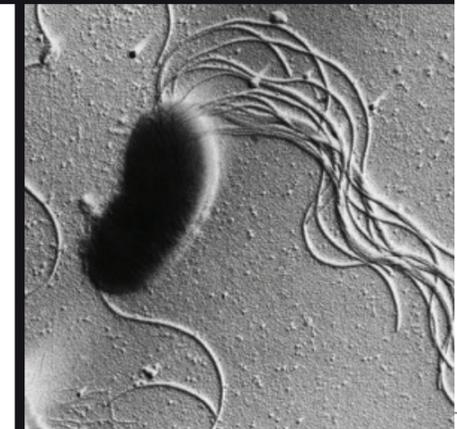
In PA: (215) 412-8400 Toll-Free (800) 523-5874

Fax (215) 412-8450 or 8452

email: [sgkcck@aol.com](mailto:sgkcck@aol.com) or [stacie@ems-secure.com](mailto:stacie@ems-secure.com)

[www.emsdiasum.com](http://www.emsdiasum.com)

**Electron  
Microscopy  
Sciences**





# Equipment Applications and Techniques

2012 EDITION



## Electron Microscopy Sciences

In PA: (215) 412-8400  
Toll-Free (800) 523-5874  
Fax (215) 412-8450 or 8452  
email: sgkcck@aol.com or stacie@ems-secure.com  
www.emsdiasum.com

**Dear Fellow Researchers,**

Here at Electron Microscopy Sciences we are very proud to present the 2nd Edition of our *Applications and Techniques for Equipment*. Our goal has been to put together a convenient reference source for sample preparation and cryo-preparation for EM equipment, featuring extensive information about scanning electron microscopy and different preparation techniques. Applications include sputter coating, vacuum evaporation, plasma etching, critical point drying, and cryo-SEM.

Here also you will find a full range of all the latest equipment for electron microscopy, as well as replacement parts, consumables, and sputter targets. EMS is committed to providing the highest quality products along with competitive pricing, prompt delivery and outstanding customer service.

We hope we have provided something for everyone in the field of electron microscopy.

**Sincerely,  
The EMS Team**



## Electron Microscopy Sciences Announces their State of the Art Scientific Equipment Repair Center

**No equipment is too small or impossible to either Repair, Refurbish, or Upgrade!**

Our facility is equipped to handle the following Manufacturers:

- Balzers/Baltech
- Denton
- Edwards
- Emitech
- Emscope
- Bio-Rad
- Leica
- Reichert-Jung
- LKB
- RMC-Boeckeler
- and many more...

Our unique service allows us to come to you, or you send the equipment to us, depending on the level of Repair/Refurbishment needed.

Equipment such as:

- Vacuum Systems
- Microtomes
- Ultramicrotomes
- Tissue Slicers
- Vibratomes
- Ovens
- Baths
- Shakers
- Tissue Processors
- Lynx
- Freeze Fracture Instruments
- High Pressure Freezers/Slammers/Propane Jet Freezers
- and much more...

For more information on any of our services, or a comprehensive quote, please call, write, or e-mail us today.

## Electron Microscopy Sciences

In PA: (215) 412-8400 Toll-Free (800) 523-5874  
Fax (215) 412-8450 or 8452  
email: sgkcck@aol.com or stacie@ems-secure.com  
www.emsdiasum.com



# table of contents

<b>Cooling Stages</b> .....	<b>2–3</b>	<b>Freeze Dryers</b> .....	<b>56–59</b>
The EMS Coolstage for SEM, LV or VP.....	2–3	Techniques and Applications.....	56–57
<b>Recirculating Heaters and Chillers</b> .....	<b>4–5</b>	EMS 750 Freeze Dryer.....	58
EMS 3500 Thermocirculator.....	4	EMS 775 Turbo Freeze Dryer.....	59
EMS 4800 Recirculating Heater/Chillers.....	5	<b>Cryo-SEM Preparation Systems</b> .....	<b>60–65</b>
<b>Sputter Coaters/ SEM/TEM Carbon Coaters</b> .....	<b>6–37</b>	Techniques and Applications.....	60
Techniques and Applications.....	6–11	PP3000T Cryo-SEM Preparation System.....	62–65
EMS 7620 “Mini” Sputter Coater/ Glow Discharge.....	12–13	<b>Vacuum Pumps and Accessories</b> .....	<b>66</b>
Techniques and Applications.....	14–16	<b>Evaporation Supplies</b> .....	<b>67–70</b>
EMS 7620-CF Carbon Accessory Power Supplies.....	17	<b>Sputter Targets</b> .....	<b>71</b>
EMS 150T Turbo-Pumped Sputter Coater/ Carbon Coater.....	18–21	<b>Lab Safety</b> .....	<b>72</b>
EMS150R Rotary Pumped Carbon and Sputter Coating System.....	22–25		
EMS300RT Triple Target, Large Chamber, Rotary-Pumped Sputter Coater.....	26–29		
EMS300TT Triple Target, Large Chamber, Turbo-Pumped Sputter Coater.....	30–33		
EMS300TD Dual Target, Large Chamber, Turbo-Pumped Sputter Coater.....	34–37		
<b>Vacuum Evaporators (Large Chamber)</b> .....	<b>30–39</b>		
EMS300TT Triple Target, Large Chamber, Turbo-Pumped Sputter Coater.....	30–33		
EMS300TD Dual Target, Large Chamber, Turbo-Pumped Sputter Coater.....	34–37		
EMS 975 Large Chamber Turbo Evaporator & EMS 975S for Semiconductor Wafer Coating...	38–39		
<b>RF Plasma Etchers/Plasma Reactors</b> ...	<b>40–47</b>		
Techniques and Applications.....	40–45		
EMS 1050 Plasma Asher.....	46–47		
<b>Critical Point Dryers</b> .....	<b>48–55</b>		
Techniques and Applications.....	48–51		
EMS 3000 and EMS 3100 Critical Point Dryers.....	52		
EMS 850 Critical Point Dryer with Thermoelectronic Heating and Adiabatic Cooling.....	54		
EMS 850WM Large Chamber Critical Point Dryer.....	55		

**Electron Microscopy Sciences**  
 In PA: (215) 412-8400  
 Toll-Free (800) 523-5874  
 Fax (215) 412-8450 or 8452  
 email: [sgkcck@aol.com](mailto:sgkcck@aol.com)  
 or [stacie@ems-secure.com](mailto:stacie@ems-secure.com)  
[www.emsdiasum.com](http://www.emsdiasum.com)

## Cooling Stages

### Why cool?

Low vacuum (LV) or variable pressure (VP) modes are now standard on most scanning electron microscopes (SEMs). For this reason it has become important to control water evaporation from wet specimens. Cooling such specimens reduces the loss of water by evaporation, or - depending on chamber pressure - can prevent it altogether.

Saturated vapour pressure of water decreases considerably with temperature. At room temperature, water will very quickly evaporate - causing considerable damage to specimen composition and ultra-structure. This is due to high forces of surface tension at the drying front as it passing through the specimen. In most biological systems this will result in distortion and collapse of membranes and other structures.

At 300Pa, the specimen temperature needs to be less than  $-9.5^{\circ}\text{C}$ , and at 85Pa less than  $-25^{\circ}\text{C}$  to stop water evaporation. Therefore, by cooling a specimen to  $-25^{\circ}\text{C}$ , chamber pressures up to 85Pa can be used with little or no water loss by evaporation. In this way, changes in specimen structure can be minimised. In addition, being able to operate at higher vacuum gives a better signal-to-noise ratio and clearer images.

## ● The EMS Coolstage for SEM, LV or VP

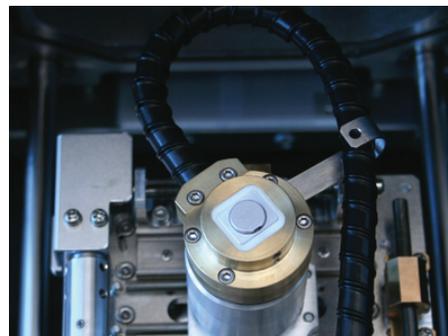
### Overview

The Coolstage is a Peltier-driven SEM cooling stage for scanning electron microscopy (SEM), low vacuum (LV) or variable pressure (VP) applications. The stage can be cooled to sub-zero temperatures for specimens that may be sensitive at ambient temperature, subject to beam damage, or may otherwise 'sublime' (lose water) at ambient temperatures.

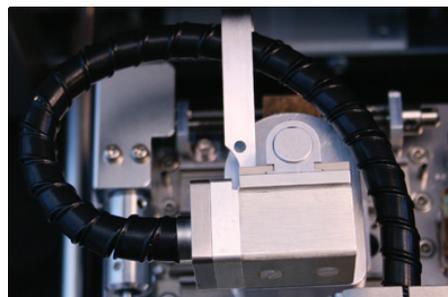
There are three versions of coolstage - Standard, Enhanced and Ultra - to cover differing specimen requirements.

### Features - Standard Coolstage

- Temperature range  $-30^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  at 300Pa
- Self contained cooling - no additional external cooling water needed
- Temperature accuracy  $\pm 1.5^{\circ}\text{C}$  or 2% - whichever is greater
- Minimal image drift
- Cooling and heating rates of up to  $30^{\circ}\text{C}$  per minute
- Keypad control - with simultaneous display of actual and target temperature
- Supplied with SEM chamber port feedthrough - specify when ordering
- One-year warranty



Standard Coolstage. Range:  $-30^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  at 300Pa



Ultra Coolstage. Range:  $-50^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  at 300Pa

### Features - Enhanced Coolstage

- Temperature range  $-30^{\circ}\text{C}$  to  $+160^{\circ}\text{C}$  at 300Pa
- All other specifications as per Standard Coolstage

### Features - Ultra Coolstage

- Temperature range  $-50^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  at 300Pa
- All other specifications as per Standard Coolstage

### Product Description

The Coolstage is a temperature-controlled specimen stage that can be fitted to any low vacuum (LV) or variable pressure (VP) scanning electron microscope (SEM).

The Standard Coolstage consists of a single stage Peltier device, onto which a thermally isolated specimen holder and dual temperature sensor is mounted. The Coolstage assembly is mounted onto the SEM stage using an adaptor plate specific to the microscope. Cooling pipes and electrical wires connect to the SEM feedthrough flange. External components are a recirculating water chiller and power supply case, and a compact keypad for digital temperature readout and control.

### Compact, efficient cooling and temperature control

The temperature range of the Standard Coolstage is  $-30^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  at 300Pa. The specimen holder is water-cooled using a small, self-contained closed loop recirculating chiller that is normally positioned approx 2m from the SEM. A microprocessor controls and monitors the temperature of the cold stage. A small keypad is used to set the required temperature and display target and current temperatures.

The specimen holder has been designed to minimise image drift due to temperature change, giving a stable image at high magnification. An integrated RS-232 interface allows temperature to be set and read from the SEM.

# Cooling Stages

## ● The EMS Coolstage (Continued)

### Rapid specimen exchange

To exchange a specimen it is necessary to increase the specimen stage temperature to ensure that condensation does not form on the specimen or specimen stage. The keypad controller has a convenient 'exchange' button that will automatically take the specimen holder temperature to a programmable temperature from between +5°C to +20°C. Typical cooling and heating rates are up to 30°C per minute.

When not in use, the major parts of the system can be left in situ and the cooling stage is very easily removed when reverting to 'normal' use. A convenient storage block is provided for Coolstage stage assembly and vacuum feedthrough for when the system is not in use.

### Specifications

Specimen size	.....10mm Ø (adaptor stub for 12" Hitachi stubs can be supplied on request)
Stage Temperature Range	....NOTE: Higher vacuum will allow for cooler temperatures, compatible with high vacuum levels to 1x10 <sup>-5</sup> Pa <b>Standard Coolstage:</b> -30°C to +50°C at 300Pa with no external cooling water from SEM (at ambient +20°C) <b>Enhanced Coolstage:</b> 30°C to +160°C at 300Pa with no external cooling water from SEM (at ambient +20°C) <b>Ultra Coolstage:</b> -50°C to +50°C at 300Pa with no external cooling water from SEM (at ambient +20°C)
Temperature display resolution	.....-0.1°C
Temperature stability	.....+/- 0.2°C
Temperature accuracy	.....+/- 1.5°C or 2% (whichever is greater)
Stage movement	.....Normal x, y and z movements maintained. Tilt maintained for X-ray analysis (up to 45°). No rotation
Working distance	.....As on SEM, Coolstage is set to the SEM Eucentric height
Operating voltage	.....100V or 115V or 230V @ 100VA, voltage tolerance +/- 10%
Size and weight	.....Operation/display unit: 90mm L x 112mm W x 350mm H, 300g Power supply/cooling unit: 305mm L x 245mm W x 330mm H, 15kg
Packed size and weight	.....550mm L x 580mm W x 400mm H, 28kg
Supplied with:	.....Operating manual, one set of interconnecting cables and mains supply lead, storage block for specimen cooling unit when not in use, specimen holders: 15 x flat 10mm Ø stubs and 15 x dish 10mm Ø stubs



Coolstage and vacuum feed through connected to the storage block (stage protected by plastic shutter)



Coolstage and vacuum feed through connected to the storage block (with protection shutter open)



Dish-style specimen stub, showing flat bottom side (left) and dish side (right)

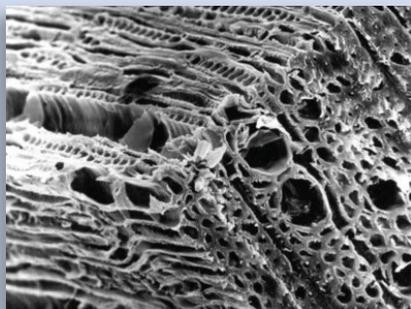
### Ordering Information

<b>90100</b>	EMS Standard Coolstage	each
<b>90101</b>	EMS Enhanced Coolstage	each
<b>90102</b>	EMS Ultra Coolstage	each
<b>90103</b>	Flat specimen stubs, 10mm diameter	10/pk
<b>90104</b>	Dish style specimen stubs, 10mm external diameter	10/pk

## Recirculating Heaters and Chillers

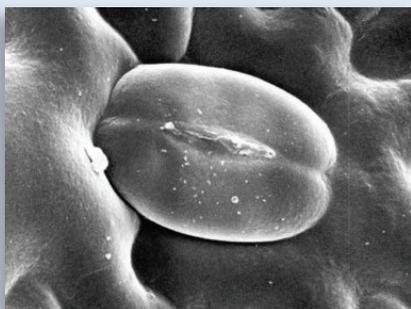
### Using the EMS 3500 with Critical Point Dryers

The EMS 3500 will give controlled heating of EMS 3000 and EMS 3100 Critical Point Dryers. It is connected directly to the inlet and outlet of the EMS 3000 or EMS 3100 water jacket. The temperature of the circulating fluid is pre-set, typically set to 37°C (eg just above the critical temperature of carbon dioxide).



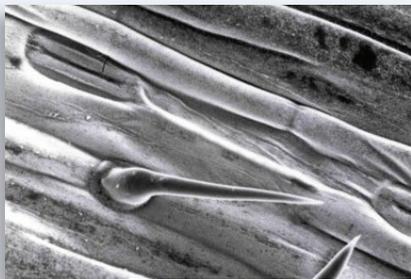
### Mature Spruce Wood

Critical point dried block of mature spruce wood block, demonstrating transverse, tangential and radial views of tracheids and vessels.



### Stomatal Pore on Xerophyte Leaf Surface

Critical point dried epidermis of a xerophyte (cactus), demonstrating raised stomatal pores.



### Barley Leaf

Trichomes and stomatal pores on the epidermal surface of a barley (*Hordeum vulgare*) leaf. Some very fine wax crystalites are also just visible on the surface of the leaf.

## ● EMS 3500 Thermocirculator

The EMS 3500 Thermocirculator is a low cost, portable water circulating system for supplying a constant temperature supply for closed and open loop applications at near ambient to +60°C.

### Features

- Precise control of heating
- Compact
- Robust and reliable
- No running water to waste
- Ideal for use with the EMS 3000 and EMS 3100 Critical Point Dryers



The EMS 3500 is suitable for open and closed loop applications and has all controls and liquid connections on the front panel. The temperature controller shows not only the 'set' temperature (between ambient and +65°C), but also the actual liquid temperature. The fluid is circulated by a glandless magnetic pump, through an aluminum reservoir.

A cooling coil is mounted in the liquid reservoir. This coil can be directly connected to the main water supply and is primarily used when temperature control near to ambient is required. The electronic control system features a zero voltage switching unit which minimizes mains electrical interference, enabling the EMS 3500 to be used and sited with other sensitive electronic instruments.

### Specifications

Working temperature range	Ambient to +65°C
Heating rating	500W
Reservoir capacity	2.2L / 3.5 Pints
Pump type	Glandless magnetic drive
Pump motor	Shaded pole
Pump flow	270L/h (zero head) / 60 Gal/hr (zero head)
Pump pressure	0.25kg/cm <sup>2</sup> / 3.5psi
Max pump head (unrestricted)	2.5m / 7.5ft
Size (unpacked)	330 x 280 x 150mm / 13 x 11 x 6"
Weight	8kg / 17.6lbs
Electrical	220-240V/50Hz, 115V/60Hz

### Ordering Information

91095	EMS 3500 Thermocirculator	each
-------	---------------------------	------

# Recirculating Heaters and Chillers



## Some typical applications

- Vacuum coating equipment
- Critical point dryers (EMS 3000 and EMS 3100)
- Electron microscopes
- Chromatography equipment
- Electrophoresis baths
- Environmental chambers
- Crystal growth apparatus
- Fermentation equipment
- Interferometers
- Photographic baths
- X-ray equipment
- Polarimeters, refractometers
- ...and many others

## Optional Attachments

- High pressure pump for EMS 4860 and EMS 4870 (standard in EMS 4880 and EMS 4890)
  - Water failure alarm
  - Over and under temperature cut out
- NOTE: Larger capacity heater/chillers (6kW and 12kW) are available on request - please contact us for further information.
- Custom-made heater/chiller units

## Ordering Information

- 91098 EMS 4860 1/5 HP Recirculating Heater/Chiller
- 91099 EMS 4870 1/2 HP Recirculating Heater/Chiller
- 91090 EMS 4880 3/4 HP Recirculating Heater/Chiller
- 91095 EMS 4890 1 HP Recirculating Heater/Chiller

## ● EMS 4800 Recirculating Heater/Chillers

Recommended for open and closed loop applications, offering simplicity, reliability and quiet operation. The range includes the EMS 4860, 4870, 4880 and 4890.

### Features

- Precise temperature control
- Quiet, efficient operation
- Proven reliability
- Low maintenance
- Environmentally friendly- avoids running water to waste

### Temperature control

Many instruments measuring physical properties depend on accurate control of temperature and in some processes optimum temperature is essential. With the EMS 4800 series, over-cooling (which affects efficiency) is prevented and the water temperature can be accurately controlled over the range -10 to +60°C.

A commonly misunderstood feature of refrigerated systems is in applications where the control temperature is other than at or near room temperature. When the instruments are to be operated at controlled temperatures below ambient, the extraction deteriorates significantly and, as a guide, the compounded change is 4% per degree Celsius. In practice, the refrigerant gas pressure has to be adjusted to optimize the performance at any particular temperature. However, the EMS 4800 series incorporate automatic adjustment valves in the systems.

The EMS 4800 series are of the 'closed loop' type and therefore efficiencies are dramatically improved compared with open bath models. They are simple to set up and to operate, and essentially maintenance free.

### Choosing the correct heater/chiller

In order to optimize performance from a heater/chiller system, the correct specification must be selected for a particular application. To cool or heat any instrument or system it is important to obtain the following information from the manufacturer:

- Heat load to be dissipated to water, eg for an electron microscope: diffusion pump heater, lenses, etc
- Flow rate and size of tubing
- Minimum pressure

With this information, consult the Specifications below and select the appropriate heater/chiller. The basic heat load calculation formula is as follows:

$$\text{Flow rate} \times \text{weight of fluid} \times \text{specific heat} \times \Delta T = \text{Heat Extraction.}$$

### Specifications

#### Heat Extraction Rates (in Watts)

	- 20°C	- 10°C	0°C	+10°C	+ 20°C
EMS 4860	75W	105W	180W	300W	420W
EMS 4870	125W	250W	500W	900W	1.2kW
EMS 4880	200W	425W	700W	1.6kW	2.2kW
EMS 4890	350W	600W	1.2kW	2kW	3kW
4.5kW Recirculator	700W	1kW	2kW	3kW	4.5kW
6kW Recirculator	800W	1.3kW	2.6kW	4.5kW	6kW

Model	EMS 4860	EMS 4870	EMS 4880	EMS 4890
Extraction rate at 20°C	400W	1.4kW	2.2kW	3kW
Temperature range	-20°C to +70°C	-20°C to +70°C	-20°C to +70°C	-20°C to +70°C
Refrigeration (HP)	1/5	1/2	3/4	1
Heater rating	1kW	1.5/2.0kW	2.5kW	2.5kW
Max pump flow	450L/h	450L/h	900L/h	900L/h
200Gal/hr	200Gal/hr	275Gal/hr	275Gal/hr	
Tank capacity	1.2L	1.7L	2.3L	3.0L
Max pump pressure psi/bar	12/60psi	12/60psi	60psi	60psi
0.7/3bar	0.7/3bar	1.5/3bar	1.5/3bar	
Height	37cm	45cm	50cm	50cm
Width	32cm	38cm	45cm	45cm
Depth	46cm	61cm	62cm	62cm
Weight	40kg	62kg	70kg	82kg
Water connections	16mm hose or 1/8 BSP			
Temperature sensor	R.T. Probe	R.T. Probe	R.T. Probe	R.T. Probe

## Sputter Coaters/SEM/TEM Carbon Coaters

### What is... Sputter Coating?

When a glow discharge is formed between a Cathode and Anode using a suitable gas (typically Argon), and Cathode target material (commonly Gold) the bombardment of the target with gas ions will erode this target material, this process being termed 'Sputtering'.

The resulting omni-directional deposition of sputtered atoms will form an even coating on the surface of the specimen. It will inhibit charging, reduce thermal damage, and improve secondary electron emission which are beneficial for Scanning Electron Microscopy.

The Cathode target material is commonly Gold. However, to achieve finer grain size, and thinner continuous coatings, it is advantageous to use cathode target materials such as Chromium. To achieve sputtering with this target material requires vacuums somewhat better than those achievable with a Rotary Vacuum Pump.

## Techniques and Applications

### Introduction

When a target is bombarded with fast heavy particles, erosion of the target material occurs. The process, when occurring in the conditions of a gaseous glow discharge between an anode and cathode is termed sputtering. Enhancement of this process for scanning electron microscopy (SEM) sample coating is obtained by the choice of a suitable ionization gas and target material. Sputtered metal coatings offer the following benefits for SEM samples:

- *Reduced microscope beam damage.*
- *Increased thermal conduction*
- *Reduced sample charging (increased conduction).*
- *Improved secondary electron emission*
- *Reduced beam penetration with improved edge resolution*
- *Protects beam sensitive specimens*

Increase in electrical conductivity of a sample is probably the single most common requirement for SEM, though all factors come into play with FEG SEM. Low voltage SEM operation can still benefit in many cases from a thin coating.

The development of Sputter Coater systems embodies significant empirical design, however, an understanding in classical terms of glow discharge characteristics enhance such designs and may assist in the comparison of differing systems.

### Gaseous Condition

If an inert gas such as argon is included in a cathode gas tube, the free ions and electrons are attracted to opposite electrodes and a small current is produced. See **Figure 1**.

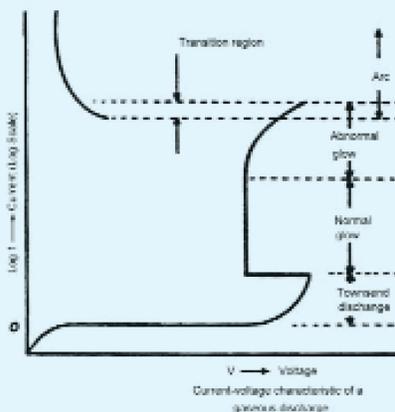
As voltage is increased some ionization is produced by collision of electrons with gas atoms, named the "Townsend" discharge. When the voltage across the tube exceeds the breakdown potential, a self sustaining glow discharge occurs - characterized by a luminous glow.

The current density and voltage drop remains relatively constant, the increase in total current being satisfied by the area of the glow increasing. Increasing the supply voltage increases current density and voltage drop, this is the abnormal glow region.

Further increase in supply voltage concentrates the glow into a cathode spot and arc discharge is apparent. The operating parameters of sputter coaters are within the glow discharge regions of the characteristic described.

**Figure 1: Circuit to determine the current-voltage characteristics of a cold cathode gas tube**

A = Ammeter V = Voltmeter



### Glow Discharge

Once the condition for a sustained discharge is met, the tube exhibits the characteristic glow discharge, so called because of the associated luminous glow. It has been established that free ions and electrons are attracted to opposite electrodes producing a discharge - however for a discharge to be self-sustaining requires regeneration of the electrons by the positive ion bombardment of the cathode. This produces secondary electrons and enhances ionization. The resulting positive ion excess creates a positive space charge near the cathode. The voltage drop experienced is termed the cathode fall. If the discharge is established in a long narrow tube it exhibits the characteristics indicated.

The positive ion density in the "Crookes dark space" is very high; as a result a significant voltage drop is experienced between it and the cathode.

The resulting electric field accelerates the positive ions which produce secondary electron emission from the cathode.

These electrons accelerated in the direction of the anode cause ionization, generating positive ions to sustain the discharge. Subsequently, excitation of the gas results in intense illumination

## Sputter Coaters/SEM/TEM Carbon Coaters

### Techniques and Applications

in the negative glow region. From this stage the electrons have insufficient exciting or ionizing energy, resulting in the "Faraday dark space". Towards the anode a small accelerating field can produce ionization and excitation, the gas again becoming luminous. See **Figure 2**.

#### Sputter Coating

It has been indicated that under conditions of glow discharge, ion bombardment of the cathode will occur. This results in the erosion of the cathode material and is termed plasma sputtering, with the subsequent omni-directional deposition of the sputtered atoms forming coatings of the original cathode material on the surface of the sample and work chamber. This process is enhanced in sputter coaters for use in Scanning Electron Microscopy where one objective is to provide an electrically conductive thin film representative of the specimen to be viewed. Such films inhibit "charging", reduce thermal damage, and enhance secondary electron emission.

The most common arrangement for a D.C. (Direct Current) sputter coater is to make the negative cathode the target material to be sputtered (typically gold, platinum or with high vacuum sputter coaters, metals such as chromium and iridium), and to locate the specimens to be coated on the anode (which is usually "earthed" to the system, so the specimens are effectively at "ground" potential).

The desired operating pressure is obtained by a pump (usually a two-stage rotary vacuum pump, or a turbomolecular pumped "backed" by a rotary pump), with an inert gas, such as argon admitted to the chamber by a fine control (leak) valve.

#### Operating Characteristics

The glow discharge in sputtering is significantly dependent on the work function of the target material and pressure of the environmental gas. A range of target materials are used including gold, gold-palladium, platinum and silver. Although gold is still a common sputtering metal, having the most effective electrical conduction characteristics, it does however, have the largest grain size and is not always suitable for high resolution coating. For this reason gold-palladium and platinum are now widely used as their grain sizes are smaller than gold. Films with even smaller grain sizes can be achieved using metals such as chromium and iridium, but both require the use of a high vacuum (turbomolecular pumped) sputtering system.

The sputter head and sputter power supply should be effective over a range of anticipated target materials.

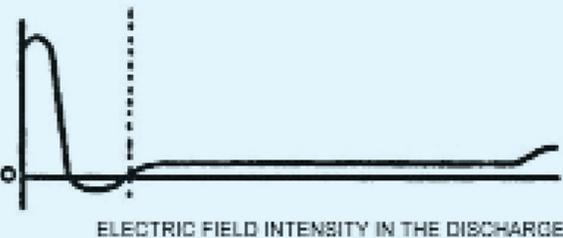
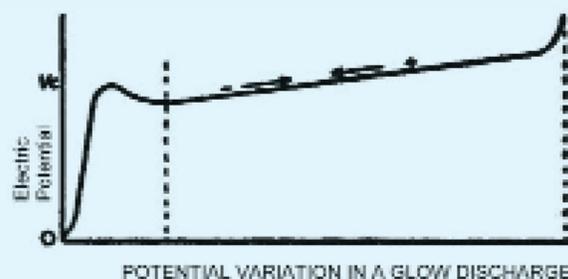
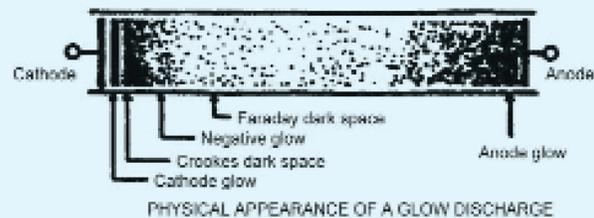
The deposition rate is current dependant, and if we operate in the correct glow region of the characteristic plasma discharge, as previously described, several fold changes in current should be available for a relatively small change in sputtering voltage. The deposition rate should not be sensitive to small changes in pressure which may be experienced in the system.

If the sputtering head is well designed and operating at low voltage and as a result, low energy input, then radiant heating from the target and high energy electrons (potentially the most significant sources of damage to delicate specimens) should be considerably reduced. There is also evidence to suggest that such a sputter head system may also produce finer grain size for a given target material.

The presence of an inert gas which will not decompose in the glow discharge is obviously desirable. Argon, having a relatively high atomic weight, provides a suitable source of ions for effective bombardment of the target material. Sputtering in air is best avoided.

The effectiveness is also dependent on the "mean free path" (m.f.p.) which is inversely proportional to pressure. If the m.f.p. is too short, insufficient

Figure 2



energy will be gained for effective bombardment and will inhibit movement of sputtered material from the target.

If the m.f.p. is too long, insufficient collisions occur and, in addition, the flow of sputtered material may change from diffusion in the gas to free molecular flow with a reduction in the effectiveness of omni-directional deposition.

If these characteristics for sputter heads are achieved, then it should not be necessary to cool the specimen stage for the majority of applications. If not, however, such cooling will only serve to reduce the baseline temperature, the thermal conductivity of most specimens we are considering being relatively poor.

For sensitive specimens pre-cooling (Peltier, water or cryo cooled) and subsequent reduction of the baseline may still be desirable and there is also evidence to suggest a reduction in grain size of the coating. It may be apparent that Scanning Electron Microscopy requires a versatile system without compromising performance. Specifically, fine grain size, uniform coating and low heat input. Consideration of these characteristics in design and development should enable a suitable coating system to be realised.

A major disadvantage of simple diode sputter coaters in SEM is the excessive amount of heat generated in the sample. To overcome this problem, permanent magnets are utilized to deflect the high energy electrons generated in the glow discharge away from the sample.

The magnetic lines of force cause enclosed loops at the target surface,

# Sputter Coaters/SEM/TEM Carbon Coaters

## Techniques and Applications

increasing the interaction path length of the high energy electrons in the discharge. Deflection and retardation of electrons result in increased ion yield and sputtering efficiency.

It was indicated previously that while imperial design may be in evidence, it should now be apparent that effective production of positive ions for glow discharge is required. The sputter head and its associated power supply should be a primary objective of design and development.

All modern SEM sputter coaters use heads fitted with an arrangement of magnets and often an associated shroud assembly, with a disc target. Power supplies generally employ solid state switching for applied voltage control. See **Figure 3**.

The overall result is a low mean voltage head with low energy input. The possibility of thermal damage due to radiant heating and electron bombardment is considered negligible.

For a typical modern magnetron sputter coater

<b>Vacuum</b>	8 x 10 <sup>-2</sup> to	2 x 10 <sup>-2</sup> mbar
<b>Sputtering Voltage</b>	100V to	3Kv
<b>Current</b>	0 to	50mA
<b>Deposition</b>	0 to	25 nm/min
<b>Grain size</b>	Less than 5nm	
<b>Temperature rise</b>	Less than 10C	

It is, of course, possible to satisfy very precise

parameters by the selection of target material, 'voltage' 'deposition', 'current' and 'vacuum'. Under these conditions, it is possible to achieve thin films to 10nm with grain sizes better than 2nm and temperature rises of less than 1°C.

### Choice of Sputtering Material

As stated many times, metal coating is an indispensable technique for SEM. The development of high resolution FEG SEMs has brought about more wide spread use of specialized techniques such as Ion Beam Sputtering, Penning Sputtering, E-Beam Evaporation and Planar magnetron ion-sputtering. More lately Chromium coating has become the "fashionable" material to use. It offers a thin continuous film and emits less back scattered electrons than other sputter materials. However it is not free of its own problems. To operate it requires a high vacuum and ideally vacuum transfer (or vacuum storage) of the sample to avoid oxidation problem. Cr coated samples may often have a "see through" look as there is the possibility of images generated from electrons from sub surface structures. More recently iridium films have been shown to give excellent fine grain (sub nanometer) films that compare favorably with those generated with Cr. Both metals require high vacuum sputter coaters for effective deposition.

Application data collected has shown that a high quality well designed rotary pumped magnetron sputter coater, such as the EMS 550X, is capable of producing a continuous Pt (platinum) film with a grain size in the order of 2 nm. It also has the benefit of being a good secondary electron emitter, unlike chromium. Some images of chromium show bright high contrast images. Many workers, and our own studies have led us to consider the possibility of each grain of chromium being oxidised before sample is coated and hence the film is not truly continuous and indeed each metal grain is individually charging. This is another reason to consider iridium as an alternative.

Silver as a sputter material is often ignored but is a very satisfactory method for ensuring conductivity of the SEM sample but has a major advantage the whole process is reversible as the metal may be removed by the neutral aqueous reagent known as "Farmers reducer". This enables many samples to be viewed and then returned to their original condition. Beware. ...Silver may form crystalline deposits on the surface of the sample in the presence of active Halogens

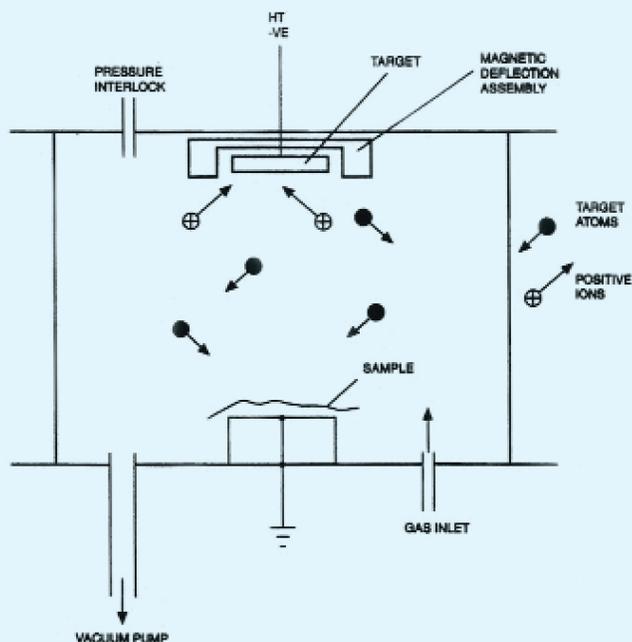
- *Sputtered silver offers smaller grain size than evaporated silver.*
- *Sputtered Gold and Silver have similar grain size but the silver has larger reticulation after storage.*
- *Silver is the most conductive metal known.*
- *Silver has a high secondary electron coefficient.*
- *X-ray emission lines are well separated from the biologically important sulphur and phosphorous.*
- *Cost effective.*

Gold/Palladium (80:20) targets are now a popular standard choice for the routine coating of a wide range of samples. The idea behind using this alloy is that the palladium will act as a physical barrier to the gold which will attempt to conglomerate into large islands and restrict ultimate resolution performance.

The minimal loss in secondary electron emission performance from the palladium is not seen as significant with current SEMs.

Other target choices are generally made based on the requirement for X-ray analysis of samples or back scattered electron detection.

**Figure 3 – Diagram of a "cool" sputtering head**



# Sputter Coaters/SEM/TEM Carbon Coaters

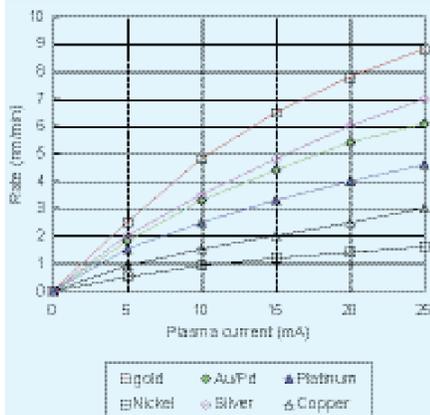
## Techniques and Applications

### Rates of Sputtering

A question regularly asked is what difference is there in sputtering rates for each of the target materials. The following list gives the variance of the materials in relation to gold, assuming gold to be: 1, it is impossible to give actual coating rates as this varies with sputtering conditions.

Au	Gold	1.0
Ag	Silver	1.2
Co	Cobolt	0.5
Cr	Chromium	0.5
Cu	Copper	0.7
Fe	Iron	0.5
Mo	Molybdenum	0.3
Ni	Nickel	0.5
Pd	Palladium	0.85
Pt	Platinum	0.6
Ta	Tantalum	0.2
W	Tungsten	0.2

Figure 4 - Sputtering Rates for the EMS 7620



### Thickness of Coating

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

$$Th = 7.5 I t \text{ (angstroms) } (V = 2.5KV, \text{ target to specimen distance} = 50mm)$$

t = time in minutes

I = current in mA

Th = thickness in angstroms

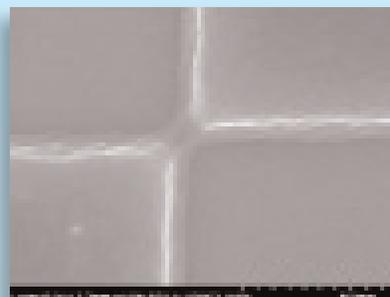
Average coating times will be of the order of 2 -3

minutes using V = 2.5KV and I = 20 mA

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

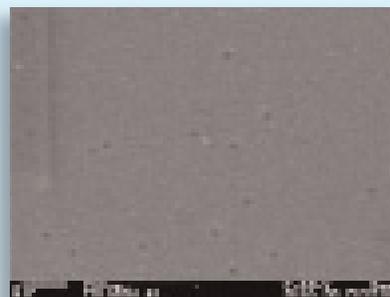
### General Points for Improving Performance

1. Cleanliness, the work chamber must be kept clean! We advise that a separate carbon coater be used in applications where the maximum performance of the sputter coater is required
  - Clean the glass chamber with hot soapy water and dry thoroughly, solvents can be used but we have found this unnecessary and having greater danger to health and safety. If the deposit is stubborn, use a kitchen scouring pad such as the green Scotch Bright variety.
  - Use Isopropyl alcohol on metal surfaces, not acetone which has greater danger to health and safety. It will also take longer to out gas and reduce the vacuum performance.
2. Vacuum, Never leave the chamber under vacuum without isolating the roughing pump from the coater, this is usually done with a manual valve (Quorum high vacuum sputter coaters have useful "pump hold" facility that allows the vacuum chamber to be held under vacuum when the instrument is not in use). Failure to do so will increase the risk of suck back of hydrocarbons (pump oil) in to the sputter chamber and increase contamination.
  - Always ensure the system is dry and pumping to its correct vacuum level before working with samples, failure to do so will result in poor sputter rate and contamination.
  - Ballast rotary pumps on a regular basis and ensure they are serviced at regular intervals.
3. Sputter gas, Always use high purity argon gas of the grade known as "White spot" this will ensure fast sputter rate and good pump down time.
4. Rotary planetary specimen stages are essential for ensuring even coatings on specimens with irregular surfaces.



### Gold/palladium coating of 6" wafer

This wafer was wafer-coated with 3nm of gold/palladium (Au/Pd) using the EMS 7640 Sputter Coater. Settings: 800V 12mA using argon gas and vacuum of 0.004 bar. Further tests revealed that coating was of an even thickness right to the edge of the 6" wafer. Work was done by Dr. Jost Gabler of Gala Instrumente GmbH.



### Platinum coating using SC7640

Borosilicate glass with surface imperfections (dark spots). Coated with 3nm of platinum (Pt) using the EMS 7640 Sputter Coater. Settings: 800V 12mA using argon gas and vacuum of 0.004 bar. Image provided by Gala Instrumente GmbH.



### TEM image of 2nm sputtered platinum film

Carbon-coated Formvar film. Coated with 2nm of platinum (Pt) using the EMS 7640 Sputter Coater. Settings: 800V 10mA using argon gas and vacuum of 0.004 bar. Image courtesy of Topcon Electron Beam Services Corporation.

## Sputter Coaters/SEM/TEM Carbon Coaters

### Techniques and Applications

# Silver as a removable coating for scanning electron microscopy

Acknowledgement: The following abstract and method results (introduction only) is reproduced by kind permission of A.A. Mills, Scanning Microscopy, Vol. 2, No.3, 1988 (Pages 1265-1271)

#### Abstract

A thin film of silver, applied by sputtering or vacuum evaporation, provides an excellent conformal conductive coating for scanning electron microscopy of insulating specimens. When no longer required it is easily removed with Farmer's Reducer - a dilute aqueous solution of potassium ferricyanide and sodium thiosulphate. No damage was apparent to fine structure in the calcite matrix of ostracode shells, or to other biological tissues. No problems have been encountered with grain in the silver film at magnifications up to x15,000, or in the storage of coated specimens in a desiccator for periods exceeding six months.

#### Introduction

Many specimens for which scanning electron microscopy (SEM) is invaluable are electrical insulators, for example microfossils and dried biological preparations. To promote the emission of secondary electrons, and to prevent charging of the surface (with consequent repulsion of both incoming and secondary electrons) it is usual to coat such specimens with a very thin layer of metal.

Nowadays gold (sometimes over a thin undercoat of carbon) is commonly employed for the majority of work, although refractory metals have been recommended for the very highest magnifications. These coatings are normally applied by sputtering in a glow discharge, for this technique is omni-directional and tends to give a fine-grained deposit, while the apparatus required is comparatively simple and inexpensive since a high vacuum is not required.

An alternative, older technique (which also allows aluminum to be deposited) is evaporation of a molten bead of the chosen metal in a high vacuum. The inherent directionality of this method means that specimens must generally be moved continuously by a rotating/nodding table.

Problems arise when it is desired to return a specimen to its original uncoated condition, for example to allow successive treatments or because too thick a coating has been accidentally applied. Even specimens which have been

correctly coated may be rendered unsuitable for subsequent optical and analytical examination, due to the highly reflective nature of the gold film and its interference with x-ray emission. For these reasons there is frequently a reluctance to allow SEM examination of certain material, eg type specimens and archaeological artifacts.

#### Removal of Gold and Aluminum Coatings

Attempts have therefore been made to remove the metal film by suitable reagents, which must obviously not attack the substrate. It is well-known that gold is recovered from siliceous ores by complexing with aqueous cyanide under oxidizing (aerobic) conditions, and two groups have independently utilized this reaction.

A major obstacle is the highly toxic nature of cyanides, necessitating efficient fume hoods and a high degree of supervision and control unwanted in most laboratories. A less objectionable reagent is ferric chloride in alcohol, but it requires some six hours on a gold/palladium film from a smooth PTFE surface, and appears likely to attach many specimens. Mercury amalgamates gold, but does not remove it completely and adds its own background.

Aluminum dissolves in weak acids and alkalies with the evolution of hydrogen. Sylvester and Bradley therefore hoped that soaking in a dilute solution of sodium hydroxide would enable this metal to be removed from calcite microfossils without damage to the matrix. Unfortunately, they were later obliged to acknowledge that insufficiently careful exposure to alkali could result in dissolution of fine structure.

#### Advantages of a silver film

Silver would appear to have much to commend it as an alternative to gold. It is the most conductive metal known, possesses a high secondary electron coefficient, and is readily applied by sputtering or evaporation to follow irregular contours better than any other material.

Unlike gold, its x-ray emission lines are well-separated from those of the biologically important sulphur and phosphorus. Its cost is only a fraction of gold and the platinum metals. The unique applicability of silver to photography has resulted in extensive research upon its complex ions and their solubility.

Quite early in the history of photography it was found that a dark, over-exposed negative could

be rendered less opaque ('reduced') by aqueous oxidizing agents in the presence of sodium thiosulphate. The metallic silver forms the Ag ion, which is promptly complexed by the thiosulphate so that still more silver dissolves. No gas is evolved. The negative would be removed from the reagent and thoroughly washed when a sufficient amount of silver had been abstracted from the image.

#### Materials and methods

One of the mildest of these 'reducers' is that formulated by Farmer in 1884, employing very dilute potassium ferricyanide as the oxidizing agent. As paper, albumen and gelatine were apparently unaffected, it was thought that this reagent might well prove suitable for dissolving silver from a variety of coated specimens without damage to the matrix. Ferricyanides do not possess the extreme toxicity of the simple cyanides, and may be purchased and used in the same way as ordinary laboratory and photographic chemicals.

#### Farmer's Reducer - the formulation used is based on that given by Jacobson:

##### Solution A

25g sodium thiosulphate (crystals)  
250ml water  
2 drops of Kodak 'Photoflo'

##### Solution B

10g potassium ferricyanide  
100ml water

These solutions appear to be stable indefinitely at room temperature if kept in securely stoppered amber glass bottles. Immediately before use, the following mixture is to be prepared:

50ml water  
50ml Solution A  
3ml Solution B

# Sputter Coaters/SEM/TEM Carbon Coaters

## Techniques and Applications

It was found that the resulting pale yellow solution had a pH of about 5, the same as the CO<sub>2</sub>-equilibrated tap water used for its preparation. It was unstable, losing activity and color after about two hours at room temperature.

A neutral mixture may be prepared by substituting pH 7 phosphate buffer (conveniently prepared from a BDH tablet) for water in the above dilution. However, all the tests to be described in the paper were conducted with the ordinary solution prepared with tap water.

It should be noted that calcium carbonate has a significant solubility in water. In nature, calcite microfossils are protected against percolating groundwater by the sacrificial dissolution of fossils above and around them. Once removed from this environment to the laboratory, such fossils should presumably be washed only with distilled water that has been allowed to stand in contact with CaCO<sub>3</sub> (eg marble chips) and filtered. Otherwise needles and similar fine structures will be particularly at risk.

This equilibrated 'hard' water could be used to prepare and dilute the Farmer's Reducer. A very brief final rinse in distilled water is probably permissible; the common practice of 'soaking overnight' is not.

### Results — silver mirror on glass

A silver mirror was made by evaporating the metal on to a microscope slide cleaned with chromic acid. Sufficient was deposited to give a semi-transparent film: silvery when placed on a dark background and viewed by reflected light, but behaving as a blue filter when examined by transmitted light.

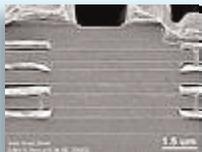
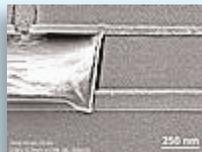
The coated glass slide was immersed in freshly-prepared Farmer's Reducer. The silver was gently dissolved in a controlled manner, as shown by the gradual and uniform loss of color in transmitted light, until none remained after three minutes. No gas was evolved. It was decided that a 10 minute immersion should allow an ample margin to deal with specimens with convoluted surfaces. The reagent had no effect upon gold films. Alloys of silver and gold have not been investigated.

## Comparative Sputter Data

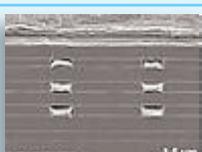
### Iridium and other materials

Samples were coated using an EMS 575X Sputter Coater and were examined using a Hitachi S-5200 Field Emission SEM.

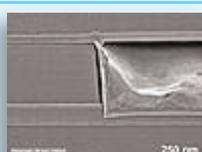
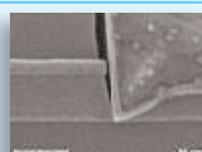
#### Gold

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	10 seconds	10 seconds	10 seconds
Current Used:	20 mA	20 mA	20 mA

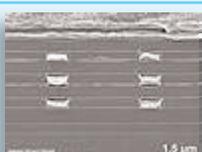
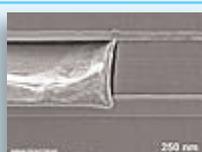
#### Gold/Palladium

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	10 seconds	10 seconds	10 seconds
Current Used:	20 mA	20 mA	20 mA

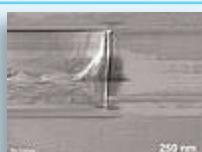
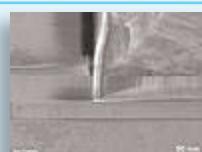
#### Chromium

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	30 seconds	30 seconds	30 seconds
Current Used:	100 mA	100 mA	100 mA

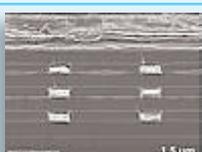
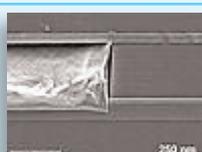
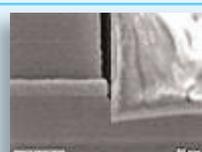
#### Iridium

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	10 seconds	10 seconds	10 seconds
Current Used:	20 mA	20 mA	20 mA

#### No Coating

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	N/A	N/A	N/A
Current Used:	N/A	N/A	N/A

#### Platinum

			
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	N/A	N/A	N/A
Current Used:	N/A	N/A	N/A

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS 7620 "Mini" Sputter Coater/Glow Discharge

The EMS 7620 is a compact, low cost SEM sputter coater that comes complete with a glow discharge option as a standard. When combined with the optional carbon attachment EMS 7640-CF it makes the ideal low cost SEM sputtering and carbon coating system package. The EMS 7620 is robust, easy to operate and is backed up with a three-year warranty.

#### Features

- Low cost
- Simple operation
- Magnetic deflection sputter coating head
- Compact design
- Carbon fiber evaporation option
- Adjustable height specimen stage
- Easy to change sputter targets - gold/palladium (Au/Pd) standard
- Built to all the latest safety standards - features include positive break electro-mechanical interlock which ensures the sputter coating head is electrically isolated when the optional carbon attachment is in use
- Robust and reliable
- Three-year warranty
- Glow discharge as a standard

#### Easy operation

The EMS 7620 is ideally suited to the budget-conscious user who none-the-less demands quality results from an easy-to-use instrument. Designed for routine applications, the EMS 7620 uses a basic magnetron sputter head with a simple-to-replace disc target (gold/palladium (Au/Pd) as standard). The head is hinged for easy operation and fitted with electrical safety interlocks.

To prevent accidental damage the high voltage lead is shielded. The plasma current is variable by adjustment of the vacuum level using an argon leak valve with the plasma voltage pre-set. For maximum sputter coating efficiency the gas injector system ensures that argon gas enters the chamber close to the plasma discharge. Venting is to argon.

#### Fast cycle times

The 100mm/4" diameter Pyrex cylinder is mounted on an aluminium collar and sealed with O rings. The small vacuum chamber means pump-down times and cycle times are fast; it also allows a small economical rotary pump to be used.

The specimen stage is height-adjustable over a wide range and can easily be removed to accommodate larger specimens. The system is controlled manually by a 180-second timer with 15-second resolution. Pressure and plasma current are monitored by analogue meters.

#### Quality coating

The EMS 7620 will deposit a quality coating and has the advantage of simple operation, ensuring reliability and suitability for general use. It can be simply converted to deposit carbon by the addition of an optional carbon evaporation attachment, consisting of a switchable voltage power supply and a carbon fiber head (see EMS 7620-CF below). The carbon attachment includes an additional glass cylinder. This is taller than the standard sputter coater cylinder and ensures that the distance between the carbon fiber and the specimens is optimal.

The EMS 7620 also comes complete with 1m x 12mm bore vacuum hose and fittings, and requires only the addition of a rotary pump with a capacity of 50L/m or greater - see Options and Accessories.

#### Requirements

A 50L/m two-stage rotary pump with oil mist filter is recommended. If intending to use an existing rotary pump it is important that its capacity is 50L/m or greater.



#### Options and Accessories

A carbon coating accessory (EMS 7640-CF) which consists of an evaporation power supply and carbon fiber head is available. Fitting the optional carbon evaporation attachment is simple. The normal sputtering head is tilted back and replaced with the carbon fiber head. Connection is then made to the power supply. To ensure that the exposed sputtering head cannot be powered when the add-on carbon head is under vacuum, a positive-break mechanical interlock ensures electrical isolation of the sputtering head. We also offer a 'stand alone' SEM carbon coater, see the EMS 450X.

#### Choosing a target

Gold/Palladium (Au/Pd):

Supplied as standard. Has the same properties (sputtering rate, secondary electron yield, cost) as gold but the sputtered grain size is smaller.

Gold (Au): Gold sputter coating is still widely used in many laboratories

Platinum (Pt): The sputtered grain size is smaller than gold or gold/palladium. Platinum has a slower sputtering rate and is more expensive than gold or gold/palladium.

Silver (Ag): Compared to the other metals, it is relatively easy to remove silver. Therefore it is useful for museum and forensic specimens.

Palladium (Pd): Sometimes used instead of gold, gold/palladium and platinum for x-ray microanalysis.

All targets are 57mm Ø x 0.1mm thick (unless specified otherwise)

#### Ordering Information

Part number	Description	
EMS 7620	'Mini' Sputter Coater/Glow Discharge	each
91003	RV-3 two-stage rotary pump with oil mist filter (115/230V 50/60Hz)	each
96000	Replacement oil mist filter	each
91040	Carbon evaporation attachment — EMS 7640	each
91017-AU	Gold (Au) target	each
91017-AP	Gold/palladium (Au/Pd) target	each
91017-PT	Platinum (Pt) target	each
91017-AG	Silver (Ag) target	each
91017-PD	Palladium (Pd) target	each
91017-.2-AU	Gold target (Au) 0.2mm thick	each
91017-.2-AP	Gold/palladium (Au/Pd) target 0.2mm thick	each
91017-.2-PT	Platinum (Pt) target 0.2mm thick	each

# Sputter Coaters/SEM/TEM Carbon Coaters

## ● EMS 7620 (continued)

### Specifications

**Dimensions:**.....340mm W x 130mm D x 250mm H (unpacked) excluding chamber  
**Supplied with:**.....91017-AP gold/palladium (Au/Pd) target, 1m length of 12mm bore flexible vacuum hose, 1 x KF25 hose adapter flange and fittings to fit a rotary pump, 1 x rotary pump plug, comprehensive operating instructions

### Site Requirements:

**Electrical:** .....Ensure that a suitable mains electricity supply (110VAC - 20A or 240VAC - 13A, frequency 50/60Hz) is available. Check that the voltage label attached to the side of the cabinet is suitable for the local voltage and frequency. The units are supplied for either 230V or 110V operation at 50/60Hz. The power rating is 250VA excluding the rotary pump. The rotary pump outlet is rated at 230V at 10A or 110V at 16A. The 240V pump outlet uses either a three-pin plug (404440310) or 110V standard US plug - both supplied.

**Sputtering Gas:** .....Ensure that a suitable gas supply is available, such as a commercial cylinder of argon gas (Zero Grade) fitted with a two-stage regulator, in order to deliver gas at a pressure of around 5-10psi (0.5bar).

**Vacuum Pump:** .....Ensure that a suitable vacuum pump is available. The work chamber has to be evacuated to less than 10-2mbar. This can be achieved in a reasonable time (depending on the cleanliness of the chamber) using a floor-mounted 50L/m or 90L/m two-stage rotary pump. Alternatively you can use a 30L/m desk-top mounted two-stage rotary pump, preferably incorporating an anti suck-back device and fitted with an oil mist filter on the exhaust port. Where a rotary pump is used, ensure that it has been filled with oil, in accordance with the manufacturer's instructions. The exhaust should be filtered or expelled to a safe area. All pumps we supply are fitted with an exhaust filter.

**Carbon evaporation attachment (optional):** .....The EMS 7640-CF Carbon Accessory Power Supply can be used in conjunction with the EMS 7640 'Mini' Sputter Coater. The units are supplied for either 230V or 110V operation at 50/60Hz. Ensure that a suitable mains electricity supply (110VAC - 20A or 240VAC - 13A, frequency 50/60Hz) is available. Check that the voltage label attached to the side of the cabinet is suitable for the local voltage and frequency.

**Space requirement:** .....340mm W x 320mm D x 310mm H (including chamber and sputtering head). Weight: 14kg. Additional space is required for the rotary pump, which can be located either on the floor or on the bench with the coater.



The SC7620 can be readily switched between sputtering and glow discharge modes



Optional carbon fibre evaporation attachment and controller



Optional carbon fibre evaporation attachment and controller (fitted)



SC7620 specimen stage and sputtering head



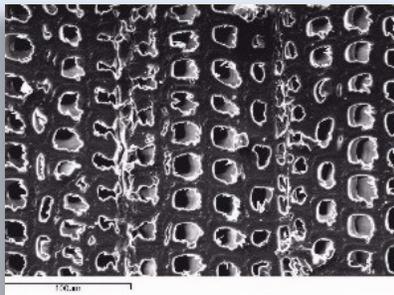
SC7620 vacuum chamber and specimen stage (adjustable height)

## Sputter Coaters/SEM/TEM Carbon Coaters

### What is... Carbon Coating?

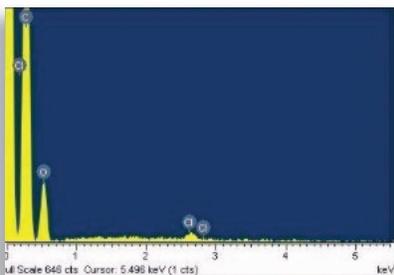
The use of carbon films in Electron Microscopy with their low background signal and relatively good electrical conductivity is well known. Thin films, nominally 5nm or 50 Angstroms, are used in TEM, while a range of somewhat thicker films, ranging from 50nm or 500 Angstroms, may be used in SEM for such applications as X-ray microanalysis.

Commonly, a high vacuum evaporator with carbon rods is used to achieve these coatings, and still has preferential applications. The use of carbon fiber however, has allowed a flash evaporation technique to be developed which can be suitable for a number of general EM requirements.



#### Pinus sylvestris (Scots pine)

Transverse section of Pinus sylvestris (Scots pine) in the first image shows the latewood portion of the growth ring. This surface shows latewood tracheids (transportation and structural cells) and also part of a ray (cells for storage of food substances). EDX spot analysis of the wood specimen using Oxford Instruments' INCA Energy shows a small chlorine peak, which results from treatment of the wood with a preservative - shown in the graph. The specimen charges excessively unless carbon coated. Other types of coating cannot be used due to the very low levels of chlorine used in the preservative, with which the wood is treated. With thanks to Oxford Instruments.



## Techniques and Applications

Procedures for the preparation of TEM carbon support films

### Section A. Preparation of normal carbon support films

NOTE: Process uses a diffusion-pumped vacuum evaporator, for turbomolecular-pumped systems please modify the process as appropriate. For optimum results, vacuum levels in the range of  $5 \times 10^{-6}$  mbar or better are recommended.

**Step 1.** Copper grids should be pre-cleaned by sonicating for 10 seconds in acetone, followed by 10 seconds of sonication in ethyl alcohol. Allow grids to dry on filter paper in a dust-free environment before use.

**Step 2.** Add 0.12g of formvar powder to 50ml of ethylene dichloride and mix well on a magnetic stirrer until dissolved. Pour the solution into a clean coplin jar.

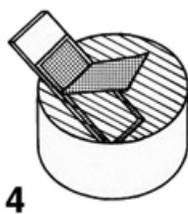
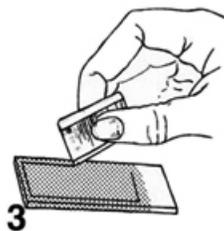
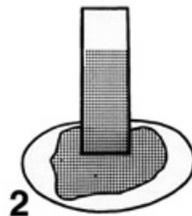
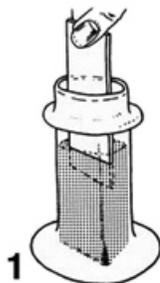
**Step 3.** Clean a glass slide with water and detergent. Rinse well to make sure that all of the detergent is removed and finally rinse in de-ionized water before drying with a paper towel. Blow off any lint on the slide with compressed air. Place the slide in a dry, dust-free environment such as on filter paper under an upturned beaker. If there are problems in getting the plastic film to be released from the slide (Step 5), using a slide that has not been as thoroughly cleaned might help.

**Step 4.** Dip the cleaned slide into the formvar solution (step 1 in picture) and touch edge to filter paper to drain off the excess fluid (step 2 in picture). Dry upright in a dust-free environment (this requires 5-10 minutes).

**Step 5.** Score the edges of the formvar film with an acetone-cleaned razor blade (step 3 in picture). Breathe on the slide to loosen the film, and slowly slide off onto a clean water surface by immersing the slide into the water at a  $-15^\circ$  angle (step 4 in picture). Place grids, dull/rough surface down, onto good (uniform, grey color, un-wrinkled) areas of the film. Place a small piece of clean, white office paper onto the surface of the grids and film and allow the paper to soak up water. Pick up the paper, grids and film and place in a covered petri dish to dry.

**Step 6.** Carbon coat film according to directions (see Section C) to desired thickness - a light-brown color indicates a thickness of 100Å.

**Step 7.** Place the paper and coated grids onto a piece of filter paper that is soaked with ethylene dichloride in a covered petri dish. 30 minutes should be sufficient time to dissolve the Formvar film and not damage the carbon support. Remove the grids and paper and allow them to dry in a dust-free area.



Figures from M. A. Hayat and S. E. Miller (1990). *Negative Staining*. McGraw Hill Publishing Co., N.Y. 253pp.

## Sputter Coaters/SEM/TEM Carbon Coaters

### Techniques and Applications

#### Section B. Preparation of perforated carbon support films

**Step 1.** Copper grids should be pre-cleaned by sonicating for 10 seconds in acetone, followed by 10 seconds of sonication in ethyl alcohol. Allow grids to dry on filter paper in a dust-free environment before use.

**Step 2.** Add 0.17g of formvar powder to 50ml of chloroform and mix well on a magnetic stirrer until dissolved. Pour the solution into a clean coplin jar.

**Step 3.** Clean a glass slide with water and detergent. Rinse well to make sure that all of the detergent is removed and finally rinse in de-ionized water before drying with a paper towel. Blow off any lint on the slide with compressed air. Place the slide in a dry, dust-free environment such as on filter paper under an upturned beaker. If there are problems in getting the plastic film to be released from the slide (Step 6), using a slide that has not been as thoroughly cleaned might help.

**Step 4.** Add about 50 drops of a 50% glycerol/water solution to the surface of the formvar solution. Place the tip of a probe sonicator onto the surface of the solution and sonicate until mixed. Sonication intensity should be great enough to 'violently' cause the solution to bubble. This often requires not much more than about five seconds. This should produce numerous holes that are 1-2 $\mu$ m in diameter and suitable for use with frozen-hydrated specimens. Sonication for longer periods of time produces smaller holes in the film.

**Step 5.** Immediately after sonicating, dip the cleaned slide into the formvar solution (step 1 in first diagram) and touch edge to filter paper to drain off the excess fluid (step 2 in first diagram). Dry upright in a dust-free environment for about 5-10 minutes.

**Step 6.** Score the edges of the formvar film with an acetone-cleaned razor blade (step 3 in first diagram). Breathe on the slide to loosen the film, and slowly slide off onto a clean water surface by immersing the slide into the water at a -15° angle (step 4 in first diagram). Place grids, dull/rough surface down, onto good (uniform, grey colour, unwrinkled) areas of the film. Place a small piece of clean, white office paper onto the surface of the grids and film and allow the paper to soak up water. Pick up the paper, grids and film and place in a covered petri dish to dry.

**Step 7.** Place the paper with the film and grids onto a methanol-soaked piece of filter paper in a covered petri dish for about 30 minutes. This should perforate any pseudo-holes that may be in the films (these occur when a small drop of glycerol was present but it was not enough to perforate the film).

After allowing the paper and film to dry, the grids may be examined in a light microscope under phase contrast to determine the quality of the films.

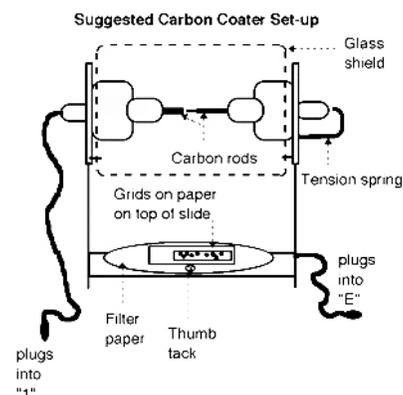
**Step 8.** Carbon coat film according to directions (see Section C) to desired thickness - a light-brown color indicates a thickness of 100Å.

**Step 9.** Place the paper and coated grids onto a piece of filter paper that is soaked with ethylene dichloride in a covered petri dish. 30 minutes should be sufficient time to dissolve the formvar film and not damage the carbon support. Remove the grids and paper and allow them to dry in a dust-free area.

#### Section C. Use of a shadow evaporator for carbon coating plastic films

**Step 1.** Turn shadow evaporator on: Turn both the main and mechanical pump switches on. Move the black-knobbed, manifold valve handle downwards to 'backing' position. Open the air inlet valve and CAREFULLY remove the implosion shield and bell jar. Set the bell jar upside down on the rest on the adjacent cabinet.

**Step 2.** Set up carbon coating apparatus: Plug one lead to ground ('E') and the other to '1' (see second diagram). Remove the cylindrical glass shield. Release the tension spring that holds the right carbon rod in place and remove the rod. File the edge of the left carbon rod flat with a piece of emery cloth. Replace the right rod with a fresh one or sharpen it by the procedure described below.



**Step 3.** Carbon rod sharpening procedure: Place the carbon rod in the chuck of the sharpener. Pull the rod out until its edge is aligned with the edge of the aligning arm and then tighten the chuck. Turn on the sharpener and run the first sharpener tool against the rod until a conical point is formed. Then run the other sharpener tool against the rod until a narrow point is formed. Turn off the sharpener and clean off all carbon dust. Put the newly sharpened rod in the chuck of the carbon coater and tighten. Replace the tension spring and then the glass shield.

**Step 4.** Set up grids: Place the grids and paper support on a piece of filter paper on top of the base of the carbon coating apparatus (see second diagram). Place a thumbtack alongside the slide. This provides a 'shadow' on the filter paper and helps you determine the relative thickness of the carbon coating.

**Step 5.** Diffusion pump warm up: Replace the bell jar and the implosion shield. Close the air inlet, and move the manifold valve handle slowly upwards to the roughing position. Allow the vacuum to reach 0.04 Torr on the bell jar gauge and then move the handle downwards to backing. IMPORTANT: Turn on the water supply. The water supply-line valve is located on the wall behind the shadow evaporator. Turn on the diffusion pump switch and allow the pump to warm up for 15 minutes before continuing.

## SEM/TEM Carbon Coaters

### Terms and Techniques

#### Carbon Fiber

Carbon fiber, normally in the form of a woven cord, can be used to thermally evaporate thin layers of carbon onto a substrate. The main application in electron microscopy (EM) is the production of thin, electrically-conducting coatings on scanning electron microscopy (SEM) specimens. Carbon fibre can be used for transmission electron microscopy (TEM) applications, but carbon rod is normally preferred due to superior control of the evaporation process.

#### Carbon Rods

Carbon (C) in the form of shaped graphite rods can be used to thermally evaporate thin layers of carbon onto a substrate. Common applications in electron microscopy (EM) include the production of carbon-coated transmission electron microscopy (TEM) grid support films and the making of TEM surface replicas. Carbon rod evaporation is also used to produce electrically-conducting surface coatings on scanning electron microscopy (SEM) specimens.

#### Glow discharge

Electric glow discharge is a type of plasma formed by passing a current at 100V to several kV through a gas at low pressure (ie in a vacuum system). The main application of glow discharge in electron microscopy (EM) is to convert naturally hydrophobic ('water-hating') carbon-coated transmission electron microscopy (TEM) support grids into a hydrophilic ('water-loving') condition. This treatment allows the collection of ultra-thin TEM resin sections from the water bath of an ultramicrotome.

Glow discharge systems can be stand-alone units or add-ons to larger vacuum evaporators.

NB: Glow discharges are sometimes considered to be 'imperfect' plasmas and cannot be used to plasma etch or plasma ash specimens – their use mainly being confined to altering surface energies, not the removal of bulk material.

### Techniques and Applications

#### Section C. Use of a shadow evaporator for carbon coating plastic films (continued)

**Step 6.** Obtaining a high vacuum: Move the manifold valve handle slowly upwards to the roughing position and allow the vacuum to reach 0.04 Torr on the bell jar gauge. While waiting for the vacuum to recover, fill the baffle with liquid nitrogen. When the bell jar vacuum has reached 0.04 Torr, move manifold valve handle down to the backing position. Depress the metal guard beneath the red mains valve knob and move the knob handle upwards to the open position. Allow the vacuum to reach a minimum of  $2 \times 10^{-5}$  Torr or better.

**Step 7.** Carbon coating: Turn the electrode selector to #1. Turn the electrode switch on. Slowly turn the electrode current control knob until there is a slight glow at the point where the two carbon rods meet. Slowly increase the current until the rods become white hot. The proper current setting should be just before the point where the carbon starts to sputter. Frequently monitor the thickness of the carbon by turning down the current, checking the darkening of the filter paper and then turning the current back up again.

**Step 8.** Diffusion pump cool down: Turn down the electrode current control knob and turn off the electrode switch. Make sure the manifold valve is set to the backing position and close the mains valve. Open the air inlet, remove the implosion shield and bell jar and remove the grids. Then replace the bell jar and implosion shield, close the air inlet and move the manifold valve handle to the roughing position. Allow the vacuum to reach 0.04 Torr on the bell jar gauge, move the manifold valve handle to the backing position, turn off the diffusion pump, and allow the pump to cool for 20 minutes.

**Step 9.** Turn shadow evaporator off: Close the manifold and turn off the mechanical pump and main power switches. Turn off the cooling water.

#### Section D. Glow discharging carbon films

**Step 1.** NOTE: Place the very edge of your carbon coated grids along the edge of a piece of double-sided tape on a glass slide. This will help to prevent your grids from flying around inside the shadow evaporator when the air release switch is opened.

**Step 2.** Turn shadow evaporator on: Turn the main power switch on, turn on the mechanical pump and move the manifold valve handle (black knob) downwards to the backing position. Open the air inlet. CAREFULLY remove the implosion shield and bell jar.

**Step 3.** Set up glow discharge unit: Plug the lead into the proper receptacle (BNC connector). Place the glass slide with your grids on the unit and replace the bell jar and implosion shield. Close the air inlet, turn the butterfly switch by the current gauges to glow discharge and move the manifold valve handle slowly upwards to the roughing position. Allow the vacuum to reach 0.2-0.15 Torr on the bell jar gauge. The manifold valve may be turned to the closed position if the vacuum rises above 0.10 Torr.

**Step 4.** Glow discharging: Turn the electrode selector to position #1 and turn the electrode switch on. Slowly turn up the electrode current until there is a bright purple glow surrounding the glow discharge unit. Maintain this setting for approximately 10 seconds while monitoring vacuum. Turn off the electrode current control knob and the electrode switch. Move the manifold valve handle to the backing position. Turn the butterfly switch back to the evaporator setting.

**Step 5.** Turn shadow evaporator off: Slowly open the air inlet to prevent your grids from being blown around the bell jar. Remove the grids, replace the shields and then close the air inlet. Move the manifold valve to the roughing position. Allow the vacuum to reach 0.04 Torr on the bell jar gauge before moving the manifold valve handle to the horizontal (closed) position. Turn off the mechanical pump and the main switch.

## SEM/TEM Carbon Coaters

### ● EMS 7620-CF Carbon Accessory Power Supplies

The EMS 7620-CF carbon attachment is a modular add-on for our sputter coater, allowing carbon fiber or carbon rod evaporation.

The attachment uses the existing chamber and vacuum system of the sputter coater and is therefore a cost-effective and efficient method for the evaporation of carbon for SEM applications. Note that the diameter of the top plate will vary according to the chamber size of the sputter coater onto which it is being fitted.

We also offer free-standing carbon evaporators.

#### Features

- Carbon rod or fiber
- Protection shutter
- Modular control electronics
- Interlocking for safe operation
- Three-year warranty

The EMS 7620-CF can be used in conjunction with the EMS 7620 (EMS 7620-CF), sputter coater. Sometimes it is also possible to retrofit one of the above onto our older models, Please contact us for information on compatibility.

The attachment consists of two components - a free-standing power supply and a carbon fiber or carbon rod head to suit the chamber size of the sputter coater onto which it is to be fitted.

The power supply is switchable between 10V/100A (for carbon rod evaporation) and 20V/50A (for carbon fiber evaporation). A vacuum interlock is provided to ensure safe operation of the sputter coater and carbon accessory system. Out-gas and coat switches are provided for complete control of the evaporation sequence.

#### Specifications

<b>Dimensions</b>	235mm W x 350mm D x 175mm H.
<b>Weight</b>	15kg
<b>Carbon source</b>	Carbon fiber, carbon cord
<b>Ammeter gauge</b>	0-50A
<b>Low voltage</b>	25V
<b>Out-gas current</b>	Selectable for carbon fiber or carbon rod
<b>Electrical supply</b>	230V/50Hz (3A max), 115V/60Hz (6A max)

#### Ordering Information

Sputter Coater	Carbon Rod Attachment	Carbon Fiber Attachment
EMS 7620 Sputter Coater	Not Available	EMS 7620-CF

#### Carbon fiber cord

<b>91046-1</b>	Carbon fiber cord - standard grade - 1cm
<b>91046-10</b>	Carbon fiber cord - standard grade - 10m
<b>91046-100</b>	Carbon fiber cord - standard grade - 100m
<b>91047-1</b>	Carbon fiber cord - high purity - 1m
<b>91047-5</b>	Carbon fiber cord - high purity - 5m
<b>91046</b>	Carbon fiber cord - standard purity, fine strands - 1m
<b>91046-4</b>	Carbon fiber cord - standard purity, fine strands - 10m
<b>91046-01</b>	Carbon fiber cord - standard purity, fine strands - 100m



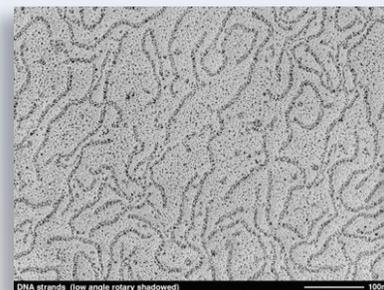
#### What is...

### Carbon Evaporation?

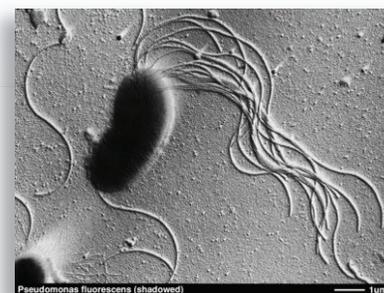
The use of Carbon Evaporation is well known in Electron Microscopy for support films and replicas in TEM, and X-Ray Microanalysis and conducting coatings in SEM.

The films are continuous for thicknesses of 2nm (or 20 Angstroms) or more, and free of significant structure.

The most common form of deposition is from resistance heated carbon, or graphite rods, spectrographically pure. The rods are shaped to achieve a high current density at the point of contact of the rods with sufficient temperature being generated to cause the evaporation. At this point, the appearance being that of small, bright, glowing particles of carbon. To achieve the required vacuum condition of  $1 \times 10^{-4}$  mbar, or better, requires the use of Diffusion or Turbo Molecular Pumps.



DNA strands



Pseudomonas fluorescens

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS 150T Turbo-Pumped Sputter Coater/Carbon Coater

The EMS150T is a compact turbomolecular-pumped coating system suitable for SEM, TEM and many thin-film applications. The EMS150T replaces previous the following models EMS575X and EMS950X.

The EMS150T is available in three formats:

**EMS150T S** — a high resolution sputter coater, suitable for oxidising and non-oxidising metals

**EMS150T E** — a high vacuum carbon coater for SEM and TEM applications

**EMS150T ES** — a high resolution sputter coater and high vacuum carbon evaporator

#### Features

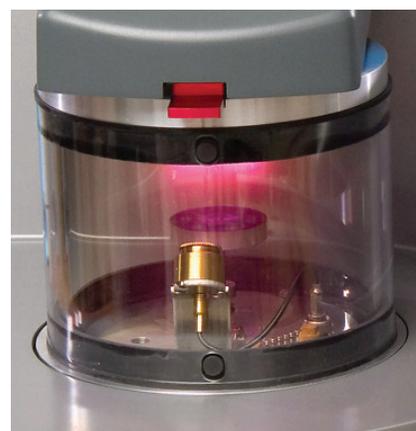
- Metal sputtering or carbon evaporation - or both - in one space saving design
- Fine grain sputtering - for advanced high resolution FESEM applications
- High vacuum turbo pumping - allows sputtering of a wide range of oxidizing and non-oxidizing metals - suitable for SEM, high resolution FESEM and also for many thin film applications
- High vacuum carbon coating - ideal for SEM and TEM carbon coating applications
- Advanced design carbon rod evaporation gun - simple operation, reproducible results
- Control of evaporation current profile - ensures consistently reproducible carbon films
- Precise thickness control using the film thickness monitor option
- Fully automatic touch screen control - rapid data input, simple operation
- Multiple, customer defined coating schedules can be stored - ideal for multi-user laboratories
- Automatic vacuum control, which can be pre-programmed to suit the process and material - no needle valve to adjust
- "Intelligent" recognition system - automatically detects the type of coating insert fitted
- Easy-to-change, drop-in style specimen stages (rotation stage as standard)
- Vacuum shutdown feature - leaves the process chamber under vacuum when not in use - improved vacuum performance
- Thick film capabilities - up to 60 minutes sputtering time without breaking vacuum
- Ergonomic one piece molded case - easy maintenance and service access
- Ethernet with local FTP server connection - simple programmer updates
- Power factor correction - complies with current legislation (CE Certification) - efficient use of power means reduced running costs
- Three-year warranty



#### Ideal for SEM, high resolution FESEM and TEM applications

EMS150T is available in three formats: sputtering, carbon evaporation or both. Depending upon the selected configuration, the EMS 150T can be a top-of-the-range sputter coater for high resolution scanning electron microscopy (SEM), a carbon coater suitable for SEM and transmission electron microscopy (TEM), or both, in a single easy to use system.

The ability of the EMS150T to rapidly sputter a wide selection of oxidizing and non-oxidizing metals also makes it an ideal platform for many thin film applications.



#### Molded case with color touch-screen

The EMS150T is presented in a custom molded, one-piece case. The color touch screen allows multiple users to input and store coating protocols. The case houses all the working components, including the efficient 70L/s air-cooled turbo molecular pump. Automatic bleed control ensures optimum vacuum conditions during sputtering.

The vacuum chamber has an external diameter of 165mm (6.5") and comes with an integral implosion guard. The EMS150T includes "vacuum shutdown" which enhances vacuum performance by allowing the chamber vacuum to be maintained when the system is not in use. A variable speed rotary specimen stage is fitted as standard, with other stages available as options.

#### Sputter coating, carbon coating or both

The EMS150T is available in three formats, each with a range of optional accessories:

- **EMS150T S** - a high resolution sputter coater for oxidizing and non-oxidizing (noble) metals. A wide selection of sputtering targets is available, including iridium and chromium, which are highly recommended for FESEM applications.

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS 150T (continued)

■ **EMS150T E** – a high vacuum carbon coater, ideal for the production of highly stable carbon films and surface replicas for transmission electron microscopy (TEM). The system uses economical 3.05mm diameter carbon rods.

■ **EMS150T ES** – a combined system with both sputtering and carbon coating. The deposition inserts can be swapped in seconds and the intelligent system logic automatically recognizes which insert is in place and displays the appropriate operating settings.

Each of the above can be fitted with a range of optional accessories (eg metal evaporation, carbon fiber coating, film thickness monitor). See options for details.

#### Rapid data entry

At the operational heart of the EMS150T is a simple color touch screen which allows even the most inexperienced or occasional operator to rapidly enter and store their own process data. To further aid ease of use, a number of typical sputtering and evaporation profiles are already stored.



#### Processes

**Sputtering:** 0-150mA to a pre-determined thickness (with optional FTM) or by the built-in timer. The maximum sputtering time is 60 minutes (without 'breaking' vacuum and with built in rest periods).



Standard Rotation Stage and FTM

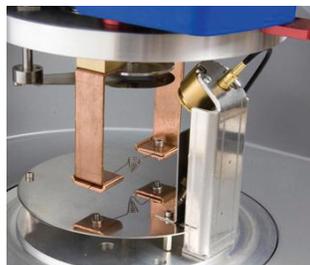
**Carbon evaporation:** A robust, ripple free DC power supply featuring pulse evaporation ensures reproducible carbon evaporation from rod or fiber sources. Current pulse: 1-90A



Carbon Rod Evaporation Insert

#### Metal evaporation and aperture cleaning insert (option):

For thermal evaporation of metals from filaments or boats. For cleaning SEM or TEM apertures a standard molybdenum boat (supplied) can be fitted. The metal evaporation head is set up for downwards evaporation, but upward evaporation can be achieved by fitting two terminal extensions (supplied). Evaporation times: up to four minutes.



Metal Evaporation Insert

#### Specifications

<b>Instrument case</b>	585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
<b>Weight</b>	33.4kg
<b>Packed dimensions</b>	725mm W x 660mm D x 680mm H (42kg)
<b>Work chamber</b>	Borosilicate glass 152mm Ø (inside) x 127mm H
<b>Safety shield</b>	Integral polyethylene terephthalate (PET) cylinder
<b>Display</b>	145mm 320 x 240 color graphic TFT (Thin Film Transistor) display
<b>User interface</b>	Intuitive full graphical interface with touch screen buttons, includes features such as a log of the last ten coatings carried out and reminders for when maintenance is due
<b>Sputter target</b>	Disc style 57mm Ø. A 0.3mm thick chromium target is fitted as standard. EMS150T S and T ES versions only
<b>Vacuum</b>	
<b>Turbomolecular pump</b>	Internally-mounted, 70L/s air-cooled turbomolecular pump
<b>Rotary pump</b>	Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter
<b>Vacuum measurement</b>	Pirani gauge as standard. A full range gauge (10428) is available as an option
<b>Typical ultimate vacuum</b>	$5 \times 10^{-5}$ mbar in a clean system after pre pumping with dry nitrogen gas
<b>Sputter vacuum range</b>	Between $5 \times 10^{-3}$ and $5 \times 10^{-1}$ mbar
<b>Specimen stage</b>	60mm Ø rotating stage. Rotation speed 8-20 rpm

#### Services and other information

**Gases:** Argon sputtering process gas, 99.999% (TS and TES versions)  
Nitrogen venting gas (optional)

**Electrical supply:** 90-250V ~ 50/60 Hz 1400 VA including RV3 rotary pump power. 110/240V voltage selectable

**Conformity:** CE conformity:

**Power factor correction:** Complies with the current legislation (CE Certification) and ensures efficient use of power, which means reduced running costs

#### Ordering information (see next page...)

## SEM/TEM Carbon Coaters

### ● EMS 150T Turbo-Pumped Sputter Coater/Carbon Coater (continued)

#### Ordering Information

- EMS150T S** High Resolution Turbomolecular-Pumped Sputter Coater, inc. a 54mm  $\varnothing$  x 0.3mm chromium (Cr) target
- EMS150T ES** High Resolution Turbomolecular-Pumped Sputter Coater and Carbon Evaporator, including a 57mm  $\varnothing$  x 0.3mm chromium (Cr) target and high vacuum carbon rod evaporation insert for 3.05mm  $\varnothing$  carbon rods. NOTE: coating inserts are interchangeable
- EMS150T E** Turbomolecular-Pumped Carbon Evaporator, suitable for TEM and SEM applications. Fitted with a carbon rod evaporation insert for 3.05mm  $\varnothing$  carbon rods. Supplied with carbon rods (3.05mm  $\varnothing$  x 100mm) and a carbon rod shaper (manual operation)

#### Rotary pump requirements (must be ordered separately)

- 91003** Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter

#### Options and Accessories

Including details of coating head inserts and specimen stages that are fitted as standard.

#### Coating Head Options

A range of interchangeable, plug-in style coating head inserts are available:

- 3200** Sputtering head insert suitable for oxidizing and non-oxidizing metals. Supplied with a 54mm  $\varnothing$  x 0.3mm thick chromium target as standard.
- 3210** Additional sputter insert for quick metal change. Note: this is an entire sputtering assembly, individual targets can also be purchased
- 3230** Carbon rod evaporation head insert (for 3.05mm  $\varnothing$  rods)
- 3240** Carbon rod evaporation head insert (for 6.15mm  $\varnothing$  rods). Note that EMS recommends 3.05mm  $\varnothing$  rods as they offer greater process control and are more economical (less wastage)
- 3250** Carbon fiber evaporation head insert
- 3260** Metal evaporation and aperture cleaning head insert, including the ability to evaporate upwards or downwards (T E and T ES versions only). Supplied with a pack of ten tungsten filaments and a molybdenum boat
- 3270** Extended height vacuum chamber (214mm high – the standard chamber is 127mm high). For increased source to sample distance and for coating large specimens
- 3280** Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS150T when bench depth is limited
- 3290** Film thickness monitor (FTM) attachment. Consists of a built in chamber mounted quartz crystal oscillator (includes crystal). As sputtered or evaporated material is deposited onto the crystal, so its frequency of oscillation is modified. This 'modification' is used to measure and control the thickness of material deposited
- 3300** Spare quartz crystals. Pack of three
- 3320** Full range vacuum gauge for low and high vacuum measurement (a low vacuum Pirani gauge is fitted as standard)
- 4513** Glow discharge inset to modify surface properties or to clean surface residues



Easy-to-change coating inserts



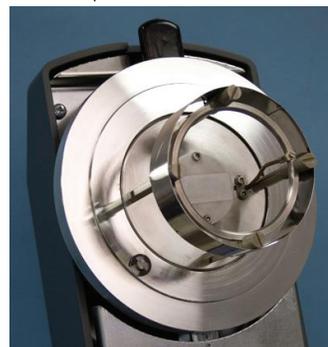
Sputter coater insert



Carbon fibre evaporation insert



Metal evaporation insert



Glow discharge attachment option

## SEM/TEM Carbon Coaters

### ● EMS 150T (continued)

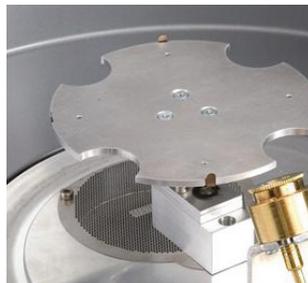
#### Specimen stages

The EMS150T has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage): Please contact us for a quote.

<b>3330</b>	Rotation stage, 50mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment
<b>3340</b>	Rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37mm-60mm). Tilt angle can be pre-set. 50mm Ø specimen platform with six stub positions for 15mm or 6.5mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm
<b>3350</b>	Variable angle "Rotacota" rotary planetary stage with 50mm Ø specimen platform. Has six stub positions for 15mm or 6.5mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm
<b>3360</b>	Flat rotation specimen stage for 100mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm
<b>3370</b>	Rotating specimen stage for glass microscope slides (up to two x 75mm x 25mm slides). Stage rotation speed variable between 8 and 20rpm. Includes gear box to allow optional FTM to be used
<b>3380</b>	EMS150T S High resolution turbomolecular pumped sputter coater, including a 54mm Ø x 0.3mm chromium target
<b>3400</b>	EMS150T ES High-resolution turbomolecular pumped sputter coater, including a 57mm Ø x 0.3mm chromium target and high vacuum carbon rod evaporation coater for 3.05mm Ø carbon rods.



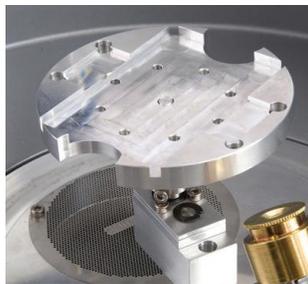
Tilt Angle Stage



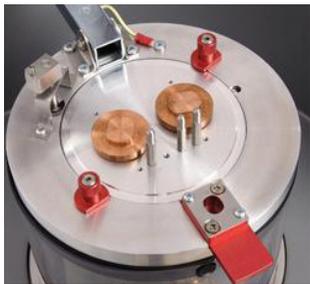
4" Wafer Stage



Rotary Planetary Stage



Glass Microscope Stage



Carbon Evaporation Insert

#### Sputtering Targets

The EMS150T S and EMS150T ES are fitted as standard with a 0.3mm thick chromium sputter target. Other optional targets:

<b>3410</b>	57mm 0 x 0.1mm Gold	each
<b>3411</b>	57mm 0 x 0.1mm Gold/Palladium (80/20)	each
<b>3412</b>	57mm 0 x 0.1mm Platinum	each
<b>3413</b>	57mm 0 x 0.1mm Nickel	each
<b>3414</b>	57mm 0 x 0.1mm Silver	each
<b>3415</b>	57mm 0 x 0.1mm Palladium	each
<b>3416</b>	57mm 0 x 0.1mm Copper	each
<b>3417</b>	57mm 0 x 0.3mm Chromium	each
<b>3418</b>	57mm 0 x 0.5mm Tungsten	each
<b>3419</b>	57mm 0 x 1.5mm Chromium	each
<b>3420</b>	57mm 0 x 0.2mm Tungsten	each
<b>3421</b>	54mm 0 x 1.5mm Carbon	each
<b>3422</b>	57mm 0 x 0.1mm Aluminium	each
<b>3423</b>	57mm 0 x 0.1mm Platinum/Palladium (80/20)	each
<b>3424</b>	57mm 0 x 1.5mm Titanium	each
<b>3425</b>	57mm 0 x 0.3mm Platinum/Palladium (80/20)	each
<b>3426</b>	57mm 0 x 0.3mm Gold	each
<b>3427</b>	57mm 0 x 0.3mm Gold/Palladium (80/20)	each
<b>3428</b>	57mm 0 x 0.3mm Platinum	each
<b>3429</b>	57mm 0 x 0.5mm Titanium	each
<b>3430</b>	57mm 0 x 0.1mm Iron	each
<b>3431</b>	57mm 0 x 0.3mm Iridium	each
<b>3432</b>	57mm 0 x 0.1mm Cobalt	each
<b>3433</b>	57mm 0 x 0.1mm Tin	each
<b>3434</b>	57mm 0 x 0.1mm Molybdenum	each
<b>3435</b>	57mm 0 x 0.3mm Magnesium	each
<b>3436</b>	57mm 0 x 0.1mm Tantalum	each
<b>3437</b>	57mm 0 x 3mm Indium Tin Oxide (90/10)	each

#### Carbon supplies

<b>3500</b>	Carbon rods – 6.15mm Ø x 100mm length (unshaped)	10/pk
<b>3510</b>	Carbon rods – 6.15mm Ø x 50mm length (shaped)	10/pk
<b>3520</b>	Carbon rods - 3.05mm Ø x 50mm length (shaped)	10/pk
<b>3530</b>	Carbon rods 3.05mm Ø x 300mm length (unshaped)	10/pk
<b>3540</b>	Carbon fiber cord - high purity - 1m	each
<b>3550</b>	Carbon fiber cord - high purity - 5m	each
<b>3560</b>	Carbon fiber cord - standard grade - 1m	each
<b>3570</b>	Carbon fiber cord - standard grade - 10m	each
<b>3580</b>	Carbon fiber cord - standard grade - 100m	each
<b>3590</b>	Manual rod shaper for 6.15mm Ø carbon rods	each
<b>3595</b>	Manual rod shaper for 3.05mm Ø carbon rods	each
<b>3540</b>	Carbon fiber cord - high purity - 1m	each
<b>3550</b>	Carbon fiber cord - high purity - 5m	each
<b>3560</b>	Carbon fiber cord - standard grade - 1m	each
<b>3570</b>	Carbon fiber cord - standard grade - 10m	each
<b>3580</b>	Carbon fiber cord - standard grade - 100m	each
<b>3590</b>	Manual rod shaper for 6.15mm Ø carbon rods	each
<b>3595</b>	Manual rod shaper for 3.05mm Ø carbon rods	each

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS150R Rotary Pumped Carbon and Sputter Coating System

#### EMS Series of Rotary Pumped Modular Coating Systems

*Innovative and versatile Sputter Coater and Carbon Evaporator for SEM Applications*

#### Quick Overview

The EMS150R is a compact rotary pumped coating system ideally suited for SEM and other coating applications. The EMS150R is available in three formats:

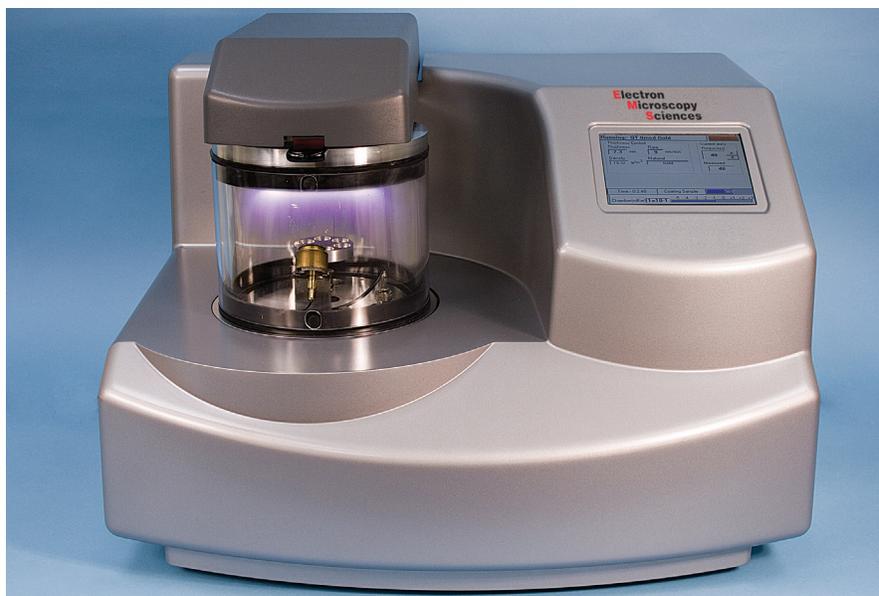
**EMS150R S** – a compact rotary pumped sputter coater, suitable for non-oxidising metals, such as gold (Au) and platinum (Pt)

**EMS150R E** – a compact rotary pumped carbon fibre coater suitable for SEM applications

**EMS150R ES** – a compact rotary pumped combined sputter coater and carbon evaporator

#### KEY FEATURES

- Metal sputtering or carbon evaporation - or both – in one space saving design
- Rotary pumped sputter coating – allows sputtering of a wide range of non-oxidising (noble) metals, such as gold (Au), platinum (Pt), silver (Ag) and palladium (Pd)
- Carbon fibre coating – ideal for SEM carbon coating applications (eg EDS and WDS)
- Advanced design carbon evaporation gun – simple operation, reproducible results
- Glow discharge option – for modification of sample surface properties (eg hydrophobic to hydrophilic conversion) or for cleaning / removal of contaminating surface residues
- Control of evaporation current profile – ensures consistently reproducible carbon films
- Repeatable thickness control using the film thickness monitor option
- Fully automatic touch screen control – rapid data input, simple operation
- Multiple, customer defined coating protocols can be stored – ideal for multi-user labs
- Automatic vacuum control, which can be pre-programmed to suit the process and material – no needle valve to adjust
- “Intelligent” recognition system – automatically detects the type of coating insert fitted
- Easy-to-change, drop-in style specimen stages (rotation stage as standard)
- Thick film capabilities – up to 60 minutes sputtering time without breaking vacuum
- Ergonomic one piece moulded case – easy maintenance and service access
- Ethernet with local FTP server connection – simple programmer updates
- Power factor correction – complies with current legislation (CE Certification) – efficient use of power means reduced running costs
- Three-year warranty



#### Product Description

Ideal for SEM applications

EMS150R is available in three formats: sputtering, carbon evaporation or both. Depending upon the selected configuration, the EMS150R can be a top-of-the-range sputter coater for scanning electron microscopy (SEM), a carbon coater suitable for SEM (eg EDS and WDS), or both, in a single easy to use system.



Color touch screen control

#### Molded case with color touch-screen

The EMS150R is presented in a custom molded, one-piece case. The color touch screen allows multiple users to input and store coating protocols. The case houses all the working components. Automatic bleed control ensures optimum vacuum conditions during sputtering.

The vacuum chamber has an external diameter of 165mm (6.5”) and comes with an integral implosion guard. A variable speed rotary specimen stage is fitted as standard, with other stages available as options.

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS150R (continued)

#### Sputter coating, carbon coating or both

The EMS150R is available in three formats, each with a range of optional accessories:

■ **EMS150R S** – a compact rotary pumped sputter coater, suitable for non-oxidising (noble) metals. A wide selection of sputtering targets is available, which are highly recommended for SEM applications.

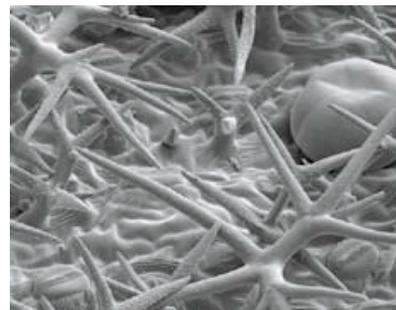
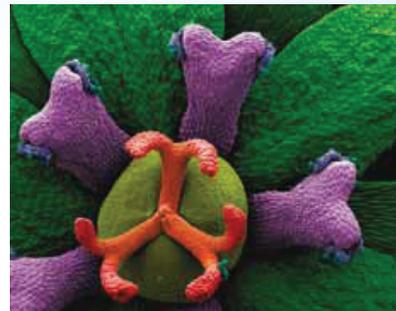
■ **EMS150R E** – a compact rotary pumped carbon fibre coater suitable for SEM applications (eg EDS and WDS). The system uses carbon fibre / cord as standard.

■ **EMS150R ES** – a combined system with both sputtering and carbon fibre coating. The deposition inserts can be swapped in seconds and the intelligent system logic automatically recognizes which insert is in place and displays the appropriate operating settings.

Each of the above can be fitted with a range of optional accessories (eg glow discharge, carbon rod coating, film thickness monitor). See options for details.



'Cool' sputtering. Optional film thickness monitor (FTM) fitted



#### Rapid data entry

At the operational heart of EMS150R is a simple color touch screen, which allows even the most inexperienced or occasional operators to rapidly enter and store their own process data. To further aid ease of use a number of typical sputtering and evaporation profiles are already stored.



#### Maintenance

The intuitive touch screen interface features maintenance prompts which highlight:

- Time of last clean
- System "on time"
- Coating time since last cleaned
- Time of last service

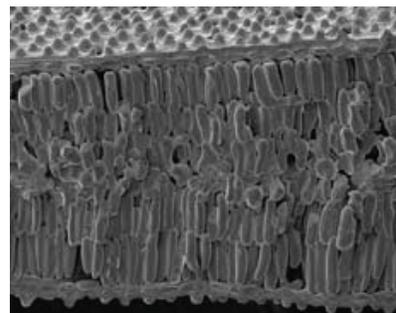
#### Glow Discharge Attachment

The primary application of glow discharge is as a technique for the surface modification, or 'wetting' or hydrophilisation of newly-evaporated transmission electron microscopy (TEM) carbon support films.

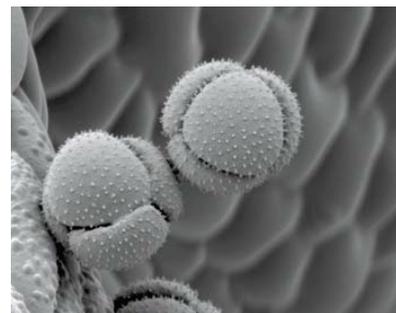
Freshly-made TEM carbon support films tend to have hydrophobic surfaces which inhibits the spreading of suspensions of particles in negative staining solutions. However, after glow discharge treatment with air, the carbon film is made hydrophilic and negatively charged, thus allowing easy spreading of aqueous suspensions. With subsequent magnesium acetate treatment the surface is made hydrophilic and positively charged.



Optional glow discharge attachment (for R S and R ES versions)



In addition to glow discharge treatment using air, other process gases may be used to modify surface properties. For example, methanol as a process gas results in the surface becoming hydrophobic and negatively charged. Such treatment can facilitate the optional absorption of selected biomolecules



continued >>>>

# Sputter Coaters/SEM/TEM Carbon Coaters

## ● EMS150R Rotary Pumped Carbon and Sputter Coating System (continued)

### Specifications

Instrument case	.585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
Weight	.28.4kg
Packed dimensions	.725mm W x 660mm D x 680mm H (36.8kg)
Work chamber	.Borosilicate glass 152mm Ø (inside) x 127mm H
Safety shield	.Integral polyethylene terephthalate (PET) cylinder
Display	.145mm 320 x 240 colour graphic TFT (Thin Film Transistor) display
User Interface	.Intuitive full graphical interface with touch screen buttons, includes features such as reminders for when maintenance is due and a log of the last ten coatings carried out
Sputtering target	.Disc style 57mm Ø. A 0.1mm thick gold target is fitted as standard. Q150R S and Q150R ES versions only
Specimen stage	.50mm Ø rotating stage with 6 stub positions for 15mm, 10mm, 6.5mm or 1/8" pin stubs. Rotation speed 8-20 rpm

### Vacuum

Rotary pump	.Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter
Vacuum measurement	.Pirani gauge fitted as standard
Typical ultimate vacuum	. $2 \times 10^{-2}$ mbar in a clean system after pre pumping with dry nitrogen gas
Sputter vacuum range	.Between $3 \times 10^{-2}$ and $5 \times 10^{-1}$ mbar

### Processes

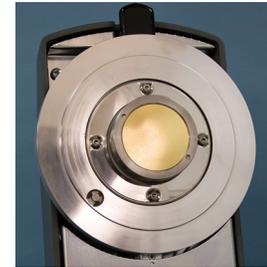
Sputtering	.0-80mA to a pre-determined thickness (with optional FTM) or by the built-in timer. The maximum sputtering time is 60 minutes (without "breaking" vacuum and with built in rest periods)
Carbon evaporation	.A robust, ripple free D.C. power supply featuring pulse evaporation ensures reproducible carbon evaporation from fibre, cord or rod sources. Current pulse for carbon cord: 1-60 Amps; current pulse for carbon rods with spigot size of 1.4mm Ø: 1-90 Amps
Glow discharge	.0-80mA operates in DC+ and DC- modes

### Services and other information

Gases	.Argon sputtering process gas, 99.999% (R S and R ES versions). Nitrogen venting gas (optional)
Electrical supply	.90-250V ~ 50/60 Hz 1400 VA including RV3 rotary pump power. 110/240V voltage selectable
Conformity	.CE conformity
Power factor correction	.Complies with the current legislation (CE Certification) and ensures efficient use of power which means reduced running costs



Carbon fibre evaporation insert and automatic source shutter



Sputtering insert. Gold (Au) fitted as standard, but other metals available

## Ordering Information

### EMS150R S

4500	EMS150R S Rotary pumped sputter coater, includes an 57mm Ø x 0.1mm gold target
4501	Quick-release sputter insert for EMS150R S and EMS150R ES – suitable for non-oxidising (noble) metals. Supplied with A 57mm Ø x 0.1mm thick gold target as standard. For additional noble metal targets (eg Platinum, Gold / Palladium, Silver) see Sputtering Targets section
4502	Rotation stage, 50mm Ø with adjustable height for target to sample distances of 38mm-79mm (supplied with 2 mounting pillars). Note: this stage does not tilt – for tilting stage see Specimen Stages section

### EMS150R E

4503	EMS150R E Rotary pumped carbon fibre evaporator, supplied with carbon fibre / cord
4504	Quick-release carbon fibre evaporation insert for EMS150R E and EMS150R ES – suitable for evaporation of carbon fibre and carbon cord
4506	Rotation stage, 50mm Ø with adjustable height for target to sample distances of 38mm-79mm (supplied with 2 mounting pillars). Note: this stage does not tilt – for tilting stage see Specimen Stages section

### EMS150R ES

4507	EMS150R ES Rotary pumped sputter coater, includes an 57mm Ø x 0.1mm gold target, and carbon fibre evaporator, supplied with carbon fibre / cord
4508	Quick-release sputter insert for EMS150R S and EMS150R ES – suitable for non-oxidising (noble) metals. Supplied 57mm Ø x 0.1mm thick gold target as standard. For additional noble metal targets (eg Platinum, Gold / Palladium, Silver) see Sputtering Targets section
4509	Quick-release carbon fibre evaporation insert for EMS150R E and EMS150R ES – suitable for evaporation of carbon fibre and carbon cord
4510	Rotation stage, 50mm Ø with adjustable height for target to sample distances of 38mm-79mm (supplied with 2 mounting pillars). Note: this stage does not tilt – for tilting stage see Specimen Stages section

## Sputter Coaters/SEM/TEM Carbon Coaters

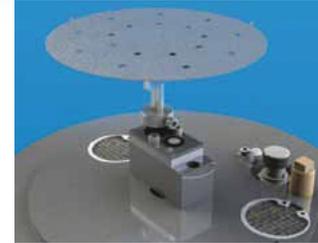
### ● EMS150R (continued)



Carbon fibre evaporation insert



Glow Discharge insert



### Options and Accessories

A range of interchangeable, plug-in style coating inserts are available:

4511	Additional sputter insert for quick metal change (R S and R ES only). Note: this is an entire sputtering assembly; individual noble metal targets can also be purchased
4512	Carbon rod evaporation insert for 3.05mm Ø rods (R E and R ES only). Includes manual rod shaper and 3.05mm Ø carbon rod
4513	Glow discharge insert to modify surface properties (eg hydrophobic to hydrophilic conversion) or to clean surface residues (R S and R ES only). Can be retrofitted
4514	Additional standard glass chamber assembly
4515	Extended height vacuum chamber (214mm high – the standard chamber is 127mm high). For increased source to sample distance and for coating large specimens
4516	Rotating vacuum spigot allows more convenient connection of the vacuum hose to the rear of the ems150R when bench depth is limited
4517	Film thickness monitor (FTM) attachment. Consists of a built in chamber mounted quartz crystal oscillator (includes crystal). As sputtered or evaporated material is deposited onto the crystal, so its frequency of oscillation is modified. This "modification" is used to measure and control the thickness of material deposited
4518	Spare quartz crystal

### Specimen stages

The EMS150R has alternative specimen stages to meet most requirements. All are easy-change, drop-in style and are height adjustable (except 10360 rotary planetary stage)

4519	50mm Ø rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37mm-60mm). Has six stub positions for 15mm or 6.5mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm
4520	90mm Ø rotating specimen stage for glass microscope slides (up to two x 75mm x 25mm slides). Stage rotation speed variable between 8 and 20rpm
4521	Variable angle "Rotacota" rotary planetary stage with 50mm Ø specimen platform. Has six stub positions for 15mm or 6.5mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm
4522	Flat rotation specimen stage for 100mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm

### Sputtering Targets

4523	57mm Ø x 0.1mm Gold	each
4524	57mm Ø x 0.2mm Gold	each
4525	57mm Ø x 0.3mm Gold	each
4526	57mm Ø x 0.1mm Gold/Palladium (80/20%)	each
4527	57mm Ø x 0.2mm Gold/Palladium (80/20%)	each
4528	57mm Ø x 0.3mm Gold/Palladium (80/20%)	each
4529	57mm Ø x 0.1mm Platinum	each
4530	57mm Ø x 0.2mm Platinum	each
4561	57mm Ø x 0.3mm Platinum	each
4532	57mm Ø x 0.1mm Nickel	each
4533	57mm Ø x 0.1mm Silver	each
4534	57mm Ø x 0.1mm Palladium	each
4535	57mm Ø x 0.1mm Copper	each
4536	57mm Ø x 0.1mm Platinum/Palladium (80/20%)	each
4537	57mm Ø x 0.3mm Platinum/Palladium (80/20%)	each

### Carbon Supplies

4538	Carbon fibre cord - high purity - 1m	each
4539	Carbon fibre cord - high purity - 5m	each
4540	Carbon fibre cord - standard grade - 1m	each
4541	Carbon fibre cord - standard grade - 10m	each
4542	Carbon fibre cord - standard grade - 100m	each
4543	Carbon rods - 3.05mm Ø x 50mm length (shaped)	each
4544	Carbon rods 3.05mm Ø x 300mm length (unshaped)	each
4546	Manual rod shaper for 3.05mm Ø carbon rods	each

### Spare kits

4547	Two-year spares kit for EMS150R S Includes: 57mm Ø x 0.1mm gold target, standard glass chamber assembly quartz crystals, O-rings	each
4548	Two-year spares kit for EMS150R E Includes: carbon fibre / cord, standard glass chamber assembly, quartz crystals, O-rings, springs	each
4549	Two-year spares kit for EMS150R ES Includes: 57mm Ø x 0.1mm gold target, / carbon fibre / cord, standard glass chamber assembly, quartz crystals, O-rings, springs	each

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300R T Triple Target, Large Chamber, Rotary-Pumped Sputter Coater

#### Quick Overview

The EMS300R T is a large chamber, rotary-pumped coating system ideally suited for sputtering a single large diameter specimen (e.g. a wafer) up to 8"/203 mm or smaller, multiple specimens over a similar diameter.

The EMS300R T is fitted with three individual sputtering heads to ensure even deposition on a range of specimen types. The system is designed to sputter non-oxidizing (noble) metals, for example gold (Au), gold/palladium (Au/Pd) and platinum (Pt). It is fitted with three individual sputtering heads to ensure even sputtering deposition over a large diameter. Gold targets are fitted as standard.

Note: for sputtering both non-oxidizing and oxidizing metals please see the EMS300T Large Chamber Turbo-Pumped sputter coater. For sequential sputtering of two different oxidizing or non-oxidizing metals please see the EMS300T D Large Chamber, Turbo-Pumped dual head sputter coater.



#### KEY FEATURES:

- **Large area sputter coating** – up to 8"/203 mm diameter
- **Triple sputtering head** – ensures even coating deposition of large specimens
- **Single target selection** – for economic coating of small specimens
- **Sputtering of a range of non-oxidising (noble) metals**, such as gold (Au), platinum (Pt) and silver (Ag). For oxidising metals see EMS300T T and EMS300T D
- **Precise thickness control using the film thickness monitor option**
- **Fully automatic touch-screen control** – rapid data input, simple operation
- **Multiple, customer-defined coating schedules can be stored** – ideal for multi-user laboratories
- **Coat logging** – details of the last 100 coatings available on screen
- **Automatic vacuum control** – can be pre-programmed to suit the process and material; no needle valve to adjust
- **Easy-to-change, drop-in style specimen stages (rotation stage as standard)**
- **Vacuum shut-down option** – leaves the process chamber under vacuum when not in use giving improved vacuum performance
- **Thick film capabilities** – up to 60 minutes sputtering time without breaking vacuum
- **Ergonomic one-piece moulded case** – allowing easy maintenance and service access
- **Ethernet with local FTP server connection** - simple programmer updates
- **Power factor correction** - complies with the current legislation (CE Certification), efficient use of power means reduced running costs
- **Three-year warranty**

#### Product Description

Ideal for sputter coating large specimens for SEM

The EMS300R T is suited for sputtering noble metals, such as gold (Au) and platinum (Pt) for the preparation of specimens for scanning electron microscopy (SEM).

#### Triple sputtering head

The EMS300R T is fitted with three individual sputtering heads to ensure even deposition of individual large specimens or multiple smaller specimens. Please note that it not possible to sequentially sputter three different sputtering metals from each sputtering head – for sequential coating please see EMS300T D dual head coater..

For economical coating of small specimens "single target" mode can be selected.



Triple sputtering head (closed)



Triple sputtering head (open)

# Sputter Coaters/SEM/TEM Carbon Coaters

## ● EMS300R T (continued)

### Molded case with color touch screen

The EMS300R T is presented in a custom molded, one-piece case, allowing easy servicing access. The color touch-screen allows multiple users to input and store coating 'recipes'. The case houses all the working components, with an automatic bleed control valve that ensures optimum vacuum conditions during sputtering.



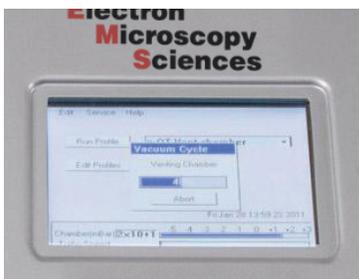
The vacuum chamber has an internal diameter of 283 mm/12" and comes with an integral implosion guard. The EMS300R T also includes 'vacuum shutdown', which enhances vacuum performance by allowing the chamber vacuum to be maintained when the system is not in use.

A variable speed rotary specimen stage is fitted as standard for 8"/203 mm and

6"/152 mm wafers, with other stages available as options, please see the Options and Accessories section below.

### Rapid data entry

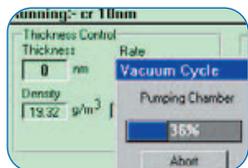
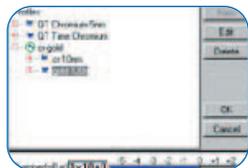
At the operational heart of the EMS300R T is a simple color touch screen, which allows even the most inexperienced or occasional operator to rapidly enter and store their own process data. To further aid ease of use, a number of typical sputtering and carbon coating profiles are already stored. For added convenience summaries of the last 100 coatings carried out can be viewed.



### Maintenance

The intuitive touch screen interface features maintenance prompts which highlight:

- Time of last clean
- Coating time since last cleaned
- System 'on time'
- Time of last service



### Additional Information

*Options and Accessories (including details the standard specimen stage)*

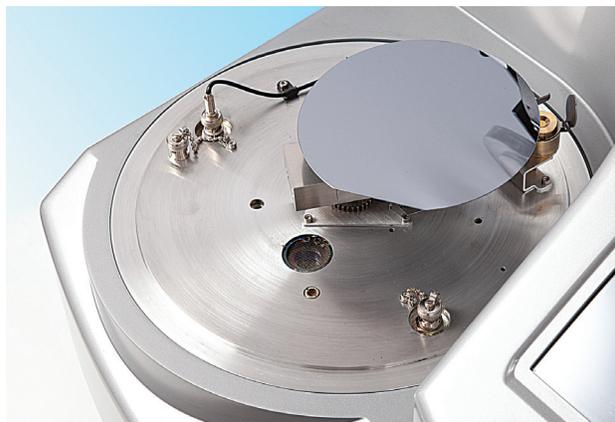
#### Specimen stages

The EMS300R T has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except for the rotary planetary stage). Rotation speed is variable between preset limits:

- Flat rotation stage for wafers - for 8"/203 mm and 6"/152 mm/wafers (fitted as standard)
- Rotation stage – 50 mm Ø. This stage only rotates - no tilt or height adjustment is possible
- Rotate-tilt stage – 50 mm Ø with height adjustment (target to stage height variable between 30-80 mm). Tilt angle can be pre-set (horizontal to 30 degrees)



- Rotation stage for glass microscope slides



EMS300R T standard specimen stage shown with a 4" wafer. Optional film thickness monitor (1152) fitted

### Other options

Extended height chamber – for taller specimens

Film thickness monitor (FTM) - this attachment consists of a controller and quartz crystal oscillator built into the EMS300R T and a vacuum feed-through, chamber-mounted crystal holder and quartz crystal. As sputtered or evaporated material is deposited onto the crystal, so its frequency of oscillation is modified. This 'modification' is used to measure and control the thickness of material deposited. Note: cannot be used when the coater is operated in "single target" mode.

continued >>>>

# Sputter Coaters/SEM/TEM Carbon Coaters

## ● EMS300R T Triple Target, Large Chamber, Rotary-Pumped Sputter Coater (continued)

### Specifications

Instrument case	585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
Weight	36.4kg
Packed dimensions	725mm W x 660mm D x 680mm H (44.8kg)
Work chamber	Borosilicate glass 283mm ID x 127mm H
Safety shield	Integral polyethylene terephthalate (PET) cylinder
Display	145mm x 320mm x 240mm colour graphic thin film transistor (TFT) display
User interface	Intuitive full graphical interface with touch screen buttons, includes fea- tures such as a log of the last 100 coatings carried out and reminders for when maintenance is due
Sputter targets	Disc style 57mm Ø with thick depending upon the targets fitted. Three 57 mm Ø x 0.1 mm thick gold (Au) targets are fitted as standard.
Vacuum	
Rotary pump	50L/m two-stage rotary pump with oil mist filter.
Typical ultimate vacuum	Vacuum measurement ..Pirani gauge ..... $2 \times 10^{-2}$ mbar in a clean system after pre-pumping with dry nitrogen gas
Specimen stage	Flat rotation stage for 200mm/8" and 150mm/6" wafers fitted as standard. Rotation speed is variable between preset options. For alternative stages see Options and Accessories

### Processes

Sputtering	0-80mA to a pre-determined thick- ness (with optional FTM) or by the built-in timer. The maximum sputter- ing time is 60 minutes (without 'breaking' vacuum and with built-in rest periods)
------------	------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

### Services and other information

Gases	Argon sputtering process gas, 99.999% Nitrogen venting gas (optional)
Electrical supply	90-250V 50/60Hz 1,400VA including RV3 rotary pump power. 110/240V voltage selectable
Conformity	CE conformity: Power factor correc- tion. Complies with the current legis- lation (CE Certification) and ensures efficient use of power, which means reduced running costs

### Ordering information

<b>EMS300R T</b>	Large-Chamber-Rotary-Pumped Sputter Coater, fitted with three sputtering heads to ensure even metal deposition. Include three 57mm Ø x 0.3mm gold (Au) sputter targets. A flat rotation stage for 8"/203 mm/ and 6"/152 mm wafers is fitted as standard	each
<b>91005</b>	Rotary pump requirements (needs to be ordered separately) Edwards RV5 90L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter	each

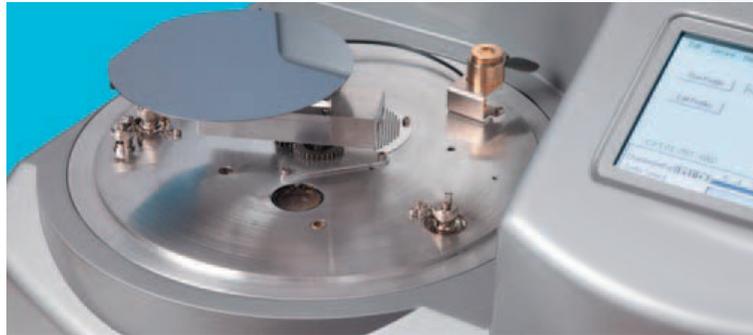
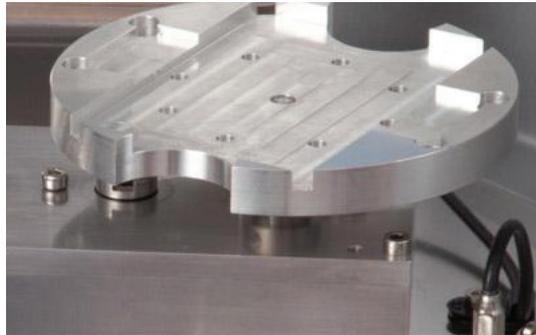
### Sputter targets

NB: The EMS300R T is fitted as standard with three 0.3mm thick gold (Au) targets. Other optional targets are available (three required):

<b>4523</b>	57mm Ø x 0.1mm Gold	each
<b>4524</b>	57mm Ø x 0.2mm Gold	each
<b>4525</b>	57mm Ø x 0.3mm Gold	each
<b>4526</b>	57mm Ø x 0.1mm Gold/Palladium (80/20%)	each
<b>4527</b>	57mm Ø x 0.2mm Gold/Palladium (80/20%)	each
<b>4528</b>	57mm Ø x 0.3mm Gold/Palladium (80/20%)	each
<b>4529</b>	57mm Ø x 0.1mm Platinum	each
<b>4530</b>	57mm Ø x 0.2mm Platinum	each
<b>4561</b>	57mm Ø x 0.3mm Platinum	each
<b>4532</b>	57mm Ø x 0.1mm Nickel	each
<b>4533</b>	57mm Ø x 0.1mm Silver	each
<b>4534</b>	57mm Ø x 0.1mm Palladium	each
<b>4535</b>	57mm Ø x 0.1mm Copper	each
<b>4536</b>	57mm Ø x 0.1mm Platinum/Palladium (80/20%)	each
<b>4537</b>	57mm Ø x 0.3mm Platinum/Palladium (80/20%)	each

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300R T (continued)



### Options and accessories

<b>6400</b>	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 6.5mm, 10 mm and 15 mm or 1/8" pin stubs. Stage rotation speed is variable between preset limits. No rotation is possible when in single target mode. Target to stage height is variable between 0 mm and 42 mm for the standard stage. When used with the extended height cylinder (optional accessory 10596) the target to stage height would be an additional 87 mm	each
<b>6401</b>	50 mm Ø variable height specimen stage with six stub positions for, 6.5 mm 10 mm and 15 mm disc stubs or 1/8" pin stubs. Stage rotation speed is variable between preset limits	each
<b>6402</b>	50 mm Ø rotary tilting stage. A rotary planetary style stage with variable tilt angle from horizontal to 30 degrees. The platform has six positions for either 6.5 mm 10 mm and 15 mm disc stubs or 1/8" pin stubs. Rotation speed is variable between preset limits. each Note: depending upon specimen height, this stage may require the optional extended height cylinder	each
<b>6403</b>	A 90mm Ø specimen stage for glass microscope slides (up to two x 75mm x 25mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pins stub. Stage rotation speed variable between preset limits. Includes a gear box to allow the optional FTM to be used	each
<b>6404</b>	Film thickness monitor (FTM) attachment. Including oscillator, feed-through, quartz crystal holder and one C5460 quartz crystal	each
<b>6405</b>	Spare quartz crystal	each
<b>6406</b>	Extended height vacuum chamber (214 mm in height - the standard chamber is 127 mm high). For increased source to specimen distance and for coating larger specimens	each
<b>6407</b>	Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the Q300R T when bench depth is limited	each
<b>6408</b>	A lockable emergency stop (e-stop) switch which can be mounted on top of the system in a position easily accessible for the operator. It is provided with a key to release the knob after activation. Note: the addition of the e-stop does not inhibit or replace the normal On/Off switch function. The e-stop can be retrofitted to existing systems	each
<b>6409</b>	Coating shields. Shields can be fitted to protect large surfaces from coating deposition, easily removable for ease of cleaning	each
<b>6410</b>	Two-year spares kit for EMS300R T. Includes: 3 x 57mm Ø x 0.3mm gold (Au) target, standard glass chamber assembly, O-rings etc.	each
<b>6411</b>	6" Wafer Specimen Stage: A flat adjustable stage capable of accepting 6" or 101.6 mm wafers	each
<b>6412</b>	4" Wafer Stage : A flat 4 1/2" 102mm flat, drop in wafer stage which accepts 2, 3, 4 " Wafers	each
<b>6413</b>	Additional Standard Glass Cylinder Assembly	each

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300TT Triple Target, Large Chamber, Turbo-Pumped Sputter Coater

#### Quick Overview

The EMS300T T is a large chamber, turbomolecular-pumped coating system ideally suited for sputtering a single large diameter specimen up to 8"/203 mm (for example a wafer) or smaller multiple specimens of a similar area.

The EMS300TT is fitted with three individual sputtering heads to ensure even deposition on a range of specimen types. The system is designed to sputter both oxidising metals e.g. chromium (Cr) and aluminium (Al) and non-oxidising (noble) metals such as gold (Au), gold/palladium (Au/Pd) and platinum (Pt). Chromium targets are fitted as standard.

If the requirement is for sputtering non-oxidising metals only then the EMS300R T large chamber, turbo-pumped sputter coater is the most suitable choice.



#### KEY FEATURES

- Large area sputter coating – up to 8"/203 mm diameter
- Triple sputtering head – ensures even coating deposition of large specimens
- Single target selection – for economic coating of small specimens for SEM
- Fine grain sputtering for advanced high resolution FE-SEM applications
- Coat logging – details of the last 100 coatings available on screen
- High vacuum turbo pumping - allows sputtering of a wide range of oxidising and non-oxidising metals suitable for SEM, high resolution FE-SEM and also for many thin film applications
- Precise thickness control using the film thickness monitor option
- Fully automatic touch-screen control – rapid data input, simple operation
- Multiple, customer-defined coating schedules can be stored – ideal for multi-user laboratories
- Automatic vacuum control – can be pre-programmed to suit the process and material; no needle valve to adjust
- Easy-to-change, drop-in style specimen stages (rotation stage supplied as standard)
- Vacuum shut-down option – leaves the process chamber under vacuum when not in use, giving improved vacuum performance
- Thick film option – up to 60 minutes sputtering time without breaking vacuum
- Ergonomic one-piece moulded case – enables easy maintenance and service access
- Ethernet with local FTP server connection – simple programmer updates
- Power factor correction – complies with the current legislation (CE Certification) – efficient use of power means reduced running costs
- Three-year warranty

#### Product Description

Ideal for sputter coating large specimens and thin film applications and SEM / FE-SEM

The EMS300T T is suited for sputtering a range of oxidising and non-oxidising (noble) metals for thin film applications and scanning electron microscopy (SEM). The range of target materials available is extensive and detailed in the Ordering Information section below.

#### High vacuum turbo molecular pumping

The EMS300T T is fitted with an internally mounted 70L/sec turbo-molecular pump, backed by a 50L/m two-stage rotary pump (which needs to be ordered separately). A Pirani vacuum measurement gauge (range: 1000 mbar to  $5 \times 10^{-4}$  mbar) is included, but a full range gauge (1000 mbar to  $5 \times 10^{-9}$  mbar) is available as an option.

Typical ultimate vacuum of around  $5 \times 10^{-5}$  mbar can be expected in a clean system after pre-pumping with dry nitrogen gas

# Sputter Coaters/SEM/TEM Carbon Coaters

## ● EMS300TT (continued)

### Triple sputtering head – for an even, consistent coating

The EMS300T T is fitted with three individual sputtering heads to ensure even deposition of individual large specimens or multiple specimens. Please note that it not possible to sequentially sputter three different sputtering metals from each sputtering head, for sequential coating of two metals then the EMS300T D is a more suitable option. For economical coating of small specimens such a SEM stubs then “single target” mode can be selected.



Triple sputtering head with automatic shutter open



Triple sputtering head with automatic shutter closed

### Moulded case with colour touch-screen

The EMS300T T is presented in a custom moulded, one-piece case allowing easy servicing access. The colour touch-screen enables multiple users to input and store coating ‘recipes’. The case houses all the working components and includes an automatic bleed control that ensures optimum vacuum conditions during sputtering.



The vacuum chamber has an internal diameter of 283 mm/12” and comes with an integral implosion guard. The vacuum shutdown option can enhance vacuum performance by allowing the chamber vacuum to be maintained when the system is not in use.

A variable speed rotary specimen stage is fitted as standard and accommodates

8”/203 mm and 6”/152 mm wafers. Other stages are available as options for this coater, please see the Options and Accessories section for more information.

### Rapid data entry

At the operational heart of the EMS300T T is a simple colour touch-screen, which allows even the most inexperienced or occasional operator to rapidly enter and store their own process data. To further aid ease of use, a number of typical sputter coating profiles are already stored. For added convenience summaries of the last 100 coatings carried out can be viewed.



### Maintenance

The intuitive touch-screen interface features a maintenance prompt facility, which highlights:

- Time of last clean
- Coating time since last cleaned
- System ‘on time’
- Time of last service.

### Additional Information

*Options and Accessories (including details of the standard specimen stage)*

### Specimen stages

The EMS300T T has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except the rotary planetary stage). Rotation speed is variable between preset limits.

- Flat rotation stage for wafers – for 8”/203 mm and 6”/152 mm wafers (fitted as standard).
- Rotation stage – 50 mm Ø. This stage only rotates - no tilt or height adjustment
- Rotate-tilt stage – 50 mm Ø with height adjustment (target to stage height variable between 30-80 mm). The tilt angle can be pre-set (horizontal to 30 degrees)
- Rotation stage for glass microscope slides



The standard specimen stage is designed to hold a 6” or 8” wafer. This image shows the non-standard 4” wafer stage and wafer and optional film thickness monitor (1152)

### Other options

- Extended height chamber – for taller specimens
- Film thickness monitor (FTM) – the optional FTM attachment consists of a controller and quartz crystal oscillator built into the EMS300TT, a vacuum feed-through, chamber-mounted crystal holder and quartz crystal. Note: the FTM cannot be used when the coater is operated in “single target” mode.

continued >>>>

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300TT Triple Target, Large Chamber, Turbo-Pumped Sputter Coater (continued)

#### Specifications

Instrument case .....	585 mm W x 470 mm D x 410 mm H (total height with coating head open -710 mm)
Weight .....	36.4kg
Packed dimensions .....	725 mm W x 660 mm D x 680 mm H (44.8kg)
Work chamber .....	Borosilicate glass 283 mm ID x 127 mm H
Safety shield .....	Integral polyethylene terephthalate (PET) cylinder
Display .....	145 mm x 320 mm x 240 mm colour graphic thin film transistor (TFT) display
User interface.....	Intuitive full graphical interface with touch-screen menus and buttons, includes features such as a log of the last 100 coatings carried out and reminders for when maintenance is due
Sputter target.....	Disc style 57 mm Ø with thickness depending upon the targets fitted. Three 57 mm Ø x 0.3 mm thick chromium (Cr) targets (TK8845) are fitted as standard.

#### Vacuum

High vacuum pumping .....	Internally-mounted, 70L/s air-cooled turbo-molecular pump
Rotary pump .....	50L/m two-stage rotary pump with oil mist filter.
Vacuum measurement .....	Pirani gauge

Typical ultimate vacuum.....	5x10 <sup>-5</sup> mbar in a clean system after pre-pumping with dry nitrogen gas
Specimen stage .....	A flat rotation stage for 8"/203 mm and 6"/152 mm wafers fitted as standard. Rotation speed is variable between preset limits. For alternative stages see Options and Accessories

#### Processes

Sputtering .....	0-80 mA to a pre-determined thickness (with optional FTM) or by the built-in timer. The maximum sputtering time is 60 minutes (without breaking vacuum and with built-in rest periods)
------------------	----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

#### Services and other information

Gases .....	Argon sputtering process gas, 99.999% Nitrogen venting gas (optional)
Electrical supply .....	90-250 V 50/60Hz 1,400 VA including an RV3 rotary pump, 110/240 V, voltage selectable
Conformity .....	Power factor correction. Complies with the current legislation (CE Certification) and ensures efficient use of power, resulting in reduced running costs

#### Ordering information

<b>EMS300 T</b>	Large-Chamber-Turbo-Pumped Sputter Coater, fitted with three sputtering heads to ensure even metal deposition. Fitted with three 57mm Ø x 0.3 mm chromium (Cr) sputtering targets as standard (TK8845). A flat rotation stage (10826) for 8"/203 mm and 6"/152 mm wafers is also supplied	each
-----------------	-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------	------

#### Rotary pump requirements (needs to be ordered separately)

<b>91003</b>	Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter	each
<b>6548</b>	XDS 5 Scroll Pump	each
<b>6550</b>	Diaphragm pump. A "dry" alternative to the standard 91003 oil-based rotary pump complete with vacuum hose, coupling kit and oil mist filter	each



Edwards RV3 Rotary Pump  
see page 66

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300TT (continued)

#### Options and accessories

##### Specimen Stages

6551	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 15 mm, 10 mm, 6.5 mm or 1/8" pin stubs. Stage rotation speed is variable between preset limits. No rotation when in single target mode. Target to stage height is variable between zero and 42 mm for the standard stage. When used with the extended height cylinder (optional accessory 10596) the target to stage height would be an additional 87 mm	each
6552	50 mm Ø variable height specimen stage with six stub positions for 15 mm, 10 mm, 6.5 mm disc stubs or 1/8" pin stubs. Stage rotation speed variable between preset limits	each
6553	50 mm Ø rotary tilting stage. A rotary planetary style stage with variable tilt angle from horizontal to 30 degrees. The platform has six positions for either, 6.5 mm, 10 mm, 15 mm disc stubs, or 1/8" pin stubs. Rotation speed is variable between preset limits. Note: depending upon specimen height, this stage may require the optional extended height cylinder	each
6554	A 90 mm Ø specimen stage for glass microscope slides (up to two 75 mm x 25 mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pins stub. Stage rotation speed is variable between preset limits. Includes gear box to allow optional FTM to be used.	each
6547	6" Wafer Specimen Stage: A flat adjustable stage capable of accepting 6" or 101.6 mm wafers	each
6549	A 4" 102mm flat drop-in-wafer stage which accepts 2", 3", and 4" wafers	each

##### Options and accessories:

6555	Film thickness monitor (FTM) attachment. Including oscillator, feed-through, quartz crystal holder and one quartz crystal	each
6556	Spare quartz crystal	each
6557	Extended height vacuum chamber (214 mm in height - the standard chamber is 127 mm high). For increased source to specimen distance and for coating large specimens	each
6558	A lockable emergency stop (e-stop) switch which can be mounted on top of the system in a position easily accessible for the operator. It is provided with a key to release the knob after activation. Note: the addition of the e-stop does not inhibit or replace the normal On/Off switch function. The e-stop can be retrofitted to existing systems	each
6559	Coating shields. Shields can be fitted to protect large surfaces from coating deposition - easily removable for ease of cleaning	each
6560	Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS300T T when bench depth is limited	each
6561	Full range, active vacuum gauge capable of measurement over the range of 1000 mbar to 5 x 10 <sup>-9</sup> mbar. Typical ultimate vacuum of system is 5 x 10 <sup>-5</sup> mbar. Note: gauge must be factory fitted	each
6562	Spares kit, including: spare standard glass cylinder, three chromium sputtering targets, vacuum tubing with coupling insert, argon gas tubing, three sputter head magnets, rotary pump oil mist filter and fuses	each

##### Sputter targets

NB: The EMS300T T is fitted as standard with three 0.3 mm chromium (Cr) targets.

Other optional targets are available (three required):

3410	57mm Ø x 0.1mm Gold	each	3424	57mm Ø x 1.5mm Titanium	each
3411	57mm Ø x 0.1mm Gold/Palladium (80/20)	each	3425	57mm Ø x 0.3mm Platinum/Palladium (80/20)	each
3412	57mm Ø x 0.1mm Platinum	each	3426	57mm Ø x 0.3mm Gold	each
3413	57mm Ø x 0.1mm Nickel	each	3427	57mm Ø x 0.3mm Gold/Palladium (80/20)	each
3414	57mm Ø x 0.1mm Silver	each	3428	57mm Ø x 0.3mm Platinum	each
3415	57mm Ø x 0.1mm Palladium	each	3429	57mm Ø x 0.5mm Titanium	each
3416	57mm Ø x 0.1mm Copper	each	3430	57mm Ø x 0.1mm Iron	each
3417	57mm Ø x 0.3mm Chromium	each	3431	57mm Ø x 0.3mm Iridium	each
3418	57mm Ø x 0.5mm Tungsten	each	3432	57mm Ø x 0.1mm Cobalt	each
3419	57mm Ø x 1.5mm Chromium	each	3433	57mm Ø x 0.1mm Tin	each
3420	57mm Ø x 0.2mm Tungsten	each	3434	57mm Ø x 0.1mm Molybdenum	each
3421	54mm Ø x 1.5mm Carbon	each	3435	57mm Ø x 0.3mm Magnesium	each
3422	57mm Ø x 0.1mm Aluminium	each	3436	57mm Ø x 0.1mm Tantalum	each
3423	57mm Ø x 0.1mm Platinum/Palladium (80/20)	each	3437	57mm Ø x 3mm Indium Tin Oxide (90/10)	each

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300TD Dual Target, Large Chamber, Turbo-Pumped Sputter Coater

#### Quick Overview

The EMS300T D is a fully automatic, free-standing sputter coater ideally suited for thin film applications and for conductive coating of scanning electron microscopy (SEM) specimens. At the operational heart of the EMS300T D is a large chamber fitted with two independent sputtering heads. This "dual-head" configuration allows two different metals to be sequentially sputtered without the need to "break" vacuum. A dual channel film thickness monitor option is available.

The system is designed to sputter a wide range of metals, both non-oxidising (noble) metals such as gold (Au), silver (Ag) and platinum (Pt) and oxidising metals such as chromium (Cr) and aluminium (Al).

The EMS300T D sputter coater has a 300 mm x 127 mm work chamber. The specimen stage accepts substrates of 2", 3" or 4" wafer sizes as standard and 6" wafers with an optional stage accessory. An optional extended height glass chamber is available to enable coating of larger specimens.



#### KEY FEATURES

- Fully automatic touch screen control - rapid data input, simple operation
- Dual sputter head – for sequential sputtering of two different metals
- Customer defined coating protocols
- Wide range of oxidising and non-oxidising targets available
- Fine grain sputtering for advanced high resolution FE-SEM applications
- Large chamber format
- Coat logging - details of the last 100 coatings available on screen
- Optional dual channel film thickness monitor (FTM) module
- High vacuum turbo pumping - allows sputtering of a wide range of oxidising and non-oxidising metals - suitable for SEM, high resolution FE-SEM and also for many thin film applications
- Automatic vacuum control - can be pre-programmed to suit the process and material - no needle valve to adjust
- Vacuum shut-down feature - leaves the process chamber under vacuum when not in use giving improved vacuum performance
- Thick film capabilities - up to 60 minutes sputtering time without breaking vacuum
- Ergonomic one-piece moulded case - easy maintenance and service access
- Ethernet with local FTP server connection - simple programme updates
- Power factor correction - complies with the current legislation (CE Certification) - efficient use of power means reduced running costs
- Three-year warranty

#### Product Description

Ideal for thin film applications or for sputter coating large specimens for SEM/FE-SEM

The EMS300T D is suited for sputtering a range of oxidising and non-oxidising (noble) metals for scanning electron microscopy (SEM) and thin film applications. The range of target materials available is extensive and detailed in the Ordering Information section below. The EMS300T D sputter coater comes with a chromium (Cr) and a gold (Au) target as standard.

#### High vacuum turbo molecular pumping

The EMS300T D is fitted with an internally mounted 70L/sec turbomolecular pump, backed by a 50L/m two-stage rotary pump (ordered separately). A Pirani vacuum measurement gauge (range: 1000 mbar to  $5 \times 10^{-4}$  mbar) is included, but a full range gauge (1000 mbar to  $5 \times 10^{-9}$  mbar) is available as an option. Typical ultimate vacuum of around  $5 \times 10^{-5}$  mbar can be expected in a clean system after pre-pumping with dry nitrogen gas.

#### Dual head sputtering – for sequential sputtering

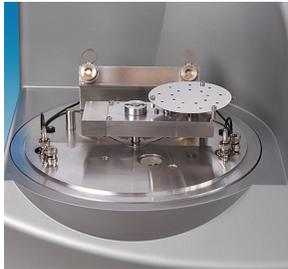
The EMS300T D is fitted with two independent sputtering heads to allow sequential sputtering of two different metals without the need to break vacuum. For example, a thin "seeding" layer of chromium followed by a deposition of gold. An automatic shutter mechanism is fitted to enable cleaning of oxidising sputter targets and to protect the second target and substrate during coatings. For single metal coating applications an individual target can be selected.

## Sputter Coaters/SEM/TEM Carbon Coaters

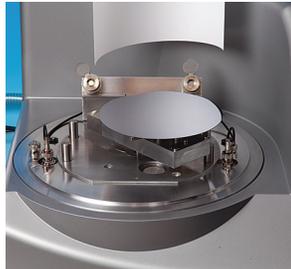
### ● EMS300TD (Continued)

#### Specimen stages

The EMS300T D is fitted with a flat rotating specimen stage capable of accepting wafers up to 4" (102 mm) in diameter. The rotation speed is variable between preset limits and the stage to sputtering head distance can be adjusted between 25 mm and 71 mm. A range of optional specimen stages can also be fitted, please see the Options and Accessories section.



Standard rotating specimen stage - for 4" wafers or individual SEM mounting stubs. Shown with optional dual-channel film thickness monitor



Standard rotating specimen stage with 4" wafer and optional dual-channel film thickness monitor

#### Moulded case with colour touch-screen

The EMS300T D is presented in a custom moulded, one-piece case, allowing for easy servicing and access. The colour touch screen enables user control of the coater functions (on various administrative levels) and multiple users to input and store coating 'recipes'. The case houses all the working components and includes an automatic bleed control that ensures optimum vacuum conditions during sputtering. The vacuum chamber has an internal diameter of 283 mm/12" and comes with an integral implosion guard. The EMS300T D can also be fitted with the optional facility for vacuum shutdown, which enhances vacuum performance by allowing the chamber vacuum to be maintained when the system is not in use.



#### Rapid data entry

At the operational heart of the EMS300T D is a simple colour touch screen, which allows even the most inexperienced or occasional operator to rapidly enter and store their own process data. To further aid ease of use, a number of typical sputter coating profiles are already stored. For added convenience summaries of the last 100 coatings carried out can be viewed on the touch screen.

#### Maintenance

The intuitive touch screen interface features maintenance prompts which highlight:

- Time of last clean
- Coating time since last cleaned
- System 'on time'
- Time of last service

#### Additional Information

*Options and Accessories (including details the standard specimen stage)*

#### Specimen stages

The EMS300T D has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage). Rotation speed is variable between 14 and 38 rpm:

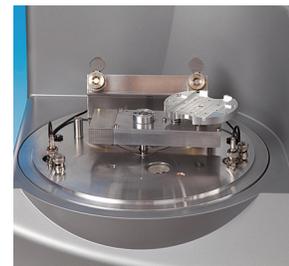
- Flat rotation stage for 4" (102 mm) wafers - fitted as standard
- Flat rotation stage for 6" (152 mm) wafers
- Rotation stage - 50 mm Ø. This stage only rotates - no tilt or height adjustment
- Rotate-tilt stage - 50 mm Ø with height adjustment (target to stage height variable between 30-80 mm). The tilt angle can be pre-set (horizontal to 30 degrees)
- Rotation stage for glass microscope slides

#### Other options

- Extended height chamber – for taller specimens
- Dual channel film thickness monitor (FTM) - the optional FTM attachment consists of a controller and quartz crystal oscillator built into the EMS300T D and a vacuum feed-through, two chamber-mounted crystal holders and quartz crystals. As sputtered material is deposited onto the crystal its frequency of oscillation is modified, this 'modification' is used to measure and control the thickness of material deposited



Rotary planetary stage and dual-channel film thickness monitor - both EMS300T D options



Optional specimen stage for glass microscope slides and dual-channel film thickness monitor option

continued >>>>

## Sputter Coaters/SEM/TEM Carbon Coaters

### ● EMS300TD Dual Target, Large Chamber, Turbo-Pumped Sputter Coater (continued)

#### Specifications

Instrument case .....	585 mm W x 470 mm D x 410 mm H (total height with coating head open-710 mm)
Weight .....	36.6 kg
Packed dimensions .....	725 mm W x 660 mm D x 680 mm H (44.8kg)
Work chamber .....	Borosilicate glass 283 mm ID x 127 mm H
Safety shield .....	Integral polyethylene terephthalate (PET) cylinder.
Display .....	145 mm x 320 mm x 240 mm colour graphic thin film transistor (TFT) display
User interface.....	Intuitive full graphical interface with touch-screen buttons, includes features such as a log of the last 100 coatings carried out and reminders for when maintenance is due
Sputter target.....	Disc style 57 mm Ø targets are fitted as standard, one 57 mm Ø x 0.3 mm thick chromium (Cr) target and one 57 mm Ø x 0.1 mm thick gold (Au) target , other targets are available

#### Vacuum

High vacuum pumping .....	Internally-mounted, 70L/s air-cooled turbomolecular pump
Rotary pump .....	50L/m two-stage rotary pump with oil mist filter.
Vacuum measurement .....	Pirani gauge (a full range gauge is available as an option)

Typical ultimate vacuum.....	5x10 <sup>-5</sup> mbar in a clean system after pre-pumping with dry nitrogen gas
Specimen stage .....	Flat rotation stage for 4" (102 mm) wafers is fitted as standard. Rotation speed is variable between preset limits. For alternative stages see Options and Accessories.

#### Processes

Sputtering .....	0-80 mA to a pre-determined thickness (with optional FTM) or by the built-in timer. The maximum sputtering time is 60 minutes (without 'breaking' vacuum and with built-in rest periods).
------------------	-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

#### Services and other information

Gases .....	Argon sputtering process gas, 99.999% Nitrogen venting gas (optional).
Electrical supply .....	90-250 V 50/60Hz 1,400 VA including RV3 rotary pump, power. 110/240 V voltage selectable.
Conformity .....	Power factor correction. Complies with the current legislation (CE Certification) and ensures efficient use of power, resulting in reduced running costs

### Ordering information

<b>EMS300T D</b>	Dual-Head, Large-Chamber-Turbo-Pumped Sputter Coater, supplied with 1 x 57 mm Ø x 0.3 mm chromium (Cr) and 1 x 57 mm Ø x 0.1 mm thick gold (Au) target. A flat rotation stage for 6" (152 mm) wafers is included.	each
------------------	-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------	------

#### Rotary pump requirements (needs to be ordered separately)

<b>91003</b>	Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter.	each
<b>6550</b>	Diaphragm pump. A "dry" alternative to the standard oil-based rotary pump. Complete with vacuum hose, coupling kit and oil mist filter.	each

#### Options and accessories

##### Specimen Stages:

<b>6790</b>	Swinging arm stage drive, a stage drive and positioning mechanism which positions the stage under the correct target. Also provides rotation drive to the stage. Rotation Speed Max 38 rpm Min 14 rpm	each
<b>6800</b>	Rotating specimen stage for 6" (152 mm) wafers, with rotation variable between preset limits.	each
<b>6801</b>	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 15 mm, 10 mm, 6.5 mm or 1/8" pin stubs. The stage rotation speed is variable between preset limits. The target to stage height is variable between 0 mm and 42 mm for the standard stage. When used with the extended height cylinder the target to stage height would be an additional 87 mm.	each
<b>6802</b>	50 mm Ø variable height specimen stage with six stub positions for 15 mm, 10 mm, 6.5 mm disc stubs or 1/8" pin stubs. Stage rotation speed variable between preset limits. Note: Target to stage height is variable between 10 mm and 53 mm for the standard stage. The stage is supplied with two mounting pillars; one provides 10 mm to 32 mm target to stage distance and the other 31 mm to 53 mm. An adjustable stop is used to set the height. When used with the extended height cylinder (optional accessory) the target to stage height would be an additional 87 mm.	each
<b>6803</b>	50 mm Ø rotary tilting stage. A rotary planetary style stage with a variable tilt angle from horizontal to 30 degrees. The platform has six positions for either 6.5 mm, 10 mm and 15 mm disc stubs or 1/8" pin stubs. Rotation speed is variable between preset limits. Note: depending upon specimen height, this stage may require the optional extended height cylinder.	each

## Sputter Coaters/SEM/TEM Carbon Coaters

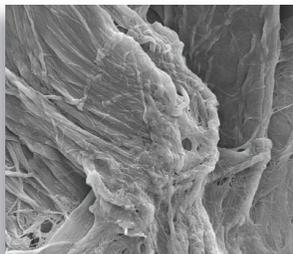
### ● EMS300TD (Continued)

<b>6804</b>	A 90 mm Ø specimen stage for glass microscope slides (up to two 75 mm x 25 mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pin stubs. The stage rotation speed is variable between preset limits. A gear box is included to allow the optional FTM to be used.	each
<b>6805</b>	Dual channel film thickness monitor (FTM). A fully integrated system using the Q300T D touch screen display for the control and display of all FTM functions. The FTM allows for the automatic termination of the metal sputtering process at a pre-selected thickness value. The rate for the sputtering processes is displayed in nm/min, with a resolution of 0.1 nm. Two FTM crystal holders are fixed in the chamber to give optimal position for both targets and to coat one material per crystal. Operating crystal frequency is in the 5 MHz to 400 kHz operating range. Includes two spare quartz crystals.	each
<b>6806</b>	Spare quartz crystal.	each
<b>6807</b>	Extended height vacuum chamber (214 mm in height, the standard chamber is 127 mm high). Ideal for increased source to specimen distance and for coating of larger specimens.	each
<b>6808</b>	Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS300T D when bench depth is limited.	each
<b>6809</b>	A lockable emergency stop (e-stop) switch which can be mounted on top of the system in a position easily accessible for the operator. It is provided with a key to release the knob after activation. Note: the addition of the e-stop does not inhibit or replace the normal On/Off switch function. The e-stop can be retrofitted to existing systems.	each
<b>6810</b>	Full range, active vacuum gauge capable of measurement over the range of 1000 mbar to $5 \times 10^{-9}$ mbar. Typical ultimate vacuum of the EMS300T D is $5 \times 10^{-6}$ mbar. Note: this must be factory fitted.	each
<b>6811</b>	Coating shields. Can be fitted to protect large surfaces from coating deposition and can be easily removable for cleaning.	
<b>6812</b>	Spares kit, including: spare standard glass cylinder, one chromium (Cr) and one (Au) sputtering target, vacuum tubing with coupling insert, argon gas tubing, two sputter head magnets, rotary pump oil mist filter, FTM quartz crystal and fuses.	each

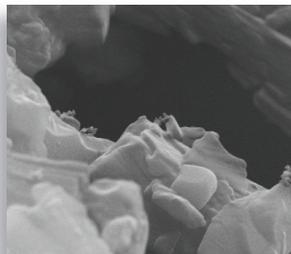
### Sputter targets

NB: The EMS300T D is fitted with a 57 mm diameter 0.3 mm thick chromium (Cr) target and a 57 mm diameter 0.1 mm thick gold (Au) target as standard. Other optional targets are available:

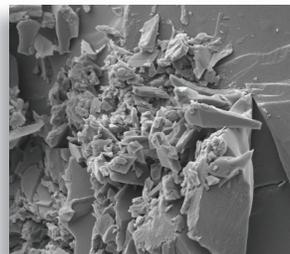
<b>3410</b>	57mm Ø x 0.1mm Gold	each	<b>3424</b>	57mm Ø x 1.5mm Titanium	each
<b>3411</b>	57mm Ø x 0.1mm Gold/Palladium (80/20)	each	<b>3425</b>	57mm Ø x 0.3mm Platinum/Palladium (80/20)	each
<b>3412</b>	57mm Ø x 0.1mm Platinum	each	<b>3426</b>	57mm Ø x 0.3mm Gold	each
<b>3413</b>	57mm Ø x 0.1mm Nickel	each	<b>3427</b>	57mm Ø x 0.3mm Gold/Palladium (80/20)	each
<b>3414</b>	57mm Ø x 0.1mm Silver	each	<b>3428</b>	57mm Ø x 0.3mm Platinum	each
<b>3415</b>	57mm Ø x 0.1mm Palladium	each	<b>3429</b>	57mm Ø x 0.5mm Titanium	each
<b>3416</b>	57mm Ø x 0.1mm Copper	each	<b>3430</b>	57mm Ø x 0.1mm Iron	each
<b>3417</b>	57mm Ø x 0.3mm Chromium	each	<b>3431</b>	57mm Ø x 0.3mm Iridium	each
<b>3418</b>	57mm Ø x 0.5mm Tungsten	each	<b>3432</b>	57mm Ø x 0.1mm Cobalt	each
<b>3419</b>	57mm Ø x 1.5mm Chromium	each	<b>3433</b>	57mm Ø x 0.1mm Tin	each
<b>3420</b>	57mm Ø x 0.2mm Tungsten	each	<b>3434</b>	57mm Ø x 0.1mm Molybdenum	each
<b>3421</b>	54mm Ø x 1.5mm Carbon	each	<b>3435</b>	57mm Ø x 0.3mm Magnesium	each
<b>3422</b>	57mm Ø x 0.1mm Aluminium	each	<b>3436</b>	57mm Ø x 0.1mm Tantalum	each
<b>3423</b>	57mm Ø x 0.1mm Platinum/Palladium (80/20)	each	<b>3437</b>	57mm Ø x 3mm Indium Tin Oxide (90/10)	each



10nm Au on Filter Paper



10nm Au on Salbutamol



10nm Au on Sugar

## Vacuum Evaporators

### ● EMS 975 Large Chamber Turbo Evaporator & EMS 975S for Semiconductor Wafer Coating

The EMS 975 Turbo Evaporator is a multiple application system to enable a range of preparation techniques to be applied with the flexibility and module expansion capability to develop new methods and prepare new specimens.

#### Quick Overview

##### EMS 975

The EMS 975 is a compact, bench-mounted, multiple application thermal evaporator for vacuum deposition of thin layers of carbon and metals. It is ideal for a wide range of techniques, including the production of carbon support films and replicas for TEM and carbon/metal thin film applications. The EMS 975 is available with a wide range of optional add-ons, including low-angle shadowing and sequential layer coating using dual-source evaporation (an additional metal evaporation source is required).

The system is fitted with an 80mm diameter flat rotation specimen stage, but this can be exchanged for optional stages and holders to meet differing user requirements. The EMS 975 vacuum system is under fully automatic or manual control and uses a 100L/s turbomolecular pump to ensure rapid pump down and clean vacuum conditions.

##### EMS 975S Carbon Evaporator for 8" wafers

The EMS 975S is similar to the EMS 975 but designed to coat an 8" wafer or similarly large specimen with carbon (C). The carbon rod evaporation source is directly mounted to the vacuum chamber top plate, allowing easy access to the carbon gun and giving the optimal source-to-specimen distance required for large diameter specimens. Unlike the EMS 975, the EMS 975S is not fitted with a metal evaporation source or associated base plate mounting pillars, and also has a larger specimen access door.



#### FEATURES

- Turbomolecular Pump.
- Automatic pumping sequence.
- Clean vacuum.
- Variable outgas control.
- Evaporation pulse button.
- Unique "anti stick" carbon rod gun evaporation assembly (patent pending).
- Rack out drawer sample loading system.
- Rotating plate specimen table with external accurate tilt control (option).
- Large sample capacity, 150mm (6") using drawer with up 200mm (8") for top loading.
- Sample holders range of options, including grid holders, stub holders.
- Carbon and Carbon/Platinum evaporation.
- Full range vacuum measuring system.
- Fast pumping cycle.
- Protective Polycarbonate Implosion Shield.
- Selectable evaporation supplies giving x 4 evaporation settings.
- Restricted or full vent control to avoid disturbances of samples.
- Microcontroller with LCD displays of status and customer data entry for control of systems.
- Modular electronics.
- Bench mounted unit.
- Sputter coating (option) for range of metal targets.
- Film thickness measurement for carbon and metal depositions.

#### Product Description

##### Coating sources

The EMS 975 is fitted with a carbon rod gun and metal filament/boat source which can also be used for cleaning TEM and SEM apertures. An optional sputtering source is available.

##### Work chamber

The borosilicate glass work chamber is 250mm diameter x 300mm and mounted on an aluminium support collar. A tough chamber implosion guard is included as standard. The chamber can accommodate specimens up to 8"/200mm in diameter. A unique rack-out specimen loading system gives the user easy specimen access and the hinged lid assembly makes other areas of the vacuum chamber readily accessible.

##### Menu-driven control

The menu-driven microcontroller allows the user access to a range of options, but readily 'defaults' to optimum operating conditions, allowing both fully-automatic and manual override as required.

##### Turbomolecular pumping and venting

The EMS 975 uses a modern 100L/s turbomolecular pump backed up by an external rotary vacuum pump (not included, see 91005) with the complete pumping sequence being under fully-automatic control.

The vacuum pump-down sequence is automatically controlled by the system microprocessor. Vacuum measurement is by a combined pirani/penning gauge and is displayed digitally.

Process gases (nitrogen for venting - if fitted - and argon for the optional EMS 350 sputtering attachment) are automatically controlled and can be programmed for use during coating sequences. The vent valve has an adjustable restrictor and programmable vent time to prevent disturbing specimens due to the inrush of gas at the end of the cycle.

A very useful feature of the EMS 975 is 'vacuum shut-down', which allows the process chamber to remain under vacuum when not in use. This helps to maintain a high level of system cleanliness and vacuum performance.



Typical chamber set up, showing carbon and metal evaporation sources, specimen stage and optional film thickness monitor (FTM)

# Vacuum Evaporators

## ● EMS 975 (Continued)

### Specimen stages

The EMS 975 is fitted with an 80mm flat stage as standard, but this may be exchanged for optional holders, such as a 3mm grid holder, low-angle shadowing attachment and a rotary planetary stage (see Options and Accessories). Specimen holders are supplied with a bayonet fixing for quick exchange.



EMS 975 specimen stage and optional film thickness monitor (FTM)



EMS 975 specimen drawer with stage in specimen exchange position

The rotary stage is mounted on a sliding access port on the side of the chamber. This allows the user to exchange specimens quickly without having to remove the glass chamber and disturb any coating set-up. The standard flat stage may be tilted. For rotary shadowing techniques, the standard stage can be tilted from 0° to +/- 180°.

### Chamber base plate and evaporation power supplies

The EMS 975 is fitted with a 0-100A evaporation power supply with base plate terminals for carbon rod evaporation (14V/100A), evaporation from a metal filament (15V/35A), carbon string evaporation (25V/35A) and a terminal rated at 5V/35A for TEM and SEM aperture cleaning using a molybdenum (Mo) boat. A wide range of add-on options is available - see Options and Accessories.



Close up of EMS 975 carbon evaporation source. The 'anti-stick' design ensures smooth movement of carbon rods during evaporation



EMS 975 metal evaporation source fitted. NB: Can also be used to evaporate carbon fiber string

### EMS 975S Thermal Evaporator

The EMS 975S is based on the EMS 975, but a special load lock door allows the loading of wafers up to 8"/200mm. Please contact us for more information.

## SPECIFICATIONS

<b>Instrument Case</b> .....	450mm(W) x 500mm(D) x 300mm(H)
<b>Work Chamber</b> .....	Borosilicate Glass 250mm(Dia.) x 300mm(H) with hinged top plate (can accommodate samples to 200mm) (8") in Dia.
<b>Polycarbonate Implosion Guard</b> .....	(To suite work chamber readily) Removable for maintenance
<b>Weight</b> .....	.65kg
<b>Carbon Gun</b> .....	Adjustable height with tilt control 0-20° uses 6.15mm Carbon rods
<b>Turbomolecular Pump</b> .....	..100L/ Sec as standard, optional sizes available
<b>Vacuum Gauges</b> .....	Active Gauge Head Atm to 1 x 10 <sup>-7</sup> mbar
<b>Working Vacuum</b> .....	Within 15 minutes
<b>Operating Vacuum</b> .....	Into x 10 <sup>-5</sup> mbar range (liquid nitrogen trap option)
<b>Evaporation Supply</b> .....	4 x selectable, low voltage (pulsed or controlled) variable volts a.c. V, 5V, 15V, 25V volts @ 25 amps. 7V @ 100 amps
<b>Specimen Table</b> .....	With tilt facility 1-45°
<b>Rotating Specimen Table Option</b> .....	.60mm (Dia) tilting 0-45°, with variable speed control 15 rpm to 45 rpm Sputtering Option
<b>Deposition Rate</b> .....	0-100mA
<b>Standard Target</b> .....	0-50nm/minute gold (Au)
<b>Timer (HT Safety ) Interlocked</b> .....	.57mm Au (options Pt, Pt/Pd, Pd, Cr, Ni, Cu, Ag) 0-4 minutes
<b>Services</b> .....	Argon (for sputtering option) – Nominal 4 psi
<b>Vacuum Pump</b> .....	No. 2 pump (2m <sup>3</sup> /hr) Pumping speed oil mist filter vacuum hose (included with instrument)
<b>Supply</b> .....	115V 60Hz (16 Amp Max incl pump) 230V 50Hz (8 Amp Max incl pump)

## Ordering Information

<b>91090</b>	EMS 975 Large Chamber Turbo Evaporator complete with carbon source	each
<b>91090-S</b>	EMS 975S Turbo Evaporator for Semiconductor Wafer Coating	each
<b>91005</b>	Rotary Vacuum Pump	each

## Replacement Source

<b>91077</b>	Carbon Rods (6.15mm Dia.)	each
--------------	---------------------------	------

## Replacement Parts

<b>91033</b>	Glass Cylinder	each
<b>91034</b>	"L" Gaskets	each

## Optional Accessories

<b>91004</b>	EMS 105 Aperture Cleaning Head	each
<b>92060</b>	EMS 190 Low Angle Shadowing	each
<b>91082</b>	EMS 350 Sputter Coater Attachment	each



6.15mm diameter rods (left)  
Carbon rod shaper supplied with the EMS 975 (above)

# RF Plasma Etchers/Plasma Reactors

## What is... The Plasma Process?

The Plasma process is accomplished through the use of a low pressure, RF induced gaseous discharge. The material or specimen is loaded into the reaction chamber. The chamber is evacuated to a vacuum pressure of 0.1-0.2 torr by a mechanical vacuum pump. A carrier gas is introduced into the chamber, raising the chamber pressure to 0.3-1.2 torr, depending on the application.

RF Power is applied around the chamber (13.56 MHz). This excites the carrier gas molecules and dissociates it into chemically active atoms and molecules. The mechanism employed in this process is one of ionization. The combustion products, which are completely dissociated and harmless are carried away in the gas stream. The unique property of this process is that it occurs near low temperatures without employing toxic chemicals.

## Applications

Asbestos and man-made mineral fiber (MMMF) detection

Coal ashing

Detection of metals in blood

Ashing of biological material, food stuffs etc.

Organic and inorganic composites

Surface treatment of plastics

Plasma polymerization

Artificial weathering

Plasma etching and plasma ashing of organic specimens for SEM and TEM examination

## Techniques and Applications Plasma Etching and Ashing

### What is a Plasma

A plasma is a partially-ionized gas consisting of equal numbers of positive and negative charges and a different number of unionized neutral molecules. When a gas is subjected to a DC or radio frequency (RF) potential at reduced pressure this is usually accompanied by glow, which is known as glow discharge. The words glow discharge and plasma tend to be used synonymously, although glow discharges are not perfect plasmas - but for the purposes of this text they will not be differentiated. The characteristic glow of these plasmas is due to electronically excited species producing optical emission in the ultraviolet or visible regions of the spectrum and is characteristic of the composition of the glow discharge gas. For example, argon gives a bright blue color and air or nitrogen gives a pink colour that is due to excited nitrogen molecules.

### Ionization

In the context of plasma-enhanced chemistry reactors, the plasma is created in a vacuum chamber, which contains a constant flow of a gas at reduced pressure - typically in the order of 1mbar. This gas is exposed to a radio frequency (RF) potential, which results in the partial ionization of the gas. In the ionization process, a bound electron in an atom is ejected from that atom. For example, the ionization of an argon atom is expressed as follows:  $- Ar \rightarrow Ar^+ + e$

### Excitation

A less dramatic transfer of energy allows the electron to jump to a higher energy level within the atom. This process is known as excitation. The excited state of an atom is conventionally shown by an asterisk:  $e + Ar \rightarrow Ar^* + e$

### Dissociation

A further process that can occur is the dissociation of a molecule. If oxygen, for example, is the gas subjected to the RF potential, the oxygen molecule can be dissociated into two oxygen atoms, whereas a monatomic gas such as argon cannot be dissociated at all:  $e + O_2 \rightarrow e + O + O$

A normal result of dissociation is an enhancement of chemical reactivity, since the products are usually more reactive than the parent molecule.

Dissociation may or may not be accompanied by ionization, for example:  $e + CF_4 \rightarrow e + CF_3 + F$  (Dissociation) or  $e + CF_4 \rightarrow 2e + CF_3 + F$  (Dissociation)

### Summary

Exposing a gas to the RF potential at reduced pressure creates a plasma which contains active species - for example, in the case of oxygen,

atomic oxygen. Oxygen atoms will oxidize organic molecules more readily than oxygen molecules. So typically a cellulose material can be converted to carbon dioxide, carbon monoxide and water at room temperature, rather than at elevated temperatures (eg burning) and furthermore the oxidation is more controllable.

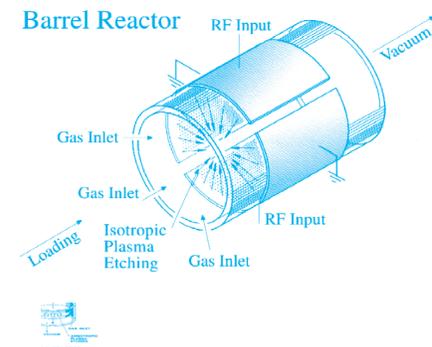
### Types of Reactor Systems

There are many types of reactors available. They are all glow discharge systems but vary considerably in terms of excitation frequency (5kHz - 5GHz), operating pressure (1mbar - atmospheric pressure) and electrode arrangement.

In addition to barrel systems there are parallel plate reactors; these usually consist of a grounded plate onto which the specimens are placed and an insulated parallel plate to which the RF power is applied. The reverse of this arrangement where the specimens are placed on the non-grounded electrode is generally known as 'Reactive ion etching' (RIE).

Etching in this type of reactor is inherently directional, whereas the former can be both directional (anisotropic) or isotropic. The barrel reactor usually etches isotropically and is favored for most plasma applications.

The barrel reactor, as the name implies, is a cylindrical container, which can be evacuated. The RF power, usually at 13.56MHz frequency is applied to the system via internal or external electrodes by capacitive or inductive coupling. This type of reactor is used for the plasma ashing process and also for the plasma etching process, although the disadvantage in the latter for some users is that the process is not completely isotonic so that undercutting can occur.



## RF Plasma Etchers/Plasma Reactors

### Techniques and Applications Plasma Applications

#### Plasma ashing

The process of plasma ashing, plasma stripping or micro-incineration is usually restricted to the total removal of organic matter by an oxygen plasma; the products being carbon oxides and water vapor, which are volatile and pumped away by the vacuum system. Historically, the first application was for the removal of photoresist in the microelectronics industry. Photoresist is composed of organic compounds, essentially consisting of carbon, plus hydrogen and oxygen. Exposure to an oxygen plasma eventually removes all the photoresist as volatiles leaving no residues, unless there are inorganic contaminants in the photoresist. The shows process is therefore totally dry and is also a means of concentrating inorganic contaminants in organic materials.

#### Asbestos and man-made mineral fiber (MMMF) detection

This is a major application for RF plasma systems. The exact protocols vary from country to country, but in the UK the following is a summary of the recommended method. The technique requires that a specimen of filtered air (or water) is collected from a known volume on MF series filter (mixed cellulose acetate and nitrate). The filter is then transferred to a microscope slide, treated with a clearing solution, dried and then transferred to the plasma unit. The asbestos fibers are then exposed by partial ashing of the filter by treatment in an oxygen plasma for typically seven minutes. This removes surface layers of the filter, leaving the asbestos fibres exposed but still attached to the collapsed filter matrix. The fibers can be counted and identified by light microscopy, light contrast microscopy, SEM and EDX. Some SEM and EDX protocols require complete ashing of the filter.

#### Coal ashing

Small specimens can be ground and distributed in a petri dish to produce maximum surface area. The coal dust can be ashed by an oxygen (or air) plasma at low temperatures, compared to the extremely high temperatures generally used in a muffle furnace for this process. Volatile elements, such as selenium, are retained and therefore a more accurate calorific and ash value can be produced. Problems in estimating the required values are a result of the structure of coal which includes organic materials (and hence convertible material) together with inert inorganic materials in the same overall matrix.

Plasma chemistry is a surface reaction, so

methods such as the ashing of coal require the exposure of new surfaces. For this reason physical stirring of the specimen is recommended every 1-2 hours. The complete ashing of a 1g specimen is typically completed in 12-24 hours.

#### Detection of metals in blood

Plasma ashing as a pre-treatment for atomic absorption analysis (AAS) is another well-established application. In this case one is normally looking for metals such as lead, cadmium, zinc and mercury in trace quantities in organic materials such as vegetables, dairy products or animal tissue. A specific example involves the treatment of multiple specimens of human blood exposed to a  $CF_4/O_2$  plasma. The organic materials in this application can be removed in 15 minutes, leaving only the metallic contaminants to be analyzed for cadmium.

#### Ashing of biological material, food stuffs etc

Plasma ashing has also been successfully used to ash materials as varied as post-mortem lung tissue (for asbestos), bread (to determine type and distribution of iron) and specimens of prepared food (for asbestos and man made mineral fibers). Specimens need to be dried prior to ashing and their size kept to a minimum.

#### Organic and inorganic composites

Similar problems are encountered in composite materials such as paints, vehicle tires and brake linings, contaminated oils and the application of clays onto paper. In paints, the organic binder can be removed to leave the inorganic pigment in its original distribution. In paper, the clay platelet distribution and adhesion can be investigated after ashing of the paper and binder. Similarly, epoxy composite materials can be investigated.

#### Plasma etching

Plasma ashing and plasma etching rely on the same basic principles. Plasma Etching is usually confined to the semiconductor industry, and more often than not, uses carbon tetrafluoride (with oxygen) as the plasma process gas. Probably the most frequent application is the etching of silicon, silicon oxides, and silicon nitrate, as well as glass passivation layers.

Failure analysis of integrated circuits is also an important application of plasma processing. Oxygen gas is used to remove epoxy encapsulates.  $CF_4/O_2$  is also used to remove glass filters in the encapsulants and so uncover

devices which have failed. Inspection by methods, such as SEM, is then possible.

When etching, it should always be remembered that not only the required surface will be removed. Careful choice of gas is made so that preferential etching of the required surface is attained.

Plasma etching is a chemical process. The RF discharge generates species which then react with the material being etched to form a volatile product. The resulting products are swept away by the gas flow. Since reactive species are being formed, the reactant gas is chosen to give the highest concentration of the etching species. For example,  $CF_4$  and  $CF_4/O_2$  mixtures produce very reactive fluorine and  $CF_3$  radicals and ions. Similarly, other gases and volatile compounds have been investigated and used to etch a wide variety of materials. These include  $CCl_4$ ,  $CFCl_3$ ,  $C_2Cl_6$ ,  $C_2F_6$ ,  $SF_6$ ,  $SiF_4$ , and mixtures of these gases with  $H_2$ ,  $O_2$ , Ar, He,  $CO_2$ , CO,  $N_2$  etc.

#### Surface treatment of plastics

A number of applications of plasma involve the surface treatment of plastic materials, prior to a subsequent process. An example is the treatment of reinforcing fibres that are to be integrated into an epoxy structure. Treatment in an oxygen plasma for say, five minutes at 50-100W, increases surface roughness. These pitted fibers enhance adhesion and a good mechanical bond is produced with enhanced rigidity and strength.

Plasma processing of plastics can also convert a hydrophobic surface to a hydrophilic surface. This type of treatment usually requires short exposure (3-5 minutes) at low power (50 watts). This sort of reaction has been applied to the assembly of ink pens to improve the speed of ink filling and transfer. Other examples include the treatment of electrical wiring so that the insulation can be printed upon with regular inks. Plasma treatment of car bumpers allows simpler and more cost-effective painting schedules and the treatment of textile fibers can improve water retention.

Plasma surface treatment in biomedical applications is expanding rapidly. For example, surface modifications of a polymer to improve blood compatibility. This involves tailoring the polymer surface to minimize blood reaction. Similarly, the internal surfaces of tubing can be modified, allowing pharmaceutical materials to be chemically bonded to the surfaces, thus allowing the drug to be slowly dispensed in a localized area.

*continues on next page*

## RF Plasma Etchers/Plasma Reactors

### Frequently Asked Questions...

#### What is the difference between RF plasma etching or ashing and glow discharge?

Glow discharge is an 'imperfect plasma' used to alter surface energies to turn hydrophobic (water hating) surfaces hydrophilic (water loving). Glow discharge will also remove adsorbed gases from the chambers of vacuum systems and in doing so improve pump down speed and ultimate vacuum levels.

This is the limit of what glow discharge can do - it will not plasma 'etch' or 'ash' specimens. RF plasma reactors can alter surface energies, de-gas and remove materials from specimens in a controlled way (etching or ashing).

#### What is the difference between etching and ashing?

Ashing is the total removal of organic matter using oxygen plasma. Mineral components of the specimen will be left behind as a residue (ash). The by-products of this process - mainly carbon oxides and water vapor - are pumped away by the vacuum pump. Etching is the controlled removal of layers or part layers of material and is usually confined to semiconductor applications.

#### How automated is the EMS 1050?

After initial set up of the operating conditions operation is fully automatic (one-button operation).

#### When must a fomblin vacuum pump be specified?

When oxygen is to be used as a process gas it has the potential to combine explosively with normal organic types of rotary pump oils. Fomblin is a synthetic oil and will not react in this way. Fomblin is also resistant to most of the highly corrosive plasma process gases.

#### What is the capacitance manometer and when should it be specified as an option?

The capacitance manometer measures vacuum. The standard plasma system uses a conventional pirani gauge for this purpose. However, if corrosive halogenated gases are used on a regular basis, they have the potential to attack (erode) the pirani vacuum gauge filament. In such cases (mainly in semiconductor conductor applications) the capacitance manometer option is strongly recommended because it is resistant to attack by corrosive gases.

## Techniques and Applications Plasma Applications (continued)

### Plasma polymerisation

Plasma polymerization refers to the polymerisation of active species generated in a plasma. For example, the introduction of polysiloxanes onto hard contact lenses improves the hydrophilic nature of the surface. An application in the soft drinks industry using CF<sub>4</sub>, to create a fluorinated surface on PET and polypropylene bottles, making bottles less pervious to carbon dioxide.

A porous surface can be produced on medical equipment, this makes it possible to sterilise the equipment with nitrous oxide while remaining impervious to air. Deposition of polymers onto the surfaces of implants is also possible and can help prevent rejection by improved bio-compatibility.

### References

An important application of plasma technique is to improve the 'wettability' and adhesion of polymers for surface coatings, inks and dyes <sup>(1)</sup>. Self-adhesion can also be markedly improved by plasma treatment <sup>(2,3)</sup>.

Plasma techniques are widely used in the electronics industry <sup>(4)</sup> particularly for

microelectronics fabrication. Although most of the materials involved in these applications are inorganic, they are of interest to polymer chemists because polymers can be used as resists, insulators or semiconductors. Operations carried out by plasma techniques include photoresist removal <sup>(5)</sup>, etching silicon compounds <sup>(6)</sup> and deposition of polymer films <sup>(7,8)</sup>. The chief virtue of plasma techniques in microelectronics fabrication is that it permits automated, multi-step processing of complex devices <sup>(9)</sup>.

Applications for plasma polymerization have included the production of protective coatings for metals and other reactive surfaces <sup>(12)</sup>, fabrication of reverse-osmosis membranes <sup>(13)</sup>, coatings for optical plastics <sup>(14)</sup> and the formation of radiation resistant coatings <sup>(15)</sup>.

Last and by no means least, is the application of plasma ashing for the analysis of inorganic materials within an organic matrix. Prime examples include the investigation of asbestos fibers in air and investigation of metal contamination of food.

1. M. Hudis, Techniques and applications of plasma chemistry, Wiley-Interscience, New York, 1974 chap. 3.
2. M. Stradi and D. A. L. Goring, Can, J. Chem. Eng., 53, 427 (1975)
3. D. K. Owens, J. Appl. Polym. Sci., 19, 265, (1975)
4. R. W. Kirk, as (1) chap. 9.
5. S. M. Irving, Kodak Photoresist Seminar, 2, 26, (1968).
6. H. Abe, Y. Sonobe, and T. Enomoto, Jpn. J. Appl. Phys., 12, 154 (1973).
7. P. J. Ozowa, IEEE Trans, Parts Mater., Pachag., PMP-5, 112 (1969)
8. A. Chida, Jpn. Kokai Tokkyo Koho, 78, 47, 393, (1978).
9. R. L. Bersin, Kodak Microelectronics seminar proc., San Diego, Calif., October 1-3 (1978).
10. P. Kassenbeck, Bull. Inst. Text, FR. 18, 7 (1963).
11. W. J. Tjhorssen, Text. Res J 38,644, (1968).
12. T. Williams J, Oil Colour Chem Assoc., 48,936 (1965).
13. H. Yasuda, Appl., Polym. Symp., 22,241 (1979).
- 14.1. R. M. Kubacki, US Patent 4096315 (1978).
- 14.2. Y. Masuda and Y. Nakagawa JPN, Kokai Tokkyo Koho 78, 56, 177, (1978).
15. M. Miyamura, JPN, Kokai Tokkyo Koho, 78, 120, 527 (1878).

# RF Plasma Etchers/Plasma Reactors

## Techniques and Applications

### Plasma chemistry

#### Photo resist removal

The removal of photo resist by plasma etching from silicon or GaAs semiconductor slices is a very safe and effective process. The thickness of resist is normally 1-3 microns and has usually been baked on for about 1 hour at 100°C. Using O<sub>2</sub> at full power, 1 micron should disappear in about 20-30 minutes. Ion implanted resist is very hard and in some cases will take longer to etch, up to two hours. One way of reducing time is by fitting a larger rotary pump, therefore allowing more gas flow for the same pressure. Normal operating pressure is between 0.4-0.2mbar.

#### Silicon etching

To etch silicon and silicon nitride from slices, a fluorinated gas is required, usually CF<sub>4</sub>. Films are normally in the order of 100-500 microns and are quickly removed; 2-5 minutes normally being sufficient. Visual colour changes make complete removal obvious. CF<sub>4</sub> will also attack the chamber and window. Quartz, which does not contain boron, is offered as standard instead of glass to avoid boron contamination.

#### Asbestos detection

Plasma is now accepted as the standard process for SEM examination of asbestos fibers caught in

acetate filter papers. Filters are surface etched in O<sub>2</sub> at approximately 100W for 2-8 minutes, this being sufficient to etch away the filter surface, leaving the asbestos fibres for SEM examination.

#### Atomic absorption spectroscopy preparation

Plasma is widely used in AAS as a convenient way of removing the organic binder from a host of specimens making trace metal determination easier. Specimens are presented dry and crushed and etched in O<sub>2</sub>. Ashing times vary but are usually 1-3 hours.

#### General cleaning

Cleaning of delicate parts, such as SEM gun assemblies or chamber parts, can be achieved using plasma. For super-clean surfaces 99.999% ultra pure argon or high purity oxygen is used.

#### Plastic treatment

There are numerous applications being tried on plastic surfaces using plasma, the most common being the requirement to convert hydrophobic surfaces to be hydrophilic (surface 'wetter'). The advantages being the ease of printing, painting, marking etc and great improvement in the bond strength of adhesives.

These reactions take place in high purity O<sub>2</sub> at relatively low temperatures and power input (10-40W). Process times are relatively short; 15 seconds – 2 minutes usually being sufficient. Interest has been shown in putting down 'barrier layers' on plastic containers to increase shelf life by stopping the ingress of CO<sub>2</sub> and stopping the contents of the container reacting with the plastic. The films are normally of inert fluorine and are produced using CF<sub>4</sub>.

Oil Generally in plasma environments the rotary pumps are required to pump large amounts of oxygen. As most rotary pump oils are organic based, there is a very remote chance of explosion. There are two ways to minimise this risk:

(a) Use a synthetic based oil - Fomblin is highly recommended.

(b) It is possible to break into the pipe between the plasma unit and pump and dilute the oxygen with argon. However, this reduces the pumping speed of the pump, requires an argon supply which can be costly and would be ineffective if the argon supply failed.

### EXAMPLES OF PLASMA GASES AND THEIR APPLICATIONS

#### O<sub>2</sub> 100%

Asbestos and man-made mineral fiber detection  
Stripping photo resist  
Removal of organic contamination  
Removal of organic material (eg coal)  
Ageing paint (quick test for likely ageing characteristics)  
Degreasing of metals and polymers  
Hydrophilation  
General oxidation  
Polymer activation

#### Ar 100 %

Degreasing and activation of metals  
Removal of epoxy bleed-out from hybrid circuits without oxidation.  
Can be used with up to 3% O<sub>2</sub> for faster reaction  
Cleaning EM parts  
Oxide removal  
Hydrophilation

#### H<sub>2</sub> 100%

Used in with carrier gas in levels of amp<10%  
Metals cleaning (without oxidation)  
Hydrophobation  
Oxide removal

#### He 100%

Degreasing and activation of metals and polymers  
Hydrophilation  
Cooling agent for O<sub>2</sub>

#### N<sub>2</sub> 100%

Polymer activation  
Removal of epoxy bleed-out on hybrid circuits  
Removal of oxides

#### C<sub>2</sub>H<sub>4</sub> 100%

Polymerization

#### CH<sub>4</sub> 100%

Polymerization

#### C<sub>2</sub>H<sub>2</sub> 100%

Polymerization

#### CF<sub>4</sub> 100%

Epilamization  
Silicon etchant

#### SF<sub>6</sub> 100%

Silicon etchant

#### FS-100 97% He 3% O<sub>2</sub>

Removal of thin film organic contamination from easily oxidised metals and synthetics  
Low temperature removal of organics from metals without oxidation  
Low temperature ashing

#### FORM-ING GAS

90-95% N 5-10% H  
Removing oxides; especially useful as a follow up process in hybrid cleaning or other oxidising processes as glass to metal seals

#### DS28 N<sub>2</sub> with 2ppm water

Removal of organics from sensitive substrates without oxidation

#### DS180 92% O<sub>2</sub> 8% CF<sub>4</sub>

Removal of thick layers of photo resist

#### DS300 97% O<sub>2</sub> 3% CF<sub>4</sub>

Photo resist removal in aluminium chambers or with faraday insert in quartz chambers  
Removal of organic contamination

#### DS16281 99% N<sub>2</sub> 1% O<sub>2</sub>

Removes photo resists films over oxidising or with TCR and resistor networks being unchanged (thin films only). May also have increased O<sub>2</sub> as designated by the last digit signifying the percentage

#### IR101 70% Ethylene Trichloride 30% 1, 1, 2 Trichlorotrifluoroethane

Removal of inorganic contamination, particularly tin from resist or contaminated chambers (used in conjunction with O<sub>2</sub>). Will also remove window oxide grown on exposed Si

#### FS100 97% He 3% O<sub>2</sub>

Low temperature organic removal  
Flash strip of photo masks

## RF Plasma Etchers/Plasma Reactors

### Techniques and Applications

#### Plasma chemistry applied to electron microscopy (EM) preparation procedures

A selection of published work covering a range of applications using plasma chemistry as a preparation technique.

##### Replica EM studies of polyamide structure

Use of oxygen plasma for differential etching of ordered and disordered regions in organic specimens. Rates of etching reduced from lower molecular weight substances through disordered (amorphous) regions to ordered (crystalline) regions. This allowed identification by replica EM of simultaneous presence of single crystals and spherulites in Polyamide 68.

##### Replica EM studies of latexes of acrylic copolymers

Using replica EM and oxygen ashing it was shown that latex particles of Polyalkylacrylates and Alkylacrylate-Methacrylic Acid Copolymers are aggregates of primary globules, the size of the globules depends upon the polymer.

##### EM studies of polyethylene tetraphthalates films and fibers

Oxygen ashing in conjunction with replica EM revealed supra molecular structure with correlation between EM and x-ray diffraction data. Oxygen ashing followed by SEM examination allowed identification of three types of internal flaw in bright fibers. Results showed high concentrations of titanium dioxide in regions containing voids, and highly ordered polymers which had previously been assumed to be defusants.

##### Quantitative bulk analysis by TEAM of biological microspecimens

100µm sections of Wistar rat hearts were oxygen plasma ashed and then dissolved and sprayed onto grids. The droplets were then individually analyzed. The method was found to retain volatiles such as sulphur and possibly chlorine. Ashing times when compared to high temperature ashing are considerably reduced.

##### Microincineration for EM localization of biological materials

A review of high temperature ashing and plasma ashing of various materials.

##### Review of techniques for SEM and electron probe microanalysis

Among many applications the following are highlighted: microelectronic failure analysis; grain boundary composition in mineralogical specimens containing silicates and carbonaceous material; discovery of microvoids and flaws in carbon reinforcing fibers; differential etching of polymers; formation of 3D ash skeletons; studies of modular graphite inclusions in cast iron; mineral staining of brain tissue followed by oxygen ashing.

##### Low temperature ultra microincineration of thin-sectioned tissue

Plasma incineration used to determine the morphological localization of

structure bound mineral and metallic elements within biological cells at TEM levels.

##### Ultrastructure of cell organelles in thick plasma-etched sections

1µm sections of fixed and embedded kidney tissue when surface etched by oxygen plasma, allowed etch resistant cell components to be imaged with clarity. Resolution was better than other preparation technique for SEM of internal structures of cells and organelles in bulk specimens of tissue.

##### TEM-EDS of silica in cell walls of rye grass

A comparison of preparation methods, including plasma ashing, to determine amounts of silicon in cell wall material.

##### Plasma ashing moths and insects prior to EM and XES

Oxygen ashing of insects allowed the removal of organic material but left the structure intact. This allowed area sectioning for subsequent analysis.

##### X-ray microanalysis of Epon sections after oxygen plasma ashing

Improved X-ray detectability of elements retained in ash by lowered background counts. Method removes osmium fixative and chlorine to reveal hidden phosphorous peak; pattern fidelity allows micro analytical resolution of 0.1µm.

##### Measuring the concentration of asbestos fibers in air specimens

Oxygen plasma used to remove high levels of airborne organic contaminants and to remove filter paper prior to TEM sizing.

##### SEM of embedded biological specimens that have been surface plasma etched

As a general technique for SEM, oxygen plasma etching thick sections of a wide variety of different types of embedded tissue yields specimens that show a resolution that is considerably better than that obtainable by most other methods; particularly for viewing internal structure of cells and organelles in bulk structure.

##### Identification second counting of asbestos fibers on membrane filters

Methods are described whereby asbestos fibers can be counted by phase contrast microscopy and identified on the same membrane filter by optical and SEM techniques. Airborne concentration of different asbestos types in mixed clouds can therefore be estimated.

#### References:

Improved gas-discharge etching techniques in the Electron Microscope studies of Polyamide structures. L I Bezruk. *Vysokomol. Soyed.* A10: No. 6 1434-1437, 1968

Particle and film structures of films of some latexes of acrylic copolymers  
V I Yeliseyeva. *Vosokomol. Soyed* A9: No 11 2478-2481, 1967

Oxygen etching method of making an Electron Microscopy study of Polyethylene Tetraphthalate films  
K Z Gumargalivva. *Vosokomol. Soyed.* 8: No. 10 1742-1744, 1966

Studies of plasma-etched Polyethylene Terephthalate fibres by SEM and energy-dispersive X Ray microanalysis  
P R Blakey & M O Alfy. *Letter to Journal of Textile Institute* 1978 No 1

Detection of inorganic materials in biological specimens  
Source unknown

Microincineration techniques for electron-microscopic localization of biological minerals  
Richard S Thomas. *W Region Research Lab, Agricultural Research Service, US Dept. of Ag. Albany Ca. USA*

Use of chemically reactive gas plasma in preparing specimens for SEM and Electron Probe Microanalysis  
Richard S Thomas. *SEM/1974 part I proc 7th SEM Symp* -April/1974

Low temperature ultra-microincineration of thin section tissue  
Wayne Hohman & Harold Shraer. *Journal of Cell Biology, Volume 55* 1972 pp 328-354

Ultra-microincineration of thin-sectioned tissue  
*Principles and Techniques of EM-1976*

Ultrastructure of cell organelles by Scanning Electron Microscopy of thick sections surface-etched by an Oxygen plasma  
W J Humphreys. *Journal of Microscopy* Vol 116 July 1979.

Silica in the mesophyll cell walls of Italian Rye Grass  
D Dinsdale *Ann. Bot* 44 73-77 1979

Ashing moths and various insects  
J Bowden (pr comm) *Rothampstead Research Station.* July 1979

X-ray microanalysis of Epon sections after Oxygen plasma microincineration  
Tudor Barnard and R S Thomas *Journal of Microscopy* Vol 113 Pt 3 Aug 1978. pp269-276

Scanning Electron Microscopy of biological specimens surface-etched by an Oxygen plasma  
W J Humphreys. *Scanning Electron Microscopy* 1979/11. Asbestos counting method using TEM. Ontario Research Foundation.

In situ identification of Asbestos fibres collected on membrane filters for counting  
N P Vaugham and S J Rooker. *Ann. Occup. Hyg.* Vol 24 No 3 pp281-290 1981.

## RF Plasma Etchers/Plasma Reactors

### Techniques and Applications

#### Plasma chemistry applied to chemical analysis routines

A selection of published work applying plasma chemistry as a preparative technique to a range of chemical analysis procedures.

##### Enhanced cell culture techniques

Using argon in a plasma barrel reactor, it is possible to treat glass or polymer dishes to achieve at least double the normal cell plating efficiency. Treatment involves 3 minutes in an argon plasma which also ensures sterilization of the containers.

##### Quantitative gravimetric determination of silicon in organo silicons

A 5 minute exposure to an oxygen plasma of 3-5mg of various materials including dyes, polymer films, plant oils, conserving agents and food concentrates allowed subsequent gravimetric determination of silicon in the form of silicon dioxide.

##### Quantitative titrimetric determination of mercury in organics

A method whereby mercury is converted into mercury oxide in an oxygen plasma and, the mercury content then evaluated by titrating the dissolved ash residue with potassium iodide. The procedure takes only 15 minutes for one mercury determination.

##### Polarography and voltammetry for surveillance of toxic metals

Various advanced methods are compared for the analysis of trace levels of cadmium, lead and copper in blood and marine specimens using oxygen ashing as a pre-treatment for biometrics. Low temperature ashing gave greatly improved accuracy and reduced contamination levels when compared to acid digestion and high temperature furnacing.

##### Low temperature ashing of bituminous coal

The plasma ashing method is compared to the standard muffle furnace (700°C) method and concludes that it removed organic matter leaving relatively unaltered mineral residues. A number of elements are preserved which are volatilized in the muffle furnace.

##### Quantitative determination of mineral content of coal

Relates a standardized plasma ashing procedure to air oxidation method (370°C) and concludes that the plasma method has advantages in applied hours and elapsed time for processing specimens. Applicable to a wide range of coals, including anthracite and high pyrite coals.

##### Infrared spectrometry of minerals in coal

Discusses a quantitative technique as applied to a range of coals and

synthetic specimens after plasma ashing and concludes that minerals are preserved relatively unaltered.

##### Recovery of radioactive tracers from various organic substances

A wide variety of organic materials including muscle tissue, fat, faecal matter, ion exchange resin, cellulose filters, activated charcoal and a rat were ashed to establish optimum ashing conditions. Whole blood was treated with radioactive tracers to establish recovery in relation to muffle furnacing.

##### AAS of tin, iron, lead and chromium in biological materials

An investigation of trace metals in various canned foodstuffs using oxygen/fluorine reagent gases for ashing. The procedure showed good recoveries of standards and also establish very real improvements in reduced man hours, reduced chemical cost, reduced hazards and lower contamination levels.

##### Cadmium in blood analysis using AAS

A method of the rapid preparation of whole blood prior to AAS. Specimen holders made from PTFE, blood then spread in holder, dried at 100°C for 2 minutes and ashed for 15 minutes in 80/20 oxygen/carbon tetrafluoride. Ash dissolved in nitric acid and transferred to AAS specimen cup for direct aspiration into furnace.

##### Recovery of tracers from ashed blood

A listing of recoveries for 15 elements relating oxygen plasma ashing to muffle furnacing at 700°C. The method shows vast improvements in recovery percentages with only two exceptions, gold and silver, which are assumed to have catalytic reactions with oxygen.

##### Destruction of organics prior to AAS of cadmium in blood

A method was established for the rapid destruction of organic material after an investigation into various mixtures of carbon tetrafluoride and oxygen as the reaction gas in a plasma. The method involves ashing blood in delves cups for 12.5 minutes in a 1+1 mixture of the two gases. After ashing the cups were transferred to an AAS for analysis of cadmium; the method provides a rapid and accurate analysis.

#### References:

##### Glow discharge surface treatment for improved cellular adhesion

L Smith, Polymer Chem Div, 170th ACS meeting, Chicago 25.8.75.

##### Quantitative determination of Silicon in Organo Silicon compounds using low temperature RF-discharge Oxygen plasma

M Velodina, Analytical Letters 10 (14), 1189-1194 (1977).

##### Quantitative determination of Mercury in organic materials by means of a low temperature, high frequency discharge plasma in Oxygen

M Velodina, Zhurnal

##### Polarography and voltammetry in environmental research and surveillance of toxic metals

H W Nurnberg, International Symposium on Industrial Electro-Chemistry, Madras December 1976.

##### Trace chemistry of toxic metals in biometrics

P Valenta, Z Anal Chem. Band 285 (1977)

##### Studies on ecotoxicological base lines and speciation of heavy metals in natural waters and rain

H W Nurnberg, Institute of Chemistry, Nuclear Research Centre (KFA), PO Box 1913, D-5170, Julich, F.R.G.

##### Electronic low-temperature ashing of bituminous coal

H J Gluskoter, Illinois State Geological Survey, Urbana, Ill USA. March 1965.

##### Quantitative determination of the mineral matter content of coal by a radio frequency-oxidation technique

Frank W Frazer. Fuel 1973.

##### Quantitative infrared multicomponent determination of minerals occurring in coal

P A Estep, Anal. Chem. Vol 34. No 11. Oct 1968 Pp1454-1457

##### Use of electrically excited Oxygen for the low temperature decomposition of organic substrate

C F Gleit. Anal. Chem Vol 34. No 11. Oct 1968 pp 1454-1457

##### Low temperature Oxygen-Fluorine RF ashing of biological materials in PTFE dishes prior to determination of Tin, Iron, Lead and Chromium by AAS

E V Williams. Analyst. Sept 1982. Vol 107. pp1006-1013

##### Cadmium in blood analysis

Francois Claeys, Institute d'Hygiene and d'Epidemologie Bruxelles.

##### Recovery of tracers from ashed blood

Dr Piduttu, Catholic University, Rome, Dept of Industrial Hygiene.

##### Determination of Cadmium in blood after destruction of organic material by low-temperature ashing

G Carter and W Yeoman. Analyst. Vol 105. March 1980 Pp 295- 297

## RF Plasma Etchers/Plasma Reactors

### ● EMS 1050 Plasma Asher

The EMS 1050 consists of a solid state RF Generator and associated tuning circuits, a vacuum system with a solenoid controlled valve, a constant feed gas supply system, and a reaction chamber system which includes two semicircular electrodes and two piece pyrex chamber. The unit has one gas control as standard.

The solid state RF Generator is a solid state crystal controlled oscillator designed to provide up to 150 watts of continuous wave 13.56 MHz power to the reaction chamber. Maximum power transfer from the power supply to the reaction chamber is accomplished by matching the output impedance of the amplifier to the input impedance of the reaction chamber.

The gas supply system consists of the gas delivery system inside the reaction chamber. This delivery system is a glass tube sealed on the inner end and perforated along its bottom surface. Connections to the delivery tube are fastened with special clips to prevent the possible leakage of contaminants into the chamber.

The EMS 1050 is often used in Asbestos Specimen Preparation as a Low Temperature Ashing Technique.

#### FEATURES

- Automatic tuning of RF power.
- Built-in rotary vacuum pump.
- Barrel chamber with isotropic etching.
- Low temperature plasma ashing, etching, and cleaning. (0-150 watts RF)
- Vacuum monitoring.
- Dual flow gauge gas control.
- Accurate process timer.
- Needle valve vent control.
- Micro controller, with default settings programmable by the operator.
- Indication of settings by LCD display of status/entry.
- Indication of conditions during cycle, vacuum, power, time.
- Location bay for backing pump filled with special "oil".
- Sample carrier for convenient loading.
- Rack-out drawer loading door for ease of sample access.
- Polycarbonate safety shield.

#### Product Description

Built to withstand heavy use - 24 hours a day for some plasma ashing schedules - the EMS 1050 features microprocessor control with automatic operation and offers durability and simplicity of operation. Barrel systems plasma etch or plasma ash isotropically (in all directions) and are suitable for the majority of applications.

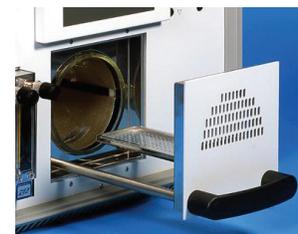
The EMS 1050 uses a low pressure, RF-induced gaseous discharge to modify specimen surfaces or remove specimen material in a gentle, controlled way. A significant advantage over alternative methods is that the plasma etching and ashing processes are dry (no wet chemicals needed) and take place at relatively low temperatures.

A wide range of surface modification methods are available, using a variety of process gases. Using oxygen (or air) as the process gas, the molecules disassociate into chemically-active atoms and molecules and the resulting 'combustion' products are conveniently carried away in the gas stream by the vacuum system.

#### Chamber, specimen handling and gas control

The EMS 1050 has a 110mm diameter x 160mm borosilicate glass chamber horizontally mounted with a slide-out specimen drawer and viewing window. Evacuation of the chamber is achieved by an optional 50L/m mechanical rotary vacuum pump. Ingress of reactive gases is controlled by two built-in flow-meters backed by solenoid valves.

NB: For applications where borosilicate glass needs to be avoided, the EMS 1050 can be fitted with a quartz chamber.



"Rack Out" Specimen Stage

### Application Example

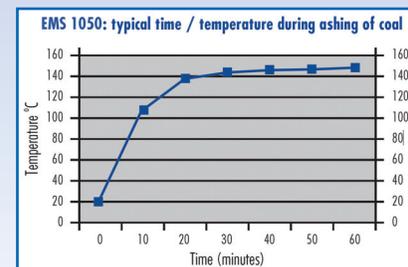
#### Low temperature plasma ashing of coal...

The EMS1050 can be used to remove the organic content from coal, leaving a residue of mineral and volatile components for subsequent analysis. The advantage of low temperature RF plasma ashing over other methods, such as heating in a muffle furnace (typically at 700°C), is that many more of the volatile components are retained.

In the following experiment oxygen gas was used with a forward power setting of 100W.

A thermocouple was introduced into the chamber via a vacuum feed through in the rear of the EMS1050 process chamber. The thermocouple was fixed with high temperature resistant tape to the base of a glass Petri dish and covered with approximately 5g of coal granules of approximately 1-2mm<sup>3</sup> in size, covering the thermocouple tip to a depth of 1.5mm.

After one hour it was apparent that the temperature had reached a maximum 150°C.



# RF Plasma Etchers/Plasma Reactors

## ● EMS 1050 (Continued)

### Power, tuning and vacuum monitoring

RF power of up to 100W at 13.56MHz is available and can be infinitely controlled and pre-set to required values. Automatic tuning of forward and reflected power is standard. Forward power and vacuum levels are indicated by the digital display.

### Automated microprocessor control

The EMS 1050 is fully automatic. Control parameters for time, power and vacuum are easy to preset and can be monitored and adjusted throughout the process run.

'Autotuning' of RF power for optimum control and reproducibility

During the plasma process the 'autotune' facility ensures that the RF power is automatically impedance-matched to any variation in the system or loading. This means conditions in the chamber are maintained at their optimum - important as it gives faster reaction times, greater reproducibility of results and protects the power supply during the RF cycle.

### Pumping options

A working system requires only the addition of a specified rotary pump. A fomblinised rotary pump (91005-F) is strongly recommended for safety reasons when applications involve the use of oxygen as a process gas. Where oil-based rotary pumps need to be avoided, we offer dry pumping options (see Specifications).



EMS 1050 during operation



EMS 1050 during operation - close up

### Specifications

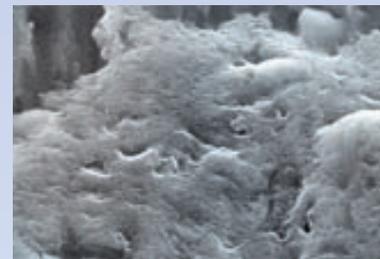
<b>Instrument Case</b> .....	450mm(W) x 350mm(D) x 300mm(H)
<b>Barrel Work Chamber</b> .....	'Pyrex' 160mm(L) x 110mm(Dia.) (Borosilicate Glass as standard)
<b>Weight</b> .....	25Kg
<b>Plasma Output</b> .....	Solid State Power Supply: 0-150 watts continuously variable at 13.56 MHz with Tuning Control of forward and reflected power to optimize RF power transfer
<b>Vacuum Gauge</b> .....	ATM to $1 \times 10^{-5}$ mbar Full scale normal 0.5 mbar to 1.0 mbar
<b>Digital Timer Unit</b> .....	Displays elapsed time with range select: 0-99 min. 99 sec. 0-99 hours. Automatic termination of Ashing Process
<b>Dual Gas Flow Gauge</b> .....	Dual Needle Valve flow control selectable for 1,2 or both gases
<b>Supply</b> .....	115V 60Hz (6 Amp Max) 230V 50Hz (3 Amp Max)
<b>Services</b> .....	Process Gas at nominal 5 psi (0.33 bar)

CAUTION: For Oxygen or Corrosive Process Gases Vacuum Pump should use a Synthetic Oil 'Fomblin Oil', or similar.

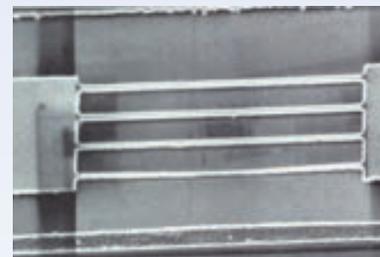
### Ordering Information

<b>93000</b>	EMS 1050 Plasma Asher	each
<b>91005-F</b>	Rotary Vacuum Pump (Fomblin)	each

## Application Example



These two SEM micrographs show before and after results comparing identical areas of a metal photo litho plate on which extraneous lines can be visualised. Treatment in a barrel plasma reactor, with oxygen as the process gas, removed the ink - which is essentially a carbon pigment in a binder - without disturbing anything that was present underneath. Subsequent SEM examination shows a scattering of particulate material made up of irregular platelets 0.2 to 2µm in diameter. X-ray microanalysis gave a spectrum characteristic of a clay mineral.



This SEM micrograph shows a set of free-standing single crystal silicon wires for studying thermal transport. The wires were fabricated in silicon-on-insulator material using electron beam lithography and CF4 plasma etching in a barrel reactor. The wires are 40µm long, 1µm wide and 0.5µm thick and are suspended above a silicon substrate. (Image courtesy of the Microelectronics Research Centre, Cavendish Laboratory, University of Cambridge).

## Critical Point Dryers

### What is... Critical Point Drying?

Critical Point Drying is so named as it includes, as part of its process, the occurrence known as the continuity of state for which there is no apparent difference between the liquid and gas state of a medium, the surface tension between this interface reducing to zero. This occurs at a specific temperature and pressure with resulting density, and is known as the Critical Point. This condition of zero surface tension can be used to dry Biological Specimens, avoiding the damaging effects of surface tension.

In biological specimens we are mainly concerned with the removal of water. Unfortunately, the critical point for water of +374°C and 3212 p.s.i. is inconvenient, and would cause heat damage to the specimen. The most common and convenient transitional medium for critical point drying is Carbon Dioxide (CO<sub>2</sub>), which has a critical point at 31°C and 1072 p.s.i. However, it is not miscible with water, and therefore, we have to involve a third medium, commonly Acetone, which is termed the intermediate fluid. We can now convert our transitional fluid, typically CO<sub>2</sub>, from liquid to gas without surface tension at the critical point.



#### Mature Spruce Wood

Critical point dried block of mature spruce wood block, demonstrating transverse, tangential and radial views of tracheids and vessels.

## Techniques and Applications

### A summary of the critical point drying method

Critical point drying is an established method of dehydrating biological tissue prior to examination in the Scanning Electron Microscope. The technique was first introduced commercially for SEM specimen preparation by Polaron Ltd in 1971. The original design concepts, which included a horizontal chamber, are still embodied in the design of the EMS 3000 and EMS 3100 CPD models.

In recent years we have introduced two further models: the EMS 850, which features built-in chamber cooling and heating, and the EMS 850WM, which is designed for drying a 100mm/4" silicon wafer.

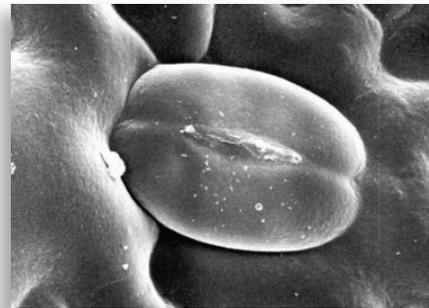
All three models have found general acceptance in many laboratories throughout the world. Together, these critical point dryers offer the user a choice most suited to the particular specimen preparation requirements.

The phase diagram shows the pressure to temperature ranges where solid, liquid and vapor exist. The boundaries between the phases meet at a point on the graph called the triple point. Along the boundary between the liquid and vapor phases it is possible to choose a particular temperature and corresponding pressure, where liquid and vapor can co-exist and hence have the same density. This is the critical temperature and pressure.

Critical point drying relies on this physical principle. The water in biological tissue is replaced with a suitable inert fluid whose critical temperature for a realizable pressure is just above ambient. The choice of fluids is severely limited and CO<sub>2</sub> is universally used today, despite early work with Freon 13 and nitrous oxide.

With CO<sub>2</sub> a critical point of approximately 35°C can be achieved at a pressure of around 1,200psi. Therefore if the water is replaced with liquid CO<sub>2</sub> and the temperature then raised to above the critical temperature, the liquid CO<sub>2</sub> changes to vapor without change of density and therefore without surface tension effects which distort morphology and ultra structure.

Since liquid CO<sub>2</sub> is not sufficiently miscible with water, it is necessary to use an intermediate fluid which is miscible with both water and liquid CO<sub>2</sub>. In practice intermediate fluids commonly used are methanol, ethanol, amyl acetate and acetone.



#### Stomatal Pore on Xerophyte Leaf Surface

Critical point dried epidermis of a xerophyte (cactus), demonstrating raised stomatal pores.



#### Barley Leaf

Trichomes and stomatal pores on the epidermal surface of a barley (*Hordeum vulgare*) leaf. Some very fine wax crystallites are also just visible on the surface of the leaf.

The advent of Scanning Electron Microscopy (SEM) in the study of surface morphology in biological applications made it imperative that the surface detail of a specimen was preserved. Air (evaporative) drying of specimens can cause severe deformation and collapse of structure - the primary cause of such damage being the effects of surface tension. The specimen is subject to considerable forces, which are present at the phase boundary as the liquid evaporates. The most common specimen medium, water, has a high surface tension to air; by comparison that for acetone is considerably lower. The surface tension could be reduced by substitution of a liquid with a lower surface tension with thereby reduced damage during air-drying. However, the occurrence of what is known as 'continuity of state' suggests a drying technique for which the surface tension can be reduced to zero. If the temperature of liquefied gas is increased the meniscus becomes flatter indicating a reduction in the surface tension. If the surface tension becomes very small the liquid surface becomes very unsteady and ultimately disappears.

## Critical Point Dryers

### Techniques and Applications

## Critical Point Drying Principles

When this 'critical point' is reached, it is possible to pass from liquid to gas without any abrupt change in state.

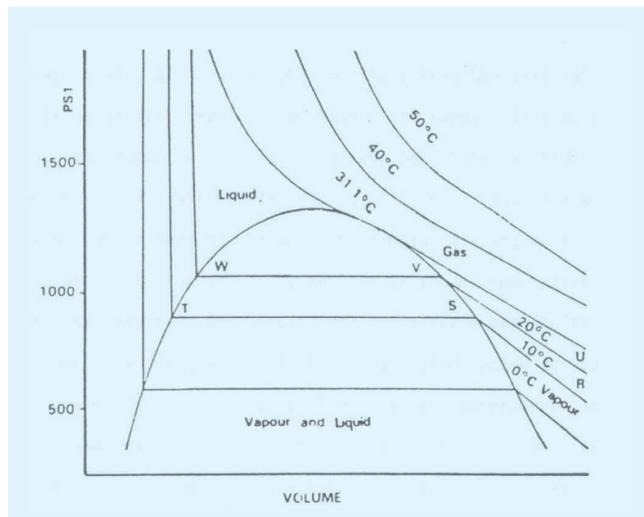
If a specimen had been in the liquid during this process it would have experienced a transition from a 'wet' to a 'dry' gas environment without being in contact with a surface, in this way avoiding the damaging effects of surface tension.

This is termed Critical Point Drying (CPD), the basis of which are the classic experiments carried out over 100 years ago during investigations on the liquification of gases.

### The Critical Phenomena

The principle of the experiments, which were initially carried out using carbon dioxide (CO<sub>2</sub>), was to measure the change in volume with the application of pressure, of a fixed mass of gas, while maintaining a constant temperature. This was repeated for a range of different temperatures.

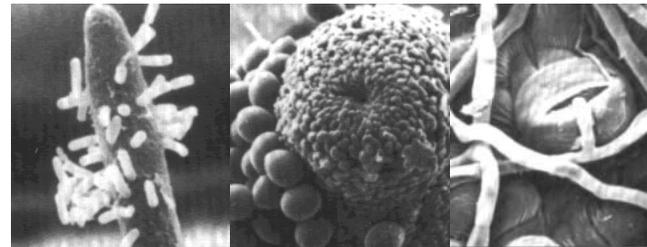
The results are best understood by considering the graph obtained from plotting pressure (P) against volume (V) for the series. This is shown in Figure 1; the curves obtained are termed 'isothermals'



Consider first the 10° C isothermal at low applied pressure. The CO<sub>2</sub> is gaseous (vapor) and generally exhibits the characteristics of a gas (Boyle's Law) over the range from 'r' to 's'. From point 's' a very slight increase in pressure results in a change from vapor state to the liquid state. This is the phenomena of saturation. From 's' to 't' the pressure is virtually constant while the volume is decreasing and at 't' the substance is all liquid.

From point 't' the graph becomes almost vertical indicating significant application of pressures for very little change in volume, liquids being virtually incompressible.

The 20° C isothermal has similar general characteristics, however there is less difference between points 'v' to 'w' compared to the difference between equivalent points 's' to 't' on the 10° C isothermal; these points representing the difference in volume occupied between the vapor phase and the liquid phase.



Fruit body neck with spores adhering to the sides

Bacteria adhering to the tip of a fungus.

Powdery Mildew, hyphal filament on leaf surface.

This indicates that the densities of the saturated vapor and liquid are approaching each other, also the slight departure from the vertical 'w' shows the compressibility is greater than that at higher pressures. This shows that the properties of the liquid and gas states of the substance are becoming similar and will ultimately coincide. This in fact is realized at the 31.1° C isothermal, which does not show any horizontal discontinuity. The temperature at which this occurs is termed the *Critical Temperature* and has an associated *Critical Pressure and density* and hence for a particular mass of gas, a *Critical Volume*. If a liquid was heated in a closed system so that the critical pressure could be attained, at the critical temperature, any visible meniscus would disappear; the surface tension would be zero and it would not be possible to distinguish between the properties of a liquid or a gas. We therefore have continuity of state. Above this temperature the gas cannot be liquified by the addition of pressure and strictly speaking a substance should only be classified as a gas above its critical temperature, below this temperature where it could possibly be liquified by the application of pressure, it is more precisely termed a vapor.

The critical phenomena can be utilized as a drying technique as it achieves a phase change from liquid to dry gas without the effects of surface tension and is therefore suitable for delicate biological specimens.

However, it is not surprising that the initial investigations were on CO<sub>2</sub> as will be apparent from Figure 2, showing a table of critical constants for some common substances. Even the practical achievement of the critical conditions would not assist the biologist, as the specimens would suffer significant thermal damage if we attempted to apply the technique direct for the removal of water from specimens.

### CRITICAL CONSTANTS

Substance	Temp. C	P.S.I
HYDROGEN	-234.5	294
OXYGEN	-118	735
NITROGEN	-146	485
CARBON DIOXIDE	+31.1	1072
CARBON MONOXIDE	+141.1	528
WATER	+374	3212

continues on page 50

# Critical Point Dryers

## Techniques and Applications Critical Point Drying Principles (continued)

Therefore CO<sub>2</sub> remains the most common medium for the CPD procedure and is termed the 'Transitional Fluid'. However, CO<sub>2</sub> is not miscible with water and therefore water has to be replaced in the specimen with another fluid which is miscible with CO<sub>2</sub>, this is termed the 'Intermediate Fluid'.

Ideally it will be able to replace the water in the specimen, and also serve as the 'Dehydration Fluid'. This is not exclusively the case, and additional steps may be used for particular circumstances.

However, where it is being utilized for both processes, texts may refer to it under the different headings, dehydration and intermediate, depending at what stage it is being used in the specimen preparation schedule. Prior to any of these stages chemical fixation of the specimen must be carried out (normally using glutaraldehyde -osmium procedures).

### NOTE

The whole discipline of specimen preparation (chemical or vapor fixation) prior to the transitional stage is only mentioned in its most basic terms, procedures vary according to the type and nature of the specimens. Further references should be obtained.

### a) Intermediate Stage

As mentioned previously this involves dehydration and intermediate fluids, the following is a possible schedule.

**(Wet Specimen) H<sub>2</sub>O → Acetone → CO<sub>2</sub> → C.P.D. (Dry Specimen)**

The specimen is usually processed through varying concentrations of dehydration fluid, culminating in complete replacement of the water with this intermediate fluid. Because it has a low surface tension the specimen is less likely to experience damage due to evaporation while transferring to the chamber, also being miscible with CO<sub>2</sub> (the Transitional Fluid) ensures satisfactory conditions after flushing (purging) for the CPD process to commence.

**(Wet Specimen) H<sub>2</sub>O → Acetone → 30%\* 100% → CO<sub>2</sub>\*\* → CPD (Dry Specimen)**

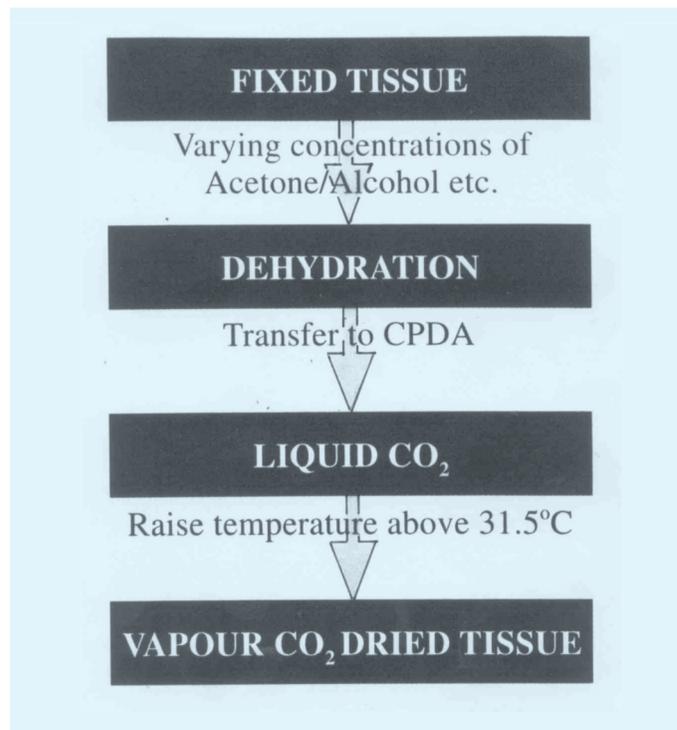
Note:

\*50/60/70/80/90 typically 10 minutes each

\*\* Flush Typically 3 times

The table (Figure 3) gives an indication of some intermediate fluids. (Water is 73 Dynes/cm.)

### Critical point drying stages



**Figure 3: Dehydration Intermediate Fluids for CPD**

SUBSTANCE	SURFACE TENSION (DYNES/CM)
ETHANOL	23
ACETONE	24
FREON (113)	19

Having transferred the specimen to the chamber in the Intermediate Fluid, the chamber is flushed several times to replace it with the Transitional Fluid. The process from which the complete techniques derives its name CPD can now be initiated.

## CO<sub>2</sub> grades required for critical point drying

Generally speaking, the grade we recommend is 'normal' grade - that is the one most commonly offered by industrial gas suppliers.

In most parts of the world, 'normal' grade of CO<sub>2</sub>, from the suppliers, is specified as 'N4.5' or 99.995% minimum purity with a maximum level of 50ppm of impurities.

There are, however, two other grades available by special order from most gas suppliers. One is 'N4.0' or 99.99% (less pure than 'normal' grade). The other is 'N5.5' or 99.9995% minimum purity. The N5.5 purity is easier to find in those parts of the world where there is high level of activity in electronics, since these customers often demand gases with higher purities.

We are not aware of anyone who has ever reported either superior results using N5.5 purity vs N4.5, or inferior results using N4.5 vs N5.5. However, we do want our customers to have the benefit of such detail about liquid carbon CO<sub>2</sub> procurement in the event they should ever find reason to believe that their particular specimens might benefit from the higher purity product.

Remember, the requirement is for LIQUID carbon dioxide and NOT gaseous carbon dioxide. For this reason a cylinder with an internal 'siphon' must be specified. A siphon cylinder is normally denoted by a white stripe painted along its length. No pressure regular is needed.

## Critical Point Dryers

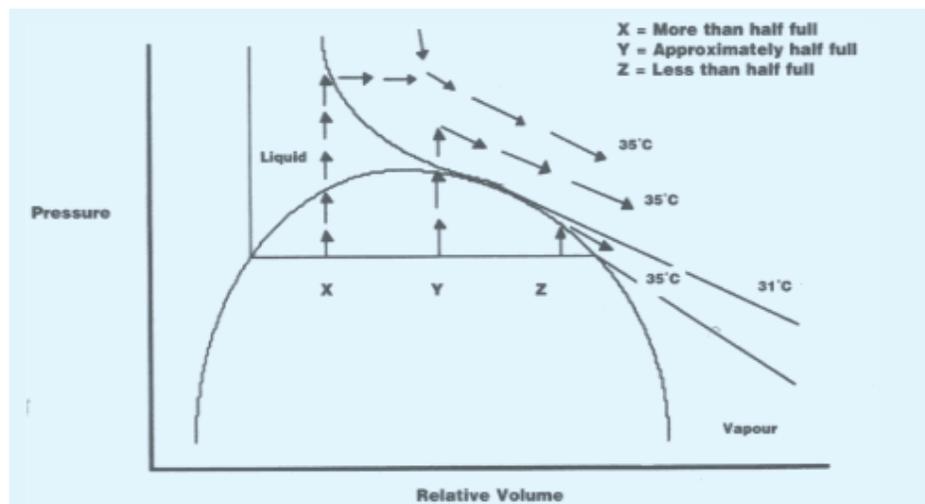
### Techniques and Applications Critical Point Drying Principles (continued)

#### (b) Transitional Stage

As discussed previously (see Figure 1) the conditions for which the critical point passage can be obtained for CO<sub>2</sub> are 31.1°C and 1072 psi. However, it must be remembered that these isothermals are obtained from a fixed mass of gas and an applied pressure for a series of constant temperatures.

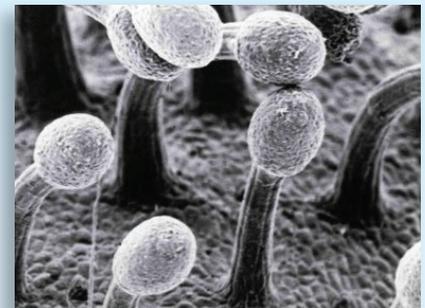
In the laboratory application of CPD we have a fixed volume which is filled with the transitional fluid. Some typical examples of which are given in figure 4.

Pressure is obtained by the effect of applying heat and while it can be readily appreciated that we can take a liquid from below its critical temperature and obtain the transition to gas above its critical temperature, an understanding of the relevant 'start' and 'end' points and the cycle involved is required in evaluating the design and performance of CPD equipment. It is still useful however, to utilize these CO<sub>2</sub> isothermals as indicated in figure 5 with the Superimposed 'arrows' showing differing conditions for the CPD device.



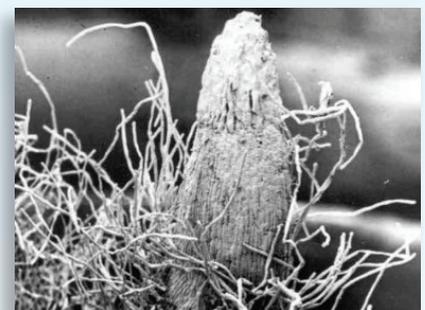
#### Starch Grains in Potato Tuber

Critical point dried fractured cell from the tuber of potato (*Solanum tuberosum*), demonstrating thin cell walls starch grains (amyloplasts).



#### Glandular Trichomes on Modified Leaf Surface of Butterwort

An insectivorous plant, the butterwort (*Pinguicula vulgaris*) has modified leaves which bear tiny granular trichomes which trap insects. The capitate head of the trichome then secretes protease enzymes to digest the insect parts.



#### Barley Root Tip

Critical point dried tip of barley (*Hordeum vulgare*) root, demonstrating root cap cells (calyptra) and slightly deformed (compressed) root hairs (Pili).

It is already acknowledged that these circumstances are not exactly comparable. For example, during operation of the CPD we would fill at CO<sub>2</sub> cylinder pressure and at ambient temperature: not at saturated vapor pressure. At a lower temperature decompression is as a result of venting and the subsequent reduction in mass of gas, not reduction in externally applied pressure. The relative volume is determined by the initial level of liquid in relationship to the total free volume available (this being the chamber plus sample "boat" etc.)

If we consider 'X' with the liquid CO<sub>2</sub> more than half filling the total available volume and we heat from 10°C to 35°C then we will make the transition from liquid to gas. The pressure rise will be rapid as the liquid will expand and the level will increase before the critical temperature is reached. This is termed 'going around' the critical point. Usually (as in the case of instruments supplied by Electron Microscopy Sciences) a pressure-bursting disc is employed to prevent excessive pressure increase.

For condition 'Y' with approximately a full pressure chamber, the liquid level will remain relatively constant, its density decreasing and that of the vapor increasing, and becoming the same when its critical temperature has been reached, together with the corresponding critical pressure.

Looking at condition 'Z' with the pressure chamber less than half full. The level will fall and vaporization will occur before the critical temperature is reached, also the specimens may be uncovered and subjected to unwanted evaporation.

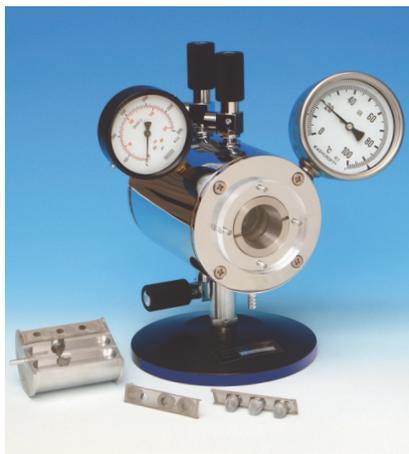
Ideally, we wish have a situation where the liquid fills the specimen chamber, while still only accounting for approximately 50% of the total volume available. This will ensure that specimens are not uncovered during initial flushing stages and in addition this should enable critical constants of temperature, pressure, and density to be achieved relatively simultaneously without excessive pressure or evaporation conditions occurring.

It is also advisable to maintain a temperature somewhat above the critical temperature during decompression, this will avoid the possibility of gas recondensing. It is also important to control the decompression rate itself as there is evidence that rapid pressure equalization can cause specimen.

## Critical Point Dryers

### ● EMS 3000 and EMS 3100 Critical Point Dryers

*The design of the EMS 3000 gives unequalled visibility of the critical point drying process and an unsurpassed view of the fluid level in the chamber. Unlike many of the more complex critical point dryer designs, it is much easier to see the phase change at the critical point.*



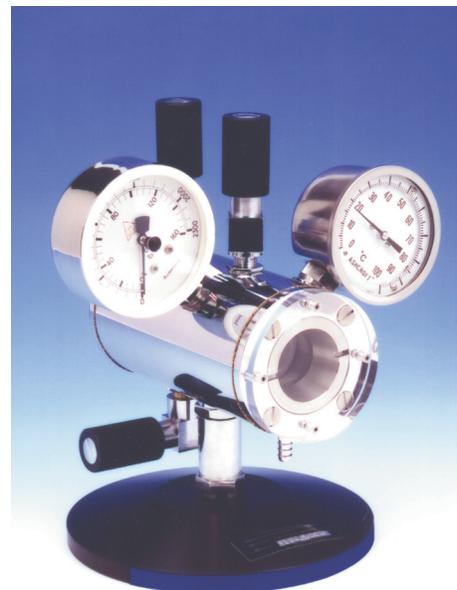
EMS 3100

#### EMS 3000

The design of the EMS 3000 features a horizontal pressure chamber measuring 30.1mm internal diameter x 82mm in length. The chamber has an external water jacket for temperature control and specimens are introduced via a removable rear door. The front of the chamber is fitted with a 25mm diameter window which permits easy viewing of the liquid level.

#### EMS 3100

Where increased chamber volume is required, for either size or quantity of specimens to be dried, the large capacity model EMS 3100 is available. The chamber dimensions are 63.5mm internal diameter x 82mm in length, giving approximately three times the volume of the EMS 3000. The transfer boat will also accept three times the number of specimen baskets.



EMS 3000

### FEATURES

- Proven reliability - over 6,000 critical point dryer installations world-wide
- Simple robust construction - easy to maintain - many critical point dryer users carry out their own routine maintenance
- Horizontal chamber and large viewing window - excellent visibility of the fluid level and drying process
- Large robust valves for draining of fluids, ingress of CO<sub>2</sub> and venting of gas - very durable; the rapid ingress of CO<sub>2</sub> helps prevent pre-drying of specimens
- Safety - every critical point dryer unit is pressure tested to 2,500psi and a certificate is issued. A pressure bursting disc is also fitted to safeguard against misuse
- Specimen handling - optional specimen holders for coverslips and TEM grids. Porous pots are available for fragile or very small specimens
- Three-year warranty

### Temperature control

Dial gauges display pressure in the chamber and the temperature of water circulating through the jacket. Three pressure valves permit easy connection to the liquid CO<sub>2</sub> cylinder and allow liquid agitation and venting of the chamber. A source of hot running water is essential. Cooling is also useful, especially for sequential process runs or in hot climates.

The temperature of the EMS 3000 and EMS 3100 chamber is raised with a hot water supply. Mains water can be used but a more elegant method involves the use of the optional EMS 3500 Thermocirculator, which is connected directly to the inlet and outlet of the water jacket. The temperature of the circulatory fluid can be pre-set (eg at 37°C, just above the critical temperature).

A second alternative is the model EMS 4860 Recirculating Heater/Chiller, which can be used to pre-cool the chamber to below ambient prior to loading specimens and then to heat the chamber to the critical temperature.

### Safety

Safety is, of course, an important consideration with all pressure vessels. Should critical pressure and temperature be inadvertently exceeded, a bursting disc is incorporated in the chamber support. The critical point drying chamber itself is tested to 2,500psi, which is approximately twice the working pressure. A guard is also fitted over the viewing window.

### Specimen holder (boat)

An important feature is the design of the transfer boat. This permits specimens in the intermediate fluid to be transferred to the critical point dryer. On sealing the chamber, the intermediate fluid begins to drain and can be replaced with liquid CO<sub>2</sub>. In this way the specimens are never allowed to dry out during the specimen loading and transfer stage of the process.

Both the EMS 3000 and EMS 3100 are supplied with a standard tissue holder (boat). The EMS 3000 comes with the EMS 3000-01 tissue boat and has a single slot with three tissue baskets. Specimens are loaded into each basket and the gauze lid moved laterally to seal the top.

The EMS 3100 is supplied with the EMS 3100-01 tissue boat and has three slots each with three tissue baskets, making a total of nine tissue baskets. Other choices of holders are listed below under Options and Accessories.

### Bonded chamber seals – Nitrile or EPDM?

All models of EMS 3000 and EMS 3100 are fitted with a standard with nitrile bonded window and door seals. Nitrile is a good general material due to its ability to withstand attack by solvents, such as ethanol.



EMS 3000 Specimen Boat



EMS 3100 Specimen Boat

## Critical Point Dryers

### ● EMS 3000 and EMS 3100 (continued)

However, if acetone is used as the transition fluid then the EPDM seals have been found to be more resistant to chemical attack by that solvent.

If you are ordering an EMS 3000 or EMS 3100 and are planning to use acetone as the transition fluid, please state this on the order and EPDM bonded seals will be fitted.

For existing instruments, both Nitrile and EPDM bonded seal can be ordered as spare parts.

### Options and Accessories

#### Glass microscope coverslip holder (option):

Specially adapted boats allow glass coverslips to be held firmly during drying. The EMS 3000-02 is designed for the EMS 3000 and has a maximum capacity of seven coverslips. Likewise, the larger EMS 3100-02 coverslip boat is available for the EMS 3100 'Jumbo' Critical Point Dryer and has a carrying capacity of 21 coverslips.



Coverslip Holder

#### TEM grid holder (option):

The EMS 3000-1 holder for 3.05mm grids and the EMS 3000-2 grid holder for 2.3mm grids can be used with all Electron Microscopy Sciences critical point dryers. Maximum number of grids is three.



TEM Grid Holder

#### Porous pots with lids (option):

EMS 800A solvent-resistant porous pots (12.7mm x 15.5mm) with lids are ideal for very small or very delicate specimens.



Porous Pots

#### For the EMS 3000 (NOTE: EMS 3000-01 is included as standard):

- EMS 3000-1 Specimen holder for 3.05mm grids
- EMS 3000-2 Specimen holder for 2.3mm grids
- EMS 3000-01 Specimen holder for tissue (boat)
- EMS 3000-02 Specimen holder for coverslips
- EMS 800A Porous pots with lids 12.7mm x 15.5mm (pack of 10) for micro-specimens
- EMS 3500 Thermocirculator for control of heating cycle
- EMS 4860 Recirculating Heater/Chiller to control heating and cooling cycle (please specify voltage)

#### For the EMS 3100 (NOTE: EMS 3100-01 is included as standard):

- EMS 3100-1 Specimen holder for 3.05mm grids
- EMS 3100-2 Specimen holder for 2.3mm grids
- EMS 3100-01 Specimen holder for tissue (boat)
- EMS 3100-02 Specimen holder for coverslips
- EMS 800A Porous pots with lids 12.7mm x 15.5mm (pack of 10) for micro-specimens
- EMS 3500 Thermocirculator for control of heating cycle
- EMS 4860 Recirculating Heater/Chiller to control heating and cooling cycle (please specify voltage)

### Site Requirements

**Site selection:** The apparatus should be positioned in the laboratory with convenient access to:

- Hot and cold water supply (if the optional EMS 3500 Thermocirculator or EMS 4860 Recirculating Heater/Chiller are not used)
- Mains power supply (for EMS 3500 and EMS 4860 only)
- Fume cupboard or window, or an area of good ventilation
- Space for CO<sub>2</sub> siphon cylinder

**CO<sub>2</sub> Cylinder:** The EMS 3000 and EMS 3100 require a cylinder of liquid CO<sub>2</sub> fitted with a siphon tube (indicated by a vertical white stripe on the cylinder). If there is any doubt regarding the presence of a siphon tube, advice should be sought from the gas supplier.

Cylinder connection threads vary from country to country and even between manufacturers in the same country. For example, the transfer pipe supplied is fitted with ¼" British Standard Pipe (BSP) and 0.86" x 14 TPI union. These are standard threads for the UK and generally in the rest of the world, but will not fit cylinders in the USA.

An EMS 3000-US kit should be specified for use within the USA; this includes a transfer pipe adaptor which will fit USA cylinders. If it is deemed necessary to fabricate another transfer pipe, advice should be sought from a local supplier of high-pressure fittings.

**Heating and cooling:** Use a mixer to the laboratory hot and cold water outlets, terminating with a 6mm/¼" hose connection for the PVC tubing supplied. A 'Y' piece connected to the hot and cold water taps is also suitable.

The EMS 3000 and EMS 3100 require both hot and cold water during the operating cycle. Cooling facilitates filling of the work chamber with liquid CO<sub>2</sub>, and heating is required to take the liquid above its critical point.

Good control of the water temperature is essential for good results, hence the recommended use of the EMS 3500 Thermocirculator or, more conveniently, the EMS 4860 Recirculating Heater/Chiller which gives precise control of cooling and heating.

**Space requirement:** A minimum bench space of approximately 230 x 230mm is required.

### Ordering Information

<b>EMS 3000</b>	Critical Point Dryer Chamber dimensions: 30.1mm Ø x 82mm length	each
<b>Supplied with:</b>	EMS 3000-01 Specimen holder for tissue (boat) 1m liquid CO <sub>2</sub> delivery tube O ring and L gasket set (including window and door bonded seals) Spare bursting disc and retaining copper (Cu) washer Steel bar for tightening/untightening the door Flat wrench (for removing the window retaining ring) Comprehensive manual Pressure test certificate	
<b>EMS 3100</b>	Large Chamber Critical Point Dryer Chamber dimensions: 63.5mm Ø x 82mm length	each
<b>Supplied with:</b>	EMS 3100-01 Specimen holder for tissue (boat) 1m liquid CO <sub>2</sub> delivery tube O ring and L gasket set (including window and door bonded seals) Spare bursting disc and retaining copper (Cu) washer Steel bar for tightening/untightening the door Flat wrench (for removing the window retaining ring) Comprehensive manual Pressure test certificate	

## Critical Point Dryers

### ● EMS 850 Critical Point Dryer with Thermoelectronic Heating and Adiabatic Cooling

The EMS 850 Critical Point Dryer is designed for use with CO<sub>2</sub>, having first replaced any water in the specimen by a series of dehydration, often in the same fluid such as Acetone, which will also be the intermediate fluid.

*(Wet Specimen)–Water–Acetone–30%–100%–CO<sub>2</sub>–  
C.P.D.–(Dry Specimen)*

The specimens for critical point drying are located in the pressure chamber of the EMS 850. The chamber is pre-cooled to allow it to be readily filled with liquid CO<sub>2</sub> from a gas cylinder. The chamber is then heated to just above the critical temperature with subsequent critical pressure being achieved. The CO<sub>2</sub> gas is vented through a needle valve, to avoid specimen distortion.

The EMS 850 is fitted with thermoelectronic Heating and Cooling and Temperature control of +5°C on Cooling, and +35°C on Heating. This ensures the critical point is accurately obtained, avoiding excess pressures or temperatures, or the need to rely on pressure relief valves to control pressure during the heating cycle. The chamber is vertical, with top loading, to ensure specimens do not become uncovered during the drying process, with a side viewing port to locate the meniscus for the correct level when initially filling the chamber.

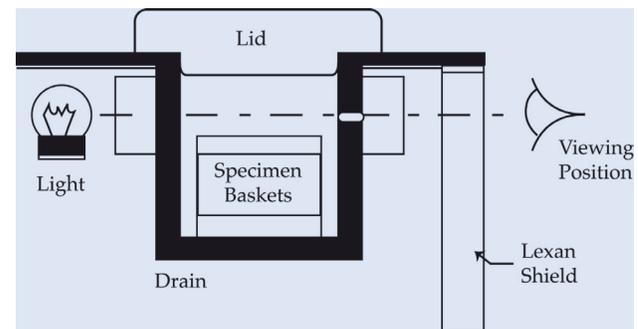
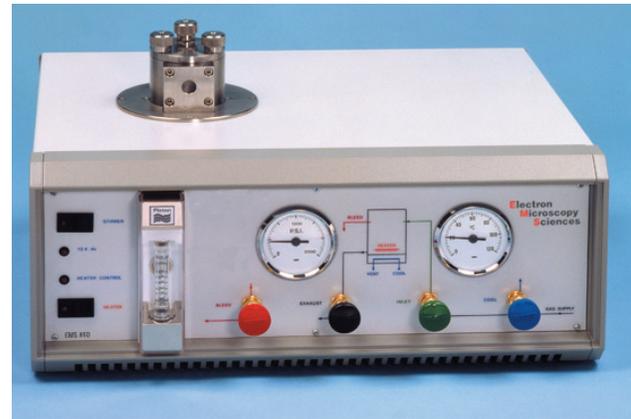
The EMS 850 is fitted with a fine let down needle as standard and flow gauge is no longer required.

#### Features

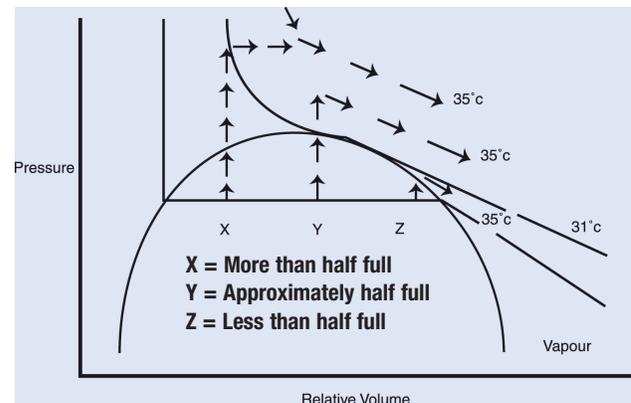
- Vertical chamber with top filling and bottom draining.
- Normal operating temperature 35°C pressure 1500 psi.
- Thermoelectric Peltier cooling and heating.
- Fine control needle valve pressure letdown.
- Illuminated chamber with side viewing port and protective 'Lexan' shield.
- Stirrer system for enhanced solvent exchange.
- Temperature monitoring and control with thermal cut-out protection.
- Pressure monitoring with pressure relief valve and rupture disc protection.
- Polycarbonate safety shield.

#### Specifications

<b>Instrument Case</b>	450mm (W) x 350mm (D) x 175mm (H)
<b>Weight</b>	12Kg
<b>Specimen Chamber</b>	30mm (Dia.) x 40mm (H) (Tested to 3000 psi)
<b>Temperature Gauge</b>	0-120°C
<b>Thermal Cut-Out</b>	40°C
<b>Pressure Gauge</b>	0-3000 psi
<b>Pressure Relief</b>	At 1500 psi
<b>Rupture Disc</b>	At 1900 psi
<b>Peltier Cooling/Heating</b>	+5°C to +35°C
<b>Two On/Off Valves</b>	Inlet/Outlet/Cool/Vent Needle Valve Letdown
<b>Supply</b>	115V 60Hz (6 Amp Max) 230V 50Hz (3 Amp Max)
<b>Services</b>	Requires CO <sub>2</sub> gas cylinder direct connection by high pressure hose. (High pressure hose included with instrument.)



Cross-sectional view of chamber, showing meniscus viewing position.



#### Ordering Information

91090 EMS 850 Critical Point Dryer complete each

#### Replacement Parts

91091 Mesh Basket each

91092 Porous Spec Pots 10/pack

## Critical Point Dryers

### ● EMS 850WM Large Chamber Critical Point Dryer

The EMS 850WM is compact, bench-top instrument designed to critical point dry a complete 150mm/6" wafer. A convenient wafer holder allows rapid transfer and ensures that pre-drying does not occur.



#### Features

- 170mm diameter chamber - optimized for wafer/MEMS drying
- Vertical chamber with top-loading and bottom draining - ensures specimens do not become uncovered during drying
- Thermoelectric heating - accurate temperature control
- Fine control needle valve pressure let down - precise control
- Temperature monitoring and control with thermal cut-out protection
- Pressure monitoring with safety cut-out for over pressure
- Three-year warranty

The EMS 850WM has built-in heating and water cooling using the EMS 4860 Recirculating Heater/Chiller. This combination will give temperature control of +5°C cooling and +35°C during heating. This ensures the critical point is accurately obtained, avoiding excess pressures or temperatures, or the need to rely on pressure relief valves to control pressure during the heating cycle.

The EMS 850WM has a vertical chamber which allows top-loading of specimens. A viewing port is fitted in the top plate for specimen observation. The specimen exchange mechanism is simple to use and ensures the specimen remains under liquid during loading.

#### Specimen handling

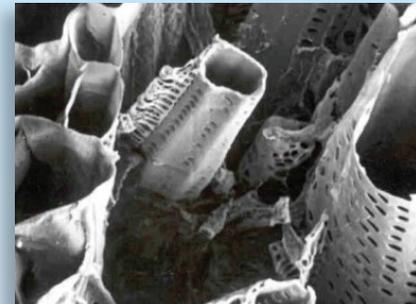
100mm or 150mm diameter wafers are held in a PTFE holding tray. The tray including wafer is immersed in acetone in order to remove all moisture from the specimen. After dehydration, the wafer and holder are transferred into the pre-cooled specimen chamber using the wafer transfer device. On completion of the critical point drying process, the wafer is removed from the chamber using the transfer device prior to further processing.

#### Ordering Information

<b>91090-WM</b>	EMS 850WM Large Chamber Critical Point Dryer	each
-----------------	----------------------------------------------	------

#### Requirements

<b>EMS 4860</b>	Recirculating Heater/Chiller (for cooling chamber)	each
<b>EMS 3102</b>	Carbon Dioxide bottle heating system	each



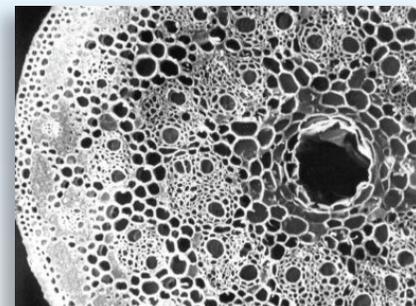
#### Mature Barley Root

Critical point dried transverse fracture of a mature barley (*Hordeum vulgare*) root, demonstrating central stele and surrounding cortical cells. Root hairs (Pili) are also obvious.



#### Aphid

Critical point dried aphid on a leaf surface.



#### Bamboo Stem

Transverse fracture of the stem of young bamboo (*Bambusa* sp), demonstrating xylem and phloem bundles and heavily thickened (lignified) epidermal and hypodermal cells.

# Freeze Dryers

## What is... Freeze Drying?

The application of freeze drying for specimen preparation for TEM and SEM is a well established practice.

Its application is to reduce the distortion which occurs when a wet specimen dries by normal evaporation. This distortion is due to the forces of surface tension going from a liquid to a vapor phase such as water to water vapor, commonly the situation in a Biological Specimen. However, if we freeze the specimen and maintain it frozen, then by applying a vacuum, we can obtain a situation where we can remove the frozen water by sublimation, avoiding the liquid phase, and reducing the distortion. This rate of sublimation is very much a function of temperature and vacuum, and associated drying time which is on the order of several hours or longer. Ideally freeze drying could be carried out at temperatures below the recrystallization of ice, which will require an inordinately long drying period. In practice, temperatures of  $-60^{\circ}\text{C}$  have been found to give reasonable results under vacuums achievable with two stage rotary pumps, having ensured that good, fast freezing of the specimen has been carried out initially.

For particular applications, however, it can be necessary to freeze dry at temperatures below  $-80^{\circ}\text{C}$  with lower sublimation rates for delicate specimens. This requires a better vacuum than can be obtained using a rotary vacuum pump.

At normal temperatures and pressures the water in the specimen would dry by evaporation and would experience forces due to the effects of surface tension between the liquid and vapor interface, however, at pressures below the Triple Point, the solid phase can be changed to the vapor phase at a rate depending on the temperature, without the liquid being present. In the case of a frozen Biological Specimen where we are considering water ( $\text{H}_2\text{O}$ ) from solid to vapor phase the Triple Point pressure is a relative vacuum. There are notable exceptions such as Carbon Dioxide ( $\text{CO}_2$ ) where the Triple Point is higher than atmospheric pressure.

## Techniques and Applications

### Freeze Drying Principles

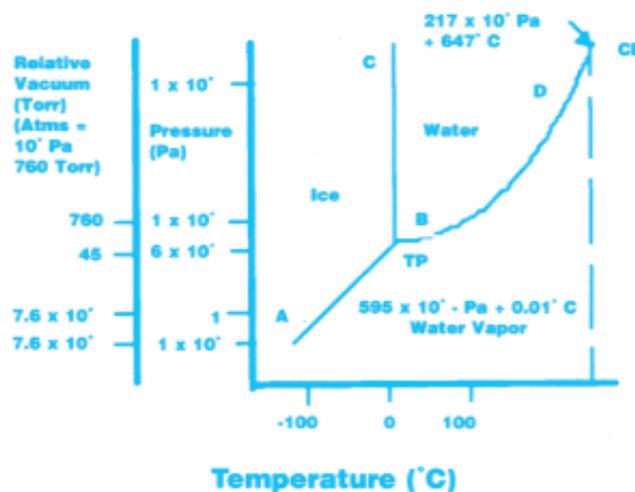
#### Sublimation consideration

The Figure shows the phase diagram for the ICE/WATER/WATER VAPOR system. The Curve A-B is termed the Sublimation Curve, at which the Solid and Vapor are in equilibrium. Similarly for Curve B-C the Melting Curve and B-D the Evaporation Curve, terminating at C-P the Critical Point. The point of intersection of the Curves T-P is called the Triple Point, at which the three phases exist in equilibrium. It has a unique value for pressure and temperature, and is a reference point, for which the Celsius temperature scale is now defined.

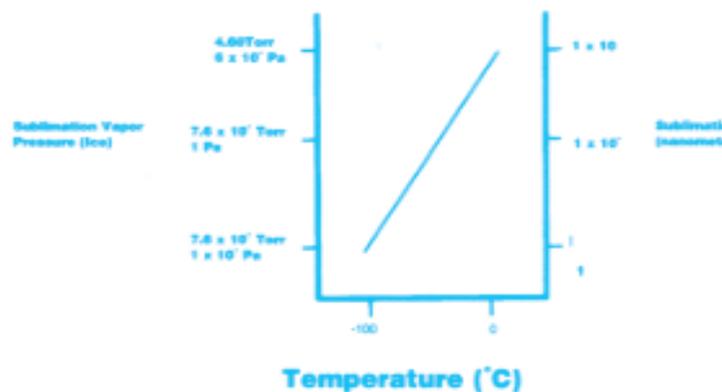
It should now be apparent that for any pressure in a system below the Triple point pressure, we can change water from the solid to vapor phase without the liquid phase being present, if the temperature at which we are allowed to do this does not have any restrictions.

In the freeze drying of Biological Specimens there are however limitations on the initial temperature to which the specimen should be allowed to warm when drying is commenced.

Phase Diagram (not to scale)



Sublimation Curve (Vacuum)



# Freeze Dryers

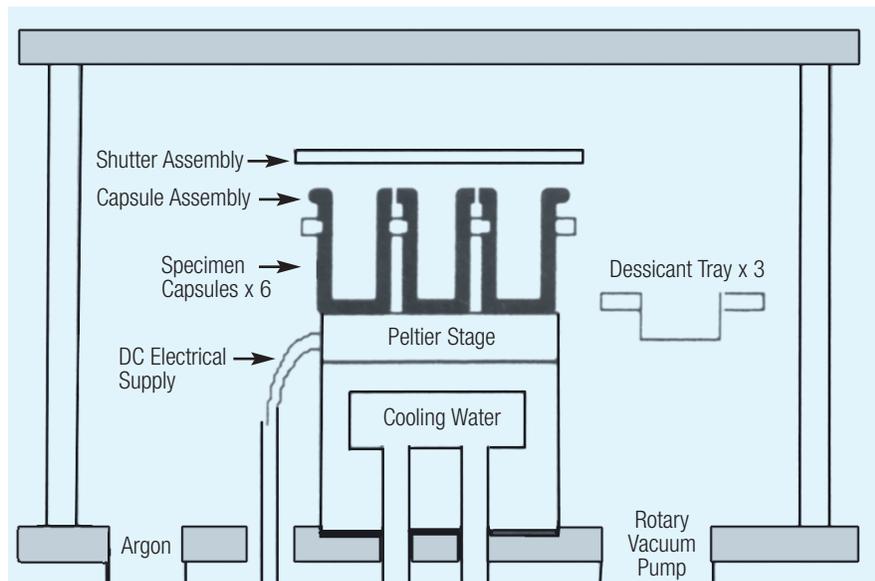
## Techniques and Applications

In practice therefore, the system pressure (relative vacuum) used, is much lower than the Triple Point pressure. The Fig. gives an indication of the sublimation pressure (Saturated Vapor pressure at differing temperatures, and satisfactory freeze drying will be determined by this, and the partial pressure (Unsaturated Vapor pressure) of the water vapor in the vacuum system, which is required to be lower. The right hand scale gives an indication of Sublimation rates, (assuming partial pressure of H<sub>2</sub>O is not limiting) which are totally temperature dependent.

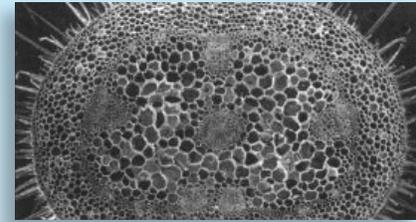
### Freezing Considerations

The presentation and nature of a specimen prior to freeze drying depends on the user and the application. Irrespective of this, however, there is a pro-requisite for good (FAST) freezing, this is probably the most singularly influential factor on the final result, within the limitations previously considered of the drying routine.

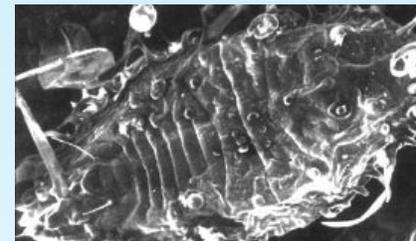
A range of cryogens and freezing techniques are being applied to the freezing of specimens, the understanding of the mechanisms of which, are still at an early stage. However, with exceptions, (Hyperbaric freezing containment of ice crystals is generally less than 20 micrometers from the surface, and Liquid Nitrogen (LN<sub>2</sub>) usually readily available and unsophisticated in its application, should be satisfactory for a range of specimen freezing. The technique for its application is that of plunge freezing by hand or mechanical device.



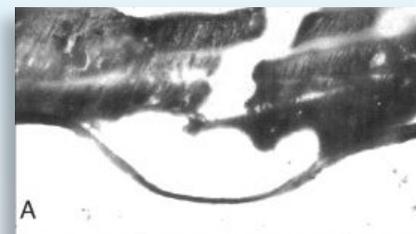
Cross-sectional View of Peltier Cooled Stage



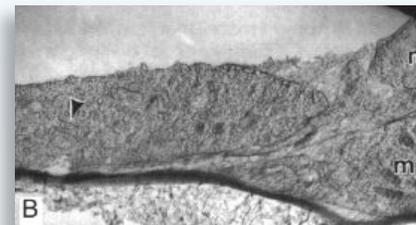
Cells of the petiole assumed their normal isodiametric shape after drying.



Scanning Micrograph of an Aphid, Freeze dried using the EMS 750 system and Sputter Coated with Gold. Aphids secrete wax ribbons of which show good preservation, normally 'Lost' in Drying methods involving solvents.



A. Preparation of ASM which has been cryo-sectioned and freeze dried at low vacuum, note rehydrated appearance.



B. Preparation of ASM which has been freeze dried under controlled conditions (using EMS 775). subcellular structures such as nucleus (n), mitochondria (m) and PER can be seen, Marker=1.μm

Photos courtesy of Dr. Alice Warley. Division of Ophthalmology, The Raines Institute, U.M.D.S., Lambeth Place Road London, SE 17EH

## Freeze Dryers

### ● EMS 750 Freeze Dryer

The EMS 750 Freeze Dryer operates at rotary pump vacuum using a 'Peltier' Thermoelectric stage, with drying temperatures of the order of  $-60^{\circ}\text{C}$ , with back-up water cooling at a nominal  $15^{\circ}\text{C}$ . Both the Temperature and the Timer can be pre-selected, and the drying cycle will be completed automatically.

Provision is made at the end of the drying cycle to allow the specimen to assume room temperature, or be subsequently warmed prior to embedding. Disposable desiccant containers are located in the preparation chamber to enhance the water vapor removal, and with a suitable container, the vacuum chamber can be utilized to prepare liquid nitrogen 'slush' for fast freezing.

#### Features

- Thermoelectric cooling and heating.
- Convenient to use cold stage.
- Accurate temperature and time monitoring and control.
- Automatic drying cycle.
- Modular electronics.
- Clean line design.
- Polycarbonate safety shield.

#### Specifications

<b>Instrument Case</b>	450mm(W) x 350mm(D) x 175mm(H)
<b>Work Chamber</b>	Borosilicate Glass 165mm(Dia.) x 125mm(H)
<b>Weight</b>	18Kg
<b>Specimen Stage</b>	$-60^{\circ}\text{C}$ to $+60^{\circ}\text{C}$
<b>Vacuum Gauge</b>	ATM $-1 \times 10^{-2}$ mbar
<b>Temperature Controller &amp; Monitor</b>	$-90^{\circ}\text{C}$ to $+90^{\circ}\text{C}$ Display Resolution to $0.1^{\circ}\text{C}$
<b>Timer</b>	0-999 Hours
<b>Supply</b>	115V 60Hz (12 Amp Max incl pump) 230V 50Hz (6 Amp Max incl pump)
<b>Services</b>	Water Cooling at nominal $15^{\circ}\text{C}$
<b>Vacuum Pump (Recommended)</b>	Complete with vacuum hose and oil mist filter 85L/Min
<b>Size</b>	470mm(L) x 150mm(W) x 250mm(H)
<b>Weight</b>	20Kg

#### Ordering Information

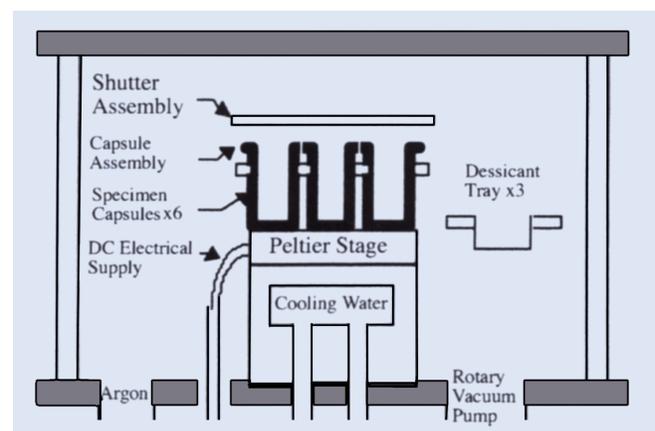
91080	EMS 750 Freeze Dryer complete with accessories	each
91005	Rotary Vacuum Pump	each

#### Replacement Parts

91013	Glass cylinder 165mm (6")	each
91014	"L" Gasket to suit 165mm (6") cylinder (1 pair)	each
91085	Desiccant Containers (set of 3)	each
91086	Polystyrene Slushing Pot	each

#### Optional Accessories

91040	EMS 7640 Carbon Coating Attachment	each
-------	------------------------------------	------



Cross-sectional View of Peltier Cooled Stage

## Freeze Dryers

### ● EMS 775 Turbo Freeze Dryer

The EMS 775 Turbo Freeze Dryer operates at temperatures down to  $-140^{\circ}\text{C}$  and employs a Turbomolecular pumping unit, backed by a Rotary Vacuum pump. The lower temperatures are achieved by using a Liquid Nitrogen fed Cold Stage.

The pre-frozen specimens are admitted into the cooled stage of the drying chamber through a vacuum gate valve which interfaces to the specimen transfer chamber.

The system incorporates both Time and Temperature Control, and at the end of the drying period, the stage may be heated prior to specimen removal. The system also has facilities for purging with Nitrogen Gas.

For extended drying periods, an auto top-up device can be provided as an option to continuously 'fill' the chamber's liquid nitrogen dewar.

The option of a custom designed specimen freezing chamber to produce 'slushy' sub cooled liquid nitrogen is available which also interfaces to the specimen transfer chamber.

The option for a carbon or sputter coating attachment without breaking vacuum is available.

#### Features

- Programmable multi-segment sequence control with 10 times and 10 temperatures.
- Built-in penning head and gauge.
- Liquid Nitrogen fed cold stage.
- Vacuum gate valve to chamber.
- Vacuum specimen transfer chamber.
- Accurate time and temperature monitoring.
- Modular electronics.
- Clean line design.
- Polycarbonate safety shield.

#### Specifications

<b>Instrument Case</b>	450mm(W) x 350mm(D) x 175mm(H)
<b>Work Chamber</b>	Borosilicate Glass
<b>Plus</b>	165mm(Dia.) x 125mm(H)
<b>Stainless Steel Base</b>	110mm(Dia.) x 115mm(H)
<b>Weight</b>	42Kg
<b>Specimen Stage</b>	$-140^{\circ}\text{C}$ to $+40^{\circ}\text{C}$
<b>Temperature Monitor</b>	$-140^{\circ}\text{C}$ to $+40^{\circ}\text{C}$
<b>Sequence Controller</b>	10 times and 10 temperatures
<b>Timer</b>	0 to 999 Hours
<b>Vacuum Gauge Range</b>	$1 \times 10^{-2}$ mbar to $1 \times 10^{-7}$ mbar
<b>Operating Vacuum</b>	$1 \times 10^{-2}$ mbar to $1 \times 10^{-5}$ mbar
<b>Turbomolecular Pump</b>	60 litres/Sec (Ultimate vacuum $1 \times 10^{-6}$ mbar)
<b>Cooling</b>	By liquid nitrogen conductive cooling from 1 liter dewar fitted to the chamber of the unit.
<b>Chamber Dewar</b>	1 Litre Capacity
<b>Supply</b>	115V 60Hz (12 Amp Max incl pump) 230V 50Hz (6 Amp Max incl pump)
<b>Vacuum Pump</b>	Complete with vacuum Hose and oil mist filter. 85L/Min
<b>Size</b>	470mm(L) x 150mm(W) x 250mm(H)
<b>Weight</b>	20Kg



#### Ordering Information

<b>93123</b>	EMS 775 Turbo Freeze Dryer complete	each
<b>91005</b>	Rotary Vacuum Pump	each
<b>95126</b>	Supply Dewar (25L)	each

#### Replacement Parts

<b>91013</b>	Glass Cylinder 165mm (6")	each
<b>91014</b>	"L" Gaskets to suit 165mm (6")	each
<b>90032</b>	Copper Discs	each
<b>90034</b>	Aluminum Planchettes (1")	each

#### Optional Accessories

<b>95128</b>	EMS 170 Slushing Chamber	each
<b>95127</b>	EMS 175 Auto Top Up Unit +Controller	each
<b>91040</b>	EMS 7640 Carbon Coating Attachment	each

## Cryo-SEM Preparation Systems

### What is... Cryogenic Specimen Preparation?

In this instance we are referring to Frozen Hydrated Bulk Specimens for Scanning Electron Microscopy, commonly termed L.T.S.E.M. (Low Temperature Scanning Electron Microscopy). When Biological specimens are prepared by conventional methods, they may collapse and distort. In addition to which, there may be a loss of the diffusible elements that they may normally contain, and therefore affecting the validity of the X-Ray Microanalysis.

The use of L.T.S.E.M. offers a solution to this, and in addition allows viewing and analysis of 'liquid' specimens such as emulsions and suspensions, which was not previously practical. In preparing the specimen, we first want to freeze it as quickly as possible. This will reduce the morphological distortion important for structural observations, and also minimize any redistribution of solutes which is essential for X-Ray Microanalysis.

The aim of fast freezing is to reduce the size of ice crystals by reaching as quickly as possible the point at which recrystallization takes place, which is on the order of  $-130^{\circ}\text{C}$  (for pure water), and maintaining the specimen below this temperature. Rapid freezing is commonly obtained by plunging the specimen into a cryogenic fluid. This is commonly liquid nitrogen, usually in the form of 'slushy' nitrogen at  $-210^{\circ}\text{C}$ , and for the types of specimen we are considering, this has proved to be an effective cryogen.

Having frozen the specimen, we need to maintain it below  $-130^{\circ}\text{C}$  (the recrystallization temperature) and prevent sublimation (below  $-130^{\circ}\text{C}$  the rate of sublimation of ice is very low, of the order of  $0.001\text{nm/sec}$ ). We should, therefore, maintain the specimen with cryoprotection in a good clean vacuum to avoid contamination. Frozen specimens can subsequently be fractured, etched, coated with gold or carbon, using a preparation system, and then viewed and analyzed on the cold stage of the S.E.M.

## Techniques and Applications

### Cryo-SEM — the advantages

The Scanning Electron Microscopist is faced with the inescapable fact that liquid is a fundamental part of practically all lifesciences – and many materials – specimens. Since water occupies up to 98% of some animal and plant tissues it represents a most formidable specimen problem to most Scanning Electron Microscopists.

Cryo-SEM is a quick, reliable and effective way to overcome these not inconsiderable SEM preparation problems. Additionally the technique is widely used for observing 'difficult' samples, such as those with greater beam sensitivity and of an unstable nature. An important application, often overlooked, is the ability to use cryo-SEM to study dynamic processes (industrial or otherwise) by using a series of time resolved samples.

Naturally the advent of various "higher pressure" modes, such as VP, LV and ESEM has allowed such samples examined in SEM without resorting to freezing or drying methods. However, cryo-SEM is still by far the most effective method of preventing sample water loss, which will in fact occur at any vacuum level – even with Peltier stages fitted to the SEM and the careful addition of water vapor in the SEM chamber. Cryo-SEM also a number of additional advantages, including the ability to fracture and selectively remove surface water (ice) by controlled specimen sublimation.

#### Why choose cryo-SEM?

The limitations of conventional 'wet processing' include:

- Shrinkage and distortion
- Extraction of soluble materials
- Relocation of highly diffusible elements
- Mechanical damage (fragile specimens can be damaged during conventional processing)
- Slow (24 hours or longer)
- Toxic reagents are required (fixatives, buffers etc)

#### Advantages of cryo-SEM:

- Specimen viewed in fully hydrated state
- Soluble materials are retained
- Less relocation of highly diffusible elements
- Little or no mechanical damage
- Time lapse experiments and evaluating industrial processes at timed intervals
- Usually no exposure to toxic reagents
- Rapid process
- High resolution capability (compared to low-vacuum techniques)
- Extra information obtained by low-temperature fracturing (compared with conventional and low-vacuum methods)
- Good for liquid, semi-liquids and beam sensitive specimens
- Ability to selectively etch (sublimate to reveal information)
- Ability to 'rework' specimen (eg re-fracture and coat)

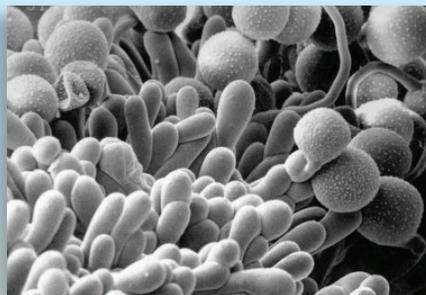
### Zoological



#### Frozen hydrated aphid

In comparison with the critical point dried aphid, this image shows that there is no distortion of the abdomen nor any other parts of the aphid following freeze drying.

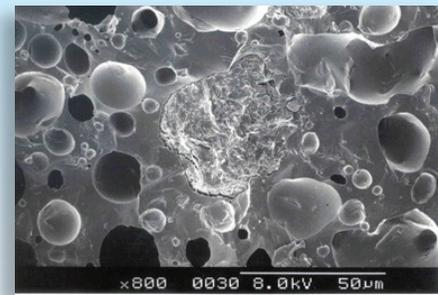
### Botanical



#### Pollen of cactus *Zygocactus truncatus*

Germinating pollen grains of *Zygocactus truncatus*.

### Foodstuffs



#### Chocolate Bar

# Cryo-SEM Preparation Systems

## Techniques and Applications

### A summary of the cryo-SEM preparation technique

Cryo preparation techniques for scanning electron microscopy (SEM) have become essential for the observation of wet or 'beam sensitive' specimens. Using such techniques removes the need for conventional preparation techniques, such as critical point drying or freeze-drying, and allows observation of the specimen in its 'natural' hydrated state.

The specimen is rapidly cooled and transferred under vacuum to the cold stage of the preparation chamber, which is mounted onto the SEM chamber. The preparation chamber is pumped either with a rotary pump (PP2000) or by a specially designed turbomolecular pumping system (PP2000T). The specimen can be fractured, sublimated ('etched') to reveal greater detail, and coated with metal by sputtering or with carbon by thermal evaporation.

Finally, the specimen can be moved under vacuum into the SEM chamber where it is easily located on a cold stage specifically tailored to the SEM. At all stages of the procedure the specimen is maintained at a 'safe' temperature of typically lower than -140°C.

#### Typical applications

Biological sciences including botany, mycology, zoology, biotechnology and biomedical – plus economically important agricultural sciences.

More recently cryo-SEM is becoming an essential tool for pharmaceutical, cosmetics and healthcare industries, where it is used in basic applied research and for routine QA of many products, such as creams, cosmetics and drug delivery systems.

Cryo-SEM has long been a standard preparation method in the food industry. Of interest are multi-

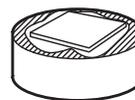
phase products, such as ice cream, confectionery and dairy products.

Botanical: Cryo-SEM is the perfect method for highly hydrated botanical material.

#### Some specimen mounting techniques for cryo-SEM

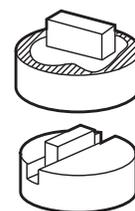
##### Surface mounting

This technique is used for leaf specimens etc. Roughen stub surface with fine emery paper. Specimen is laid on top of mounting media.



##### Edge mounting

This technique is used for edge observation and fracture. Roughen surface of stub with fine emery paper. Specimen is placed on its edge in a machined slot and secured with mounting media.



##### Film emulsion mounting

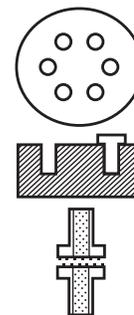
This technique is useful when a small specimen would be obscured by the Tissue-Tek mounting media, or when specimens need to be recovered. Specimens need to be slightly damp to use this method (good for nematode worms).



The specimen is laid on surface so that its dampness slightly dissolves the film emulsion allowing the specimen to adhere to the film surface. Exposed unused film with the emulsion side uppermost is secured to the stub with mounting media. It may be useful to scrape off the protective coating of the film emulsion first to assist conductivity.

#### Rivet mounting

For liquids and for when specimens need to be frozen off the stub to achieve fast freezing rates. The rivet is placed in the hole and filled with liquid prior to freezing. If the specimen needs to be frozen away from the stub, two liquid-filled rivets are held together and then frozen prior to transfer onto the stub.

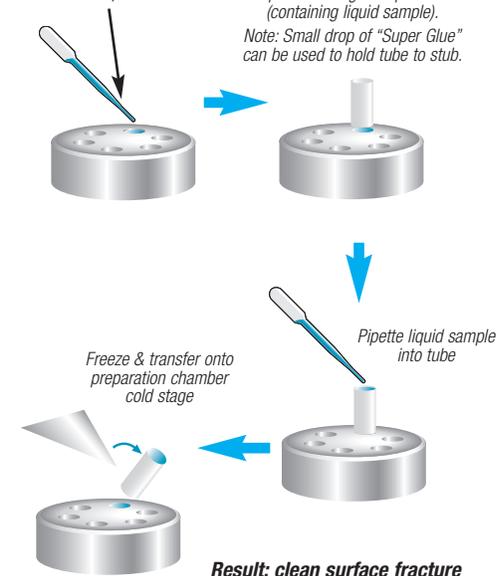


#### Alternative rivet mounting method

Pipette liquid sample into hole in sample stub

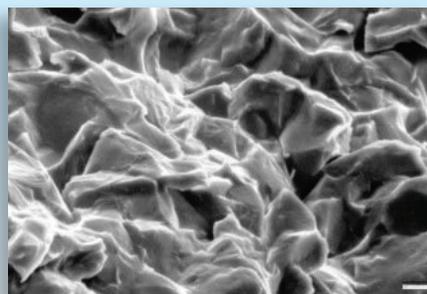
Place metal rivet or small piece of plastic tubing on top of hole (containing liquid sample).

Note: Small drop of "Super Glue" can be used to hold tube to stub.



Result: clean surface fracture

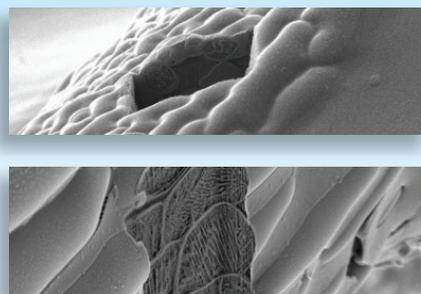
#### Geological



##### Wax crystals in gas oil

When cooled to a temperature below about 2°C, the waxes in fuel oils such as this tend to crystallize out. Wax crystal size and shape can be varied by altering the rate at which the oil is cooled.

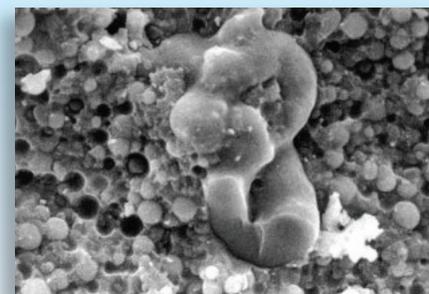
#### Cryo-DualBeam



##### Arabidopsis plant

Cryo-FIB/SEM. Image courtesy of Hannah Edwards and Arabidopsis plants provided by Darren Wells, Centre for Plant Integrative Biology, School of Biosciences, University of Nottingham, UK.

#### Polymers



##### Stable emulsion of a hydrophobic polymer

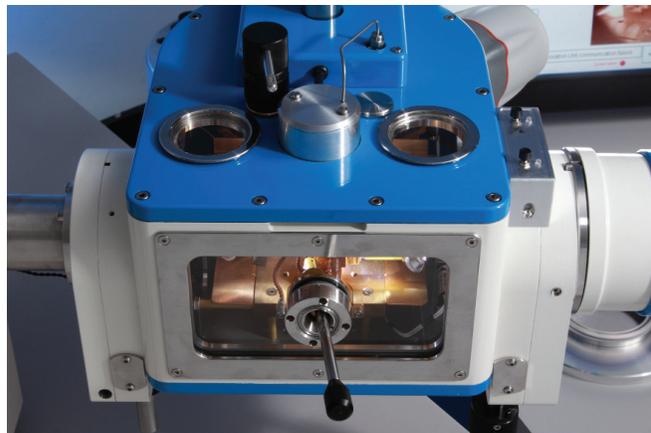
This image illustrates a stable emulsion of a synthetic liquid polymer dispersed in an aqueous continuous phase.

## Cryo-SEM Preparation Systems

### ● PP3000T Cryo-SEM Preparation System

#### Quick Overview

The PP3000T is a highly automated, easy to use, column-mounted, gas-cooled cryo preparation system suitable for most makes and models of SEM, FE-SEM and FIB/SEM. The PP3000T has all the facilities needed to rapidly freeze and transfer specimens. The cryo preparation chamber is turbomolecular pumped and includes tools for cold fracturing, controlled sublimation and specimen coating. The specimen can then be transferred onto a highly stable SEM cold stage for observation. Cold trapping in the cryo preparation chamber and SEM chamber ensures the whole process is frost free.



#### KEY FEATURES

- High resolution performance
- Large 'recipe' driven touch screen user interface
- Easy to use - extensive automation, on-screen help, videos etc
- Column-mounted preparation chamber - essential for frost-free transfer and ease of use
- Cold stage temperature down to  $-190^{\circ}\text{C}$ , plus comprehensive cold trapping
- Turbo pumping system mounted off-column - less mass on the SEM
- Unsurpassed specimen visibility - large front window, top viewing ports, multiple LED chamber lighting
- Cameras in the preparation chamber and SEM - cumbersome binocular not needed
- Preparation chamber cooling - eight-hour hold time with a single fill of liquid nitrogen
- Independent gas-cooling of the SEM cold stage and cold trap - temperature range down to  $-192^{\circ}\text{C}$  (not possible with conduction cooling)
- Off-column cooling of SEM stage and cold trap - 24 hour hold time
- Actively cooled fracturing and manipulation tools - micrometer knife and probe
- Twin liquid nitrogen slushing and specimen handling system - ideal for handling pre-frozen specimens
- Specialized support backed up with a three-year warranty

#### Product Description

*The PP3000T is a great leap forward in cryo-SEM technology. It combines the highest quality results with unparalleled ease of use*

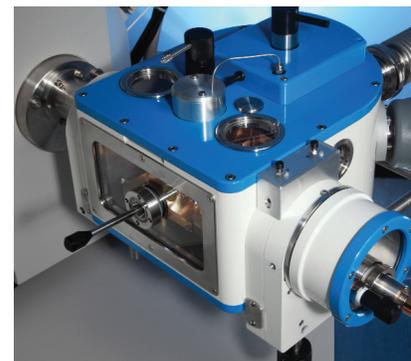
The PP3000T is a column-mounted, gas-cooled cryo preparation system suitable for use with SEM, FE-SEM and FIB/SEM instruments. Control is via a large and intuitive touch screen mounted on the spacious Prepdek™ workstation, giving the operator instant access to, and control of, all the key operating parameters.

Visibility is a key feature throughout the whole system. Chamberscope images from the preparation chamber and the SEM are displayed on the control screen - the image can be expanded to full screen when required. Five preparation chamber viewing windows give unsurpassed visibility of the specimen and chamber interior.

#### On-column preparation chamber with off-column cooling and pumping

The PP3000T conveniently combines the advantages of what are often referred to as 'on-column' and 'off-column' cryo preparation systems. The preparation chamber is directly attached to the SEM, but with the turbomolecular pumping and advanced SEM cooling system mounted remotely from the SEM. In this way, the mass and volume attached directly to SEM is kept to a minimum.

There are significant advantages of having the preparation chamber attached directly to the SEM. In particular, specimen transfer is always from high vacuum to high vacuum, which greatly reduces the risk of specimen contamination (frosting). In addition, it makes the system easier to use and allows the operator a more flexible approach to specimen preparation and observation. This is because during a single processing run it may be useful to move the specimen between the preparation chamber and the SEM cold stage - and vice versa - on a number of occasions.



PP3000T preparation chamber

## Cryo-SEM Preparation Systems

### ● PP3000T (Continued)

#### Prepdek™ workstation and touch screen user interface

The PP3000T is controlled using an intuitive colour touch screen, mounted on the user-friendly Prepdek™ workstation. The touch screen allows user-defined 'recipes' to be rapidly entered and stored for instant future access. The screen can be set to suit different operator levels and preferences - eg analogue or digital vacuum measurements.

Chamberscope images of both the preparation chamber and SEM cold stages are displayed and can be expanded to fill the screen. Although many of the key steps in the specimen preparation process are automated (airlock pumping, sublimation, sputter coating, etc), further help is instantly available through user-friendly videos. These guide the operator through the system set-up and then each specimen processing step in a concise and logical way.

#### Handling and transferring specimens

The PP3000T Prepdek™ workstation is fitted with a slushy nitrogen freezing station, connected to the pumping system. Rapid freezing reduces ice crystal damage, which results in improved ultra-structural preservation.

For handling pre-frozen material, the Prepdek™ is also fitted with the Advanced Specimen Handling System, which allows specimens that have been frozen by alternative freezing methods (or stored field specimens) to be manipulated in liquid nitrogen and then transferred under vacuum into the PP3000T preparation chamber for subsequent processing and observation.

The vacuum transfer device is compact, vacuum tight and has a convenient bayonet connection to the specimen shuttle to ensure rapid transfer. In line with the automatic design of the PP3000T, when the vacuum transfer device is located on the preparation chamber, the airlock is automatically pumped.

The PP3000T is supplied with universal 10mm specimen stubs with surface slots, holes and a flat area - useful for most specimen types, because the holes and slots can be used for liquids and to hold solid material for cross-section fracturing. Blank stubs are also included. A range of optional holders are available, including shuttles for large specimens and top-loading holders for high pressure freezing rivets and planchettes.

#### Cryo preparation chamber

The PP3000T preparation chamber is connected directly to the SEM and includes facilities for preparing all types of specimens. Specimen stage cooling is by an integral liquid nitrogen dewar which has an all-day run time on a single fill (0.75L) of liquid nitrogen. The cold stage is connected directly to the dewar and includes an integrated heater and sensor to precisely control the temperature from -190°C to over 50°C. Comprehensive cold traps surround the cold stage.

#### High visibility

The PP3000T has superb chamber visibility. In addition to the large front window there are additional top and side windows. The specimen stage is lit by four LEDs, which avoids the problem of the fracturing knife 'shadowing' the specimen during cryo fracturing.

A chamberscope allows the specimen stage to be viewed on the control touch screen. Twin manipulators (actively cooled) allow a range of specimen types to be fractured.

#### Automatic sublimation and sputtering

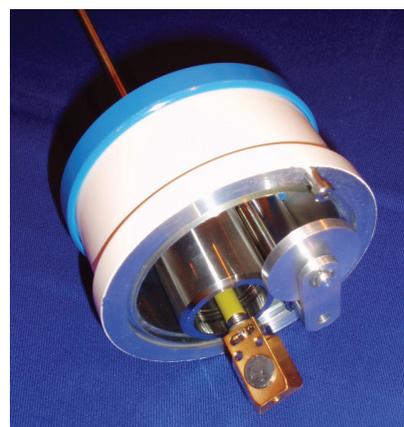
Sublimation and sputtering are fully automatic. The high resolution sputter coater is specifically designed for cryo applications and will give fine grain films that are essential for FE-SEM applications. A platinum (Pt) target is fitted as standard; other metals include gold (Au), gold/palladium (Au/Pd), chromium (Cr) and iridium (Ir). An optional carbon fibre evaporation head can be fitted.

#### Cryo preparation chamber pumping

The preparation chamber is pumped by a remotely-positioned 70L/s turbomolecular pumping system. Typical preparation chamber vacuums during operation are in the region of  $10^{-6}$  mbar or better. Positioning the turbomolecular pump away from the SEM ensures total elimination of mechanical vibration and significantly reduces the cryo system mass that is connected to the SEM. A vacuum buffer tank allows the rotary pump to be automatically switched off for most of the time. The pumping system is connected to the preparation chamber by flexible stainless-steel bellows.



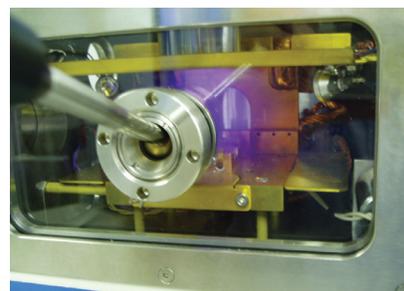
PP3000T Cryo-SEM Preparation System chamber and touch screen control monitor



Cryo transfer device with specimen shuttle and stub insert



Fracturing Knife



Front window and fracturing knife

continued >>>>

## Cryo-SEM Preparation Systems

### ● PP3000T Cryo-SEM Preparation System (Continued)

#### SEM cold stage, cold trap and cooling system

A highly stable, thermally isolated, liquid nitrogen gas-cooled stage attaches to the SEM stage. The SEM stage and cold trap are cooled by two separate cold gas circuits - both capable of reaching temperatures down to  $-192^{\circ}\text{C}$ . This configuration allows the operator to select stage and cold trap temperatures that are optimised for specific specimens. For example, for some non-biological materials it is useful to hold the specimen at very low temperatures - a cold stage temperature of  $-175^{\circ}\text{C}$  and a cold trap temperature of  $-192^{\circ}\text{C}$ .

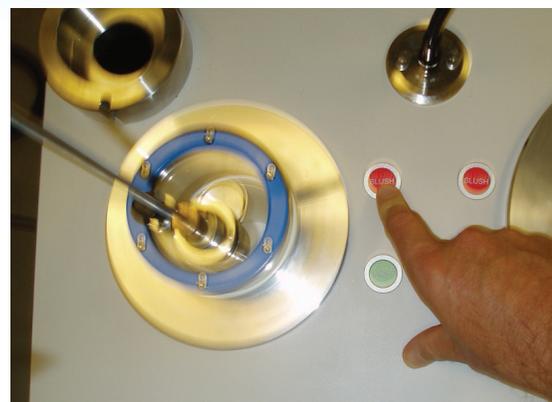
#### Off-column cooling

The cold nitrogen gas-cooling dewar for the SEM stage and cold trap is remotely positioned (typically on the floor behind the SEM). The system will run for up to 24 hours between fills.

#### Additional Information:

#### Specifications:

PP3000T	Fitted as standard?
Cryo preparation chamber (column-mounted). Consisting of:	
Built-in liquid nitrogen cooling dewar with an eight-hour run time between fills	Yes
Two integral gate valves (loading and SEM) with appropriate electrical interlocks. Air lock pumping is automatically activated when the specimen transfer device is attached	Yes
Variable temperature conduction cooled specimen stage ( $-185^{\circ}\text{C}$ to $50^{\circ}\text{C}$ )	Yes
Cold stage 'parking space' for a second specimen shuttle (allows a second specimen to be prepared in advance)	Yes
Large anti-contaminator (cold trap) plates above and below cold stage	Yes
Robust micrometer-fed fracturing knife (actively cooled)	Yes
Side-mounted surface knife/probe (actively cooled). A range of scalpel blades can be fitted to suit different specimen requirements	Yes
Automatic sublimation (controlled and viewed on the touch screen)	Yes
Fully automatic, high resolution sputter coater with platinum (Pt) target. (Other targets, including gold (Au), gold/palladium (Au/Pd), chromium (Cr) and iridium (Ir), are available as options.) Sputtering controlled and viewed on the user touch screen	Yes
High purity argon gas canister (1L)	Yes
Carbon fibre evaporation head and power supply	Option
Large front viewing window (138 x 73mm) plus top and side viewing ports	Yes
Preparation chamber camera (chamberscope) mounted on a side port	Yes
Chamberscope viewing port shutter - automatically closes during sputtering to ensure the port window is clear of sputtered material	Yes
Vacuum transfer device	Yes
Chamber illumination - four LEDs	Yes
Pumping system	
Remotely-mounted turbomolecular-pumping system (70L/s). Includes: anti-vibration base, vacuum buffer tank, vacuum valves and stainless-steel bellows connection to the preparation chamber. Typical preparation chamber vacuum: 10-6 mbar	Yes
Single 90L/m rotary pump required	Order separately
SEM cooling dewar, SEM cold stage and cold trap (anti-contaminator)	
Gas-cooled nitrogen cold stage assembly ( $-192^{\circ}\text{C}$ to $50^{\circ}\text{C}$ ). Temperature stability of $> 1^{\circ}\text{C}$	Yes
Separate gas-cooling circuits for SEM stage and SEM anti-contaminator	Yes
CHE2000 12L capacity, off-column cooling dewar with run time between fills of up to 24 hours	Yes
SEM chamberscope - viewed on the system control screen	Yes
LED lighting (interlocked)	Yes
System control and specimen handling	
Control via a colour user touch screen monitor (300mm/12") mounted on the Prepdek® specimen preparation station.	
<b>Featuring:</b>	
■ Multi-ability user interface screen (expert/novice)	
■ Quick, easy overview of system status	
■ User-definable 'recipes' can be stored	
■ Quick access to videos outlining preparation techniques and system maintenance	
■ Fully automatic sputtering	
■ Automatic sublimation	
■ Quick, easy overview of system status	
■ CCD camera image of preparation chamber and microscope chamber	
Twin liquid nitrogen slushing and specimen handling system - ideal for handling pre-frozen specimens. Mounted on the Prepdek®	Yes
System electronics stored in a ventilated, sealed unit under the Prepdek®	Yes
Specimen shuttles (x2). E7449-9 multi-specimen stubs (pack of 10) and E7402 blank aluminium (Al) stubs (pack of 10).	Yes
<b>Other shuttles and stubs available - see Ordering Information</b>	



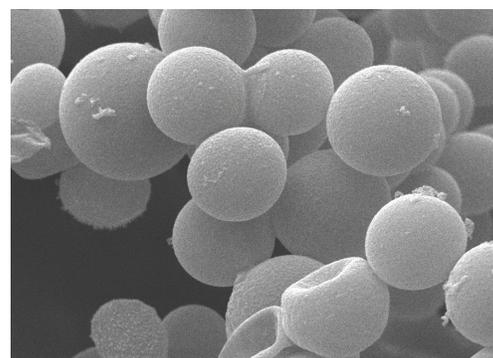
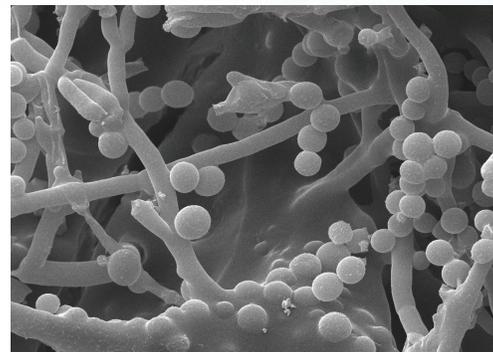
# Cryo-SEM Preparation Systems

## ● PP3000T (Continued)

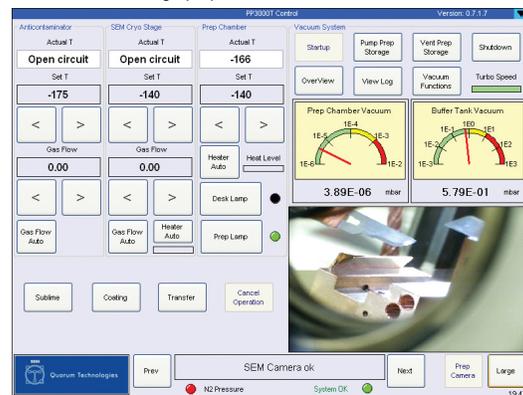
Ordering Information		
<b>NB: For a full quotation, including on-site installation and customer training, please contact us.</b>		
<b>PP3000T</b>	Cryo-SEM preparation system for SEM, FE-SEM and DualBeam applications. Including: column-mounted cryo-preparation chamber with off-column turbo pumping system. SEM cold stage and cold trap, Prepdek™ workstation with dual freezing and specimen manipulation facilities, touch screen user interface mounted on the Prepdek™ workstation. Transfer device, 2 x AL200077B and 1 x 10246 specimen shuttles, E7449-5 and E7402 specimen stubs. Microscope interfaces, start-up kit and operation manual	each
<b>Pumping</b>	The PP3000T requires one 90L/m rotary pump (dry pumps available on request)	each
<b>E5005G</b>	RV5 90L/m 115/230V 50/60Hz rotary vacuum pump with oil mist filter	each

Options and accessories		
<b>PP7450</b>	Pressurised dewar (30L) for LN <sub>2</sub> storage and venting gas supply	each
<b>10998</b>	Carbon fiber evaporation head including 1m high purity carbon fiber	each
<b>10999</b>	Film thickness monitor (FTM)	each
Specimen holders		
<b>10245</b>	Top-loading specimen shuttle for planchettes	each
<b>10246</b>	Top-loading specimen shuttle, to take a 10mm stub	each
<b>10247</b>	Top-loading specimen shuttle for rivets (vice style)	each
<b>E7433</b>	Rivet holder specimen stub, screw-down style (for use with 10246)	each
<b>E7449-5</b>	Universal specimen stub with surface holes and slots (pack of 5)	5/pk
<b>AL200077B</b>	Specimen stub shuttle (spare)	each
<b>E7402</b>	Aluminum (Al) stubs (pack of 10)	10/pk
<b>E7403</b>	Copper (Cu) stubs (pack of 10)	10/pk
<b>E7405</b>	Screw down stub for thin, hard specimens	each
<b>E7406</b>	Copper (Cu) stubs with 3 x 3mm slots (pack of 5)	5/pk
<b>E7407</b>	Copper (Cu) stubs with 1 x 3mm slot (pack of 5)	5/pk
<b>32816510</b>	Brass rivets for fracturing liquids (pack of 100)	100/pk
Sputter targets		
<b>E7400-314A</b>	Gold (Au) target 0.2mm thick	each
<b>E7400-314B</b>	Gold/palladium (Au/Pd) (80:20) target 0.2mm thick	each
<b>E7400-314C</b>	Platinum (Pt) target 0.2mm thick	each
<b>E7400-314IR</b>	Iridium (Ir) target 0.3mm thick	each
<b>E7400-314CR</b>	Chromium (Cr) target 0.3mm thick	each

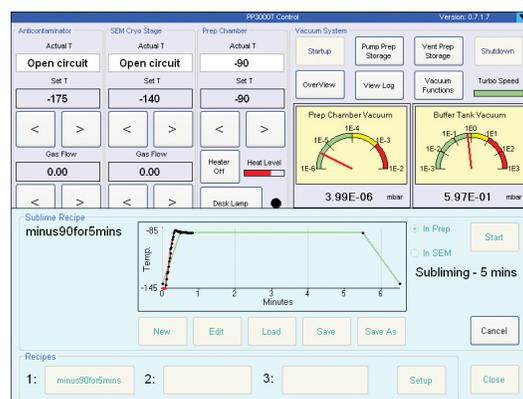
Support and other information	
Installation and training	
Installation and training at the customer site	Yes
Support and other information	
Comprehensive start-up kit with key spares	Yes
Three-year warranty	Yes
SEM column interfaces and SEM stage adaptor (tailored to each microscope)	Yes
Some options and accessories (see Ordering Information for full list)	
Terminating film thickness monitor (FTM)	Option
Self-pressurising LN <sub>2</sub> dewar and regulator (for storage and venting)	Option
Carbon fibre evaporation head	Option
Wide range of specimen holders and specimen stubs	Option



Cryo prepared image of Blue stilton cheese (Penicillium roqueforti). Image prepared on a CamScan SEM.



Main display screen with all operational parameters visible to the user at all times



Sublimation - touch screen control allows specific profiles to be set

## Vacuum Pumps and Accessories

### ● Edwards RV Series Vacuum Pumps

*Rotary vacuum pumps — double stage.*



### ● Edwards E2M1.5 Vacuum Pumps

This miniature two stage pump features an alternative inlet connection position at the side of the pump so that overall installation height can be reduced to a minimum when required.

- 1.3 cfm (190L/min) displacement
- Ultimate vacuum (without gas ballast):  $1.1 \times 10^{-3}$  torr.
- Max. inlet pressure for water vapor: 11.3 torr
- Max. water vapor pumping rate: 0.035 lb/hr
- Oil capacity Max/Min: 0.28L/0.2L

Ultra Grade 19 Oil is recommended for use with the E2M1.5. Weight: 50 lbs (22kg)

91004	Vacuum Pump E2M1.5	each
91004-E	Vacuum Pump E2M1.5, 220 Volts	each

### ● RV3 Vacuum Pump

- Speed - (Pneurop 6602)
- 50 Hz operation - 2 ft3min-1 (3.3 m3h-1)
- 60 Hz operation - 2.3 ft3min-1 (3.9 m3h-1)
- Ultimate Vacuum (Total Pressure) - High Vacuum Mode -  $2 \times 10^{-3}$  mbar
- Inlet connection - NW25

In high vacuum mode it is ideal for backing turbo pumps, analytical instruments, and electron microscopes. Configurable for high throughput mode. Weight: 43 lbs (19.6 kg).

91003	Vacuum Pump RV3	each
91003-E	Vacuum Pump RV3, 220 Volts	each

### ● RV5 Vacuum Pump

- 4.1 cfm (117L/min) displacement
- Ultimate vacuum (without gas ballast)  $1.5 \times 10^{-3}$  torr.
- Max. inlet pressure for water vapor: 38 torr
- Max. water vapor pumping rate: 0.48 lb/h
- Motor: 1/2 hp
- Oil capacity Max/Min: 0.7L/0.42L

Ultra Grade 19 Oil is recommended for use with the RV5.

Weight 43 lbs (19.6kg)

91005	Vacuum Pump RV5	each
91005-E	Vacuum Pump RV5, 220 Volts	each

### ● RV8 Vacuum Pump

- 6.9 cfm (195 L/min) displacement
- Ultimate vacuum (without gas ballast)  $1.5 \times 10^{-3}$  torr.
- Max. inlet water pressure vapor: 29 torr
- Max. water vapor pumping rate: .48 lb/h
- Motor: 3/4 hp
- Oil capacity Max/Min: 0.75L/0.43L



Ultra Grade 19 Oil is recommended for use with the RV8. Weight 50 lbs (24 kg).

91025	Vacuum Pump RV8	each
91025-E	Vacuum Pump RV8, 220 Volts	each

### ● Foreline Traps

These rechargeable in-line traps effectively block oil back-streaming from mechanical pumps. Any mechanically pumped system, even one with a turbo pump, should have a foreline trap. The trap is placed in the foreline between the roughing pump and the diffusion or turbo pump. It uses a disposable, oxygen-free copper maze absorbent which requires no liquid nitrogen and will not hold water. Typically the maze is replaced every 1-2 years. The seamless body is available in aluminum.



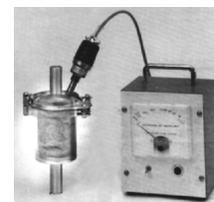
- Quick change clamp for replacement of maze
- Viton "O" ring end cap
- Recessed screen
- Available in five pipe diameters
- Replacement elements (Copper Maze) also available

Catalog Number	Pipe Diameter	Body* Material	Pump Capacity	Body Diameter
91050-10	1/2"	Aluminum	5 CFM	2 1/2"
91050-15	3/4"	Aluminum	5 CFM	2 1/2"
91050-20	1"	Aluminum	10 CFM	3"
91050-25	1 1/2"	Aluminum	10 CFM	3"
91050-30	1 3/4"	Aluminum	15 CFM	4"

\* Available in stainless steel upon special request.

### ● Vacuum Monitor

This vacuum monitor allows you to monitor the vacuum level in the foreline between the roughing pump and the diffusion (or turbo) pump. A high value would indicate a leak in the system or possibly an inefficient roughing pump. Comprised of a thermocouple gauge control module and cable. The unit can mount directly to the Foreline Trap via a thermocouple (TC) port supplied at no charge when ordered with a Foreline Trap. The 3-station version of the Vacuum Monitor allows the vacuum levels of several pumps to be measured.



91052-01	Vacuum Monitor	each
----------	----------------	------

### ● Ultra Grade 19 Oil

Edwards Ultra Grade 19 Oil is specially recommended for use in clean pumping applications. The oil offers reduced maintenance costs, longer fluid life, and cooler running pumps.

60740	Edwards Ultra Grade 19 Oil	1 L
60742	Edwards Ultra Grade 19 Oil	4 L

# Evaporation Supplies

## ● Carbon Rods/Graphite Rods

### Properties of Carbon

**Name:** Carbon      **Symbol:** C  
**Atomic number:** 6    **Atomic weight:** 12.0107  
**Group number:** 14   **Period number:** 2  
**Block:** p-block      **CAS I.D.:** 7440-44-0  
**Specific gravity (graphite):** 2.2 g/cm<sup>3</sup>  
**Specific gravity (amorphous):** 1.88 g/cm<sup>3</sup>  
**Specific gravity (diamond):** 3.51 g/cm<sup>3</sup>  
**m. p. sublimes:** ~3550°C      **Boiling point:** 4200°C  
**Evaporation Temp (at which a substance has a vapor pressure of 1.33 x 10<sup>-2</sup> mbar):** 2400°C

Graphite is the stable form of carbon. Graphite is one of the softest; diamond is one of the hardest minerals known to man. Carbon is harder than graphite. For TEM and SEM applications, graphite is recommended.

Carbon /graphite films are most usable in TEM and SEM due to its value characteristics: uniformity amorphous and high transparent to electron beams. Because of low molecular weight and their unique structure (graphite have a sheet like structure, where the atoms all lie in a plane), carbon/graphite films have excellent mechanical stability, and even film thickness is about 1 – 2 nm.

Our carbon/graphite rods are CVPTM (Chemical Vapor Process) purity and spectrographic grade, grade 1, produced to ASTM tolerances or even tighter as well we have a technical grade. The technical grade has a typical impurity level of 50ppm or greater. Technical grade may be used in applications of non critical SEM coating applications.

## ● PreSharpened and Pointed Carbon Rods



To make your work easier we invite you to order our pre-sharpened rods. All of our line of Pre Sharpened Carbon Rods are made from our Spectroscopically Pure Carbon ( Graphite)

### Single Point:

**Rod Length:** 2 3/8" (60.3mm)  
**Neck Length:** 0.205" (5.2mm)  
**Neck Diameter:** .045" (1.14 mm)

<b>70220-01</b>	Single Point Carbon Rods, 1/8" dia. x 2 3/8" L (3 x 60.3mm)	50/pack
<b>70221-01</b>	Single Point Carbon Rods, 1/4" dia. x 2 3/8" L (6.2 x 60.3mm)	50/pAck

### Double-Pointed Carbon Rods

<b>70220-45</b>	(1/8") dia. x 2 1/4" long (3.05 x 57.1mm) Tip: .039" dia. (1.0mm), neck type tips	10/pack
<b>70220-50</b>	(3/16") dia. x 2 1/2" long (4.6 x 63.5mm) Tip: .039" dia. (1.0mm), neck type tips	50/pack
<b>70221-40</b>	1/4" dia. x 2" long (6.4 x 50.8mm) Tip: .039" dia. (1.0mm), neck type tips	50/pack
<b>70221-60</b>	1/4" dia. x 1 1/2" long (6.4 x 38mm), Tip: .056" dia. (1.4mm), neck type tips	10/pack



Density g/cc .....	1.85
Shore Scleroscope Hardness.....	45
Resistivity Ohms-in .....	4.0 x 104
Ash Content, ppm .....	<2
Flexural Strength lb/in2 .....	8000
Porosity.....	16.5%

## ● A. Carbon Rods: Spectro-Grade

Carbon Rods (graphite) with an impurity level of 2ppm or less with a maximum level element of each of 1 ppm. These rods are ideal for all TEM, SEM and all other critical applications.

<b>70200</b>	Carbon Rods, Spectro Grade, 1/8"(3mm) x 12" (304mm)	12/pack
<b>70231</b>	Carbon Rods, Spectro-Grade, 3/16"(4.6mm) x 12" (304mm)	12/pack
<b>70230</b>	Carbon Rods, Spectro-Grade, 1/4"(6.2mm) x 12" (304mm)	12/Pack

## ● B. Carbon Rods: Technical Grade

<b>70301</b>	Carbon Rods, Technical Grade, 1/8"(3mm) x 12" (304mm)	12/pack
<b>70302</b>	Carbon Rods, Technical Grade, 1/4"(6.2mm) x 12" (304mm)	12/Pack

## ● Carbon Cord/Carbon Fiber

Sometimes referred to as 'braid', carbon fibre consists of eight separate strands of individual carbon fibre braided together. The fibre is manufactured so that, when cut, the ends resist unravelling. The fibre density is about 1.6g/m



Used With: Most carbon coaters where thick carbon cord is used

<b>91047-1</b>	Carbon Fibre Cord - high purity (1m)	each
<b>91047-5</b>	Carbon Fibre Cord - high purity (5m)	each

Convenient to use, giving thick coating using a single length. 1m length

<b>91046-1</b>	Carbon Fibre Cord - standard grade (1m)	each
<b>91046-10</b>	Carbon Fibre Cord - standard grade (10m)	each
<b>91046-100</b>	Carbon Fibre Cord - standard grade (100m)	each

Description: Convenient to use, giving thin to medium coating using a single strand or several lengths. 1m length

<b>91045</b>	Carbon Fibre - fine strands (1m)	each
<b>91045-10</b>	Carbon Fibre - fine strands (10m)	each
<b>91045-100</b>	Carbon Fibre - fine strands (100m)	each

## ● Pyrolytic Graphite Foil Boat

### Boat dimensions:

Thickness of pyrolytic graphite foil is 0.20 mm (0.020") Overall length is 32mm (1.25") and width is 9.6mm (0.375")

### Trough Dimension:

12.7 mm (L) x 9.0 mm (W) x 3.2 mm (D) (0.5 x 0.355 x 0.03")

<b>73808-B1</b>	Pyrolytic Graphite Boat B1	5/pack
-----------------	----------------------------	--------

## Evaporation Supplies

### ● Carbon Rod Sharpener



Produces 1mm diameter carbon tip on 1/4" or 1/8" carbon rods. Two carbide blades shape the tip. A gauge is included for adjusting the blade gap. Dust catcher is easily removed for cleaning or blade adjustment. The device is driven by a durable electric motor, producing a smoother and more uniform carbon tip than any hand sharpener.

The kit includes a blade gauge and a 1/8" rod adapter.

70305-10	Electric Carbon Rod Sharpener, 110V	each
70305-20	Electric Carbon Rod Sharpener, 220V	each

### ● Manual Carbon Rod Sharpener

Used to sharpen rods into 1 millimeter cylindrical tips



70305-60	Carbon Rod Sharpener (1/8" [3mm] rods)	each
70305-65	Carbon Rod Sharpener (1/4" [6.2mm] rods)	each
70305-70	Cutter Replacement for Carbon Rod Sharpener	each

### ● Hand Tool For Creating Two Step Carbon Rods

Comes complete with brass adjustment tool.



70305-40	Two Step Carbon Rod Sharpener, for 1/4" rods	each
70305-45	Replacement Blades for, for 1/4" rods	each

### ● Tungsten Metal Wire and Boats

Tungsten has the highest melting point and lowest vapor pressure of all metals, and at the temperatures over 1650°C has the highest tensile strength. It has excellent corrosion resistance and is attacked only slightly by most mineral acids.

Tungsten is the most useful material used in vacuum evaporation technique, due to its special properties and cost factor.

Chemical symbol: .....W

#### Atomic Structure:

Atomic Radius: .....2.02Å  
 Atomic Volume, cm<sup>3</sup>/mol: .....9.53  
 Covalent Radius: .....1.3 Å  
 Crystal Structure: ..... Body centered cube  
 Number of Electron (with no charge): .....74  
 Atomic Number of Protons: .....74  
 Number of Neutrons (most common/stable nuclide): .....110  
 Ionic Radius: .....0.62Å  
 Cross Section: .....18.5barns ± 0.5

#### Chemical Properties:

Electrochemical Equivalent: .....1.1432g/amp-hr  
 Electron Work Function: .....4.55eV  
 Electronegativity (Pauling): .....2.36  
 Heat of Function: .....35.4kJ/mol  
 Incompatibilities: ..... Bromine trifluoride, Chlorine trifluoride, Fluorine, Iodine pentafluoride  
 Valence Electron Potential (-eV): .....140

#### Physical Properties:

Atomic Weight: .....183.86  
 Boiling Point: .....5928K, 5655°C, 10211°F  
 Coefficient of Lineal Thermal Expansion: .....0.0000046cm/cm/°C (0°C)  
 Electrical Conductivity: .....0.189 106/cm  
 Thermal Conductivity: .....1.74 W/cmK  
 Density @ 20°C (gm/cc @ 300°K): .....19.35  
 Melting Point/Freezing Point: .....3680K, 3407°C, 6165°F  
 Enthalpy of Atomization: .....837 kJ/mole @ 25°C  
 Enthalpy of Fusion: .....35.23kJ/mole  
 Enthalpy of Vaporization: .....799.1 kJ/mole  
 Hardness Scale: ..... Brinell 2570MN m<sup>-2</sup>, Mohs 7.5; Vickers 3430 MN m<sup>-2</sup>  
 Heat of Vaporization: .....824 kJ/mol  
 Optical Reflectivity: .....62%  
 Vapor Pressure = .....4.27Pa@3407°C  
 CAS Number: .....7440-33-7  
 RTECS: .....Y0717500  
 OSHA Permissible Exposure Limit (PEL): .....No limits set by OSHA  
 TWA: .....5 mg/m<sup>3</sup>; STEL: 10mg/m<sup>3</sup>

#### Route of Exposure:

Inhalation, Ingestion, skin, and/or eye

#### Target Organs:

Eyes, Skin, Respiratory system, Blood

# Evaporation Supplies

## 1. Tungsten Wire

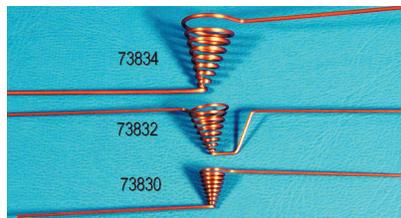
We are offering tungsten wire in 99.90% purity. Available in 0.025" (.64mm), 0.02" (.51mm), and 0.009" (0.23mm) diameters.



<b>73800</b>	Tungsten Wire, 0.020" (0.51mm) Diameter	20 ft
<b>73801</b>	Tungsten Wire, 0.020" (0.51mm) Diameter	100 ft
<b>73802</b>	Tungsten Wire, 0.025" (0.64mm) Diameter	20ft
<b>73803</b>	Tungsten Wire, 0.025" (0.64mm) Diameter	100ft
<b>73804</b>	Tungsten Wire, 0.009" (0.25mm) Diameter	20ft
<b>73805</b>	Tungsten Wire, 0.009" (0.25mm) Diameter	100ft

## 2. Tungsten Wire Baskets

Clean, vacuum grade, tungsten wire.

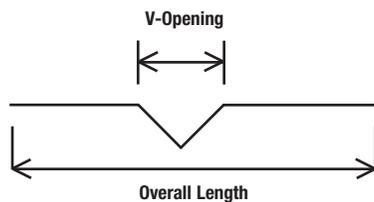


Basket Type	Turns	I.D. mm	Height mm	Wire Lead cm	Wire Dia. inches
A	9	4	7	3.5	0.020
C	8	7	9	5.0	0.020
D	8	9	14	4.0	0.030

<b>73830</b>	Tungsten Wire basket A	50/pack
<b>73832</b>	Tungsten Wire basket C	50/pack
<b>73834</b>	Tungsten Wire basket D	50/pack

## 3. Tungsten Wire Filaments



### V-Shaped Filaments

High purity, vacuum grade tungsten wire 0.02" (0.50mm) diameter, V-shaped filaments 1/2" (12.7mm) deep, with a bottom angle of 45°, either single wire or triple strand.

Cat. #	Overall Length	No. Strand	V-Opening	Qty
<b>73840</b>	3" (76.2mm)	Single	3/4" (19mm)	10/pk
<b>73842</b>	3" (76.2mm)	Triple	3/4" (16mm)	10/pk
<b>73844</b>	3 1/2" (88.9mm)	Triple	3/4" (19mm)	10/pk

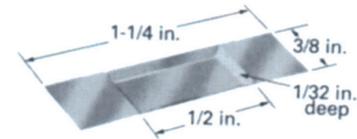
### Coiled Filaments

Single or 3-strand coiled tungsten filaments, 0.5mm (0.002") diameter wires.

Cat.#	Description	Qty
<b>73938-1</b>	4" long Horizontal Helix, Coil length 45mm (1 3/4") with 6 turns, 6.3mm (1/4") diameter coil. Single strand	5/pk
<b>73938-3</b>	4" long Horizontal Helix, Coil length 45mm (1 3/4") with 6 turns, 6.3mm (1/4") diameter coil. Triple strand	5/pk
<b>73939-1</b>	5" long Horizontal Helix, Coil length 51mm (2") with 12 turns, 9.5mm (3/8") diameter coil. Triple strand	5/pk

## Tungsten Foil Boats

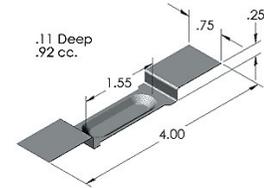
Tungsten Boat B1: Thickness of tungsten foil is 0.05mm (0.002"), overall length is 32mm (1 1/4").



The trough: 12.7mm(L) x 9.5mm(W) x 3.2mm Deep (1/2" x 3/8" x 1/32")

<b>73816</b>	Tungsten Boat B1	5/pk
--------------	------------------	------

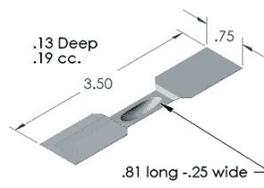
Tungsten Boat B2: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 19.05mm (4" x 3/4").



Trough: 39.37mm (L) x 2.79mm Deep, 0.92 ml

<b>73816-B2</b>	Tungsten Boat B2	5/pk
-----------------	------------------	------

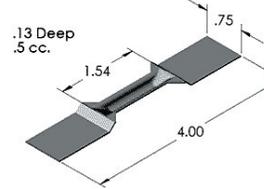
Tungsten Boat B3: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 19.05mm (4" x 3/4").



Trough: 39.1mm(L) x 6.35mm(W) x 3.3mm Deep, 0.5ml

<b>73816-B3</b>	Tungsten Boat B3	5/pk
-----------------	------------------	------

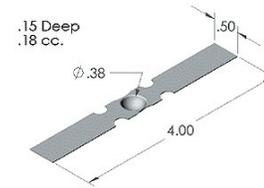
Tungsten Boat B4: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 100.5mm x 19.05mm (3 1/2" x 3/4").



Trough: 28.19mm(L) x 15.87(W) x 3.3mm Deep, 0.99ml

<b>73816-B4</b>	Tungsten Boat B	5/pk
-----------------	-----------------	------

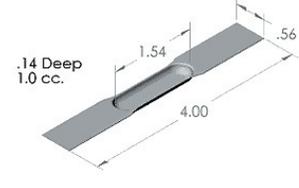
Tungsten Boat B5: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 12.7mm (4" x 1/2").



Trough: 9.65mm Diameter x 3.81mm Deep, 0.18ml

<b>73816-B5</b>	Tungsten Boat B5	5/pk
-----------------	------------------	------

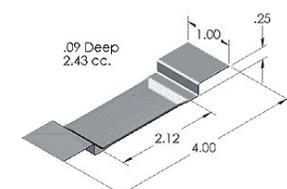
Tungsten Boat B6: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 39.1mm(L) (4" x 1 1/2")



Trough: 39.1mm(L) x 12mm(W) x 3.55mm Deep, 1.0ml

<b>73816-B6</b>	Tungsten Boat B6	5/pk
-----------------	------------------	------

Tungsten Boat B7: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 25.4mm(L) (4" x 1")



Trough: 53.84mm(L)x25mm(W) x 2.28mm Deep, 2.43ml

<b>73816-B7</b>	Tungsten Boat B7	5/pk
-----------------	------------------	------

## Evaporation Supplies

### ● Metal Boats

For disc aperture cleaning, vacuum evaporation. Thickness: 0.05mm (0.002"). Overall Length: 75mm (3").  
Trough measures: 12mm (1/2") long x 5mm (3/16") wide x 1mm deep.



73810	Molybdenum Boat	5/pack
73812	Platinum Boat	1/pack
73814	Tantalum boat	5/pack

### ● Metal Foils

For aperture cleaning, vacuum evaporation.

73820	Molybdenum Foil 0.002" (0.05mm) thick; size 6x6" (15.2x15.2cm)	each
73822	Tantalum Foil 0.003" (0.075mm) thick; size 6x6" (15.2x15.2cm)	each

### ● Heavy Duty Wire Bending Pliers

These extra heavy-duty pliers are used for bending any metal wire at a 'V' shaped angle, allowing for the formation of filaments in accordance to your specification.



62185	Wire Bending Pliers, 6 1/2" (165mm) Long	each
-------	------------------------------------------	------

### ● Precious Metal Wires

High purity, vacuum grade. These metal wires are suitable for scanning and electron microscopy techniques.



#### Aluminum Wire: Purity: 99.99%

73000	Aluminum Wire, Diameter: 0.010" (0.25mm)	10 ft
73001	Aluminum Wire, Diameter: 0.010" (0.25mm)	50 ft

#### Gold Wire: Purity: 99.99%

73100	Gold Wire, Diameter: 0.008" (0.20mm)	10 ft
73101	Gold Wire, Diameter: 0.008" (0.20mm)	50 ft

#### Molybdenum Wire: Purity: 99.95%

73200	Molybdenum Wire, Diameter: 0.008" (0.20mm)	10 ft
73201	Molybdenum Wire, Diameter: 0.008" (0.20mm)	50 ft

#### Palladium Wire: Purity: 99.90%

73300	Palladium Wire, Diameter: 0.008" (0.20mm)	10 ft
73301	Palladium Wire, Diameter: 0.008" (0.20mm)	50 ft

#### Palladium/Gold 40:60, Purity: 99.90%

73400	Palladium/Gold wire, Diameter: 0.008" (.20mm)	10 ft
73401	Palladium/Gold wire, Diameter: 0.008" (.20mm)	50 ft

#### Platinum Wire, Purity: 99.95%

73500	Platinum Wire, Diameter: 0.008" (0.20mm)	10 ft
73501	Platinum Wire, Diameter: 0.008" (0.20mm)	50 ft

#### Platinum/Palladium, 80:20, Purity 99.95%

73600	Platinum/Palladium, Diameter: 0.008" (0.2mm)	10 ft
73601	Platinum/Palladium, Diameter: 0.008" (0.2mm)	50 ft

#### Silver Wire, Purity: 99.99%

73700	Silver wire, Diameter: 0.008" (0.2mm)	10 ft
73701	Silver wire, Diameter: 0.008" (0.2mm)	50 ft

#### Tungsten Wire, Purity: 99.90%

73800	Tungsten wire, Diameter: 0.02" (0.51mm)	10 ft
73801	Tungsten wire, Diameter: 0.02" (0.51mm)	100 ft

# Sputter Targets

## ● Sputter Targets

EMS precious metal sputtering targets are made from high purity metals, starting 99.99% with most standard disc sizes, for use on most sputtering units from many manufacturers: Emitech, Emscope, Bio-Rad, Polaron, Edwards, Balzers, Plasma Sciences, Technics Hummers, Denton, Cressington, and much more.

Our targets comes in a standard thickness of 0.1mm (100µm). Other thickness, ranging from 0.05 to 6mm, are available upon request. Targets are available mounted or unmounted whichever you prefer. For mounting please always state the make and the model of the sputtering unit so we can tend to it for you . Targets in other materials such as Chromium, Iridium etc are available upon request. We also can manufacture targets with diameters from 4 mm to 304.8mm (12"). Call for pricing.

All other materials and sizes are available upon request for all other machines. The standard thickness for all targets is 0.1mm All other thicknesses are available on request The most commonly requested is 0.2, 0.3mm but all others can be made on special request



Target Diameter, mm	EMS Catalog Number								
	Gold (Au)	Platinum (Pt)	Palladium (Pd)	Au/Pd 60:40	Pt/Pd 80:20	Silver (Ag)	Nickel (Ni)	Iridium (Ir)	Copper (Cu)
20.0	91006-Au	91006-Pt	91006-Pd	91006-AP	91006-PP	91006-Ag	91006-Ni	91006-Ir	91006-Cu
20.4	91007-Au	91007-Pt	91007-Pd	91007-AP	91007-PP	91007-Ag	91007-Ni	91007-Ir	91007-Cu
32.0	91008-Au	91008-Pt	91008-Pd	91008-AP	91008-PP	91008-Ag	91008-Ni	91008-Ir	91008-Cu
39.0	91009-Au	91009-Pt	91009-Pd	91009-AP	91009-PP	91009-Ag	91009-Ni	91009-Ir	91009-Cu
42.0	91013-Au	91013-Pt	91013-Pd	91013-AP	91013-PP	91013-Ag	91013-Ni	91013-Ir	91013-Cu
50.0	91014-Au	91014-Pt	91014-Pd	91014-AP	91014-PP	91014-Ag	91014-Ni	91014-Ir	91014-Cu
50.8	91015-Au	91015-Pt	91015-Pd	91015-AP	91015-PP	91015-Ag	91015-Ni	91015-Ir	91015-Cu
54.0	91016-Au	91016-Pt	91016-Pd	91016-AP	91016-PP	91016-Ag	91016-Ni	91016-Ir	91016-Cu
57.0	91017-Au	91017-Pt	91017-Pd	91017-AP	91017-PP	91017-Ag	91017-Ni	91017-Ir	91017-Cu
60.0	91010	91012	91010-Pd	91011	91010-PP	91010-Ag	91010-Ni	91010-Ir	91010-Cu
63.5	91018-Au	91018-Pt	91018-Pd	91018-AP	91018-PP	91018-Ag	91018-Ni	91018-Ir	91018-Cu
75.0	91019-Au	91019-Pt	91019-Pd	91019-AP	91019-PP	91019-Ag	91019-Ni	91019-Ir	91019-Cu
76.0	91020-Au	91020-Pt	91020-Pd	91020-AP	91020-PP	91020-Ag	91020-Ni	91020-Ir	91020-Cu
<b>Annular Targets (Outside Diameter x Inside Diameter, mm)</b>									
1170D x 89 ID	91030-Au	91030-Pt	91030-Pd	91030-AP	91030-PP	91030-Ag	91030-Ni	91030-Ir	91030-Cu
87 OD x 57 ID	91031-Au	91031-Pt	91031-Pd	91031-AP	91031-PP	91031-Ag	91031-Ni	91031-Ir	91031-Cu
59 OD x 33 ID	91032-Au	91032-Pt	91032-Pd	91032-AP	91032-PP	91032-Ag	91032-Ni	91032-Ir	91032-Cu
57 OD x 40 ID	91033-Au	91033-Pt	91033-Pd	91033-AP	91033-PP	91033-Ag	91033-Ni	91033-Ir	91033-Cu

Target Diameter, mm	EMS Catalog Number								
	Chromium (Cr)	Tungsten (W)	Carbon (C)	Titanium (Ti)	Iron (Fe)	Cobalt (Co)	Molybdenum (Mo)	Magnesium (Mg)	Tantalum (Ta)
20.0	91006-Cr	91006-W	91006-C	91006-Ti	91006-Fe	91006-Co	91006-Mo	91006-Mg	91006-Ta
20.4	91007-Cr	91007-W	91007-C	91007-Ti	91007-Fe	91007-Co	91007-Mo	91007-Mg	91007-Ta
32.0	91008-Cr	91008-W	91008-C	91008-Ti	91008-Fe	91008-Co	91008-Mo	91008-Mg	91008-Ta
39.0	91009-Cr	91009-W	91009-C	91009-Ti	91009-Fe	91009-Co	91009-Mo	91009-Mg	91009-Ta
42.0	91013-Cr	91013-W	91013-C	91013-Ti	91013-Fe	91013-Co	91013-Mo	91013-Mg	91013-Ta
50.0	91014-Cr	91014-W	91014-C	91014-Ti	91014-Fe	91014-Co	91014-Mo	91014-Mg	91014-Ta
50.8	91015-Cr	91015-W	91015-C	91015-Ti	91015-Fe	91015-Co	91015-Mo	91015-Mg	91015-Ta
54.0	91016-Cr	91016-W	91016-C	91016-Ti	91016-Fe	91016-Co	91016-Mo	91016-Mg	91016-Ta
57.0	91017-Cr	91017-W	91017-C	91017-Ti	91017-Fe	91017-Co	91017-Mo	91017-Mg	91017-Ta
60.0	91010-Cr	91012-W	91010-C	91011-Ti	91010-Fe	91010-Co	91010-Mo	91010-Mg	91010-Ta
63.5	91018-Cr	91018-W	91018-C	91018-Ti	91018-Fe	91018-Co	91018-Mo	91018-Mg	91018-Ta
75.0	91019-Cr	91019-W	91019-C	91019-Ti	91019-Fe	91019-Co	91019-Mo	91019-Mg	91019-Ta
76.0	91020-Cr	91020-W	91020-C	91020-Ti	91020-Fe	91020-Co	91020-Mo	91020-Mg	91020-Ta
<b>Annular Targets (Outside Diameter x Inside Diameter, mm)</b>									
1170D x 89 ID	91030-Cr	91030-W	91030-C	91030-Ti	91030-Fe	91030-Co	91030-Mo	91030-Mg	91030-Ta
87 OD x 57 ID	91031-Cr	91031-W	91031-C	91031-Ti	91031-Fe	91031-Co	91031-Mo	91031-Mg	91031-Ta
59 OD x 33 ID	91032-Cr	91032-W	91032-C	91032-Ti	91032-Fe	91032-Co	91032-Mo	91032-Mg	91032-Ta
57 OD x 40 ID	91033-Cr	91033-W	91033-C	91033-Ti	91033-Fe	91033-Co	91033-Mo	91033-Mg	91033-Ta

## Lab Safety

### Neutralization and Disposal of Osmium Tetroxide

#### Introduction

A 2% solution of Osmium Tetroxide can be fully neutralized by twice the volume of oil (corn oil is preferred because of its high percentage of unsaturated bonds)<sup>1</sup>. That is to say, for every 10ml of 2% Osmium Solution, 20ml of corn oil is required.

#### Procedure

1. Do all work under fume hood.
2. Pour twice the volume of corn oil into used Osmium Tetroxide Solution.
3. Wait for the oil to completely turn black.
4. Test to check that complete neutralization has taken place. (To confirm neutralization; take either a glass cover-slip coated in corn oil or a piece of filter paper soaked in corn oil and suspend it over the solution. Blackening indicates  $OsO_4$  is still present.)<sup>2</sup>
5. Dispose in accordance to local regulations. Contact your Disposal Agency or Environmental Health & Safety Office at your Institute to obtain local regulations.

#### References

1. Cooper, K. Neutralization of Osmium Tetroxide in case of accidental spillage and for disposal. Bulletin of The Microscopical Society of Canada. 1988. 8: 24-28
2. Lunn, G.; Sansone, E.B. Osmium Tetroxide. Destruction of Hazardous Chemicals in the Laboratory; Program Resources, Inc. Frederick, MD; p 211 - 213.

### Safety Precautions in Electron Microscopy Laboratories

#### Chemicals

Treat all fixatives with respect, they will fix your tissue too.

##### Osmium Tetroxide

1. Handle ampoules with disposable gloves.
2. Use double bottles and seal with parafilm.
3. Open only in a fume hood, and well-ventilated room.
4. Do not hold your breath when using  $OsO_4$ . Your nose is a very sensitive detector of dangerous fumes.

##### Aldehydes

1. Handle in fume hood.
2. Acrolein-See Osmium precautions.

##### Buffers

Some buffers (e.g. Cacodylate and Veronal) contain toxic components, and must be handled with caution. Use gloves at all times.

##### Propylene Oxide

A very volatile solution. Must be opened and used at room temperature. Never heat, store cool.

#### Embedding Resins

Most embedding resins may cause dermatitis.

1. When preparing the mixtures and during embedding procedures, ALWAYS use disposable gloves.
2. Cover working areas with paper towel or diapers and wipe spills immediately with alcohol.
3. Do not use alcohol to remove resins from your skin, it increases penetration. Use soap & water.
4. Get medical attention for any suspicious skin rash.
5. Harden all waste resin before disposal, wrap discarded containers, beakers, vials, pipettes, etc. carefully.
6. Never pour any plastic containing solutions (e.g., propylene oxide-epon mixture) down the drain. They will harden.
7. Store in fume hood, until disposal, all hardened waste: RESINS, RESIN MIXTURE AND FILM MAKING SOLUTIONS (A coffee can or large bottle can be used).

#### Sharp Objects

Glass knives, razor blades and scalpel knives are still sharp after being used. Discard only in containers provided for that purpose, or well sealed, and labeled cardboard boxes, so cleaning personnel will not be injured.

#### Equipment

1. Most vacuum evaporators do not have a safety switch to turn off power before opening the bell jar. Follow the instructions on the equipment. Never observe metal evaporation without goggles. The intense brightness can burn your retina.
2. Critical Point Dryers are potentially lethal. Do not observe them in use unless you understand the danger.

**Note: Never attempt to operate any equipment without prior instruction by other people in the lab or without reading the operation instructions carefully.**