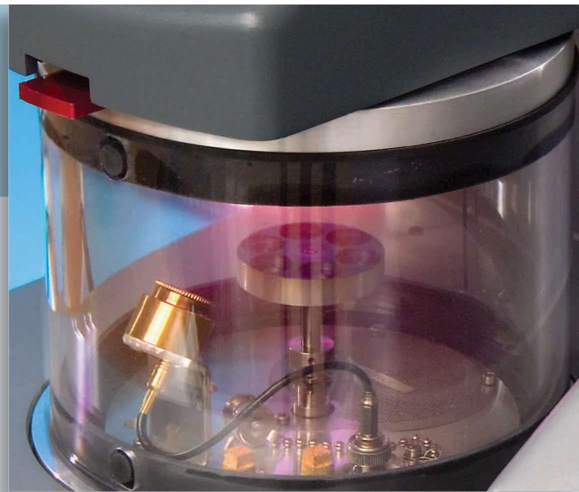

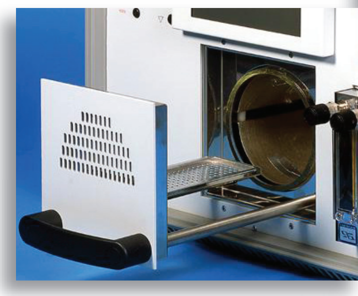

Sputter Coating

Glow Discharge

Carbon Coating

Vacuum Evaporation

Plasma Etching & Ashing

Critical Point Drying

Freeze Drying

Cryo-SEM Preparation

Equipment & Techniques

Electron
Microscopy
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Table of Contents

EMS 25 Cooling Stage for ESEM, VP, EP, and SEM

The EMS25X is a Peltier Driven Cooling Stage for SEM primarily VP & ESEM. It allows the stage to be cooled to sub zero temperatures for samples, which may be sensitive at ambient temperature, subject to beam damage, or may be sublime at ambient temperatures.

The Cooling Stage is located on the existing SEM Stage, and is designed to accept the standard range of stubs used with the SEM.

The Cooling Stage on the SEM Stage has secondary cooling via a liquid cooling circuit, through a 'spare' access port on the SEM, to the external heat exchanger unit, which is fully vacuum, isolated from the SEM chamber. This arrangement allows the majority of the x,y,z and tilt features to be maintained to an effective operating level.

The system is self-contained and employs a small power supply and temperature control system, the unit is supplied from standard supply socket.

The major parts of the system can be left in situ, and the Cooling Stage is very easily removed when reverting to 'normal' use.

Features

- *Temperature range: -35°C to 75°C*
- *Ambient to -25°C in 3 minutes*
- *Can be used with all leading SEMs – specific interface plate supplied*
- *Display of actual and target temperatures*
- *Small footprint of control unit*

Benefits

- *Versatile and compact system for use with VP/EP & ESEM*
- *Rapid cooling of samples*
- *Easy to see when system is ready*

System Items

Peltier Cooled Stage and secondary liquid cooling

Vacuum Interface Port with Integral Electrical Lead Through for Peltier Supply, cooling and Temperature Monitoring

Optional chiller for cooling circuit (SEM cooling water or ambient water flow may be used).

Control Unit with Digital P.I.D. (Proportional, Integral, Derivative) Control, with SET Point Input Temperature and MEASURED Temperature Display

Low Voltage Ultra smooth DC Power Supply for Peltier Drive.

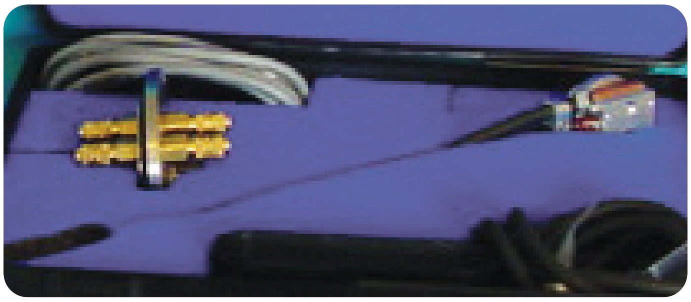
EMS25X is available for any Hitachi, Leo, Joel or FEI SEM (Other SEM vendors also available). Keypad and dual display for temperature control and display, Simultaneous display of Actual and Target temperatures, Vacuum Interface plate with all connections, Dual Microprocessor Control.

Ordering Information

10100	EMS 25 Cooling Stage	each

Specifications

Stage Temperature Control Range	+70°C to -30°C Resolution +/- 1°C
Typical Operating Temperature	-25°C within 3 minutes from ambient +25°C
Temperature Display Resolution	0.1°C
Temperature Stability	0.2°C
Control Unit Overall Dimensions	137mm H x 235mm W x 260mm D Complete with Set of 1 Meter interconnecting leads, and mains Supply lead
Sample Size	Normal Range of Specimen Stubs, Normally to 25mm Max. Dia.)
X,Y Normal Stage	Movements Maintained
Tilt	Normal Movement Maintained for X-Ray Analysis (Typically to 45°)
Working Distance	Variable +/- 25mm
Rotate	Greater than 180°
SERVICES	
Electrical Supply	230 Volts or 115 Volts 50/60Hz, Single Phase, 5 Amp max.



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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

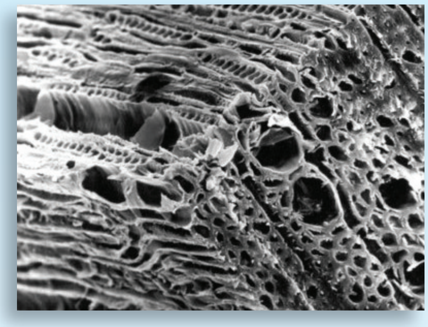
Working temperature range	Ambient to +65°C
Heating rating	500W
Reservoir capacity	2.2L / 3.5 Pints
Pump type	Glandless magnetic drive
Pump motor	Shaded pole
Pump flow	270L/h (zero head) / 60 Gal/hr (zero head)
Pump pressure	0.25kg/cm^2 / 3.5psi
Max pump head (unrestricted)	2.5m / 7.5ft
Size (unpacked)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

Ordering Information

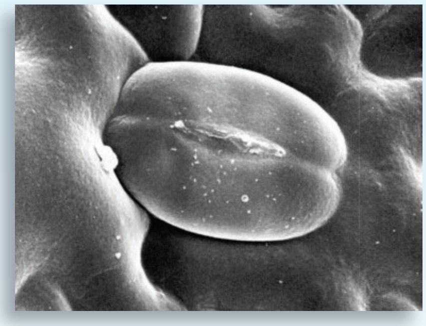
91095	EMS 3500 Thermocirculator	each

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 crystallites are also just visible on the surface of the leaf.

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:

Flow rate x weight of fluid x specific heat x D T = Heat Extraction.

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

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	16mm hose or 1/8 BSP	16mm hose or 1/8 BSP	16mm hose or 1/8 BSP
Temperature sensor	R.T. Probe	R.T. Probe	R.T. Probe	R.T. Probe

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

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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:

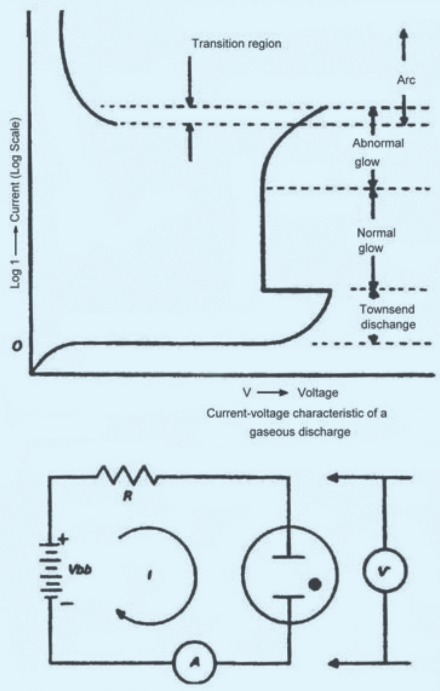
- ***Reduced microscope beam damage.***
- ***Increased thermal conduction***
- ***Reduced sample charging (increased conduction).***
- ***Improved secondary electron emission***
- ***Reduced beam penetration with improved edge resolution***
- ***Protects beam sensitive specimens***

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.

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

A = Ammeter V = Voltmeter



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.

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 ionixation, generating positive ions to sustain the discharge. Subsequently, excitation of

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Techniques and Applications

the gas results in intense illumination 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, platinium 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 grains 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.

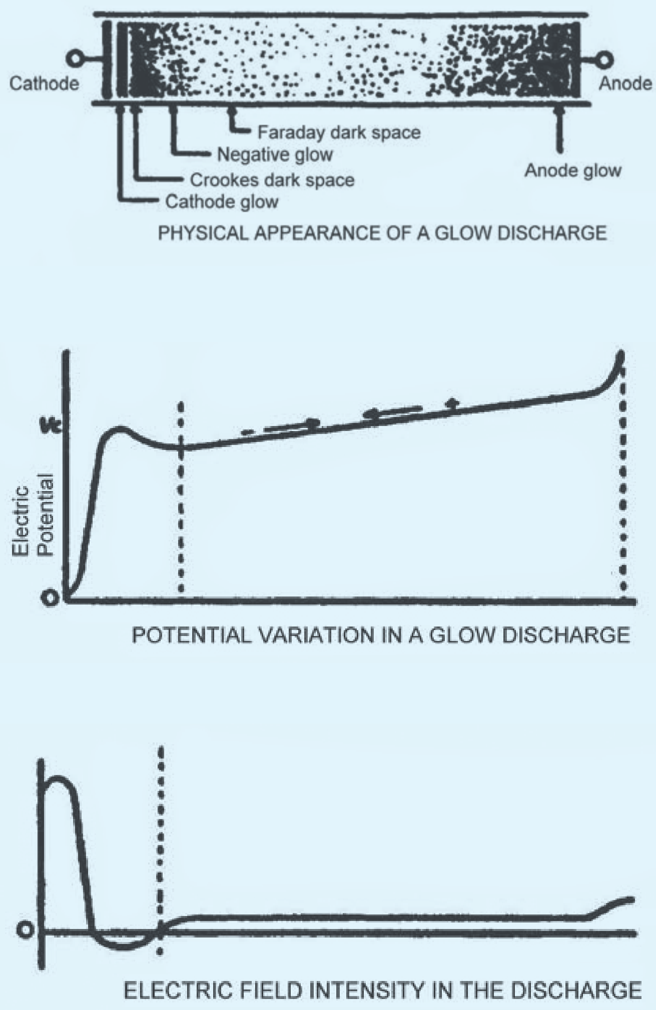
The deposition rate is current dependant, and if we operate in the correct glow region of the characteristic plasma discharge, as previously described, several fold changes in current should be available for a relatively small change in sputtering voltage. The deposition rate should not be sensitive 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 energy will be gained for effective bombardment and will inhibit movement of sputtered material from the target.

Figure 2



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.

Techniques and Applications

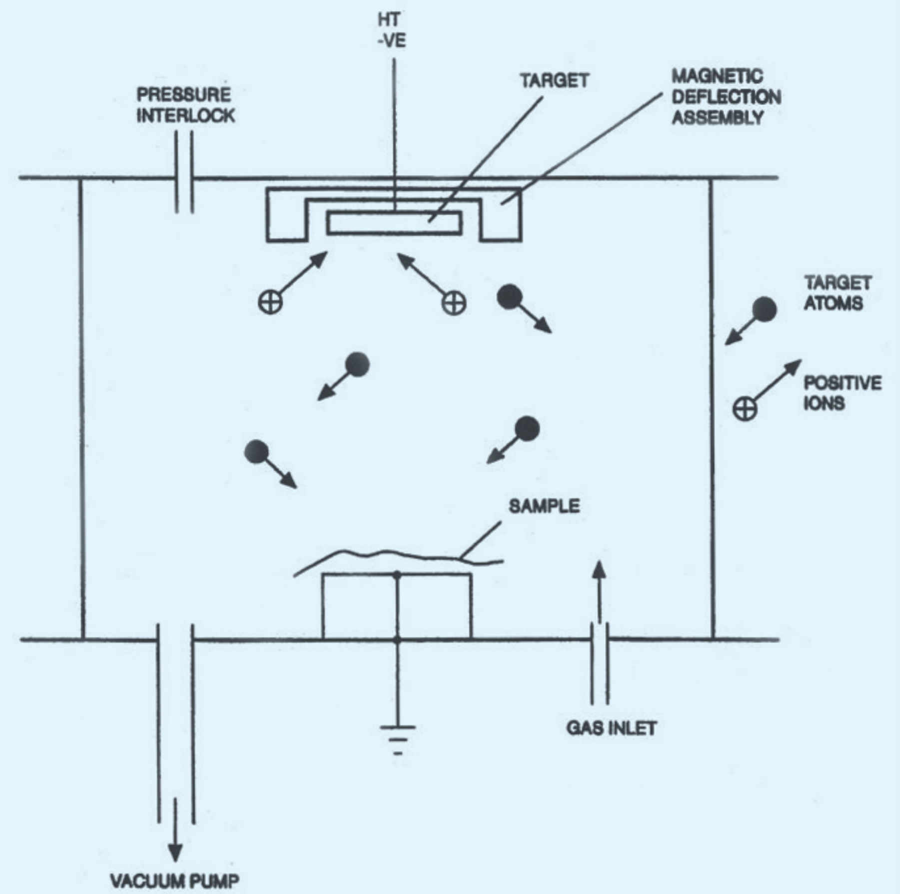
A major disadvantage of simple diode sputter coaters in SEM is the excessive amount of heat generated in the sample. To overcome this problem, permanent magnets are utilized to deflect the high energy electrons generated in the glow discharge away from the sample.

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

Figure 3 – Diagram of a "cool" sputtering head



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

Vacuum	8 x 10^{-2}	to	2 x 10^{-2} mbar
Sputtering Voltage	100V	to	3Kv
Current	0	to	50mA
Deposition	0	to	25 nm/min
Grain size	Less than 5nm		
Temperature rise	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 l0nm 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 Quorum K550X, 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.*

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Techniques and Applications

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.

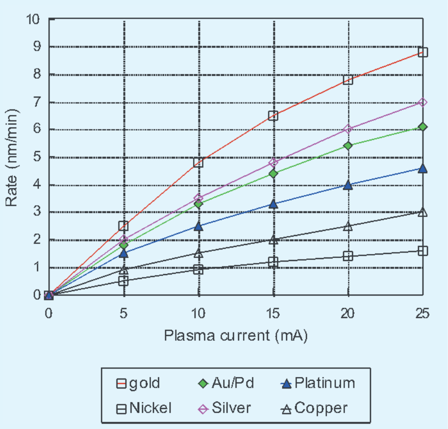
Other target choices are generally made based on the requirement for X-ray analysis of samples or back scattered electron detection.

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 (angstroms) (V = 2.5KV, 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

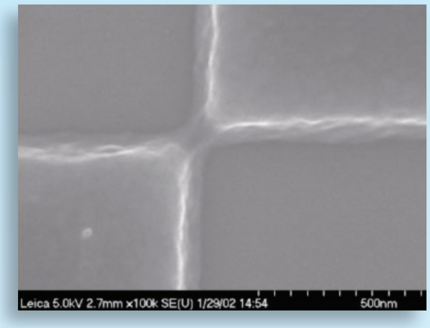
- Clean the glass chamber with hot soapy water and dry thoroughly, solvents can be used but we have found this unnecessary and having greater danger to health and safety. If the deposit is stubborn, use a kitchen scouring pad such as the green Scotch Bright variety.
- Use Isopropyl alcohol on metal surfaces, 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.

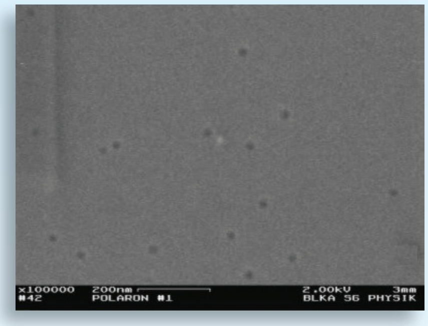
3. Sputter gas, Always use high purity argon gas of the grade known as “White spot” this will ensure fast sputter rate and good pump down time.

4. Rotary planetary specimen stages are essential for ensuring even coatings on specimens with irregular surfaces.



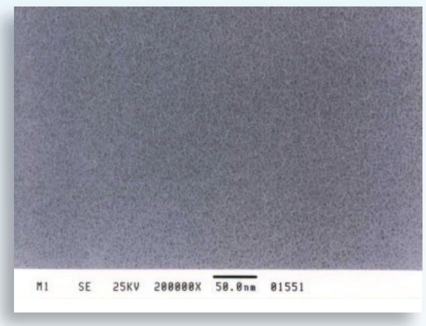
Gold/palladium coating of 6" wafer

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.

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 bath 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

It was found that the resulting pale yellow solution had a pH of about 5, the same as the CO_2-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

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Techniques and Applications

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 (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.

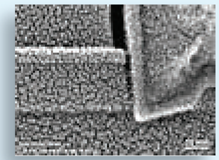
Comparative Sputter Data

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





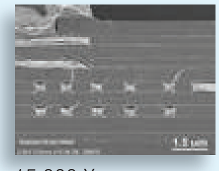
Magnification:	15,000 X	100,000 X	300,000 X
Coating Time:	10 seconds	10 seconds	10 seconds
Current Used:	20 mA	20 mA	20 mA

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

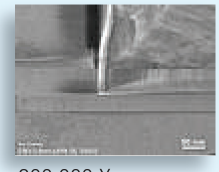
Iridium



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

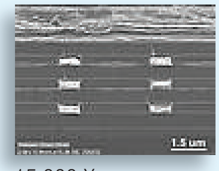
No Coating





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

EMS 7620 "Mini" Sputter Coater

The EMS 7620 is a compact, low cost SEM sputter coater. 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*

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.

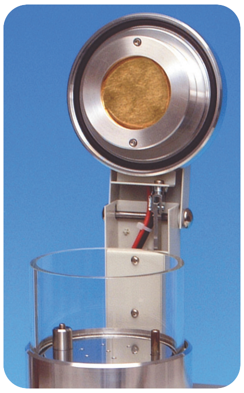
Quality coating

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

Pumps

91003 RV-3 two-stage rotary pump with oil mist filter (115/230V 50/60Hz)

96000 Replacement oil mist filter

91040 Carbon evaporation attachment — EMS 7640

91006 Film Thickness Monitor

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)

Part number	Description
91017-AU	Gold (Au) target
91017-AP	Gold/palladium (Au/Pd) target
91017-PT	Platinum (Pt) target
91017-AG	Silver (Ag) target
91017-PD	Palladium (Pd) target
91017-.2-AU	Gold target (Au) 0.2mm thick
91017-.2-AP	Gold/palladium (Au/Pd) target 0.2mm thick
91017-.2-PT	Platinum (Pt) target 0.2mm thick

Ordering Information

EMS 7620	'Mini' Sputter Coater	each

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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.

EMS 7640-CF, EMS 7640-CR and EMS 7620-CF Carbon Accessory Power Supplies

The EMS 7640-CF, EMS 7640-CR and EMS 7620-CF carbon attachments are modular add-ons for our sputter coaters, allowing carbon fiber or carbon rod evaporation.

Each 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 - see the EMS 450X and EMS 950X.

Features

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

The EMS 7640-CF, EMS 7640-CR and EMS 7620-CF can be used in conjunction with the EMS 7620 (EMS 7620-CF), EMS 500X, EMS 550X, EMS 575X, EMS 650X and EMS 675X sputter coaters. 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

Dimensions	235mm W x 350mm D x 175mm H.
Weight	15kg
Carbon source	Carbon fiber, carbon cord
Ammeter gauge	0-50A
Low voltage	25V
Out-gas current	Selectable for carbon fiber or carbon rod
Electrical supply	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 7640-CF
EMS 500X, EMS 550X, EMS 575X, EMS 675X	EMS 7640-CR	EMS 7640-CF

Please see individual sputter coater models for further information.

Carbon fiber cord

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

Carbon rods

NOTE: 3.05mm diameter carbon rods are used with the EMS 950X, EMS 450X and EMS 350. 6.15mm diameter carbon rods are used with the K975X and with most older Polaron-branded carbon evaporators.

70210-10	Shaped ('stepped') carbon rods - high purity - 3.05mm Ø x 50mm	(pack of 10)
70210-25	Shaped ('stepped') carbon rods - high purity - 6.15mm Ø x 50mm	(pack of 10)

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

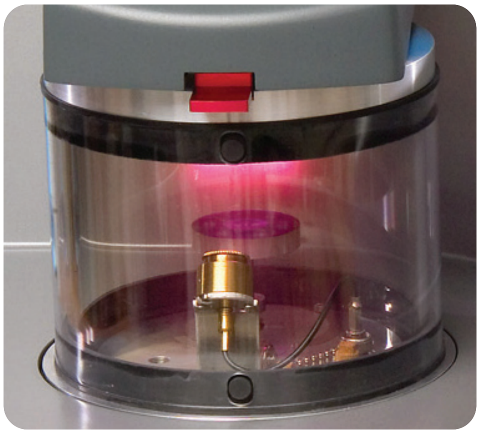
EMS150T E — a high vacuum carbon coater for SEM and TEM applications

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 evaation gun – simple operation, reproducible results*
- *Control of evaation 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.

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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.

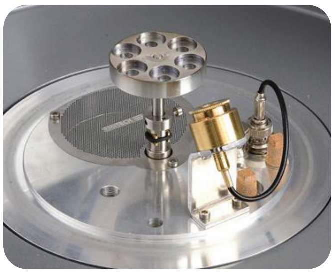
Each of the above can be fitted with a range of optional accessories (eg metal evaporation, carbon fiber coating, film thickness monitor). See options for details.

Rapid data entry

At the operational heart of the EMS 150T 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.

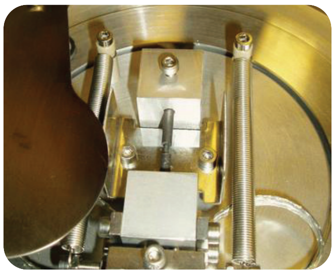
Processes

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



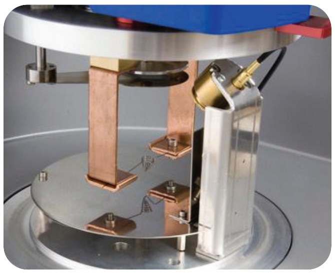
Standard Rotation Stage and FTM

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



Standard Rotation Stage and FTM

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.



Standard Rotation Stage and FTM

Specifications

Instrument case	585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
Weight	33.4kg
Packed dimensions	725mm W x 660mm D x 680mm H (42kg)
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 a log of the last ten coatings carried out and reminders for when maintenance is due
Sputter target	Disc style 57mm Ø. A 0.3mm thick chromium target is fitted as standard. EMS150T S and T ES versions only
Vacuum	
Turbomolecular pump	Internally-mounted, 70L/s air-cooled turbomolecular pump
Rotary pump	Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter
Vacuum measurement	Pirani gauge as standard. A full range gauge (10428) is available as an option
Typical ultimate vacuum	5 x 10^{-5} mbar in a clean system after pre pumping with dry nitrogen gas
Sputter vacuum range	Between $5x10^{-3}$ and $5x10^{-1}$ mbar
Specimen stage	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...)

EMS 150T (continued)


Tilt Angle Stage


Rotary Planetary Stage


4” Wafer Stage


Glass Microscope Stage


Carbon Evaporation Insert

Ordering Information

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

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 Ø 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 Ø rods)
3240	Carbon rod evaporation head insert (for 6.15mm Ø rods). Note that EMS recommends 3.05mm Ø 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, includes ability to evaporate downwards or upwards (T E and T ES 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 insert to modify surface properties (eg hydrophobic to hydrophilic conversion) or to clean surface residues (TS and T ES only). Can be retrofitted

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.

3330	Rotation stage, 50mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment
3340	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
3350	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
3360	Flat rotation specimen stage for 100mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm
3370	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
3380	EMS150T S High resolution turbomolecular pumped sputter coater, including a 54mm Ø x 0.3mm chromium target
3400	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.

Sputtering Targets

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

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

Carbon supplies

3500	Carbon rods – 6.15mm 0 x 100mm length (unshaped) pack of 10
3510	Carbon rods – 6.15mm 0 x 50mm length (shaped) pack of 10
3520	Carbon rods - 3.05mm 0 x 50mm length (shaped) pack of 10
3530	Carbon rods 3.05mm 0 x 300mm length (unshaped) pack of 10)
3540	Carbon fiber cord - high purity - 1m
3550	Carbon fiber cord - high purity - 5m
3560	Carbon fiber cord - standard grade - 1m
3570	Carbon fiber cord - standard grade - 10m
3580	Carbon fiber cord - standard grade - 100m
3590	Manual rod shaper for 6.15mm Ø carbon rods
3595	Manual rod shaper for 3.05mm Ø carbon rods
3540	Carbon fiber cord - high purity - 1m
3550	Carbon fiber cord - high purity - 5m
3560	Carbon fiber cord - standard grade - 1m
3570	Carbon fiber cord - standard grade - 10m
3580	Carbon fiber cord - standard grade - 100m
3590	Manual rod shaper for 6.15mm Ø carbon rods
3595	Manual rod shaper for 3.05mm Ø carbon rods

EMS 500 & 550 Sputter Coater

The EMS 550 system employs a Magnetron Target Assembly which enhances the efficiency of the process using low voltages, and giving a Fine Grain, Cool Sputtering, without the need to cool the target or the specimen stage.

The specimen stage height is adjustable in discreet steps, accommodates a range of specimens and stubs, which together with pre-selectable parameters and Automatic Control, gives defined and repeatable film thickness depositions.

The instrument is fitted with 60mm (Dia) and 0.1mm (T) gold (or Customer choice) quick change target, giving optimum consumable cost performance.

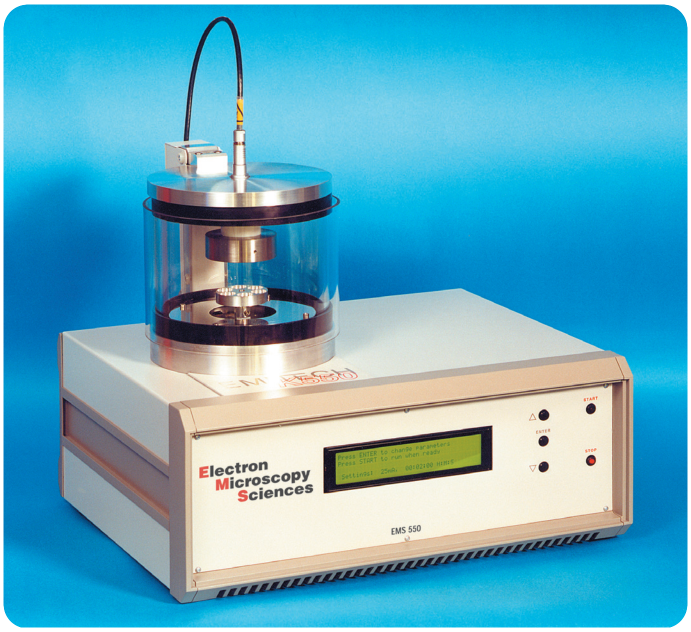
The integrated instrument panel and plug-in electronics, maximize 'up-time' and, with user friendly designs, ensures satisfactory multi-user discipline.

The sputtering parameters can be pre-set, including the gas bleed needle valve which has electromagnetic valve back-up.

The sputtering head is interlocked, and the system can easily accommodate the EMS 250 Carbon Coating Attachment.

The independent vacuum pump is controlled by the instrument throughout the fully automatic coating cycle.

The EMS 500 is a manual version of the EMS 550 without a rotary stage. All other features and specifications are the same.



Features

- *Built-in rotary and tilt stage.*
- *Fully automatic control.*
- *Low energy 'cool' system (order of 2 Watts for cycle of 2 mins is 240 joules).*
- *Low voltage sputtering (Order of 100V mean DC temperature increase less than 2°C/minute).*
- *High resolution fine coating (Fine grain size).*
- *Precise reproducible coatings.*
- *Even thickness deposition (Typically 20 nm or 20 Angstroms for SEM work).*
- *Modular control electronics.*
- *Multi specimen holder*
- *Polycarbonate safety shield.*

Specifications

Instrument Case	450mm (W) x 350mm (D) x 175mm (H)
Work Chamber	Borosilicate Glass 165mm (Dia.) x 125mm (H)
Weight	18Kg
Target	60mm (Dia.) x 0.1mm (Thick) (Gold fitted as standard)
Specimen Stage	60mm (Dia.) Adjustable height spacing to target 25mm to 45mm
Vacuum Gauge Range	ATM –1x10^{-2} mbar
Deposition Range	0-50mA

Deposition Rate	0-25nm/minute
Sputter Timer	0-4 minutes
Supply	115V 60Hz 230V 50Hz (6 Amp Max incl pump)
Services	Argon – Nominal 4 psi
Vacuum Pump (Recommended)	Complete with vacuum hose and oil mist filter 85L/Min
Size	470mm (L) x 150mm (W) x 250mm (H)
Weight	20Kg

Ordering Information

91000	EMS 550 Sputter Coater complete with Target	each
91000-MA	EMS 500 Manual Sputter Coater	each
91005	Rotary Vacuum Pump Target 60mm (Dia.) x 0.1mm (Thick) fitted inclusive as standard	each

Replacement Targets

91010	Gold Target	each
91011	Gold/Palladium Target	each
91012	Platinum Target	each

Replacement Parts

91013	Glass Cylinder 165mm (6")	each
91014	"L" Gaskets to suite 165mm (6") cylinder (1 pair)	each

Optional Accessories

91006	EMS 150 Film Thickness Monitor	each
91040	EMS 7640 Carbon Coating Attachment	each

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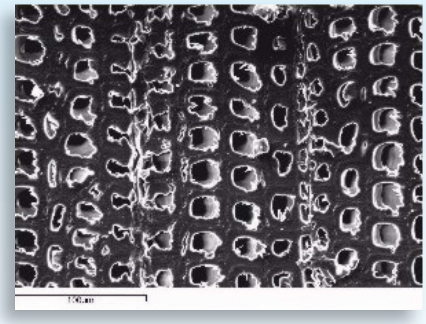
What is... Carbon Coating?

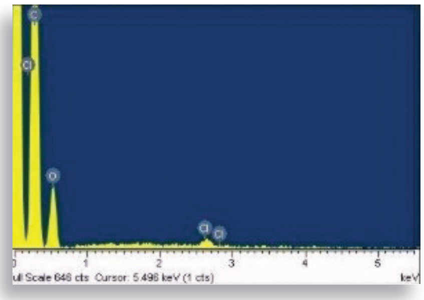
The use of carbon films in Electron Microscopy with their low background signal and relatively good electrical conductivity is well known. Thin films, nominally 5nm or 50 Angstroms, are used in TEM, while a range of somewhat thicker films, ranging from 50nm or 500 Angstroms, may be used in SEM for such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



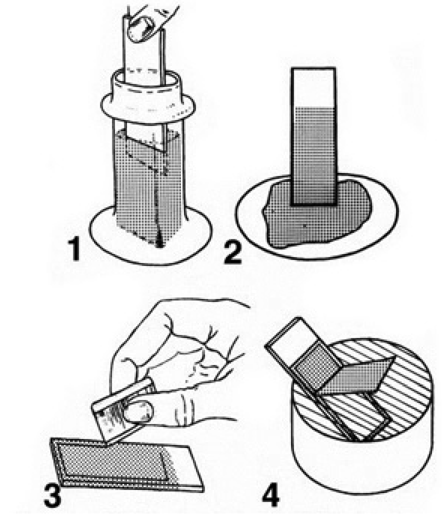
Pinus sylvestris (Scots pine)

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



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

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 $5x10^{-5}$ 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° 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.

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 1-2µm in diameter and suitable for use with frozen-hydrated specimens. Sonicating 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

Techniques and Applications

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.

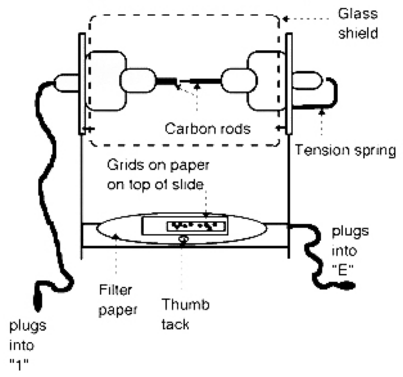
Step 9. Place the paper and coated grids onto a piece of filter paper that is soaked with ethylene dichloride in a covered petri dish. 30 minutes should be sufficient time to dissolve the formvar film and not damage the carbon support. Remove the grids and paper and allow them to dry in a dust-free area.

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

Suggested Carbon Coater Set-up



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.

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 $2x10^{-5}$ 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.

EMS 150T Turbo-Pumped Carbon Coater/Sputter 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

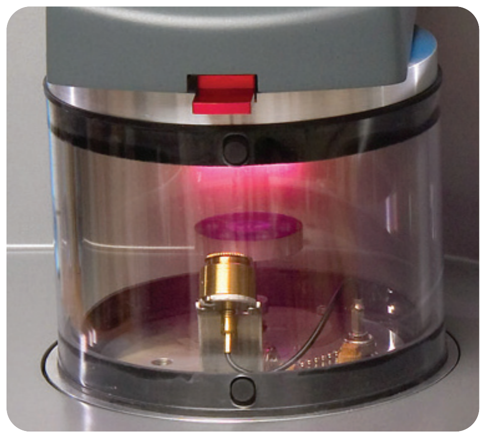
Features

- *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 evaation gun – simple operation, reproducible results*
- *Control of evaation 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.

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EMS 150T *(continued)*

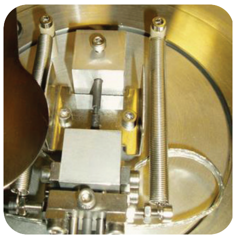
- **EMS150T E –** a high vacuum carbon coater, ideal for the production of highly stable carbon films and surface replicas for transmission electron microscopy (TEM). The system uses economical 3.05mm diameter carbon rods.
- **EMS150T ES** – a combined system with both sputtering and carbon coating.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

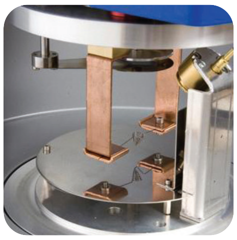
At the operational heart of the EMS150T is a simple color touch screen which allows even the most inexperienced or occasional operator to rapidly enter and store their own process data. To further aid ease of use, a number of typical sputtering and evaporation profiles are already stored.

Processes



Carbon Rod Evaporation Insert

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



Metal 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.

Specifications

Instrument case	585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
Weight	33.4kg
Packed dimensions	725mm W x 660mm D x 680mm H (42kg)
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 a log of the last ten coatings carried out and reminders for when maintenance is due
Sputter target	Disc style 57mm Ø. A 0.3mm thick chromium target is fitted as standard. EMS150T S and T ES versions only
Vacuum	
Turbomolecular pump	Internally-mounted, 70L/s air-cooled turbomolecular pump
Rotary pump	Edwards RV3 50L/s two-stage rotary pump, with vacuum hose, coupling kit and oil mist filter
Vacuum measurement	Pirani gauge as standard. A full range gauge (10428) is available as an option
Typical ultimate vacuum	5 x 10^{-5} mbar in a clean system after pre pumping with dry nitrogen gas
Sputter vacuum range	Between $5x10^{-3}$ and $5x10^{-1}$ mbar
Specimen stage	60mm Ø rotating stage. Rotation speed 8-20 rpm

Services and other information

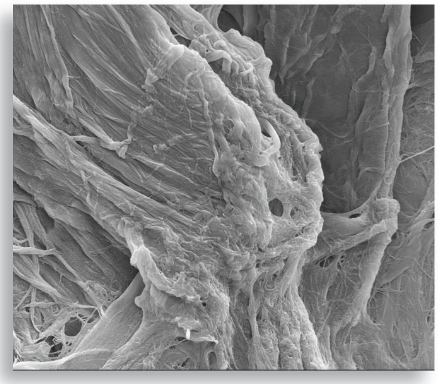
Gases: Argon sputtering process gas, 99.999% (TS and TES versions) Nitrogen venting gas (optional)

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

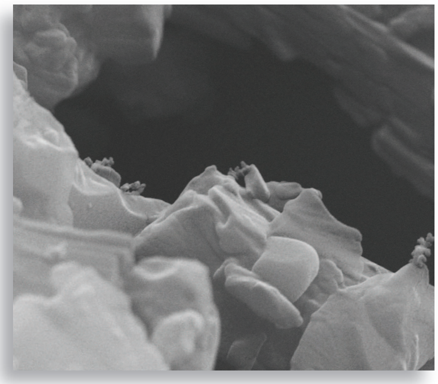
Conformity: CE conformity:

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

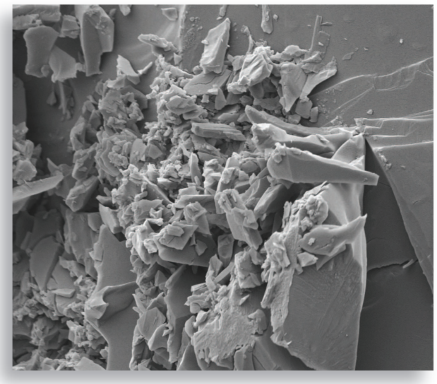
Ordering information (see next page...)



10nm Au on Filter Paper



10nm Au on Salbutamol



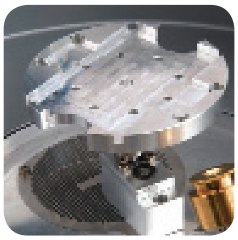
10nm Au on Sugar

EMS 150T (continued)


Tilt Angle Stage


Rotary Planetary Stage


4” Wafer Stage


Glass Microscope Stage


Carbon Evaporation Insert

Ordering Information

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

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 Ø 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 Ø rods)
3240	Carbon rod evaporation head insert (for 6.15mm Ø rods). Note that EMS recommends 3.05mm Ø 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, includes ability to evaporate downwards or upwards (T E and T ES 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 insert to modify surface properties (eg hydrophobic to hydrophilic conversion) or to clean surface residues (TS and T ES only). Can be retrofitted

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.

3330	Rotation stage, 50mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment
3340	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
3350	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
3360	Flat rotation specimen stage for 100mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm
3370	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
3380	EMS150T S High resolution turbomolecular pumped sputter coater, including a 54mm Ø x 0.3mm chromium target
3400	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.

Sputtering Targets

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

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

Carbon supplies

3500	Carbon rods – 6.15mm 0 x 100mm length (unshaped) pack of 10
3510	Carbon rods – 6.15mm 0 x 50mm length (shaped) pack of 10
3520	Carbon rods - 3.05mm 0 x 50mm length (shaped) pack of 10
3530	Carbon rods 3.05mm 0 x 300mm length (unshaped) pack of 10)
3540	Carbon fiber cord - high purity - 1m
3550	Carbon fiber cord - high purity - 5m
3560	Carbon fiber cord - standard grade - 1m
3570	Carbon fiber cord - standard grade - 10m
3580	Carbon fiber cord - standard grade - 100m
3590	Manual rod shaper for 6.15mm Ø carbon rods
3595	Manual rod shaper for 3.05mm Ø carbon rods
3540	Carbon fiber cord - high purity - 1m
3550	Carbon fiber cord - high purity - 5m
3560	Carbon fiber cord - standard grade - 1m
3570	Carbon fiber cord - standard grade - 10m
3580	Carbon fiber cord - standard grade - 100m
3590	Manual rod shaper for 6.15mm Ø carbon rods
3595	Manual rod shaper for 3.05mm Ø carbon rods

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EMS Series of Rotary Pumped Modular Coating Systems

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

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

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*
- *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*

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.

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 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.

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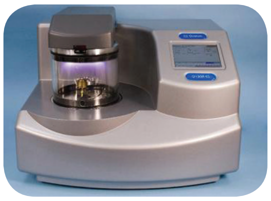
EMS 150T (continued)

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

Coating time since last cleaned

System „on time

Time of last service 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.

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)

To the right: Sputtering insert. Gold (Au) fitted as standard, but other metals available

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

To the right: Carbon fibre evaporation insert and automatic source shutter

Glow discharge

0-80mA operates in DC+ and DC- modes

To the right: Optional glow discharge attachment (for R S and R ES versions)

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
User interface	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
Sputter 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 x 10-2mbar in a clean system after pre pumping with dry nitrogen gas
Sputter vacuum range	Between 3 x 10-2 and 5 x 10-1mbar
Typical ultimate vacuum	5 x 10-5 mbar in a clean system after pre-pumping with dry nitrogen gas
Sputter vacuum range	Between 5x10-3 and 5x10-1 mbar
Specimen stage	60mm Ø rotating stage. Rotation speed 8-20 rpm

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

Ordering information (see next page...)

EMS 150R (continued)

Ordering Information

EMS150R S	Rotary pumped sputter coater, includes an 57mm Ø x 0.1mm gold target. Fitted with 4501 quick-release sputter insert suitable for non-oxidising (noble) metals. and 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	Rotary pumped carbon fibre evaporator, supplied with carbon fibre / cord4504. Fitted with quick-release carbon fibre evaporation insert for suitable for evaporation of carbon fibre and carbon cord and 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	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. Fitted with 4509 quick-release carbon fibre evaporation insert suitable for evaporation of carbon fibre and carbon cord and 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

Optional Accesories -

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 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 (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, 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

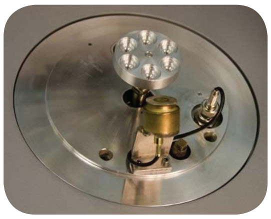
	The EMS150R has alternative specimen stages to meet most requirements. All are easy-change, drop-in style and are height adjustable (except 10360 rotary planetary stage):
4519	50mm Ø rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37mm-60mm). Has six stub positions for 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
4524	57mm Ø x 0.2mm Gold
4525	57mm Ø x 0.3mm Gold
4526	57mm Ø x 0.1mm Gold/Palladium (80/20%)
4527	57mm Ø x 0.2mm Gold/Palladium (80/20%)
4528	57mm Ø x 0.3mm Gold/Palladium (80/20%)
4529	57mm Ø x 0.1mm Platinum
4530	57mm Ø x 0.2mm Platinum
4561	57mm Ø x 0.3mm Platinum
4532	57mm Ø x 0.1mm Nickel
4533	57mm Ø x 0.1mm Silver
4534	57mm Ø x 0.1mm Palladium
4535	57mm Ø x 0.1mm Copper
4536	57mm Ø x 0.1mm Platinum/Palladium (80/20%)
4537	57mm Ø x 0.3mm Platinum/Palladium (80/20%)

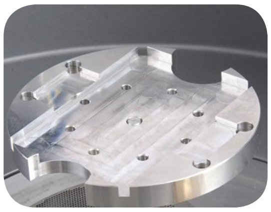
Carbon supplies

3500	Carbon rods – 6.15mm 0 x 100mm length
4538	Carbon fibre cord - high purity - 1m
4539	Carbon fibre cord - high purity - 5m
4540	Carbon fibre cord - standard grade - 1m
4541	Carbon fibre cord - standard grade - 10m
4542	Carbon fibre cord - standard grade - 100m
4543	Carbon rods - 3.05mm Ø x 50mm length (shaped) pack of 10
4544	Carbon rods 3.05mm Ø x 300mm length (unshaped) pack of 10)
4546	Manual rod shaper for 3.05mm Ø carbon rods

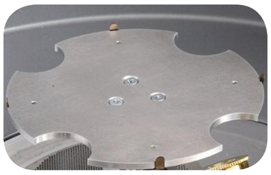


Base plate, standard specimen stage and optional film thickness monitor (FTM)

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



Optional rotation stage for glass microscope slides



Optional flat rotation stage for 100mm/4" wafers

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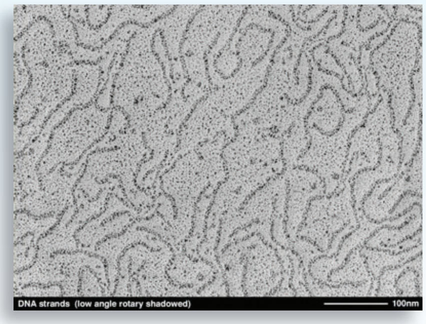
EMS 7640-CF, EMS 7640-CR and EMS 7620-CF Carbon Accessory Power Supplies

What is... Carbon Evaporation?

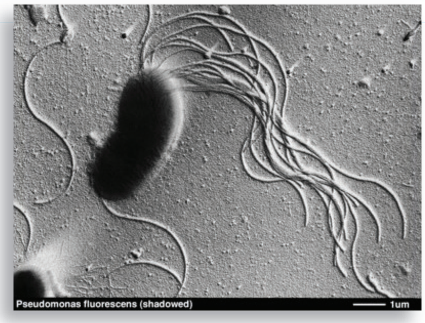
The use of Carbon Evaporation is well known in Electron Microscopy for support films and replicas in TEM, and X-Ray Microanalysis and conducting coatings in SEM.

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 1x 10^{-4} mbar, or better, requires the use of Diffusion or Turbo Molecular Pumps.



DNA strands



Pseudomonas fluorescens

The EMS 7640-CF, EMS 7640-CR and EMS 7620-CF carbon attachments are modular add-ons for our sputter coaters, allowing carbon fiber or carbon rod evaporation.

Each 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 - see the EMS 450X and EMS 950X.

Features

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

The EMS 7640-CF, EMS 7640-CR and EMS 7620-CF can be used in conjunction with the EMS 7620 (EMS 7620-CF), EMS 500X, EMS 550X, EMS 575X, EMS 650X and EMS 675X sputter coaters. 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

Dimensions	235mm W x 350mm D x 175mm H.
Weight	15kg
Carbon source	Carbon fiber, carbon cord
Ammeter gauge	0-50A
Low voltage	25V
Out-gas current	Selectable for carbon fiber or carbon rod
Electrical supply	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 7640-CF
EMS 500X, EMS 550X, EMS 575X, EMS 675X	EMS 7640-CR	EMS 7640-CF

Please see individual sputter coater models for further information.

Carbon fiber cord

C5421	Carbon fiber cord - standard grade - 1cm
C5421-10	Carbon fiber cord - standard grade - 10m
C5421-100	Carbon fiber cord - standard grade - 100m
A0819	Carbon fiber cord - high purity - 1m
A0819-5	Carbon fiber cord - high purity - 5m
C5461	Carbon fiber cord - standard purity, fine strands - 1m
C5461-10	Carbon fiber cord - standard purity, fine strands - 10m
C5461-100	Carbon fiber cord - standard purity, fine strands - 100m

Carbon rods

NOTE: 3.05mm diameter carbon rods are used with the EMS 950X, EMS 450X and EMS 350. 6.15mm diameter carbon rods are used with the EMS 975X and with most older Polaron-branded carbon evaporators.

A0834A	Shaped ('stepped') carbon rods - high purity - 3.05mm Ø x 50mm	(pack of 10)
A0832A	Shaped ('stepped') carbon rods - high purity - 6.15mm Ø x 50mm	(pack of 10)

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EMS 100 Glow Discharge Unit

The EMS 100 is a simple, free-standing glow discharge system, typically used for hydrophobic to hydrophilic conversion of carbon coated TEM grids. The EMS 100 requires only a suitable rotary pump.

Freshly made 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.

In addition to Glow Discharge treatment using air, other process gases may be used to modify surface properties. For example, if alkylamine is used as a process gas, the carbon film surface will become hydrophilic and positively charged. On the other hand, using methanol as a process gas results in the surface becoming hydrophobic and negatively charged. Such treatments can facilitate the optional absorption of selected biomolecules.

Features

- *Polarity of the head can be changed from positive to negative with respect to earth.*
- *H.T. vacuum interlock.*
- *Needle valve bleed control.*
- *Surface treatment and cleaning.*
- *Polycarbonate safety shield.*

Glow Discharge Summary

Atmosphere	Surface	Charge
Air	Hydrophilic	Negative
Air	Hydrophilic	Positive with subsequent Magnesium Acetate Treatment
Alkylamine	Hydrophobic	Positive
Methanol	Hydrophobic	Negative

Surface Cleaning

In many instances, surfaces need to be completely cleared of contamination films or deposits. This applies particularly to EM components where such deposits impair the maintenance of a clean vacuum system.

A Glow Discharge treatment can be used to clean such components of undesirable residues.

D.C. Glow Discharge

In the DC+ Mode, the Glow Discharge system can draw up 1.5Kv. This allows high efficiency ion etching of the specimen surface to remove, for example, oxide or resist layers.

Glow Discharge

The Polarity of the Head can be switched from Positive to Negative with respect to Earth, for Carbon Film Surface Treatment or Surface Etching of Metallic Specimens.

Specifications

Plasma Voltage	0-1000V Variable DC @100mA.
Electrode Polarity	+DC or -DC with Stainless Steel electrode (60mm Dia.)
Vacuum Chamber	165mm (D) x 125mm (H) Glass
Instrument Case	450mm (W) x 350mm (D) x 170mm (H)
Weight	15Kg
Supply	115V 60Hz (6 Amp Max) 230V 50Hz (3 Amp Max)

Ordering Information

94000	EMS 100 Glow Discharge Unit	each
94100	EMS 100 Glow Discharge Attachment for the EMS 450 & EMS 950	each
91005	RV-3 Rotary Vacuum Pump	each

Electron Microscopy Sciences
P.O. Box 550 • 1560 Industry Rd. • Hatfield, Pa 19440 • Tel: (215) 412-8400 • Fax: (215) 412-8450 • email: sgkcck@aol.com • www.emsdiasum.com

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 -> 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 -> 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 + 02 -> e + 0 + 0

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 + CF4 -> e + CF3 + F (Dissociation) or e + CF4 -> 2e + CF3+ + 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.

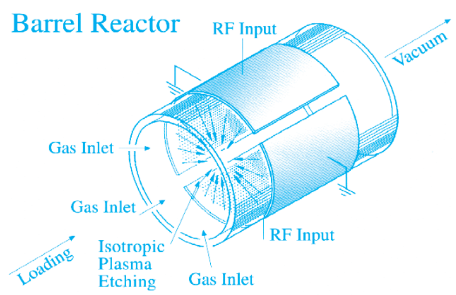
Types of Reactor Systems

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.

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 or 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.

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

continues on next page

Techniques and Applications

Plasma Applications (continued)

using CF_4, 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 [1]. Self-adhesion can also be markedly improved by plasma treatment [2, 3].

Plasma techniques are widely used in the electronics industry [4] 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 use as resists, insulators or semiconductors. Operations carried out by plasma techniques include photoresist removal [5], etching silicon compounds [6] and deposition of polymer films [7, 8]. The chief virtue of plasma techniques in microelectronics fabrication is that it permits automated, multi-step processing of complex devices [9].

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

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.

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EXAMPLES OF PLASMA GASES AND THEIR APPLICATIONS

O_2 100%
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_2 for faster reaction
Cleaning EM parts
Oxide removal
Hydrophilation

H_2 100%
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_2

N_2 100%
Polymer activation
Removal of epoxy bleed-out on hybrid circuits
Removal of oxides

C_2H_4 100%
Polymerization

CH_4 100%
Polymerization

C_2H_2 100%
Polymerization

CF_4 100%
Epilamization
Silicon etchant

SF_6 100%
Silicon etchant

FS-100 97% He 3% O_2
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_2 with 2ppm water
Removal of organics from sensitive substrates without oxidation

DS180 92% O_2 8% CF_4
Removal of thick layers of photo resist

DS100 99.78% (40% O_2 to 60% He) 22% CF_4
Removal of photo resist from chrome masks wit

DS300 97% O_2 3% CF_4
Photo resist removal in aluminium chambers or with faraday insert in quartz chambers
Removal of organic contamination

DS16281 99% N_2 1% O_2
Removes photo resists films over oxidising or with TCR and resistor networks being unchanged (thin films only). May also have increased O_2 as designated by the last digit signifying the percentage

FREON MIXES
4% O_2 96% CF_4
8.5% O_2 bal CF_4
17.5% O_2 bal CF_4
97% (99.5% He .5% O_2) 3% CF_4
Etching SiO_2, Si3N4, Si, molybdenum, tantalum, tantalum nitride, tungsten
DE100, will not etch aluminium, ceramic, GaAs, indium antimonide, or sapphire
PDE100, more common etchant than DE100, etches 20-30% faster
DE101, etches SiO_2 and Si_3N_4 without etching silicon

IR101 70% Ethylene Trichloride 30% 1, 1, 2 Trichlorotrifloroethane
Removal of inorganic contamination, particularly tin from resist or contaminated chambers (used in conjunction with O_2). Will also remove window oxide grown on exposed Si

FS100 97% He 3% O_2
Low temperature organic removal
Flash strip of photo masks

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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 spherilites in Polamide 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
100um 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 if 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
1um 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 that 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 0.1um.

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.

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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.

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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.

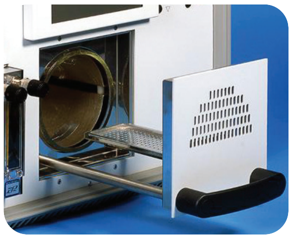
The solid state RF Generator is a solid state crystal controlled oscillator designed to provide up to 150 watts of continuous wave 13.56 MHz power to the reaction chamber. Maximum power transfer from the power supply to the reaction chamber is accomplished by matching the output impedance of the amplifier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



"Rack Out" Specimen Stage

Specifications

Instrument Case	450mm(W) x 350mm(D) x 300mm(H)
Barrel Work Chamber	'Pyrex' 160mm(L) x 110mm(Dia.) (Borosilicate Glass as standard)
Weight	25Kg
Plasma Output	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
Vacuum Gauge	ATM to 1 x 10^{-5} mbar Full scale normal 0.5 mbar to 1.0 mbar
Digital Timer Unit	Displays elapsed time with range select: 0-99 min. 99 sec. 0-99 hours. Automatic termination of Ashing Process
Dual Gas Flow Gauge	Dual Needle Valve flow control selectable for 1,2 or both gases
Supply	115V 60Hz (6 Amp Max) 230V 50Hz (3 Amp Max)
Services	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

93000	EMS 1050 Plasma Asher	each
91005-F	Rotary Vacuum Pump (Fomblin)	each

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).

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Techniques and Applications

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_2), 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_2, from liquid to gas without surface tension at the critical point.

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.

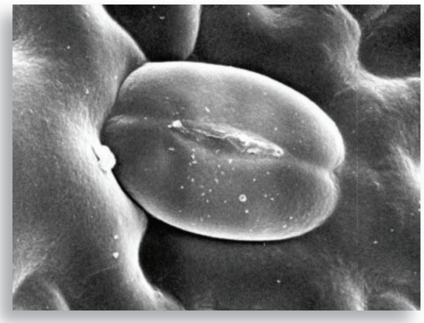
The phase diagram shows the pressure to temperature ranges where solid, liquid and vapor exist. The boundaries between the phases meet at a point on the graph called the triple point. Along the boundary between the liquid and vapor phases it is possible to choose a particular temperature and corresponding 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_2 is universally used today, despite early work with Freon 13 and nitrous oxide.

With CO_2 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_2 and the temperature then raised to above the critical temperature, the liquid CO_2 changes to vapour without change of density and therefore without surface tension effects which distort morphology and ultra structure.

Since liquid CO_2 is not sufficiently miscible with water, it is necessary to use an intermediate fluid which is miscible with both water and liquid CO_2. In practice intermediate fluids commonly used are methanol, ethanol, amyl acetate and acetone.

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 crystallites are also just visible on the surface of the leaf.

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Techniques and Applications

Critical Point Drying Principles

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.

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

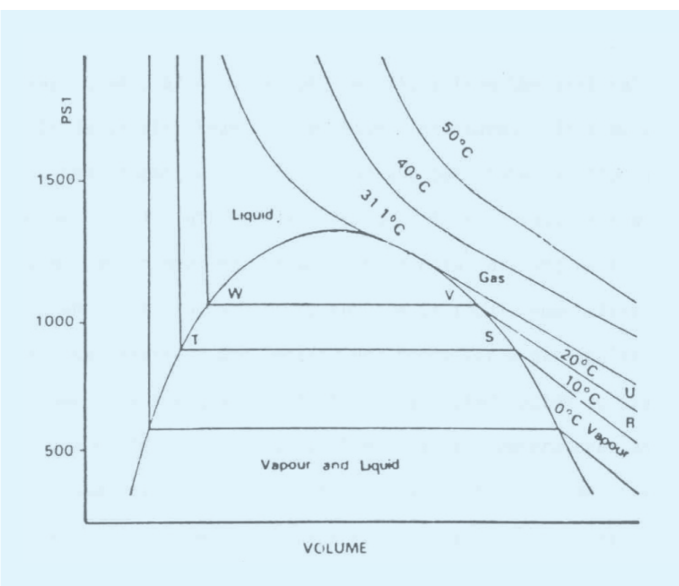
If a specimen had been in the liquid during this process it would have experienced a transition from a ‘wet’ to a 'dry' gas environment without being in contact with a surface, in this way avoiding the damaging effects of surface tension.

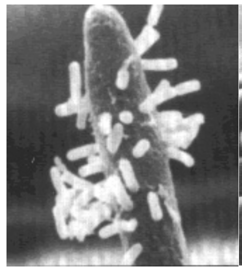
This is termed Critical Point Drying (CPD), the basis of which are the classic experiments carried out over 100 years ago during investigations on the liquification of gases.

The Critical Phenomena

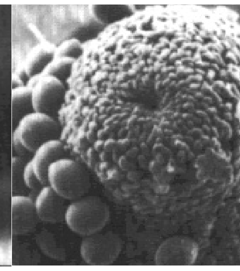
The principle of the experiments, which were initially carried out using carbon dioxide (CO_2), was to measure the change in volume with the application of pressure, of a fixed mass of gas, while maintaining a constant temperature. This was repeated for a range of different temperatures.

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’





Fruit body neck with spores adhering to the sides



Bacteria adhering to the tip of a fungus.

Powdery Mildew, hyphal filament on leaf surface.

Consider first the 10˚ C isothermal at low applied pressure. The CO_2 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.

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.

continues on page 40

Techniques and Applications

Critical Point Drying Principles (continued)

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_2 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

Therefore CO_2 remains the most common medium for the CPD procedure and is termed the 'Transitional Fluid'. However, CO_2 is not miscible with water and therefore water has to be replaced in the specimen with another fluid which is miscible with CO_2, this is termed the 'Intermediate Fluid'.

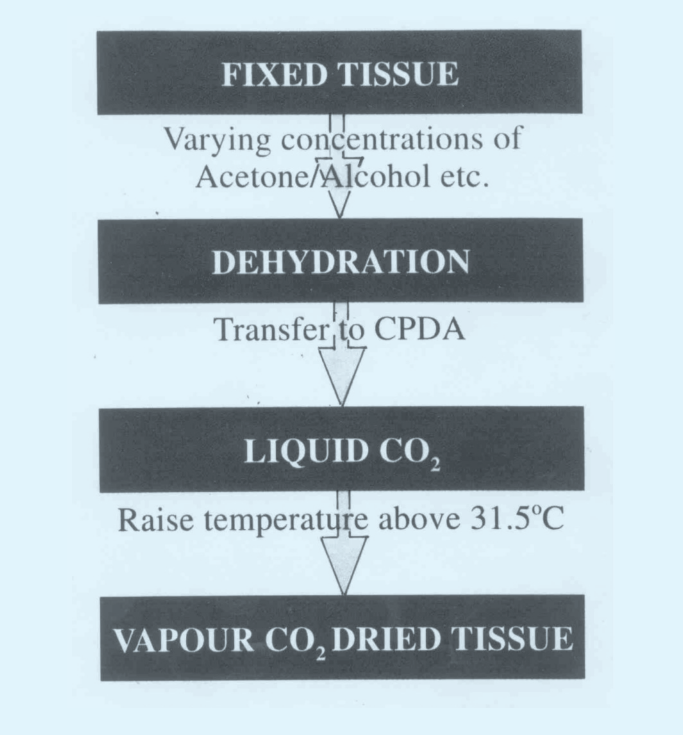
Ideally it will be able to replace the water in the specimen, and also serve as the 'Dehydration Fluid'. This is not exclusively the case, and additional steps may be used for particular circumstances.

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 vapour 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.

Critical point drying stages



a) Intermediate Stage

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

(Wet Specimen) H_2O ➡ Acetone ➡ CO_2 ➡ 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_2 (the Transitional Fluid) ensures satisfactory conditions after flushing (purging) for the CPD process to commence.

(Wet Specimen) H_2O ➡ Acetone ➡ 30%* 100% ➡ CO_2 ➡ 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.)

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.

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Techniques and Applications

Critical Point Drying Principles (continued)

(b) Transitional Stage

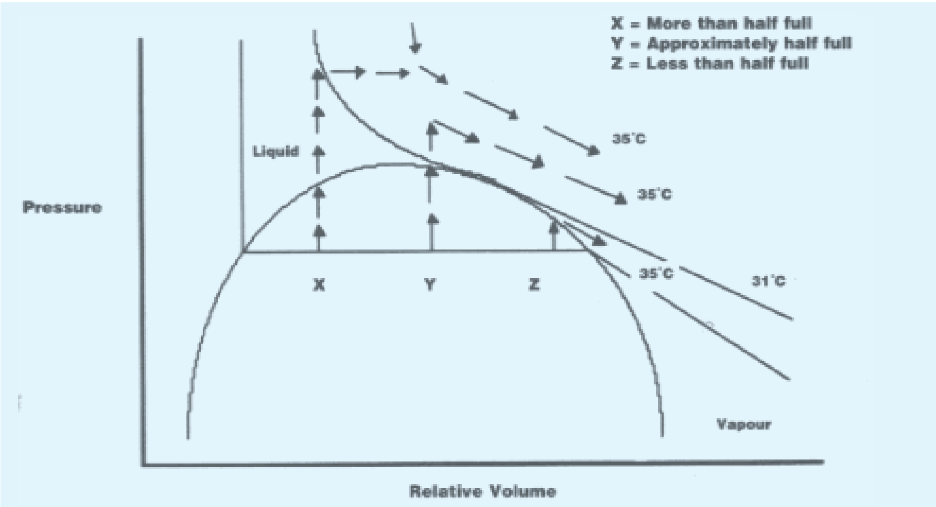
As discussed previously (see Figure 1) the conditions for which the critical point passage can be obtained for CO_2 are 31.1°C and 1072 psi. However, it must be remembered that these isothermals are obtained from a fixed mass of gas and an applied pressure for a series of constant temperatures.

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.

Figure 4: Transitional Fluids for CPD

Substance	Temp. C	P.S.I
CARBON DIOXIDE	+31.1	1072
FREON 13	+28.9	562
FREON 23	+25.9	495
WATER	+374	3212

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_2 isothermals as indicated in figure 5 with the Superimposed 'arrows' showing differing conditions for the CPD device.



It is already acknowledged that these circumstances are not exactly comparable. For example, during operation of the CPD we would fill at CO_2 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.)

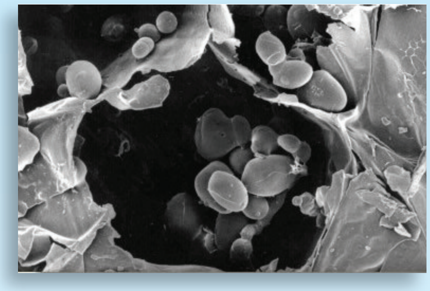
If we consider 'X' with the liquid CO_2 more than half filling the total available volume and we heat from 10°C to 35°C then we will make the transition from liquid to gas. The pressure rise will be rapid as the liquid will expand and the level will increase before the critical temperature is reached. This is termed 'going around' the critical point. Usually (as in the case of instruments supplied by Quorum Technologies) 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.

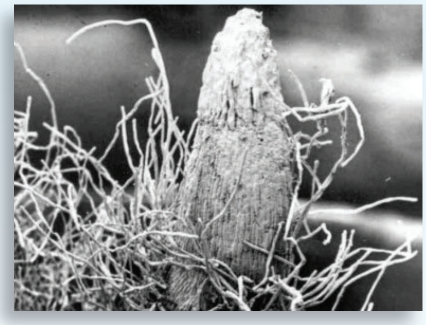


Starch Grains in Potato Tuber

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.

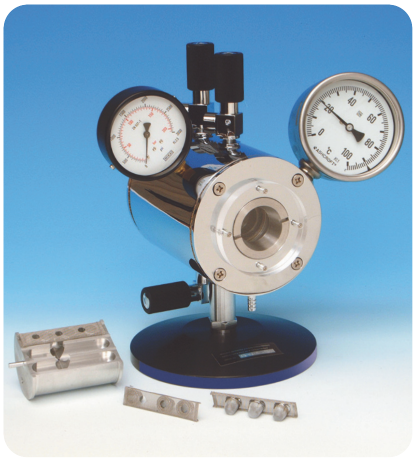


Barley Root Tip

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

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_2 and venting of gas - very durable; the rapid ingress of CO_2 helps prevents 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

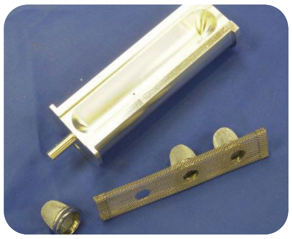
Dial gauges display pressure in the chamber and the temperature of water circulating through the jacket. Three pressure valves permit easy connection to the liquid CO_2 cylinder and allow liquid agitation and venting of the chamber. A source of hot running water is essential. Cooling is also useful, 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

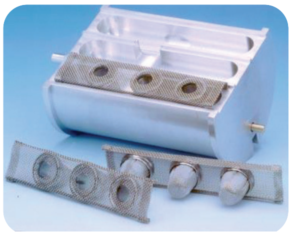
Safety is, of course, an important consideration with all pressure vessels. Should critical pressure and temperature be inadvertently exceeded, a bursting disc is incorporated in the chamber support. The critical point drying chamber itself is tested to 2,500psi, which is approximately twice the working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



EMS 3000 Specimen Boat

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_2. In this way the specimens are never allowed to dry out during the specimen loading and transfer stage of the process.



EMS 3100 Specimen Boat

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

EMS 3000 and EMS 3100 *(continued)*

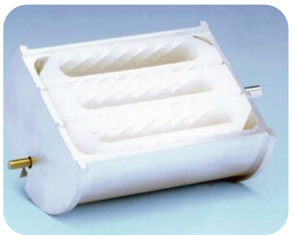
ability to withstand attack by solvents, such as ethanol. However, if acetone is used as the transition fluid then the EPDM seals have been found to be more resistant to chemical attack by that solvent.

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.

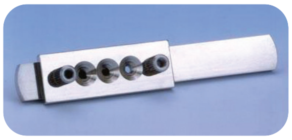
Options and Accessories

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_2 siphon cylinder

CO_2 Cylinder: The EMS 3000 and EMS 3100 require a cylinder of liquid CO_2 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_2, 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

EMS 3000	Critical Point Dryer Chamber dimensions: 30.1mm Ø x 82mm length
Supplied with:	EMS 3000-01 Specimen holder for tissue (boat) 1m liquid CO_2 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
EMS 3100	Large Chamber Critical Point Dryer Chamber dimensions: 63.5mm Ø x 82mm length
Supplied with:	EMS 3100-01 Specimen holder for tissue (boat) 1m liquid CO_2 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

EMS 850 Critical Point Dryer with Thermoelectronic Heating and Adiabatic Cooling

The EMS 850 Critical Point Dryer is designed for use with CO_2, 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_2–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_2 from a gas cylinder. The chamber is then heated to just above the critical temperature with subsequent critical pressure being achieved. The CO_2 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 miniscus for the correct level when initially filling the chamber.

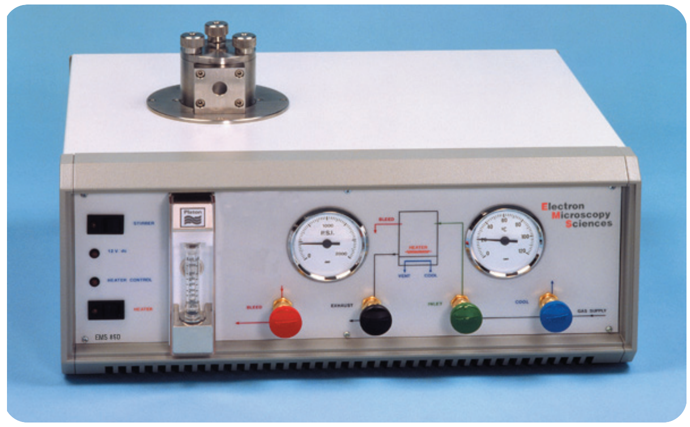
The EMS 850 is fitted with a fine let down needle as standard and flow gauge is no longer required.

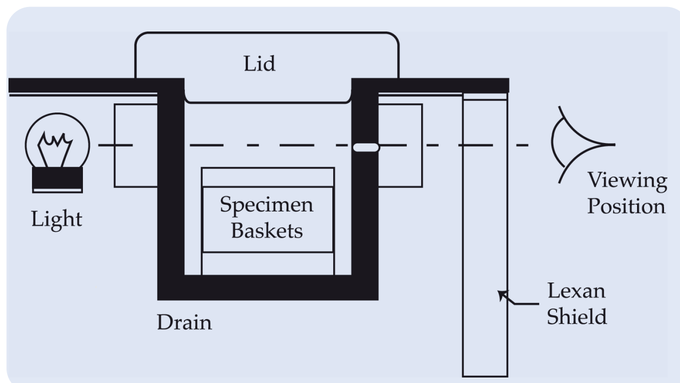
Features

- *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.*

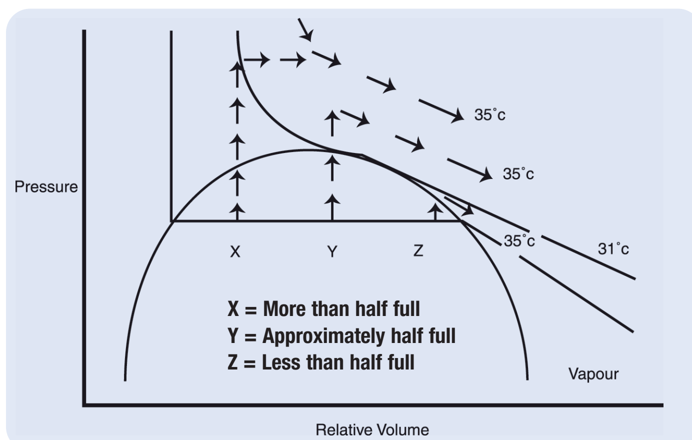
Specifications

Instrument Case	450mm (W) x 350mm (D) x 175mm (H)
Weight	12Kg
Specimen Chamber	30mm (Dia.) x 40mm (H) (Tested to 3000 psi)
Temperature Gauge	0-120°C
Thermal Cut-Out	40°C
Pressure Gauge	0-3000 psi
Pressure Relief	At 1500 psi
Rupture Disc	At 1900 psi
Peltier Cooling/Heating	+5°C to +35°C
Two On/Off Valves	Inlet/Outlet/Cool/Vent Needle Valve Letdown
Supply	115V 60Hz (6 Amp Max) 230V 50Hz (3 Amp Max)
Services	Requires CO_2 gas cylinder direct connection by high pressure hose. (High pressure hose included with instrument.)





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



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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

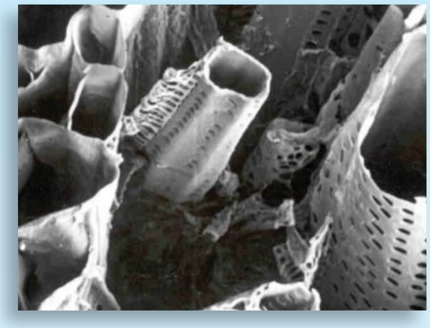
100mm or 150mm diameter wafers are held in a PTFE holding tray. The tray including wafer is immersed in acetone in order to remove all moisture from the specimen. After dehydration, the wafer and holder are transferred into the pre-cooled specimen chamber using the wafer transfer device. On completion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

Ordering Information

91090-WM	EMS 850WM Large Chamber Critical Point Dryer

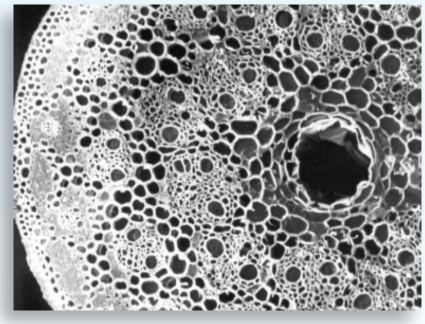
Requirements

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



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.

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°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°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 (H_2O) from solid to vapor phase the Triple Point pressure is a relative vacuum. There are notable exceptions such as Carbon Dioxide (CO_2) where the Triple Point is higher than atmospheric pressure.

Techniques and Applications

Freeze Drying Principles

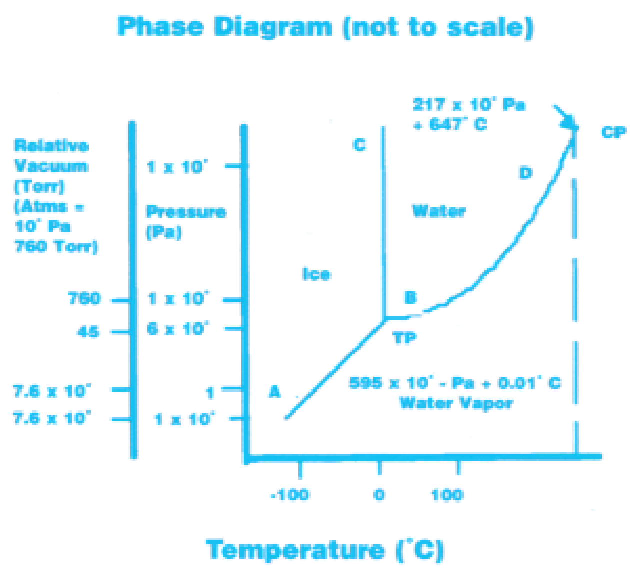
Sublimation consideration

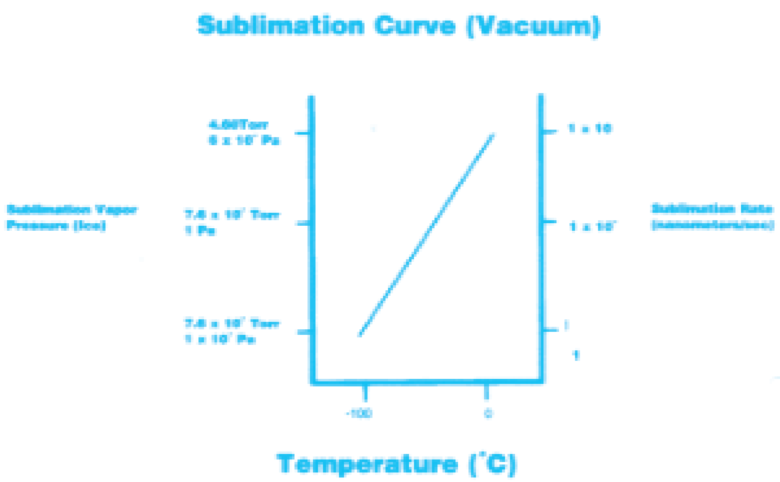
The Figure shows the phase diagram for the ICE/WATER/WATER VAPOR system. The Curve A-B is termed the Sublimation Curve, at which the Solid and Vapor fire in equilibrium. Similarly for Curve B-C the Melting Curve and B-D the Evaporation Curve, terminating at C-P the Critical Point. The point of intersection 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.

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_2O is not limiting) which are totally temperature dependent.





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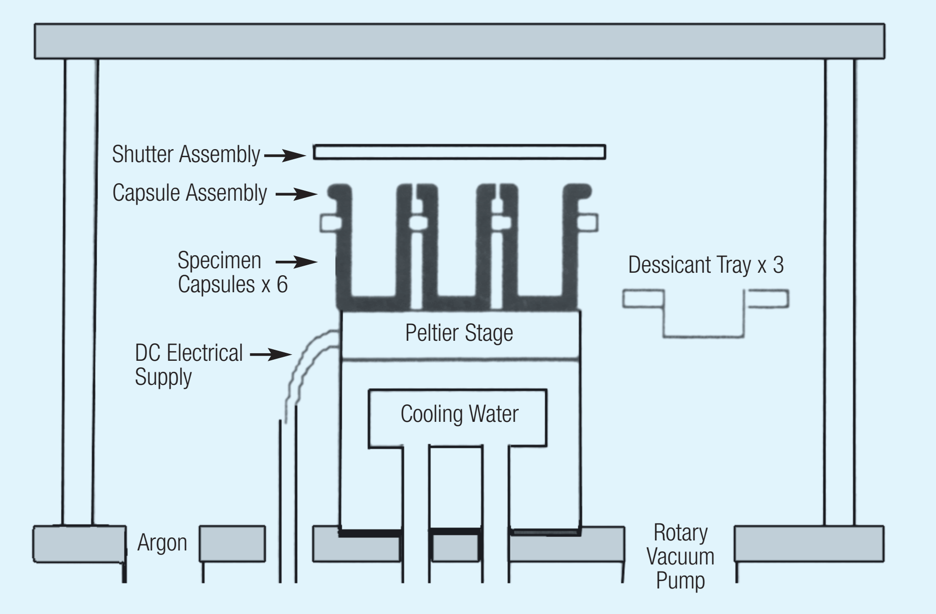
Techniques and Applications

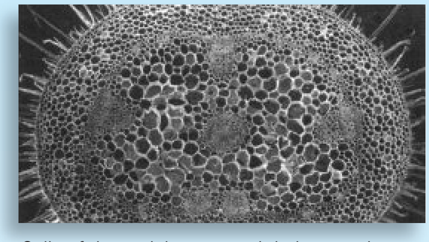
Freezing Considerations

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.

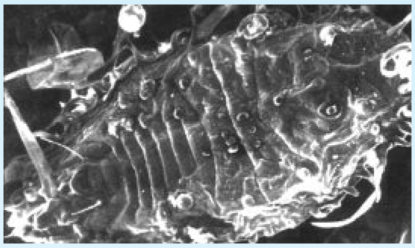
A range of cryogens and freezing techniques are being applied to the freezing of specimens, the understanding of the mechanisms of which, are still at an early stage. However, with exceptions, (Hyperbaric freezing containment of ice crystals is generally less than 20 micrometers from the surface, and Liquid Nitrogen (LN_2) 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

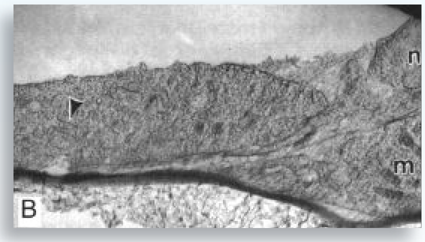


Cells of the petiole assumed their normal isodiametric shape after dying.



Scanning Micrograph of an Aphid, Freeze dried using the EMS 750 system and Sputter Coated with Gold. Aphids secret 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). subcellufar structures such as nucleus (n), michochondria (m) and PER can be seen, Marker=1.m

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

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°C, with back-up water cooling at a nominal 15°C. Both the Temperature and the Timer can be pre-selected, and the drying cycle will be completed automatically.

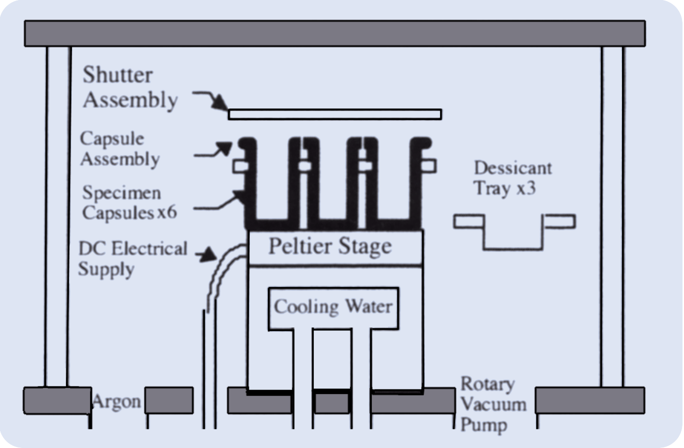
Provision is made at the end of the drying cycle to allow the specimen to assume room temperature, or be subsequently warmed prior to embedding. Disposable desiccant containers are located in the preparation chamber to enhance the water vapor removal, and with a suitable container, the vacuum chamber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

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



Cross-sectional View of Peltier Cooled Stage

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
91082	EMS 350 Sputtering Attachment	each

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EMS 775 Turbo Freeze Dryer

The EMS 775 Turbo Freeze Dryer operates at temperatures down to -140°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

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

Ordering Information

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

Replacement Parts

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

Optional Accessories

95128	EMS 170 Slushing Chamber	each
95127	EMS 175 Auto Top Up Unit +Controller	each
91040	EMS 7640 Carbon Coating Attachment	each
91082	EMS 350 Sputter Coating Attachment	each

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°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°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°C (the recrystallization temperature) and prevent sublimation (below -130°C the rate of sublimation of ice is very low, of the order of 0.001nm/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 over come 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:

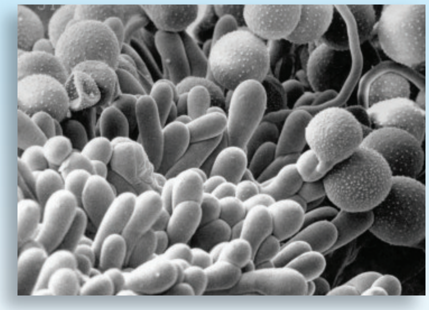
- Specimen viewed in fully hydrated state
- Soluble materials are retained
- Less relocation of highly diffusible elements
- Little or no mechanical damage
- Time lapse experiments and evaluating industrial processes at timed intervals
- Usually no exposure to toxic reagents
- Rapid process
- High resolution 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.

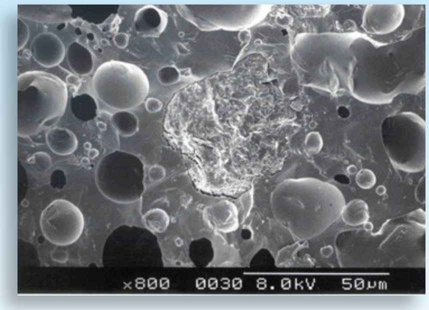
Botanical



Pollen of cactus Zygocactus truncatus

Germinating pollen grains of Zygocactus truncatus.

Foodstuffs



Chocolate Bar

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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 import agricultural sciences.

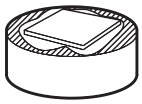
More recently cryo-SEM is becoming an essential tool for pharmaceutical, cosmetics and healthcare industries, where it is used in basic applied research and for routine QA of many products, such as creams, cosmetics and drug delivery systems.

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.

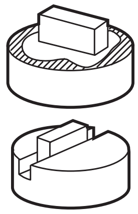
Some specimen mounting techniques for cryo-SEM

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



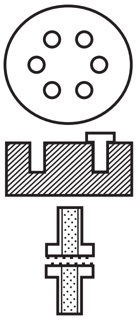
This technique is used for edge observation and fracture. Roughen surface of stub with fine emery paper. Specimen is placed on its edge in a machined slot and secured with mounting media.

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 nemotode worms).

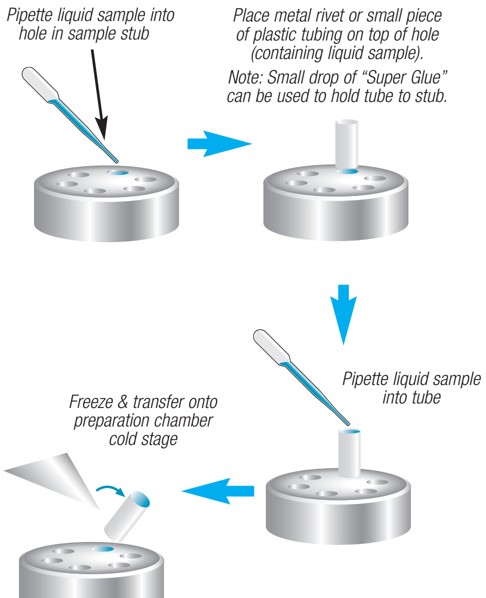
The specimen is laid on surface so that its dampness slightly dissolves the film emulsion allowing the specimen to adhere to the film surface. Exposed unused film with the emulsion side uppermost is secured to the stub with mounting media. It may be useful to scrape off the protective coating of the film emulsion first to assist conductivity.

Rivet mounting



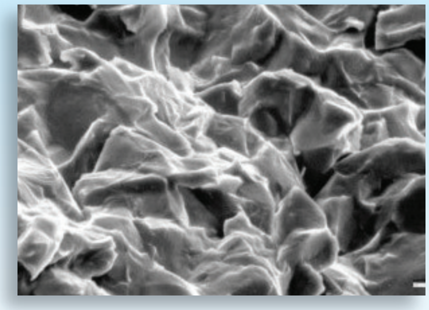
For liquids and for when specimens need to be frozen off the stub to achieve fast freezing rates. The rivet is placed in the hole and filled with liquid prior to freezing. If the specimen needs to be frozen away from the stub, two liquid-filled rivets are held together and then frozen prior to transfer onto the stub.

Alternative rivet mounting method



Result: clean surface fracture

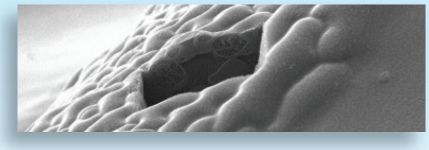
Geological

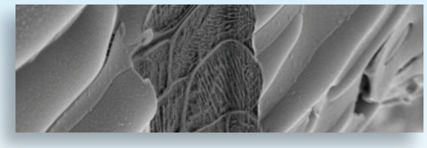


Wax crystals in gas oil

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

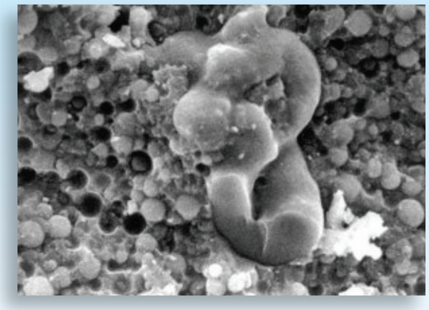




Arabidopsis plant

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.

VACUUM PUMPS & ACCESSORIES

Edwards RV Series Vacuum Pumps

Rotary vacuum pumps — double stage.

RV8 Vacuum Pump

- *6.9 cfm (195 L/min) displacement*
- *Ultimate vacuum (without gas ballast) 1.5x10^{-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*

Edwards Super Grade A Oil is recommended for use with the RV8. This pump should be used with our EMS 650 Large Sample Coater; EMS 1250 Cryogenic Preparation System. Weight 50 lbs (24 kg).

91025	Vacuum Pump RV8	each
91025-E	Vacuum Pump RV8, 220 Volts	each

RV5 Vacuum Pump

- *4.1 cfm (117L/min) displacement*
- *Ultimate vacuum (without gas ballast) 1.5x10^{-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*

Edwards Supergrade A Oil is recommended for use with the RV5.

This pump should be used with our EMS 550 Sputter Coater, EMS 450 Carbon Coater; EMS 950 Turbo Evaporator; EMS 750 Freeze Drier. Weight 43 lbs (19.6kg)

91005	Vacuum Pump RV5	each
91005-E	Vacuum Pump RV5, 220 Volts	each

RV3 Vacuum Pump

- *Speed - (Pneurop 6602)*
- *50 Hz operation - 2 ft^{3}min-1 (3.3 m^{3}h-1)*
- *60 Hz operation - 2.3 ft^{3}min-1 (3.9 m^{3}h-1)*
- *Ultimate Vacuum (Total Pressure) - High Vacuum Mode - 2 x 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. This pump can be used with our EMS 100, EMS400, EMS 450, EMS 500, EMS 550, EMS750, EMS 950. Weight: 43 lbs (19.6 kg).

91003	Vacuum Pump RV3	each
91003-E	Vacuum Pump RV3, 220 Volts	each

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): 1.1x10^{-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*

Edwards Supergrade A 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

Foreline Traps

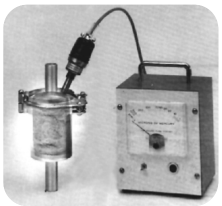
These rechargeable in-line traps effectively block oil back-streaming from mechanical pumps. Any mechanically pumped system, even one with a turbo pump, should have a foreline traps. The trap is placed in the foreline between the roughing pump and the diffusion or turbo pump. It uses a disposable, oxygen-free 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	½"	Aluminum	5 CFM	2½"
91050-15	¾"	Aluminum	5 CFM	2½"
91050-20	1"	Aluminum	10 CFM	3"
91050-25	1½"	Aluminum	10 CFM	3"
91050-30	1¾"	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

Mechanical Pump Oil

Edwards Supergrade A 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 Supergrade A Oil	1 L
60742	Edwards Supergrade A Oil	4 L

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Carbon Rods/Graphite Rods

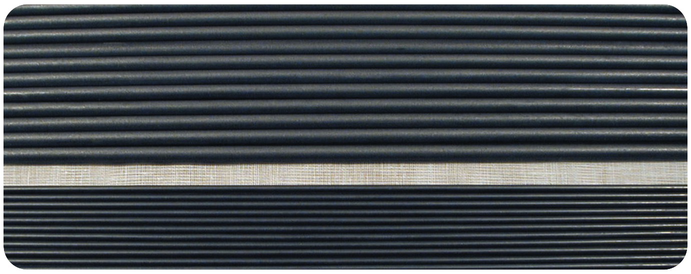
Properties of Carbon

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^3
Specific gravity (amorphous): 1.88 g/cm^3
Specific gravity (diamond): 3.51 g/cm^3
m. p. sublimes: ~3550°C **Boiling point:** 4200°C
Evaporation Temp (at which a substance has a vapor pressure of 1.33 x 10^{-2} 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 CVP™ (Chemical Vapor Process) purity and spectrographic grade, grade 1, produced to ASTM tolerances or even tighter.



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%

Cat. #	Dimensions	Qty
70200	12" (304mm) L x 1⁄8" (3mm) Diameter	12/pk
70230	12" (304mm) L x 1⁄4 " (6.2mm) Diameter	12/pk

Carbon Cord/Carbon Fiber

For carbon coating. The fiber will give you thin to medium coatings. The cord will give you single shot thick coatings.

91045	Carbon Fiber	1 M
91046	Carbon Cord	1 M

Presharpened Pointed Graphite Rods

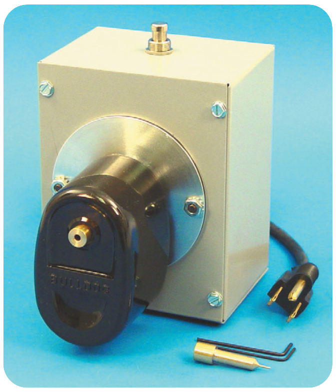
To make your work easier we invite you to order our pre-sharpened rods. (All pre-sharpened rod tips are pointed to a 60 degree angle)

Dimensions	Cat. #	Qty
SINGLE POINT		
LOA: 2" (51mm) x 1⁄8" (3.1mm) Dia. Tip Length: 3⁄16" (9mm) x Tip OD 1⁄16"(1.6mm)	**70220-01**	50/pk
LOA: 2" (51mm) x 1⁄4" (6.2mm) Dia. Tip Length: 3⁄16" (9mm) x Tip OD 1⁄16"(1.6mm)	**70221-01**	50/pk
DOUBLE POINT		
LOA: 2 3⁄8" (61mm) x 1⁄8" (3.1mm) Dia. Tip Length: 3/16" (9mm) x Tip OD: 1⁄16"(1.6mm)	**70220-02**	50/pk
LOA: 2 3⁄8" (61mm) x 1/4" (6.2mm) Dia. Tip Length: 3⁄16" (9mm) x Tip OD: 1⁄16"(1.6mm)	**70221-02**	50/pk

Carbon Rod Sharpener

Produces 1mm diameter carbon tip on ¼" or ⅛" 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 be a durable electric motor, producing a smoother and more uniform carbon tip than any hand sharpener.

The kit includes a blade gauge and a ⅛" rod adapter.



70305-10	Electric Carbon Rod Sharpener, 110V	each
70305-20	Electric Carbon Rod Sharpener, 220V	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”)

EMS #	Description	Qty
73808-B1	Pyrolytic Graphite Boat B1	5/pack

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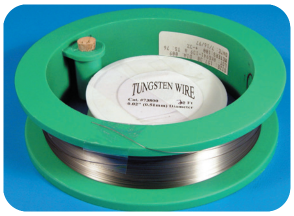
Tungsten Metal Wire and Boats

Properties of Tungsten

Name: Tungsten **Chemical symbol:** W
Atomic Radius: 2.02Å **Atomic Volume, cm³/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

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.



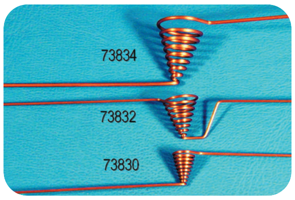
Tungsten Wire

We are offering tungsten wire in 99.90% purity. Available in 0.02" (.51mm), and 0.009" (0.23mm) diameters.

73800	Tungsten Wire, 0.020" (0.51mm) Diameter	20 ft
73801	Tungsten Wire, 0.020" (0.51mm) Diameter	100 ft
73802	Tungsten Wire, 0.025" (0.64mm) Diameter	20ft
73803	Tungsten Wire, 0.025" (0.64mm) Diameter	100ft
73804	Tungsten Wire, 0.009" (0.25mm) Diameter	20ft
73805	Tungsten Wire, 0.009" (0.25mm) Diameter	100ft

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

73830	Tungsten Wire basket A	50/pack
73832	Tungsten Wire basket C	50/pack
73834	Tungsten Wire basket D	50/pack

Tungsten Wire Filaments

V-Shaped Filaments

High purity, vacuum grade tungsten wire 0.02" (0.50mm) diameter, V-shaped filaments ½" (12.7mm) deep, with a bottom angle of 45˚, either single wire or triple strand.

Cat. #	Overall Length	No. Strand	V- Opening	Qty
73840	3" (76.2mm)	Single	¾" (19mm)	10/pk
73842	3" (76.2mm)	Triple	⅝" (16mm)	10/pk
73844	3½" (88.9mm)	Triple	¾" (19mm)	10/pk

Coiled Filaments

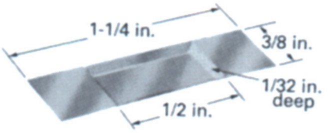
Single or 3-strand coiled tungsten filaments, 0.5mm (0.002") diameter wires.

Cat.#	Description	Qty
73938-1	4" long Horizontal Helix, Coil length 45mm (1¾") with 6 turns, 6.3mm(¼") diameter coil. Single strand	5/pk
73938-3	4" long Horizontal Helix, Coil length 45mm (1¾") with 6 turns, 6.3mm(¼") diameter coil. Triple strand	5/pk
73939-1	5" long Horizontal Helix, Coil length 51mm (2") with 12 turns, 9.5mm(½") 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").

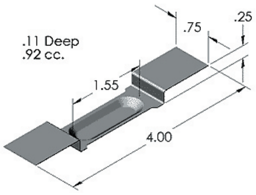
The trough: 12.7mm(L) x 9.5mm(W) x 3.2mm Deep (½" x ⅜" x 1⁄32")



73816 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").

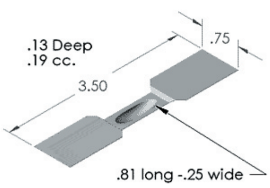
Trough: 39.37mm (L) x 2.79mmDeep, 0.92 ml



73816-B2 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").

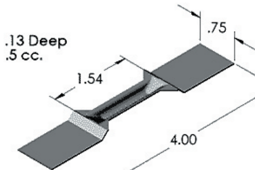
Trough: 39.1mm(L) x 6.35mm(W) x 3.3mmDeep, 0.5ml



73816-B3 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").

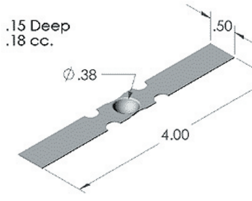
Trough: 28.19mm(L) x 15.87(W) x 3.3mmDeep, 0.99ml



73816-B4 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").

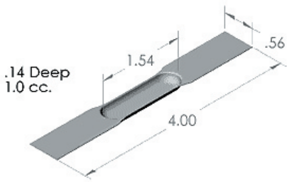
Trough: 9.65mm Diameter x 3.81mmDeep, 0.18ml



73816-B5 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 1⁄2")

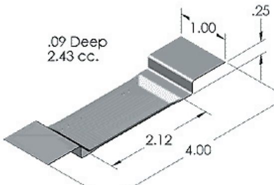
Trough: 39.1mm(L) x 12mm(W) x 3.55mmDeep, 1.0ml



73816-B6 Tungsten Boat B6 5/pk

Tungsten Boat B7: Thickness of tungsten foil is 0.25mm (0.010"). The overall length is 101.6mm x 25.4mmL x (4" x 1")

Trough: 53.84mm(L)x25mm(W) x2.28mmDeep, 2.43ml



73816-B7 Tungsten Boat B7 5/pk

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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/Paladium, Diameter: 0.008"(0.2mm)	10 ft
73601	Platinum/Paladium, Diameter: 0.008"(0.2mm)	50 ft

Silver Wire, Purity: 99.99%

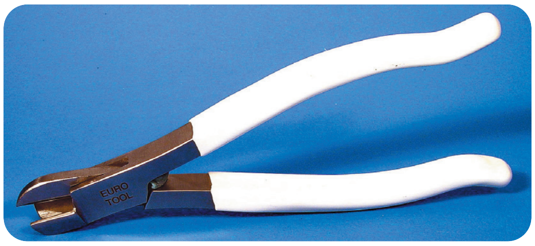
73700	Silver wire, Diameter: 0.008"(0.2mm)	10 ft
73701	Silver wire, Diameter: 0.008"(0.2mm)	50 ft

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

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

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.

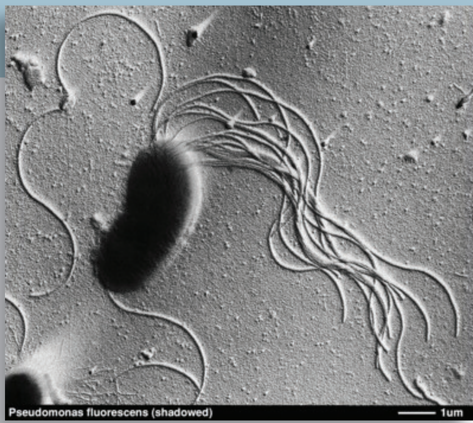
73820	Molybdenum Foil 0.002"(0.05mm) thick; size 6x6" (15.2x15.2cm)	each
73822	Tantalum Foil 0.003" (0.075mm) thick; size 6x6"(15.2x15.2cm)	each

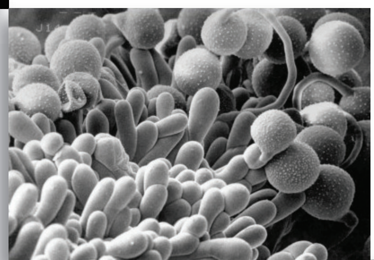
Sputter Targets

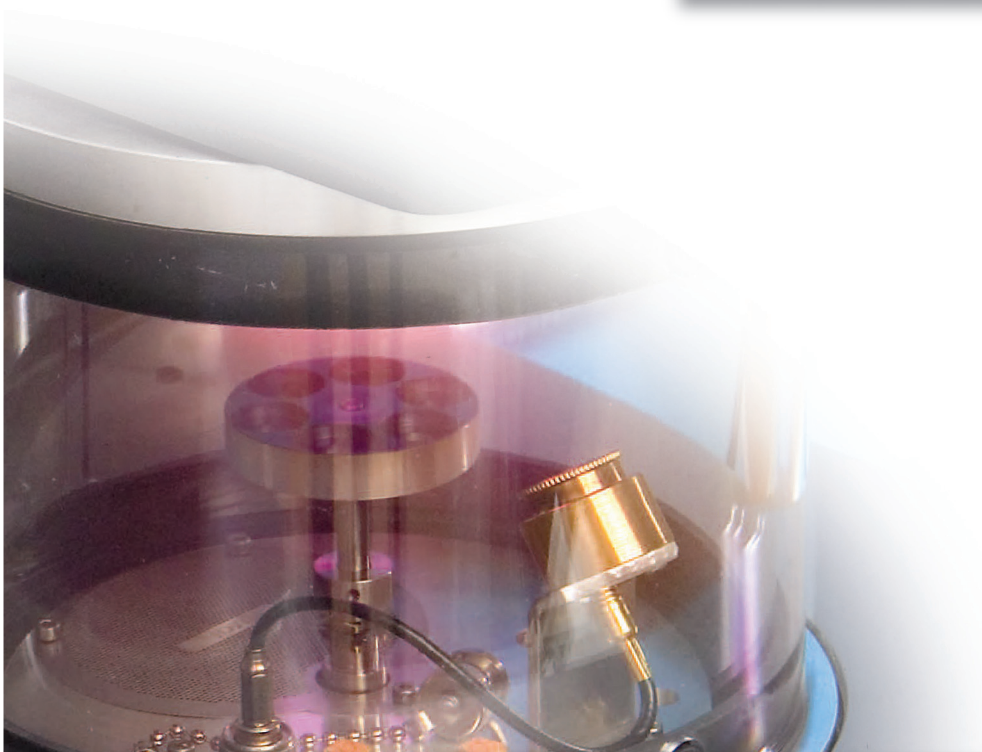
EMS precious metal sputtering targets are made from high purity metals, starting 99.99% with most standard disc sizes, for use on most sputtering units from many manufacturers: Emitech, Emscope, Bio-Rad, Polaron, Edwards, Balzers, Plasma Sciences, Technics Hummers, Denton, Cressington, and much more.

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 avaible 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.

Target Diameter, mm	EMS Catalog Number Gold (Au)	Platinum (Pt)	Palladium (Pd)	Au/Pd 60:40	Pt/Pd 80:20	Silver (Ag)
20.0	**91006-Au**	**91006-Pt**	**91006-Pd**	**91006-AP**	**91006-PP**	**91006-Ag**
20.4	**91007-Au**	**91007-Pt**	**91007-Pd**	**91007-AP**	**91007-PP**	**91007-Ag**
32.0	**91008-Au**	**91008-Pt**	**91008-Pd**	**91008-AP**	**91008-PP**	**91008-Ag**
39.0	**91009-Au**	**91009-Pt**	**91009-Pd**	**91009-AP**	**91009-PP**	**91009-Ag**
42.0	**91013-Au**	**91013-Pt**	**91013-Pd**	**91013-AP**	**91013-PP**	**91013-Ag**
50.0	**91014-Au**	**91014-Pt**	**91014-Pd**	**91014-AP**	**91014-PP**	**91014-Ag**
50.8	**91015-Au**	**91015-Pt**	**91015-Pd**	**91015-AP**	**91015-PP**	**91015-Ag**
54.0	**91016-Au**	**91016-Pt**	**91016-Pd**	**91016-AP**	**91016-PP**	**91016-Ag**
57.0	**91017-Au**	**91017-Pt**	**91017-Pd**	**91017-AP**	**91017-PP**	**91917-Ag**
60.0	**91010**	**91012**	**91010-Pd**	**91011**	**91010-PP**	**91010-Ag**
63.5	**91018-Au**	**91018-Pt**	**91018-Pd**	**91018-AP**	**91018-PP**	**91018-Ag**
75.0	**91019-Au**	**91019-Pt**	**91019-Pd**	**91019-AP**	**91019-PP**	**91019-Ag**
76.0	**91020-Au**	**91020-Pt**	**91020-Pd**	**91020-AP**	**91020-PP**	**91020-Ag**
Annular Targets (Outside Diameter x Inside Diameter, mm)						
117OD x 89 ID	**91030-Au**	**91030-Pt**	**91030-Pd**	**91030-AP**	**91030-PP**	**91030-Ag**
87 OD x 57 ID	**91031-Au**	**91031-Pt**	**91031-Pd**	**91031-AP**	**91031-PP**	**91031-Ag**
59 OD x 33 ID	**91032-Au**	**91032-Pt**	**91032-Pd**	**91032-AP**	**91032-PP**	**91032-Ag**
57 OD x 40 ID	**91033-Au**	**91033-Pt**	**91033-Pd**	**91033-AP**	**91033-PP**	**91033-Ag**







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