EQUIPMENT & ACCESSORIES CATALOG

EDITION III

COOLING STAGES, RECIRCULATING HEATERS AND CHILLERS, **GLOW DISCHARGE SYSTEMS,** SPUTTER COATERS, SEM/TEM CARBON COATERS, **LARGE CHAMBER SPUTTER COATERS,** FREEZE DRYERS, **CRITICAL POINT DRYERS,** PLASMA ASHERS, **CRYO-SEM PREPARATION SYSTEMS,** SPECIMEN TRANSFER SYSTEMS, **AND MORE...**

Electron Microscopy Sciences

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EQUIPMENT & ACCESSORIES CATALOG

EDITION III

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NEW PRODUCTS...

NEW! EMS Plus Series
Sputter Coaters/Carbon Coaters

To offer improved ultra-fine coating we have introduced the NEW EMS150V Plus, which offers an ultimate vacuum of 1×10^{-6}mbar for superior results. Large chamber sputter coaters are also available for specimens up to 200mm in diameter. Most coaters can also be configured for carbon evaporation using easy-change carbon rod and carbon fiber inserts.

See pages 18–29, 38–41, 48–51.

Electron Microscopy Sciences

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Why cool?

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

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

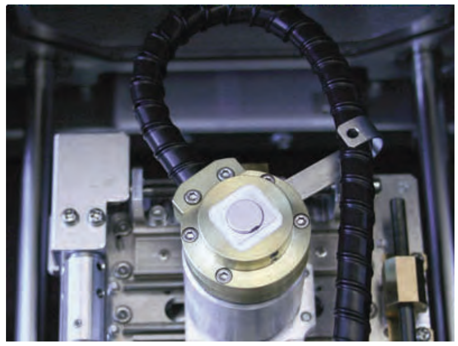
At 300Pa, the specimen temperature needs to be less than -9.5°C, and at 85Pa less than -25°C to stop water evaporation. Therefore, by cooling a specimen to -25°C, chamber pressures up to 85Pa can be used with little or no water loss by evaporation. In this way, changes in specimen structure can be minimised.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

The EMS Coolstage for SEM, LV or VP

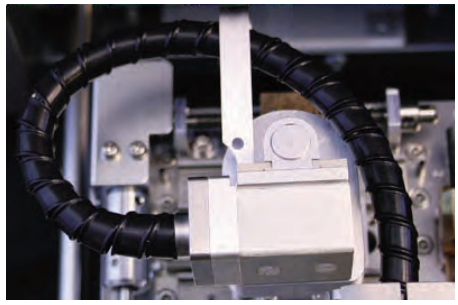
Overview

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

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



Standard Coolstage. Range: -30°C to +50°C at 300Pa



Ultra Coolstage. Range: -50ºC to +50ºC at 300Pa

Features - Standard Coolstage

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

Features - Enhanced Coolstage

- **Temperature range -30°C to +160°C at 300Pa**
- **All other specifications as per Standard Coolstage**

Features - Ultra Coolstage

- **Temperature range -50°C to +50°C at 300Pa**
- **All other specifications as per Standard Coolstage**

Product Description

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

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

Compact, efficient cooling and temperature control

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

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

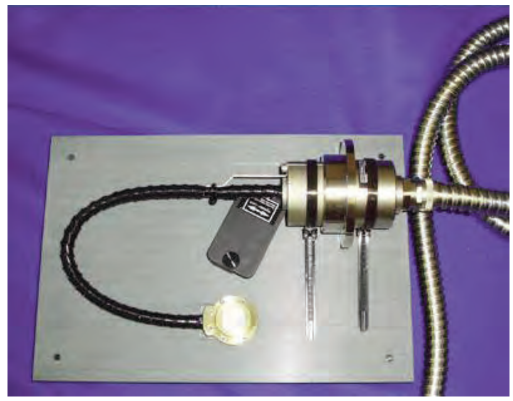
III The EMS Coolstage *(Continued)*

Rapid specimen exchange

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

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

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



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

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

SPECIFICATIONS

Specimen Size	10mm Ø (adaptor stub for 12" Hitachi stubs can be supplied on request)
Stage Temperature Range	Note: Higher vacuum will allow for cooler temperatures, compatible with high vacuum levels to $1x10^{-5}$Pa *Standard Coolstage:* -30°C to +50°C at 300Pa with no external cooling water from SEM (at ambient +20°C) *Enhanced Coolstage:* 30°C to +160°C at 300Pa with no external cooling water from SEM (at ambient +20°C) *Ultra Coolstage:* -50°C to +50°C at 300Pa with no external cooling water from SEM (at ambient +20°C)
Temperature Display Resolution	-0.1°C
Temperature Stability	+/- 0.2°C
Temperature Accuracy	+/- 1.5°C or 2% (whichever is greater)
Stage Movement	Normal x, y and z movements maintained. Tilt maintained for X-ray analysis (up to 45°). No rotation
Working Distance	As on SEM, Coolstage is set to the SEM Eucentric height
Operating Voltage	100V or 115V or 230V @ 100VA, voltage tolerance +/- 10%
Size and Weight	*Operation/display unit:* 90mm L x 112mm W x 350mm H, 300g *Power supply/cooling unit:* 305mm L x 245mm W x 330mm H, 15kg
Packed Size and Weight	550mm L x 580mm W x 400mm H, 28kg

Supplied With: Operating manual, one set of interconnecting cables and mains supply lead, storage block for specimen cooling unit when not in use, specimen holders: 15 x flat 10mm Ø stubs and 15 x dish 10mm Ø OD stubs

ORDERING INFORMATION

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

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 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

Cat No.	Description	Qty.
91098	EMS 4860 1/5 HP Recirculating Heater/Chiller	each
91099	EMS 4870 1/2 HP Recirculating Heater/Chiller	each
91090	EMS 4880 3/4 HP Recirculating Heater/Chiller	each
91095	EMS 4890 1 HP Recirculating Heater/Chiller	each

Rapid, reliable results...

EMS GloQube™ Glow Discharge System for TEM and Surface Modification

OVERVIEW

The GloQube® is a compact, easy-to-use glow discharge system primarily used for the hydrophilisation (wetting) of TEM carbon support films and grids. Other applications include surface modifications, for example for enhancing polymer bonding.

The GloQube has a convenient single drawer with two independent vacuum chambers: a clean chamber for glow discharge applications requiring hydrophobic/hydrophilic conversion and a vapour chamber designed for hydrophilic/hydrophobic (negative or positive) conversions.

FEATURES

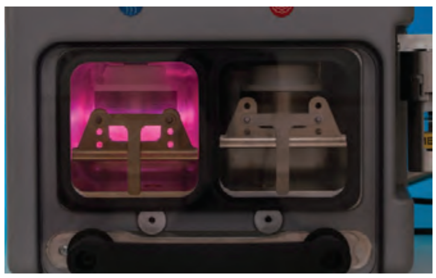
- **Dual independent chambers**
- **Hydrophilic/hydrophobic and negative/positive modes**
- **Fully automatic, short process times**
- **Intuitive touch screen control**
- **Safe vapor delivery using septum-sealed vials**
- **Automatic valving between chambers to prevent cross-contamination**
- **Quick and easy sample loading**
- **Controlled venting to prevent sample disturbance**
- **Consistent, reliable results**
- **Three-year warranty**

Unique Dual Chamber Processing, Safe Handling of Reagents

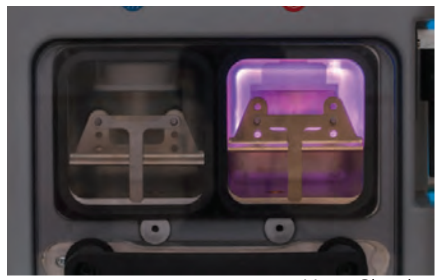
The GloQube has two independent vacuum chambers: a clean chamber, designed for applications requiring hydrophobic/hydrophilic conversion, typically using air as the process gas; and a vapor chamber, designed for use with reagents such as methanol and alkylamine. With operator safety firmly in mind, reusable septum-sealed reagent vials are used. Loading and removing reagents is convenient and reliable – the vial, located in its holder, is inserted into a shielded needle using a simple bayonet fitting.

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 being preset. 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.

The primary application of the EMS GloQube™ is the hydrophilization (wetting) of carbon-coated TEM support films and grids which otherwise have the tendency to be hydrophobic. Glow discharge treatment with air will make film surfaces negatively charged and hydrophilic and allow the easy spread of aqueous solutions. This and other processes are outlined below.



Clean Chamber



Vapor Chamber

Vapor Delivery System

Glow Discharge Process

Surface State	Charge	Atmosphere	Typical Applications
Hydrophilic	Negative	Air	Carbon coated TEM grids
Hydrophilic	Positive	Air – with magnesium acetate post-treatment	Nucleic acid adhesion to carbon films
Hydrophilic	Positive	Alkylamine	Proteins, antibodies and nucleic acids
Hydrophilic	Negative	Methanol	Positively charged protein molecules (e.g. ferritin, cytochrome c)

What is... Glow Discharge?

Electric glow discharge is a type of plasma formed by passing a current at 100 V to several kV through a gas at low pressure (i.e. in a vacuum system). The main application of glow discharge in electron microscopy (EM) is to convert naturally hydrophobic ('water-hating') carbon-coated transmission electron microscopy (TEM) support grids into a hydrophilic ('water-loving') condition. Glow discharge treatment with air will make film surfaces negatively charged and hydrophilic and allow the easy spread of aqueous solutions.

Other treatments include:

Hydrophilic-positive treatment in air with magnesium acetate post-treatment to allow nucleic acid adhesion to carbon films.

Hydrophobic-positive treatment with alkylamine for proteins, antibodies and nucleic acids.

Hydrophobic-negative treatment in air for positively charged protein molecules, (e.g. ferritin and cytochrome c).

Glow discharge can also be used for modifying surface, for example, to increase bond strength of polymers.

Glow discharges are sometimes considered to be 'imperfect' plasmas and cannot be used to plasma etch or plasma ash specimens their use mainly being confined to altering surface energies, not the removal of bulk material. For these applications the 1050X RF Plasma Barrel Reactor is recommended.

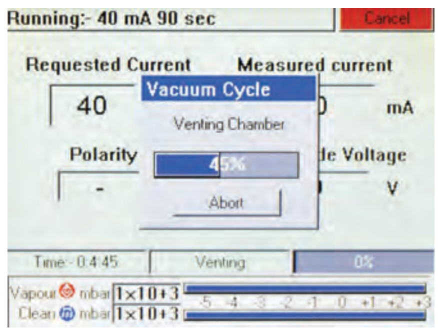
III EMS GloQube™ (continued)

Easy Sample Loading, Fast Turnaround Times

Each chamber can accommodate two 25 x 75 mm glass microscopes slides. Loading could not be easier using draw-style chamber doors and specimen stages. The stages are height adjustable and fitted with removable glass slide holders. For additional convenience – and to allow easy access for chamber cleaning – the stages can be completely removed.

Vacuum, Automatic Valving and Controlled Venting

The GloQube™ has automatic valving between chambers which maintains cleanliness by preventing cross-contamination. At the end of a process run, automatic soft venting to atmosphere through filtered inlets ensures TEM grids are not disturbed. The GloQube™ requires a single vacuum pump working in the 0.1 to 1 mbar range. A typical pump time to operational vacuum is 60 seconds.



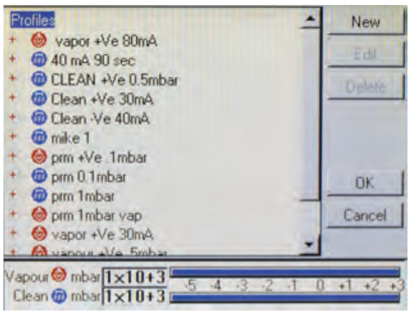
GloQube door and sample stage can be removed for cleaning chamber

EMS GloQube-D and Optional Pfeiffer DUO 6 Rotary Pump

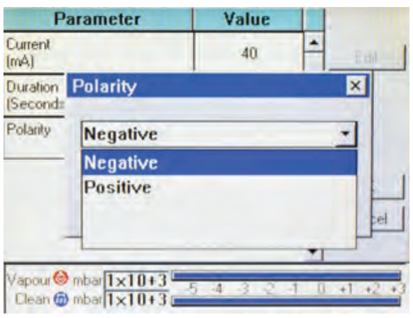
EMS GloQube™ (continued)

Touch Screen Control – Rapid Data Input, Simple Operation

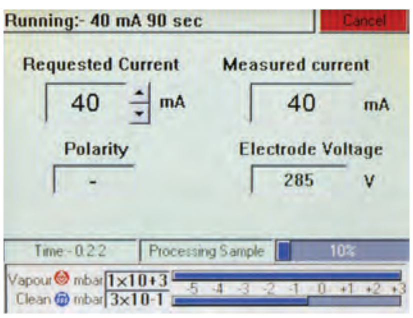
The intuitive touch screen allows multiple users to rapidly input and store preferred process "recipes". Typical default glow discharge protocols are loaded as standard. Additionally, help files and useful maintenance data such as system on time and time since last clean are readily available to the operator. An Ethernet communications port is included for software updates.



Stored Profiles



Selecting a New Profile



A Typical Process Run

SPECIFICATIONS

Power and Processes

Plasma current	1-40 mA
HV power supply	30 W
Maximum voltage	800 V
Electrode polarity –	
clean chamber	DC glow positive DC glow negative
vapor chamber	DC glow positive DC glow negative
Sample stage	125 x 100 mm with location for two 25 x 75 mm (1" x 3") glass slides
Sample stage operational height	Adjustable 12.5 mm (0.5"), 22.5 mm (0.9") or 35 mm (1.38")
Pump hold time requirement	0-24 hours
Process time	1-600 seconds

Safety

Chamber vent inlets	Filtered air inlets with slow vent to minimize sample disturbance
On-board reagent storage	Reagents (e.g. methanol or alkylamine) are contained in reusable sealed glass vials to minimize exposure to hazards. (GloQube-D only)
High voltage safety interlocks	Hardware safety interlocked and software for process control

Vacuum

Vacuum control	Integrated pirani gauge
Working vacuum range	0.1 to 1 mbar
Vacuum pump minimum requirements	6 m^3/hr, 3600 l/m, 0.03 mbar ultimate vacuum. Inlet flange: KF 16
Pumping time	Typical pump time to an operational vacuum of 0.27 mbar in 60 sec.
Vacuum isolation	Isolation valves to switch vacuum and prevent process chamber cross-contamination

User Interface

User interface	Full graphical interface with touch screen buttons and controls. In addition to displaying profiles, parameters, help screen and maintenance information are available
Profiles and profile logging	Capability to store 100 user profiles (name, date, time, vacuum, current and polarity)

Dimensions and Communications

Chamber size	100 mm W x 100 mm H x 127 mm D (3.94" x 3.94" x 5")
Instrument size	336 mm H x 364 mm D (13.2" x 14.3")
Instrument weight	19.5 kg (42.9 lbs) (GloQube-D)
Pump (optional)	391 mm W x 127 mm D x 177 mm H (15.4" x 5" x 7")
Pump weight	16 kg (35.3 lbs)
Footprint with optional pump	366 mm W x 600 mm D x 336 mm H (14.4" x 23.6" x 13.2")
Power requirements	120 V 60 Hz, 15 A or 230 V 50 Hz, 10 A
Instrument power rating	100-240 V AC 60/50 Hz 700 VA including pump, IEC inlet
Optional pump power rating	115/230 V 60/50 Hz 450 W
Communication port	Ethernet port for instrument software updates

ORDERING INFORMATION

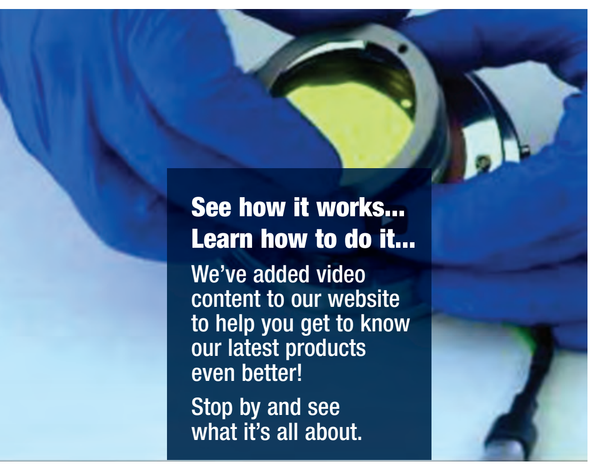
Cat No.	Description	Qty.
EMS-Glo-2	EMS GloQube, Dual chamber glow discharge system. Accessory kit, including: mains power lead, rotary pump power lead, oil mist filter and clamp, 750 mm long flexible stainless steel vacuum tube with clamps, fuses, glass vials, vial caps and sealing washers, needle (spare). Vacuum pump to be ordered separately.	each
Vacuum Pumping		
91003	5 m^3/hr Pfeiffer DUO 6 two-stage rotary vacuum pump with oil mist filter	each
96000	Oil mist filter (spare)	each
Options, accessories and spares		
EMS-Glo-11	Microscope Slide Tray	each
EMS-Glo-12	Glass Vial	10/pk
EMS-Glo-13	Glass Vial Caps	3/pk
EMS-Glo-14	Needle	each
EMS-Glo-15	Door Seal	each

What is...
Sputter Coating?

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

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

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



Techniques and Applications

Introduction

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

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

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

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

Gaseous Condition

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

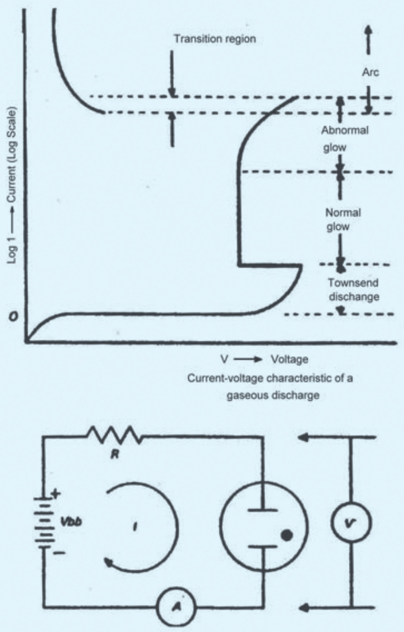
As voltage is increased some ionization is produced by collision of electrons with gas atoms, named the "Townsend" discharge. When the voltage across the tube exceeds the breakdown potential, a self sustaining glow discharge occurs - characterized by a luminous glow.

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

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

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

A = Ammeter V = Voltmeter



Glow Discharge

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

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

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

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

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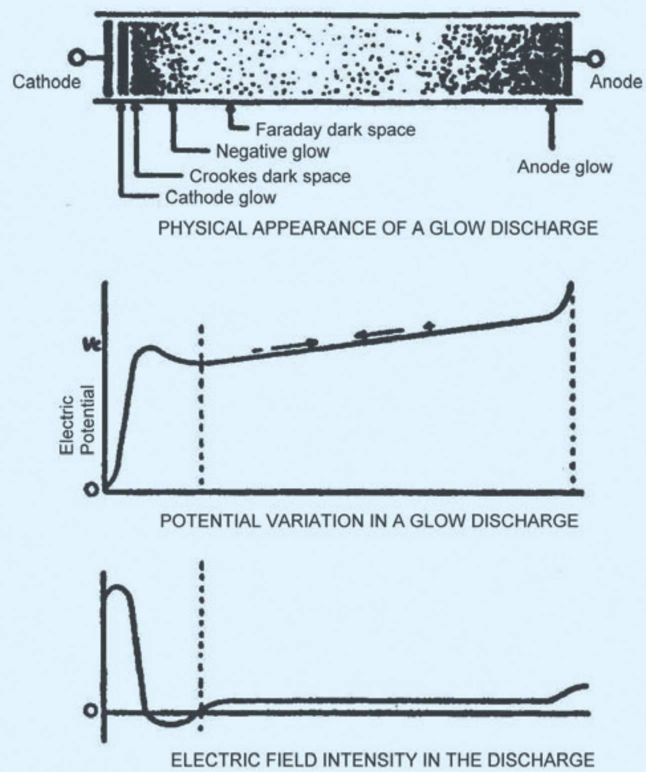
Techniques and Applications

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

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.

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

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

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

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

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

Techniques and Applications

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

It was indicated previously that while 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**.

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

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

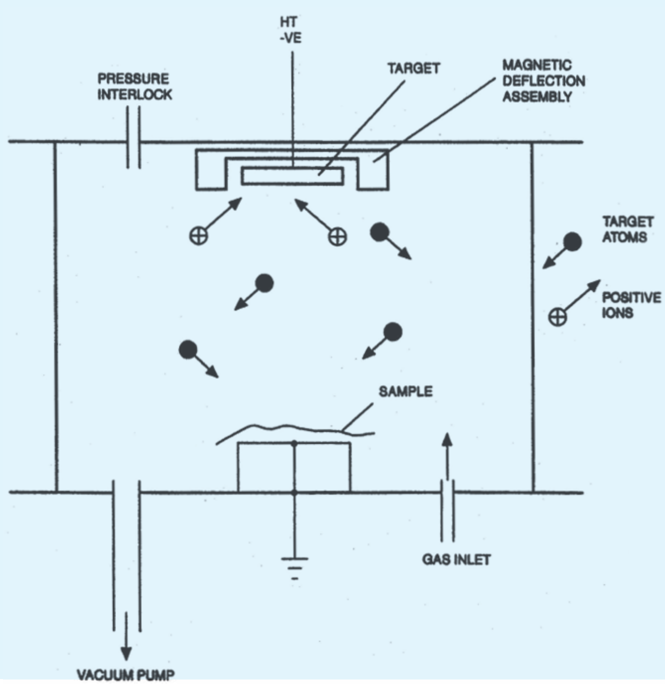
- ***Sputtered silver offers smaller grain size than evaporated silver.***
- ***Sputtered Gold and Silver have similar grain size but the silver has larger reticulation after storage.***
- ***Silver is the most conductive metal known.***
- ***Silver has a high secondary electron coefficient.***
- ***X-ray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

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

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

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

Figure 3 – Diagram of a "cool" sputtering head



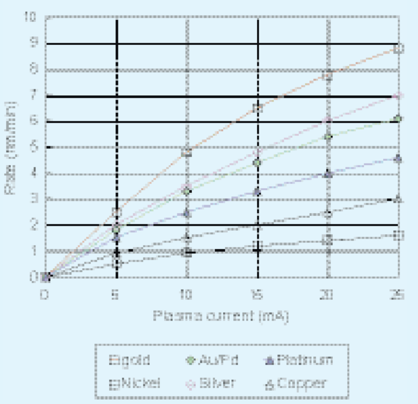
Techniques and Applications

Rates of Sputtering

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

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

Figure 4 - Sputtering Rates for the EMS 7620



Thickness of Coating

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

Th = 7.5 I t (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

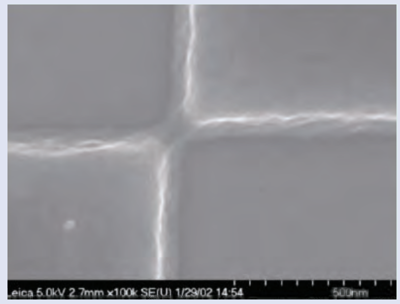
- Clean the glass chamber with hot soapy water and dry thoroughly, solvents can be used but we have found this unnecessary and having greater danger to health and safety. If the deposit is stubborn, use a kitchen scouring pad such as the green Scotch Bright variety.
- Use Isopropyl alcohol on metal surfaces,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

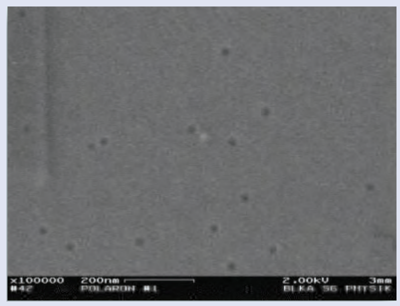
3. Sputter gas, Always use high purity argon gas of the grade known as “White spot” this will ensure fast sputter rate and good pump down time.

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



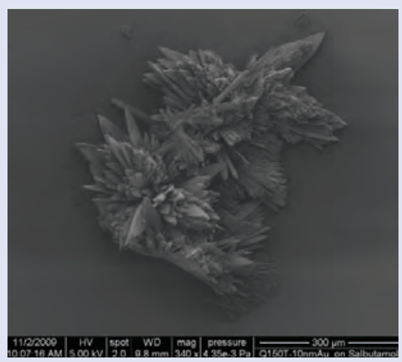
Gold/palladium coating of 6" wafer

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

Techniques and Applications

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 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.

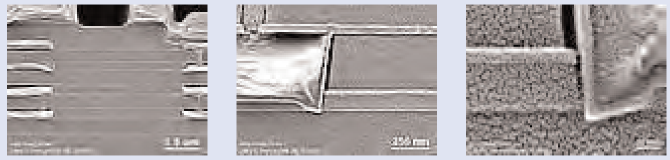
The coated glass slide was immersed in freshly-prepared Farmer's Reducer. The silver was gently dissolved in a controlled manner, as shown by the gradual and uniform loss of color in transmitted light, until none remained after three minutes. No gas was evolved. It was decided that a 10 minute immersion 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/Glow Discharge

QUICK OVERVIEW

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

FEATURES

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

Easy operation

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

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

Fast cycle times

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

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

Glow discharge (hydrophilization)

A three-way switch on the front panel allows the EMS 7620 to be switched to glow discharge mode.

Freshly-made transmission electron microscopy (TEM) carbon support films tend to have a hydrophobic surface that hinders the collection of TEM sections from the surface of water baths and prevents the spreading of suspensions of particles in negative staining solutions. However, after glow discharge treatment with air, carbon film can be made hydrophilic and negatively-charged, thus allowing collection of TEM sections and easy spreading of aqueous suspensions.

Other possible treatments include magnesium acetate treatment to create hydrophobic and positively-charged surfaces. If alkylamine is used as a process gas, the carbon film surface will become hydrophobic and positively-charged, while using methanol as a process gas results in the surface becoming hydrophobic and negatively-charged. Such treatments can allow the optional absorption of selected biomolecules.

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.

SPECIFICATIONS

Dimensions:........................340mm W x 130mm D x 250mm H (unpacked) excluding chamber

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.

Options and Accessories

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

Electron Microscopy Sciences In PA: (215) 412-8400 • Toll-Free (800) 523-5874
Fax (215) 412-8450 or 8452 • email: info@emsdiasum.com or stacie@ems-secure.com • www.emsdiasum.com

EMS 7620 (*continued*)

Choosing a target

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

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

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

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

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

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

ORDERING INFORMATION

Cat No.	Description	
EMS 7620	'Mini' Sputter Coater	each
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		
91017-Au	Gold (Au) target	each
91017-AP	Gold/palladium (Au/Pd) target	each
91017-Pt	Platinum (Pt) target	each
91017-Ag	Silver (Ag) target	each
91017-Pd	Palladium (Pd) target	eacht
91017-.2-Au	Gold target (Au) 0.2mm thick	each
91017-.2-AP	Gold/palladium (Au/Pd) target 0.2mm thick	each

Cat No.	Description	
91017-.2-Pt	Platinum (Pt) target 0.2mm thick	each
91003	RV-3 two-stage rotary pump with oil mist filter (115/230V 50/60Hz)	each
96000	Replacement compact oil mist filter	each
96001	Replacement compact oil mist filter cartridge	each
91040	Carbon evaporation attachment — EMS 7620	each

Optional carbon fiber evaporation attachment and controller

Specimen stage and sputtering head

EMS 7620-CF Carbon Accessory Power Supplies

The 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 costeffective 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 150R1 and EMS 150T.

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.

ORDERING INFORMATION

Sputter Coater	Attachment	Attachment
EMS 7620 Sputter Coater	Not Available	EMS 7620-CF
EMS 500X, EMS 550X, EMS 575X, EMS 675X	EMS 7640-CR	EMS 7640-CF

Carbon fiber cord

91046-1	Carbon fiber cord - standard grade - 1m	
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	
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

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.

FEATURES

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

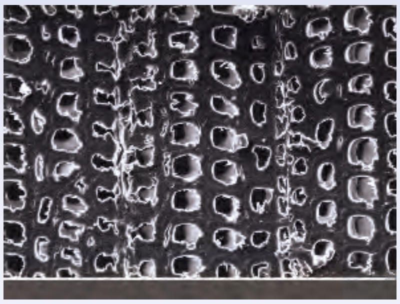
SPECIFICATIONS

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)

What is...
Carbon Coating?

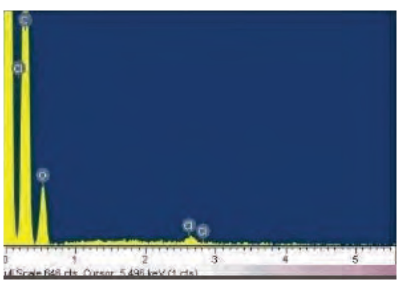
The use of carbon films in Electron Microscopy with their low background signal and relatively good electrical conductivity is well known. Thin films, nominally 5nm or 50 Angstroms, are used in TEM, while a range of somewhat thicker films, ranging from 50nm or 500 Angstroms, may be used in SEM for such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



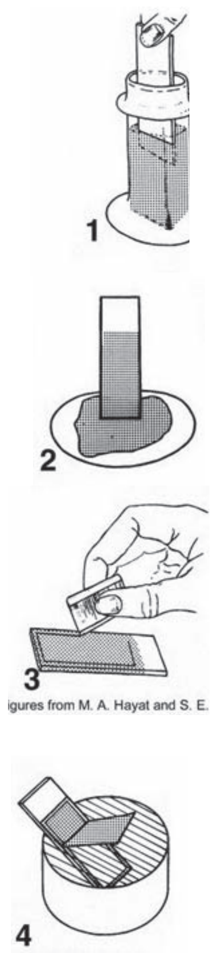
Pinus sylvestris (Scots pine)

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.

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Techniques and Applications

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

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

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

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

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

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

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

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

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

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

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

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

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 150R Plus Rotary Pumped Coater

QUICK OVERVIEW

The EMS 150R Plus is suitable for use with Tungsten/LaB_6 SEM and Benchtop SEM.

Typical uses

Sputter coating of noble metals using the EMS 150R S Plus & EMS 150R ES Plus

Recommended for magnifications:

- Up to x 50k using Au, Au/Pd
- Up to x 100k using Pt (optional)

Carbon cord coating for elemental analysis using the EMS 150R S Plus & EMS 150R ES Plus.

KEY FEATURES

- **Capable of achieving vacuum of 2 x 10^{-3} mbar**
- **New touch and swipe capacitive screen**
- **USB port for upgrades and download of log files**
- **Multiple-user profiles can be set up on one machine**
- **New software sorts recipes per user, according to recent use**
- **16GB of memory can store more than 1000 recipes**
- **New multi-color LED visual status indicator**
- **Interchangeable stage options and plug-in heads**

PRODUCT DESCRIPTION

The EMS 150R Plus is available in three configurations:

- EMS 150R S Plus – An automatic sputter coater for non-oxidizing metals. Available sputtering targets including gold, gold/palladium and platinum.
- EMS 150R E Plus – An automatic carbon cord coater for SEM applications such as EDS and WDS.
- EMS 150R ES Plus – A combined system system capable of both sputtering and carbon coating. The deposition heads can be swapped in seconds.

Improved Interface

- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help screen
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Dual-core ARM processor for a fast, responsive display

Allows multiple users to input and store coating recipes, with a new feature to sort recipes per user according to recent use.

Intelligent system logic automatically detects which insert is in place and displays the appropriate operating settings and controls for that process.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material.

Recommended applications for EMS 150 R Plus:

- **Low and medium magnifications**
- **SE signal boost (1nm or less)**
- **Table-top SEM coating**
- **Elemental analysis**
- **Copper metallization layers**

These products are for Research Use Only.

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

Automatic, controlled pulsed carbon cord evaporation

The carbon evaporation process can be terminated using the optional film thickness monitor, which incorporates a quartz crystal monitor, fitted as standard on E and ES models. This recipe ensures that carbon is evaporated in short controlled pulses, which has two effects: protecting the sample from heating and ensuring the accuracy of the film thickness monitor. Pulsing also significantly reduces the amount of debris (including large carbon fragments) associated with traditional carbon "flash" evaporation. Pulsed and ramped carbon rod recipes are supplied as standard.

Cool Magnetron Sputtering

Sputter coating is a technique widely used in various applications; it is possible to create a plasma and sputter metals with high voltage, poor vacuum and no automation. However, this is not suitable for electron

EMS 150R Plus *(continued)*

microscopy applications because it will heat the sample and can result in damage when the plasma interacts with the sample. The EMS 150R Plus series uses low temperature enhanced-plasma magnetrons optimized for the rotary pump pressures, combined with low current and deposition control, which ensures your sample is protected and uniformly coated.

The EMS 150R S Plus and EMS 150R ES Plus use easy-change, 57 mm diameter, disc-style targets which are designed to sputter non-oxidizing (noble) metals – ideal for W-SEM applications. The EMS 150R S Plus and EMS 150R ES Plus are fitted as standard with a gold (Au) sputter target.

Other targets options include Au/Pd, Pt/Pd, Pd, and Cu. Platinum (Pt) can also be sputtered with the optional Pt coating vacuum hose assembly.

Interchangeable Plug-in Heads

This allows the user to configure the system as a sputter coater, evaporator or glow discharge system – all in one space saving format. A carbon cord evaporation insert is available as an option. Automatic detection of the head type when changed.

Detachable chamber with built-in implosion guard

Removable glass chamber and easily accessible base and top plate allows for an easy cleaning process.

Users can rapidly change the chamber, if necessary, to avoid cross contamination of sensitive samples.

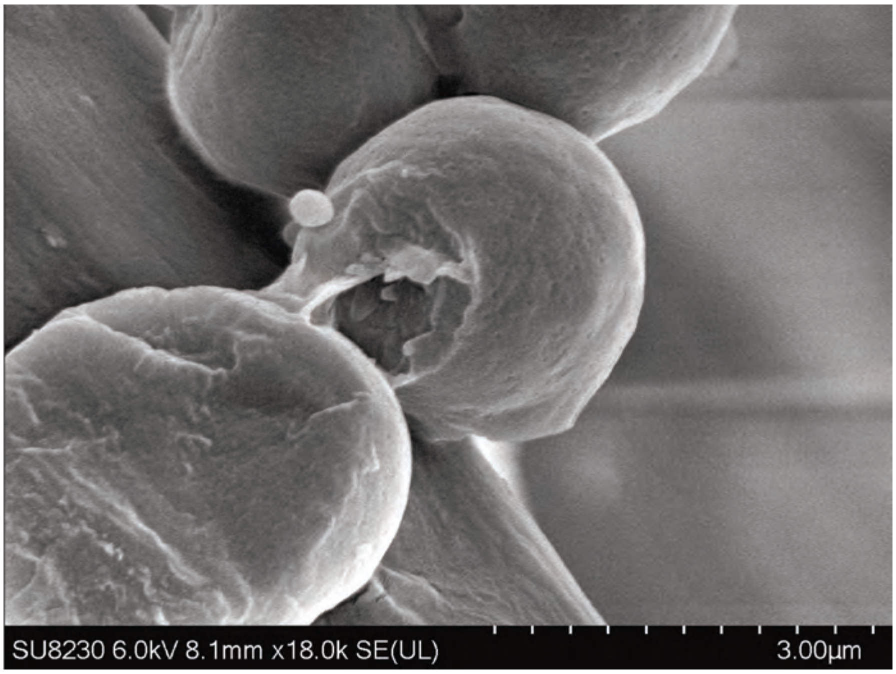
Tall chamber option is available for carbon evaporation to avoid sample heating, to improve uniformity for sputtering and to hold taller samples.

Multiple stage options

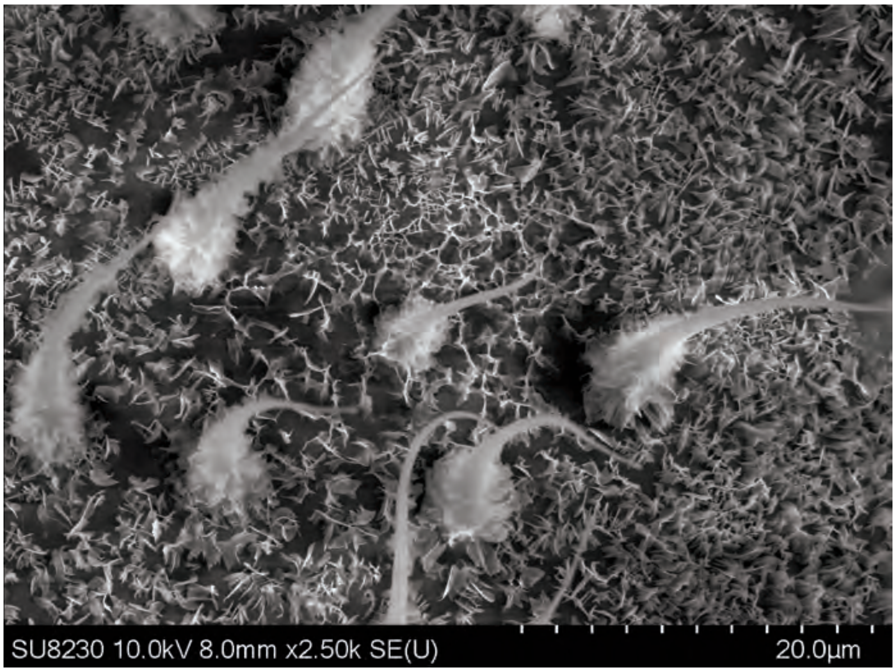
The EMS 150R Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except for the rotary planetary stage). Some examples:

- Rotation stage (supplied as standard): 50 mm Ø can accommodate six standard stubs. Height can be pre-set.
- Rotate-tilt stage for improved uniform coating: 50 mm Ø. Tilt and height can be pre-set.
- Variable angle, rotary planetary stage for heavily contoured samples
- Large flat rotation stage with offset gear box for 4"/100 mm wafers
- Rotation stage for glass microscope slides

Other options are available on request.



Pure silk coated 10nm Au x 18k magnification



Ladybird larvae hair coated 10nm Au x 2.5k magnification

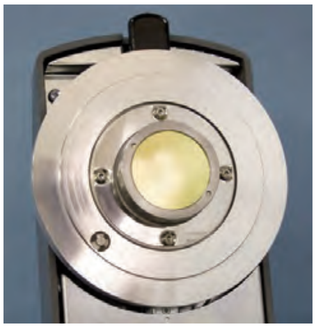
continued »»

EMS 150R Plus *(continued)*

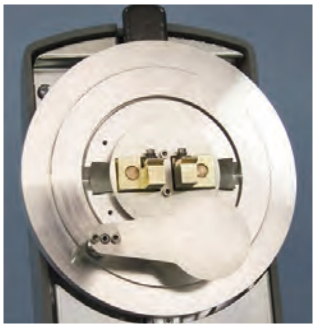
Safety

The EMS 150R Plus meets key industry CE standards:

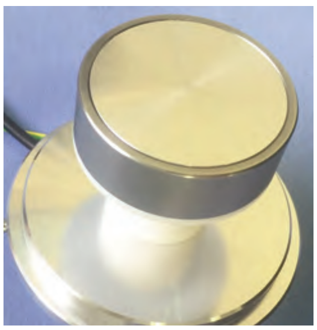
- All electronic components are protected by covers
- Implosion guard prevents user injury in event of chamber failure
- Vacuum interlocks remove power from deposition sources to prevent user exposure to high voltage in event of chamber being opened
- Electrical interlocks remove power when source head cover opened
- Overheating protection shuts down power supply



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

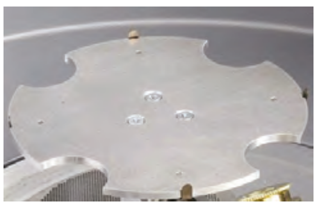


Carbon fiber evaporation insert and automatic source shutter

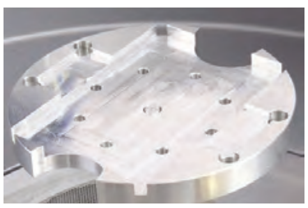


10262 optional glow discharge attachment (for R S and R ES versions)

Base plate. Standard specimen stage and optional film thickness monitor (FTM)



Optional flat rotation stage for 100 mm/4 wafers



Optional rotation stage for glass microscope slides

SPECIFICATIONS

Instrument Case	585mm W x 470mm D x 410mm H Total height with coating head open is 650 mm
Weight	28.4 kg (Packed 42 kg)
Packed Dimensions	725 mm W x 660 mm D x 680 mm H
Work Chamber	Borosilicate glass 150 mm ID x 127 mm H
Display	115.5 mm W x 86.4 mm H (active area), 640 RGB x 480 (display format), capacitive touch color display
User Interface	Full graphical interface with touch screen buttons, includes features such as a log of the last 1000 coatings and reminders for when maintenance is due
Sputter Target	Disc-style 57 mm Ø 0.1 mm thick gold (Au) target is fitted as standard. R S and R ES versions only

Specimen Stage

50 mm Ø rotation stage with rotation speed of 8-20 rpm.

Other stages available on request.

Vacuum

Rotary Pump	Optional 5 m^3/hr two-stage rotary pump with oil mist filter (order separately)
Vacuum Measurement	Pirani gauge
Ultimate Vacuum	2×10^{-2} mbar

Typical ultimate vacuum of the pumping system in a clean instrument after pre-pumping with dry nitrogen gas

Sputter Vacuum Range	Between 7×10^{-3} and 1×10^{-1} mbar for gold

Processes

Sputter Deposition Current	Single target: 1 - 140mA All targets: 60 - 420mA
Visual Status Indicator	A large status multi-color indicator light provides a visual indication of the state of the equipment, allowing users to easily identify the status of a process at distance.

The indicator LED shows the following states:

- Initialization
- Process running
- Idle
- Coating in progress
- Process completed
- Process ended in fault condition

Audio indication also sounds on completion of the process.

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EMS 150R Plus *(continued)*

ORDERING INFORMATION

EMS 150R S Plus

4500	EMS 150R S Plus Fully automatic, capacitive touch-screen controlled rotary-pumped sputter coater with status LED indicator. **Including:** quick-release sputter insert, one 57 mm diameter x 0.1 mm gold target, and 50 mm diameter rotating specimen stage.	

EMS 150R E Plus

4503	EMS 150R E Plus Fully automatic, capacitive touch-screen controlled rotary pumped carbon fiber evaporation coater with status LED indicator. **Including:** quick-release carbon fiber insert for evaporation of carbon cord, carbon fiber cord, and 50 mm diameter rotating specimen stage.	

EMS 150R ES Plus

4507	EMS 150R ES Plus Fully automatic, capacitive touch-screen controlled rotary pumped sputter coater and carbon evaporator with status LED indicator. **Including:** quick release sputter insert, one 57 mm diameter x 0.1 mm gold target, and carbon fiber evaporation coater, including quick-release carbon fiber insert for evaporation of carbon cord*, and 50 mm diameter rotating specimen stage. *Coating inserts are interchangeable and can be swapped in seconds. The intelligent system logic automatically recognises which insert is in place and displays the appropriate operating settings.	

Options and Accessories

Rotary pump requirements – order separately

91003	5m³ hr two-stage Pfeiffer Duo 6 rotary pump with oil mist filter	each

Head inserts and glow discharge

4511	Additional sputter insert for quick metal change (R E and R ES only). Note: this is an entire sputtering assembly – individual noble metal targets can also be purchased	each
4512	Carbon rod evaporation insert for 3.05 mm Ø rods (R E and R ES only). Includes manual rod shaper and 3.05 mm Ø carbon rod	each
4513	Glow discharge insert to modify surface properties (e.g. hydrophobic to hydrophilic conversion) or to clean surface residues (R S and R ES only). Can be retrofitted	each
4514	Additional standard glass chamber assembly	
4515	Extended height vacuum chamber (214 mm high – the standard chamber is 127 mm high). For increased source to sample distance and for coating large specimens	each
4516	Rotating vacuum spigot allows more convenient connectionof the vacuum hose to the rear of the EMS150R when bench depth is limited	each
4517	Film thickness monitor (FTM) attachment. Consists of a built in chamber mounted quartz crystal oscillator (includes crystal). As sputtered or evaporated material is deposited onto the crystal, so its frequency of oscillation is modified. This "modification" is used to measure and control the thickness of material deposited	each
4518	Spare quartz crystal	each
13530	Standard coating shield assembly	each
11880	Extended height coating shield assembly	each
1371	Platinum coating vacuum hose assembly	each

Specimen stages

All rotating stages have variable rotation of between 8 and 20 rpm.

4519	50 mm Ø rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37 mm-60 mm). Has six stub positions for 15 mm or 6.5 mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
4520	90 mm Ø rotating specimen stage for glass microscope slides (up to two x 75 mm x 25 mm slides). Stage rotation speed variable between 8 and 20rpm	each
4521	Variable angle "Rotacota" rotary planetary stage with 50 mm Ø specimen platform. Has six stub positions for 15 mm or 6.5 mm or 1/8" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
4522	Flat rotation specimen stage for 100 mm/4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm	each
23850	Tilt stage sub-assembly 4"	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

Carbon Supplies

4538	Carbon fiber cord – high purity – 1m	each
4539	Carbon fiber cord – high purity – 5m	each
4540	Carbon fiber cord – standard grade – 1m	each
4541	Carbon fiber cord – standard grade – 10m	each
4542	Carbon fiber cord – standard grade – 100m	each
4543	Carbon rods – 3.05 mm Ø x 50 mm length (shaped) pack of 10	10/pk
4544	Carbon rods 3.05 mm Ø x 300 mm length (unshaped) pack of 10)	10/pk
4546	Manual rod shaper for 3.05 mm Ø carbon rods	each
12097	Wedge Tool	each

Sputtering Targets

4523	57 mm Ø x 0.1 mm Gold	each
4524	57 mm Ø x 0.2 mm Gold	each
4525	57 mm Ø x 0.3 mm Gold	each
4526	57 mm Ø x 0.1 mm Gold/Palladium (80/20)	each
4527	57 mm Ø x 0.2 mm Gold/Palladium (80/20)	each
4528	57 mm Ø x 0.3 mm Gold/Palladium (80/20)	each
4529	57 mm Ø x 0.1 mm Platinum	each
4530	57 mm Ø x 0.2 mm Platinum	each
4531	57 mm Ø x 0.3 mm Platinum	each
4532	57 mm Ø x 0.1 mm Nickel	each
4533	57 mm Ø x 0.1 mm Silver	each
4534	57 mm Ø x 0.1 mm Palladium	each
4535	57 mm Ø x 0.1 mm Copper	each
4536	57 mm Ø x 0.1 mm Platinum/Palladium (80/20)	each
4537	57 mm Ø x 0.3 mm Platinum/Palladium (80/20)	each

Spare Kits

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

EMS 150T Plus Turbomolecular Pumped Coater

QUICK OVERVIEW

The EMS 150T Plus is optimized for use with a turbomolecular pump, which gives a lower vacuum down to 5 x 10-5 mbar. This enables the sputtering of oxidizing metals, which have a lower grain size suitable for high-resolution imaging. Similarly, lower scattering allows for high purity, amorphous carbon films of high density.

Typical uses

Sputter coating of noble and oxidizing metals using the EMS 150T S Plus & EMS 150T ES Plus:

Recommended for magnifications::

- Up to x 50k using Au, Au/Pd
- Up to x 100k using Pt
- above x 100k using Cr, Ir(optional)

Ideal for thin film applications such as coating with ITO, W, Al, Zn.

KEY FEATURES

- **New touch and swipe capacitive screen**
- **USB port for upgrades and download of process log files**
- **Multiple-user profiles can be set up on one machine**
- **New software sorts recipes per user according to recent use**
- **16GB of memory can store more than 1000 recipes**
- **New multi-color LED visual status indicator**
- **Capable of achieving vacuum of 5 x 10-5 mbar**

PRODUCT DESCRIPTION

The EMS 150T Plus is available in three configurations:

- EMS 150T S Plus – An automatic sputter coater for non-oxidizing metals. Sputtering targets include; Cr, Ir, W, ITO, Al. Other targets available.
- EMS 150T E Plus – An automatic carbon cord coater for SEM applications such as EDS and WDS. Metal evaporation/aperture cleaning option available.
- EMS 150T ES Plus – A combined system system capable of both sputtering and carbon coating. The deposition heads can be swapped in seconds. Metal evaporation/aperture cleaning option available.

Improved Interface

- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help screen
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Dual-core ARM processor for a fast, responsive display

Allows multiple users to input and store coating recipes, with a new feature to sort recipes per user according to recent use.

Intelligent system logic automatically detects which insert is in place and displays the appropriate operating settings and controls for that process.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material.

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

Recommended applications for EMS 150 T Plus:

- **High-resolution magnification SEM**
- **Protective platinum layers for FIB**
- **R&D of corrosion-, friction-, and wear protective layers**
- **Protective layers on medical devices**
- **BSE imaging**
- **EDX, WDS, EBSD analysis**
- **Carbon coating of replicas**

These products are for Research Use Only.

Controlled ramped carbon rod evaporation

Careful evaporation allows precise control of carbon thickness (with or without the optional film thickness monitor). The quality of the resulting carbon films is also enhanced by the eradication of "sparking" that is a common feature of less advanced coaters.

For reproducible high-quality carbon films, we would recommend the use of shaped carbon rods. Rods are higher purity, less susceptible to debris and easier to control. Pulsed and ramped carbon rod recipes are supplied as standard.

EMS 150T Plus *(continued)*

Cool Magnetron Sputtering

Sputter coating is a technique widely used in various applications; it is possible to create a plasma and sputter metals with high voltage, poor vacuum and no automation. However, this is not suitable for electron microscopy applications because it will heat the sample and can result in damage when the plasma interacts with the sample. The EMS 150T Plus series uses low temperature enhanced-plasma magnetrons optimized for the rotary pump pressures, combined with low current and deposition control, which ensures your sample is protected and uniformly coated.

The EMS 150T S Plus and EMS 150T ES Plus use easy-change, 57 mm diameter, disc-style targets which are designed to sputter oxidizing and noble metals. The EMS 150T S Plus and EMS 150T ES Plus are fitted as standard with a chromium (Cr) sputter target. Other targets options include Au, Au/Pd, Pt/Pd, Pd, Pt, Cu, Ir, W, ITO and Al. Others are available on request.

Pulsed cleaning for Aluminum sputtering

Aluminum (Al) rapidly forms an oxide layer which can be difficult to remove, but the EMS 150T ES Plus & EMS 150T S Plus have special recipes for Al that reduce the oxide removal time and prevent excessive pre-sputtering of the target.

Interchangeable plug-in heads

This allows the user to configure the system as a sputter coater, evaporator or glow discharge system – all in one space saving format. A carbon cord evaporation insert is available as an option. Automatic detection of the head type when changed.

Detachable chamber with built-in implosion guard

Removable glass chamber and easily accessible base and top plate allows for an easy cleaning process. Users can rapidly change the chamber, if necessary, to avoid cross contamination of sensitive samples. Tall chamber option is available for carbon evaporation to avoid sample heating, improved uniformity for sputtering and to hold taller samples.

Multiple stage options

The EMS 150T Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except for the rotary planetary stage).

Some examples:

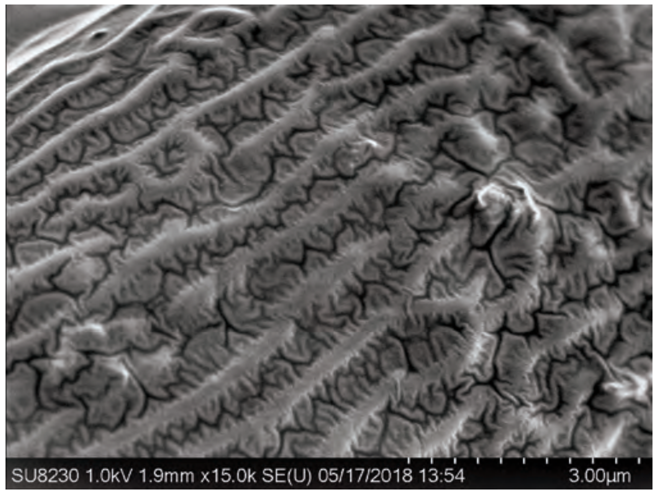
- Rotation stage (supplied as standard): 50 mm Ø can accommodate six standard stubs. Height can be pre-set.
- Rotate-tilt stage for improved uniform coating: 50 mm Ø. Tilt and height can be pre-set.
- Variable angle, rotary planetary stage for heavily contoured samples.
- Large flat rotation stage with offset gear box for 4"/100 mm wafers.
- Rotation stage for glass microscope slides.

Other options are available on request.

Safety

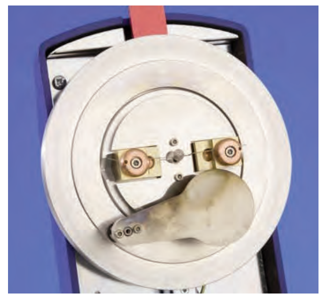
The EMS 150T Plus meets key industry CE standards

- All electronic components are protected by covers
- Implosion guard prevents user injury in event of chamber failure
- Vacuum interlocks remove power from deposition sources to prevent user exposure to high voltage in event of chamber being opened
- Electrical interlocks remove power when source head cover opened
- Overheating protection shuts down power supply

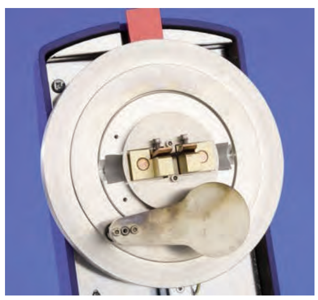


Pollen coated in 3nm Au x15k magnification on EMS 150T Plus

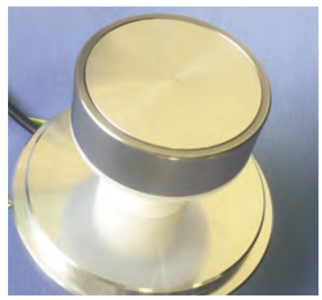
Sputter coater insert (standard with T S and T ES)



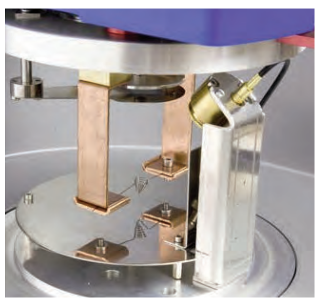
Metal evaporation insert (set up for downwards evaporation)



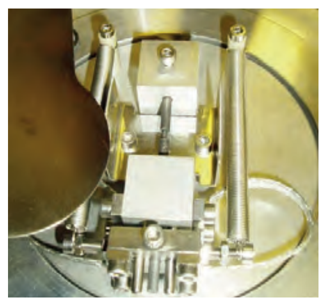
Carbon cord evaporation insert option



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



Metal evaporation insert (set up for upwards evaporation)



Carbon rod evaporation insert

EMS 150T Plus *(continued)*

SPECIFICATIONS

Instrument Case	585mm W x 470mm D x 410mm H Total height with coating head open is 650 mm
Weight	28.4 kg (Packed 42 kg)
Packed Dimensions	725 mm W x 660 mm D x 680 mm H
Work Chamber	Borosilicate glass 150 mm ID x 127 mm H
Display	115.5 mm W x 86.4 mm H (active area), 640 RGB x 480 (display format), capacitive touch color display
User Interface	Full graphical interface with touch screen buttons, includes features such as a log of the last 1000 coatings and reminders for when maintenance is due
Sputter Target	Disc-style 57 mm Ø 0.1 mm thick gold (Au) target is fitted as standard. R S and R ES versions only

Specimen Stage

50 mm Ø rotation stage with rotation speed of 8-20 rpm.

Other stages available on request.

Vacuum

Rotary Pump	Optional 5 m^3/hr two-stage rotary pump with oil mist filter (order separately)
Vacuum Measurement	Pirani gauge
Ultimate Vacuum	2 x 10^{-2} mbar

Typical ultimate vacuum of the pumping system in a clean instrument after pre-pumping with dry nitrogen gas

Sputter Vacuum Range	Between 7 x 10^{-3} and 1 x 10^{-1} mbar for gold

Processes

Sputter Deposition Current	Single target: 1 - 140mA All targets: 60 - 420mA
Visual Status Indicator	A large status multi-color indicator light provides a visual indication of the state of the equipment, allowing users to easily identify the status of a process at distance.

The indicator LED shows the following states:

- Initialization
- Process running
- Idle
- Coating in progress
- Process completed
- Process ended in fault condition

Audio indication also sounds on completion of the process.

ORDERING INFORMATION

EMS 150T S Plus

3380	EMS 150T S Plus Sputter Coater. High resolution turbomolecular pumped capacitive touch-screen controlled sputter coater with status LED indicator. Includes quick release sputter insert and one 57 mm diameter x 0.3 mm chromium target. Rotating specimen stage, 50 mm diameter, supplied as standard.	

EMS 150T E Plus

3390	EMS 150T E Plus Carbon Evaporator. Turbomolecular pumped capacitive touch-screen controlled carbon evaporator with status LED indicator suitable for TEM and SEM applications. Fitted with a carbon rod evaporation head for 3.05 mm diameter carbon rods. Supplied with carbon rods (3.05 mm x 300 mm) and a carbon rod shaper (manual operation). Rotating specimen stage, 50 mm diameter, supplied as standard.	

EMS 150T ES Plus

3400	EMS 150T ES Plus Combined Sputtering and Carbon Coating System. The deposition heads can be swapped in seconds and the intelligent system logic automatically recognizes which head is in place and displays the appropriate operating settings. Consists of high resolution turbomolecular pumped capacitive touch-screen controlled sputter coater and carbon evaporator with status LED indicator, including quick release sputter insert and one 57 mm diameter x 0.3 mm chromium target, and high vacuum carbon rod evaporation coater, including quick-release carbon rod insert for 3.05 mm diameter carbon rods. Coating inserts are interchangeable. Rotating specimen stage, 50 mm diameter, supplied as standard.	

Options and Accessories

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

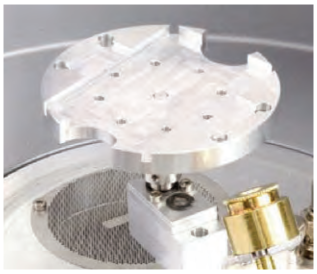
Coating head options

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

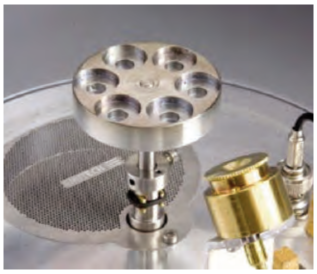
3200	Sputtering head insert suitable for oxidizing and non-oxidizing metals. Supplied with a 54 mm x 0.3 mm thick chromium (Cr) target as standard. For additional targets see Sputtering Targets section	each
3210	Additional sputter insert for quick metal change. Note: this is an entire sputtering assembly.	each
3230	Carbon rod evaporation head insert (for 3.05 mm Ø rods)	each
3240	Carbon rod evaporation head insert (for 6.15 mm Ø rods). Note that EMS recommends 3.05 mm Ø rods as they offer greater process control and are more economical (less wastage).	each
3250	Carbon fiber evaporation head insert	each
3260	Metal evaporation and aperture cleaning head insert, including the ability to evaporate upwards or downwards (T E and T ES versions only). Supplied with a pack of ten tungsten filaments and a molybdenum boat.	each
3270	Extended height vacuum chamber (214 mm high – the standard chamber is 127 mm high). For increased source to sample distance and for coating large specimens	each
3280	Rotating vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS 150T Plus when bench depth is limited	each
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	each
3300	Spare quartz crystal for FTM.	each
13530	Standard coating shield assembly	each
27691	Extended glass cylinder assembly, 220 mm H	each
3320	Full range vacuum gauge for low and high vacuum measurement (a low vacuum Pirani gauge is fitted as standard)	each

EMS 150T Plus *(continued)*

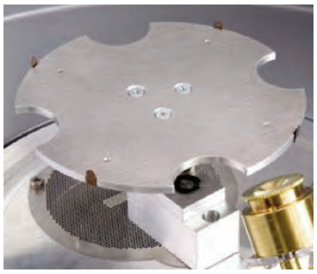
ORDERING INFORMATION (continued)



Microscope slide stage



Rotation stage



Wafer stage

Rotacota planetary stage

4513	Glow discharge insert to modify surface properties (e.g. hydrophobic to hydrophilic conversion) or to clean surface residues (TS and T ES only). Can be retrofitted	each
Specimen stages		
The EMS 150T Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage):		
3330	Rotation stage, 50 mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment	each
3340	Rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37 mm-60 mm). Tilt angle can be preset. 50 mm Ø specimen platform with six stub positions for 15 mm or 6.5 mm or ⅛" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
3350	Variable angle "Rotacota" rotary planetary stage with 50 mm Ø specimen platform. Has six stub positions for 15 mm or 6.5 mm or ⅛" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
3360	Flat rotation specimen stage for 100 mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm	each
3370	Rotating specimen stage for glass microscope slides (up to two x 75 mm x 25 mm slides). Stage rotation speed variable between 8 and 20rpm. Includes gear box to allow optional FTM to be used	each
23850	Tilt stage sub-assembly 4"	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

Evaporation Supplies		
73830-SP	Tungsten wire baskets – pack of 10	10/pk
73810-SP	Molybdenum boats – pack of 10	10/pk
Carbon supplies		
3500	Carbon rods – 6.15 mm Ø x 100 mm length (unshaped) pack of 10	10/pk
3510	Carbon rods – 6.15 mm Ø x 50 mm length (shaped) pack of 10	10/pk
3520	Carbon rods – 3.05 mm Ø x 50 mm length (shaped) pack of 10	10/pk
3525	Carbon rods – 3.05 mm Ø x 100 mm length (unshaped) pack of 10	10/pk
3530	Carbon rods 3.05 mm Ø x 300 mm length (unshaped) pack of 10	10/pk
3540	Carbon fiber cord – high purity – 1m	each
3550	Carbon fiber cord – high purity – 5m	each
3560	Carbon fiber cord – standard grade – 1m	each
3570	Carbon fiber cord – standard grade – 10m	each
3580	Carbon fiber cord – standard grade – 100m	each
3590	Manual rod shaper for 6.15 mm Ø carbon rods	each
3595	Manual rod shaper for 3.05 mm Ø carbon rods	each
12097	Wedge Tool	each
Sputtering Targets		
3410	57 mm Ø x 0.1 mm Gold	each
3410-2	57 mm Ø x 0.2 mm Gold	each
3411	57 mm Ø x 0.1 mm Gold/Palladium (80/20)	each
3411-2	57 mm Ø x 0.2 mm Gold/Palladium (80/20)	each
3412	57 mm Ø x 0.1 mm Platinum	each
3413	57 mm Ø x 0.1 mm Nickel	each
3414	57 mm Ø x 0.1 mm Silver	each
3415	57 mm Ø x 0.1 mm Palladium	each
3416	57 mm Ø x 0.1 mm Copper	each
3417	57 mm Ø x 0.3 mm Chromium	each
3418	57 mm Ø x 0.5 mm Tungsten	each
3419	57 mm Ø x 1.5 mm Chromium	each
3420	57 mm Ø x 0.2 mm Tungsten	each
3421	54 mm Ø x 1.5 mm Carbon	each
3422	57 mm Ø x 0.1 mm Aluminum	each
3423	57 mm Ø x 0.1 mm Platinum/Palladium (80/20)	each
3424	57 mm Ø x 1.5 mm Titanium	each
3425	57 mm Ø x 0.3 mm Platinum/Palladium (80/20)	each
3426	57 mm Ø x 0.3 mm Gold	each
3427	57 mm Ø x 0.3 mm Gold/Palladium (80/20)	each
3428	57 mm Ø x 0.3 mm Platinum	each
3429	57 mm Ø x 0.5 mm Titanium	each
3430	57 mm Ø x 0.1 mm Iron	each
3431	57 mm Ø x 0.3 mm Iridium	each
3432	57 mm Ø x 0.1 mm Cobalt	each
3433	57 mm Ø x 0.1 mm Tin	each
3434	57 mm Ø x 0.1 mm Molybdenum	each
3435	57 mm Ø x 0.3 mm Magnesium	each
3436	57 mm Ø x 0.1 mm Tantalum	each
3437	57 mm Ø x 3 mm Indium Tin Oxide (90/10)	each
Other consumables and spare kits		
3600	Metal evaporation basket – pack of 10 (for use with metal evaporation head)	10/pk
07803	Basic Oil Mist Filter (spare)	each
13233	Rotary Pump Oil (spare), 1L	each
13235	Rotary Pump Oil (spare), 5L	each
27607	Non-seal L Gasket	each
G6260	Glass cylinder 6"	each
10068	AdditionalÂ glass cylinder assembly	each
10429	ExtendedÂ glass cylinder assembly	each
3610	Two-year spares kit for EMS 150V S Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, quartz crystals, O-rings	kit
3620	Two-year spares kit for EMS 150V E Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, carbon fiber – fine, carbon rods 3.05 mm, quartz crystals, O-rings	kit
3630	Two-year spares kit for EMS 150V ES Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, carbon fiber – fine, carbon rods 3.05 mm, quartz crystals, O-rings	kit

EMS 150V Plus Automatic Coater

For ultra-fine coatings

QUICK OVERVIEW

The EMS 150V Plus is optimized for high-vacuum applications, with an ultimate vacuum of 1 x 10-6 mbar. Together with the use of a wide-range Penning/Pirani gauge, this enables the sputtering of oxidizing metals with ultra-fine grain sizes, which are suitable for high resolution imaging. The lower background pressure removes oxygen nitrogen and water vapour from the chamber, avoiding chemical reactions during the sputter process, which could otherwise lead to impurities or defects in the coatings. Similarly, lower scattering allows for high purity, amorphous carbon films of high density.

The EMS 150V Plus offers all the benefits of the EMS 150T Plus, but with a finer grain size and thinner coating, for ultra-high-resolution applications (above x 200,000 magnification).

KEY FEATURES

- **Ultimate vacuum of 1x10^{-6}mbar**
- **New multi-color LED visual status indicator**
- **16Gb of flash memory can store more than 1000 recipes**
- **New software sorts recipes per user according to recent use**
- **Multiple-user profiles can be set up on one machine**
- **New touch and swipe capacitive screen**

PRODUCT DESCRIPTION

The EMS 150V Plus is available in three configurations:

EMS 150V S Plus — an automatic sputter coater for oxidizing metals with ultra-fine grain size. Available sputtering targets include chromium, iridium and all noble metals

EMS 150V E Plus — an automatic carbon coater (rod/cord) for TEM applications. For carbon coating TEM grids.

EMS 150V ES Plus — a combined system capable of both sputtering and carbon coating. The deposition head inserts can be swapped in seconds. Metal evaporation/aperture cleaning option available.

New user interface has been thoroughly updated:

- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help screen
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Dual-core ARM processor for a fast, responsive display

Allows multiple users to input and store coating recipes, with a new feature to sort recipes per user according to recent use.

Intelligent system logic automatically detects which insert is in place and displays the appropriate operating settings and controls for that process.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material.

Recommended applications for EMS 150 V Plus:

- **Ultra-high-resolution magnification SEM**
- **Carbon coating of TEM grids**
- **Protective platinum layers for FIB**
- **R&D of corrosion-, friction-, and wear- protective layers**
- **Protective layers on medical devices**
- **BSE imaging**
- **EDX, WDS, EBSD analysis**
- **Carbon coating of replicas**
- **Nano-technology e.g. Zeolites, polymer nanobrushes**

These products are for Research Use Only.

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating recipes are already stored but also allows the user to create their own. Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

Automatic, controlled carbon rod evaporation for TEM applications

Careful evaporation allows precise control of carbon thickness (with or without the optional film thickness monitor). The quality of the resulting carbon films is also enhanced by the eradication of “sparking” that is a common feature of less advanced coaters.

For reproducible high-quality carbon films, we would recommend the use of shaped carbon rods. Rods are higher purity, less susceptible to debris and easier to control. Pulsed and ramped carbon rod recipes are supplied as standard.

EMS 150V Plus Automatic Coater (*continued*)

Cool magnetron sputtering

Sputter coating is a technique widely used in various applications; it is possible to create a plasma and sputter metals with high voltage, poor vacuum and no automation. However, this is not suitable for electron microscopy applications because it will heat the sample and can result in damage when the plasma interacts with the sample. The EMS 150V Plus series uses low temperature enhanced-plasma magnetrons optimized for the turbomolecular pump pressures, combined with low current and deposition control, which ensures your sample is protected and uniformly coated.

The EMS 150V S Plus and EMS 150V ES Plus use easy-change, 57 mm diameter, disc-style targets which are designed to sputter oxidizing and noble metals. The EMS 150V S Plus and EMS 150V ES Plus are fitted as standard with a chromium (Cr) sputter target. Other targets options include: Au, Au/Pd, Pt/Pd, Pd, Pt, Cu, Ir, W, ITO and Al. Others are available on request.

Pulsed cleaning for Al sputtering

Aluminum (Al) rapidly forms an oxide layer, which can be difficult to remove, but the ES & S Plus have special recipes for aluminum that reduce the oxide removal time and prevent excessive pre-sputtering of the target.

Interchangeable plug-in heads

This allows the user to configure the system as a sputter coater, evaporator or glow discharge system - all in one space saving format. A carbon cord evaporation insert is available as an option. Automatic detection of the head type when changed.

Detachable chamber with built-in implosion guard

Removable glass chamber and easily accessible base and top plate allows for an easy cleaning process.

Users can rapidly change the chamber, if necessary, to avoid cross contamination of sensitive samples.

Tall chamber option is available for carbon evaporation to avoid sample heating, to improve uniformity for sputtering and to hold taller samples.

Multiple stage options

The EMS 150V Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except for the rotary planetary stage).

Some examples:

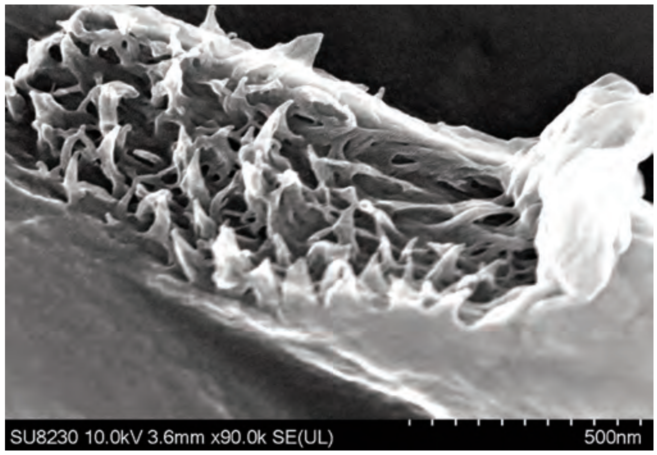
- Rotation stage (supplied as standard): 50 mm Ø can accommodate six standard stubs. Height can be pre-set.
- Rotate-tilt stage for improved uniform coating: 50 mm Ø. Tilt and height can be pre-set.
- Variable angle, rotary planetary stage for heavily contoured samples.
- Large flat rotation stage with offset gear box for 4”/100 mm wafers.
- Rotation stage for glass microscope slides.

Other options are available on request.

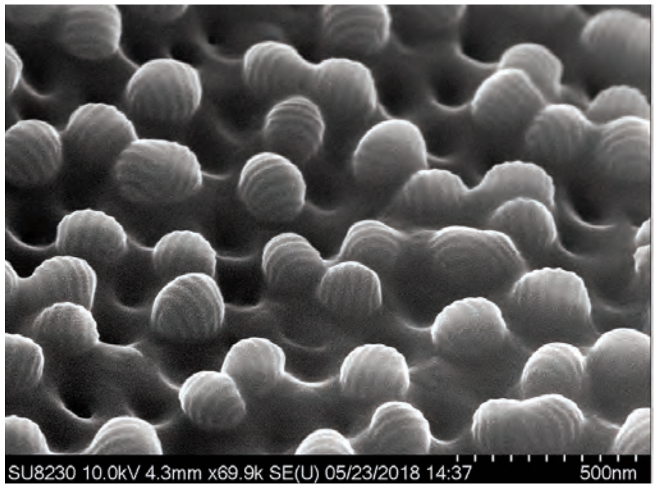
Safety

The EMS 150V Plus meets key industry CE standards

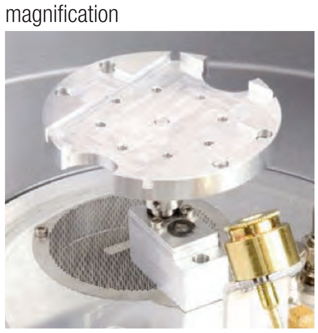
- All electronic components are protected by covers
- Implosion guard prevents user injury in event of chamber failure
- Vacuum interlocks remove power from deposition sources to prevent user exposure to high voltage in event of chamber being opened
- Electrical interlocks remove power when source head cover opened
- Overheating protection shuts down power supply



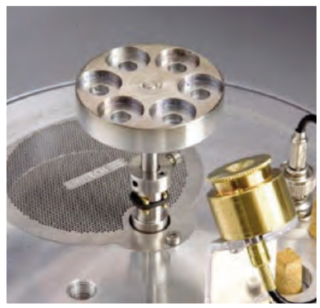
Single electrospinning fiber tear coated with 1nm Au x 90k magnification



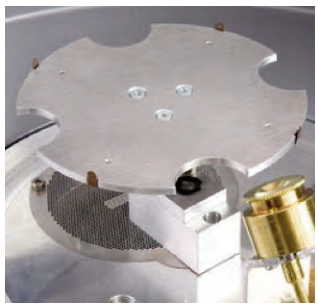
Microsporangium, spore size 150nm, coated with 3nm of Au x 70k magnification



Microscope slide stage



Rotation stage



Wafer stage

Rotacota planetary stage

EMS 150V Plus Automatic Coater (*continued*)

SPECIFICATIONS

Instrument Case	585 mm W x 470 mm D x 410 mm H (total height with coating head open: 650 mm)
Weight	33.4 kg (packed: 42 kg)
Packed Dimensions	725 mm W x 660 mm D x 680 mm H
Work Chamber	Borosilicate glass 150 mm ID x 133 mm H
Display	115.5mm W x 86.4mm H (active area), 640 RGB x 480 (display format), capacitive touch color display
User interface	Full graphical interface with touch screen buttons, includes features such as a log of the last 1000 coatings and reminders for when maintenance is due.
Sputter Target	Disc-style 57 mm Ø. A 0.3 mm thick chromium (Cr) is fitted as standard. V S and V ES versions only
Specimen stage	50 mm Ø rotation stage with rotation speed of 8-20 rpm. Other stages available on request.

Vacuum

Rotary Pump	5 m^3/hr two-stage rotary pump with oil mist filter. Hydraulically-formed bellows stainless-steel backing line.
Turbo Pump	Internally mounted 70 L/s air-cooled
Vacuum Measurement	Wide range gauge (10428)
Ultimate Vacuum	1 x 10^{-6} mbar

Typical ultimate vacuum of the pumping system in a clean instrument after pre-pumping and venting with dry nitrogen gas

Pump Down Time	5x10^{-6} mbar in 30 mins*
Sputter Vacuum Range	Between 5 x 10^{-3} and 1 x 10^{-1}mbar for gold targets

Processes

Sputtering	Sputter current 0-150 mA to a predetermined thickness (with optional FTM) or by the built-in timer. The maximum sputtering time is 60 minutes (without breaking vacuum and with automatically built-in cooling periods)
Evaporation	Carbon evaporation using rods/cord. Thermal evaporation of metals from filaments or boats. For cleaning TEM apertures a standard molybdenum boat (supplied) can be fitted

Visual Status Indicator

A large multi-colour status indicator light provides a visual indication of the state of the equipment, allowing users to easily identify the status of a progress at a distance.

The indicator LED shows the following states:

- Initialization
- Process Running
- Idle
- Coating in Progress
- Process Completed
- Process Ended in Fault Condition

Audio indication also sounds on completion of the process.

ORDERING INFORMATION

EMS 150V S Plus each

5700 EMS 150V S Plus Sputter Coater optimized for high-vacuum applications. High resolution turbomolecular pumped capacitive touch-screen controled sputter coater with status LED indicator, including quick release sputter insert and one 57 mm diameter x 0.3 mm chromium target. A rotating specimen stage, 50 mm diameter, supplied as standard.

EMS 150V E Plus each

5710 EMS 150V E Plus Carbon Evaporator optimized for high-vacuum applications. Turbomolecular pumped capacitive touch-screen controled carborn evaporator with status LED indicator suitable for TEM and SEM applications. Fitted with a carbon rod evaporation head for 3.05 mm diameter carbon rods. Supplied with carbon rods (C5422 3.05 mm x 300 mm) and a carbon rod shaper (manual operation). A rotating specimen stage, 50 mm diameter, supplied as standard.

EMS 150V ES Plus each

5720 EMS 150V ES Plus Combined Sputtering and Carbon Coating System optimized for high-vacuum applications. The deposition heads can be swapped in seconds and the intelligent system logic automatically recognizes which head is in place and displays the appropriate operating settings. Consists of high resolution turbomolecular pumped capacitive touch-screen controled sputter coater and carborn evaporator with status LED indicator, including quick release sputter insert and one 57 mm diameter x 0.3 mm chromium target, and high vacuum carbon rod evaporation coater, including quick-release carbon rod insert for 3.05 mm diameter carbon rods. Coating inserts are interchangeable. A rotating specimen stage, 50 mm diameter, supplied as standard.

Rotary pump requirements (needs to be ordered separately)

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

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 54 mm x 0.3 mm thick chromium (Cr) target as standard. For additional targets see Sputtering Targets section	each
3210	Additional sputter insert for quick metal change (V E and V ES versions only). Note: this is an entire sputtering assembly.	each
3230	Carbon rod evaporation head insert (for 3.05 mm Ø rods)	each
3240	Carbon rod evaporation head insert (for 6.15 mm Ø rods). Note that EMS recommends 3.05 mm Ø rods as they offer greater process control and are more economical (less wastage).	each
3250	Carbon fiber evaporation head insert	each
3260	Metal evaporation and aperture cleaning head insert, including the ability to evaporate upwards or downwards (V E and V ES versions only). Supplied with a pack of ten tungsten filaments and a molybdenum boat.	each
3270	Extended height vacuum chamber (214 mm high – the standard chamber is 127 mm high). For increased source to sample distance and for coating large specimens	each

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EMS 150V Plus Automatic Coater (*continued*)

ORDERING INFORMATION (continued)

3280	Rotating vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS 150V Plus when bench depth is limited	each
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	each
3300	Spare quartz crystal for FTM.	each
3320	Full range vacuum gauge for low and high vacuum measurement (a low vacuum Pirani gauge is fitted as standard)	each
4513	Glow discharge insert to modify surface properties (e.g. hydrophobic to hydrophilic conversion) or to clean surface residues (VS and V ES only). Can be retrofitted	each
13530	Standard coating shield assembly	each
27691	Extended glass cylinder assembly, 220 mm H	each
11880	Extended height coating shield assembly	each

Specimen stages

The EMS 150V Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage):

3330	Rotation stage, 50 mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment	each
3340	Rotate-tilt specimen stage with adjustable tilt (up to 90 degrees) and height (37 mm-60 mm). Tilt angle can be preset. 50 mm Ø specimen platform with six stub positions for 15 mm or 6.5 mm or ⅛" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
3350	Variable angle "Rotacota" rotary planetary stage with 50 mm Ø specimen platform. Has six stub positions for 15 mm or 6.5 mm or ⅛" pin stubs. Stage rotation speed variable between 8 and 20rpm	each
3360	Flat rotation specimen stage for 100 mm / 4" wafers, includes gearbox for increased coverage. Stage rotation speed variable between 8 and 20rpm	each
3370	Rotating specimen stage for glass microscope slides (up to two x 75 mm x 25 mm slides). Stage rotation speed variable between 8 and 20rpm. Includes gear box to allow optional FTM to be used	each
23850	Tilt stage sub-assembly 4"	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 »»»

Carbon supplies

3500	Carbon rods – 6.15 mm Ø x 100 mm length (unshaped) pack of 10	10/pk
3510	Carbon rods – 6.15 mm Ø x 50 mm length (shaped) pack of 10	10/pk
3520	Carbon rods – 3.05 mm Ø x 50 mm length (shaped) pack of 10	10/pk
3530	Carbon rods 3.05 mm Ø x 300 mm length (unshaped) pack of 10	10/pk
3540	Carbon fiber cord – high purity – 1m	each
3550	Carbon fiber cord – high purity – 5m	each
3560	Carbon fiber cord – standard grade – 1m	each
3570	Carbon fiber cord – standard grade – 10m	each
3580	Carbon fiber cord – standard grade – 100m	each
3590	Manual rod shaper for 6.15 mm Ø carbon rods	each
3595	Manual rod shaper for 3.05 mm Ø carbon rods	each
12097	Wedge Tool	each

Evaporation Supplies

The EMS 150V S Plus and EMS 150V ES Plus are fitted as standard with a 0.3 mm thick chromium (Cr) sputter target. Other optional targets:

73830-SP	Tungsten wire baskets – pack of 10	each
73810-SP	Molybdenum boats – pack of 10	each

Sputtering Targets

The EMS 150V S Plus and EMS 150V ES Plus are fitted as standard with a 0.3 mm thick chromium (Cr) sputter target. Other optional targets:

3410	57 mm Ø x 0.1 mm Gold	each
3410-2	57 mm Ø x 0.2 mm Gold	each
3411	57 mm Ø x 0.1 mm Gold/Palladium (80/20)	each
3411-2	57 mm Ø x 0.2 mm Gold/Palladium (80/20)	each
3412	57 mm Ø x 0.1 mm Platinum	each
3413	57 mm Ø x 0.1 mm Nickel	each
3414	57 mm Ø x 0.1 mm Silver	each
3415	57 mm Ø x 0.1 mm Palladium	each
3416	57 mm Ø x 0.1 mm Copper	each
3417	57 mm Ø x 0.3 mm Chromium	each
3418	57 mm Ø x 0.5 mm Tungsten	each
3419	57 mm Ø x 1.5 mm Chromium	each
3420	57 mm Ø x 0.2 mm Tungsten	each
3421	54 mm Ø x 1.5 mm Carbon	each
3422	57 mm Ø x 0.1 mm Aluminum	each
3423	57 mm Ø x 0.1 mm Platinum/Palladium (80/20)	each
3424	57 mm Ø x 1.5 mm Titanium	each
3425	57 mm Ø x 0.3 mm Platinum/Palladium (80/20)	each
3426	57 mm Ø x 0.3 mm Gold	each
3427	57 mm Ø x 0.3 mm Gold/Palladium (80/20)	each
3428	57 mm Ø x 0.3 mm Platinum	each
3429	57 mm Ø x 0.5 mm Titanium	each
3430	57 mm Ø x 0.1 mm Iron	each
3431	57 mm Ø x 0.3 mm Iridium	each
3432	57 mm Ø x 0.1 mm Cobalt	each
3433	57 mm Ø x 0.1 mm Tin	each
3434	57 mm Ø x 0.1 mm Molybdenum	each
3435	57 mm Ø x 0.3 mm Magnesium	each
3436	57 mm Ø x 0.1 mm Tantalum	each
3437	57 mm Ø x 3 mm Indium Tin Oxide (90/10)	each

Other consumables and spare kits

3600	Metal evaporation basket – pack of 10 (for use with metal evaporation head)	each
3610	Two-year spares kit for EMS 150V S Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, quartz crystals, O-rings	each
3620	Two-year spares kit for EMS 150V E Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, carbon fiber – fine, carbon rods 3.05 mm, quartz crystals, O-rings	each
3630	Two-year spares kit for EMS 150V ES Plus Includes: chromium (Cr) target, glass cylinder, carbon fiber cord, carbon fiber – fine, carbon rods 3.05 mm, quartz crystals, O-rings	each
07803	Basic Oil Mist Filter (spare)	each
13233	Rotary Pump Oil (spare), 1L	each
13235	Rotary Pump Oil (spare), 5L	each
27508	O-ring (each) 2 required per coater	each
27607	Non-seal L Gasket	each
6413	Additional Standard Glass Cylinder Assembly	each

EMS150GB Turbo-Pumped Sputter Coater/ Carbon Coater – For Glove Box

QUICK OVERVIEW

The EMS150GB is a modular glove box version of the highly successful EMS150T ES bench top turbomolecular-pumped coating system - suitable for SEM, TEM and many thin-film applications. The EMS150GB comes as standard with sputtering and carbon rod evaporation inserts and a rotating specimen stage. Options include a metal evaporation insert, glow discharge, a film thickness monitor (FTM), aperture cleaning insert and special stages to suit a range of specimen types.

FEATURES

- **Modular construction for mounting in glove boxes**
- **Integral glove box pressure monitoring**
- **Remote operation from a touch screen control panel**
- **Metal sputtering and carbon evaporation in one system**
- **Fine grain sputtering for advanced high resolution FE-SEM applications**
- **High vacuum turbo pumping - allows sputtering of a wide range oxidising and non-oxidising metals**
- **High vacuum carbon rod coating - ideal for SEM and TEM carbon coating applications (carbon fiber available as an option)**
- **Advanced "anti-stick" carbon rod evaporation gun – simple operation, reproducible results**
- **Control of evaporation current profile - ensures consistently reproducible carbon films**
- **Precise thickness control using the film thickness monitor option**
- **Fully automatic touch screen control – rapid data input, simple operation**
- **Multiple, customer defined coating schedules can be stored - ideal for multi-user laboratories**
- **Automatic vacuum control which can be pre-programmed to suit the process and material – no manual needle valve to adjust**
- **"Intelligent" recognition of system - automatically detects the type of coating insert fitted**
- **Easy-to-change, drop-in style specimen stages (flat rotation stage as standard)**
- **Vacuum shut down 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**
- **Power Factor Correction - complies with the current legislation (CE certification) - efficient use of power means reduced running costs**
- **Three-year warranty**

PRODUCT DESCRIPTION

The EMS150GB is single platform for sputtering and carbon rod evaporation. Metal evaporation using filament or boat sources is possible using an optional insert.

Depending upon user preference, the EMS150GB can be a top-of-the-range sputter coater for high resolution scanning electron microscopy (SEM), or a high vacuum carbon coater suitable for SEM and transmission electron microscopy (TEM). The flexibility of the system can be further expanded using a range of optional accessories.

The EMS150GB can rapidly sputter a wide selection of oxidising and non-oxidising metals making it ideal for many thin film applications.

The EMS150GB has an integral turbomolecular pump and additionally requires a suitable rotary pump or dry pump to "back" the turbomolecular pump (see Option and Accessories).

Flexible modular design

A modular design enables the vacuum chamber to be mounted through the base of the glove box or - when modification to the glove box floor is not possible - inside the glove box (optional feedthroughs are required).

A separate power supply housed in a rugged case is designed to be floor mounted and can be positioned conveniently beneath the glove box or bench. The touch screen user interface is housed in a robust stainless steel case and can be positioned outside of the glove box environment, if preferred.

EMS150GB mounted in a glove box

Options for glove box mounting:

Vacuum module mounted in the floor of the glove box

A cut-out is made in the floor of the glove box and the vacuum chamber fitted and sealed with the gasket supplied. External connections from the floor mounted power supply, vacuum pump and argon gas can then be made directly to the chamber.

OR

Vacuum module placed inside the glove box

The chamber assembly is placed inside the glove box and the power supply, vacuum pump and argon gas connections are made through two KF40 feedthroughs in the rear of the glove box.

Note: each EMS150GB is supplied with an accessory/configuration kit to suit either internally or externally mounted vacuum chambers. Additional kits can be designed on a case-by-case basis to allow the system to be adapted to various manufacturers' glove boxes. *Please contact EMS for more details.*

EMS150GB Turbo-Pumped Sputter Coater/Carbon Coater — For Glove Box (continued)

Touch screen user interface

Enclosed in a stainless steel case and mounted at a convenient position outside the glove box, the touch screen allows multiple users to input and store coating protocols.

Touch screen control module

Vacuum module - including "vacuum shutdown" and glove box pressure interlock

The vacuum module houses all the working components, including the efficient 70L/s air-cooled turbomolecular pump. An automatic bleed control ensures optimum vacuum conditions during sputtering and a full range active gauge is fitted as standard to monitor the vacuum.

The EMS150GB includes "vacuum shutdown", a convenient feature which enhances vacuum performance by allowing the chamber vacuum to be maintained when the coater is not in use.

A unique feature of the EMS150GB is the integral pressure interlock switch. This independently monitors the pressure inside the glove box and shuts off the vacuum pump if the glove box atmosphere is unacceptably reduced due to a vacuum leak.


Vacuum chamber assembly

The vacuum chamber is 214mm high to allow for increased source to substrate distances required for coating large specimens. It has an external diameter of 165mm and comes with an integral implosion guard. The chamber assembly is easily removed to allow specimen exchange and chamber cleaning.

A variable speed rotary specimen stage is fitted as standard, with full height adjustment from 0 to 190mm above the base plate; other stages are available as options.

Sputter coating and carbon coating as standard, plus an option for metal evaporation

"Intelligent" Quick-change deposition inserts

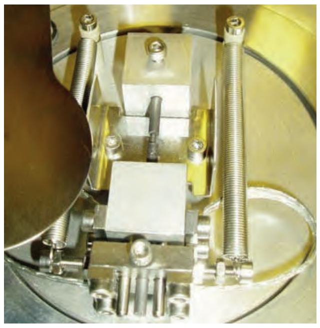
The deposition inserts can be swapped in seconds and the intelligent system logic automatically recognises which insert is in place and displays the appropriate operating settings.

High resolution sputter coating

The EMS150GB features a high-resolution sputter coater insert (3200) for oxidising and non-oxidising (noble) metals. A wide selection of sputtering targets is available, including iridium and chromium, which are highly recommended for FE-SEM applications. Please see Options and Accessories for details of available metal targets.

Carbon rod evaporation

The high vacuum carbon rod coating insert (3230) is 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 and the advanced "anti-stick" carbon rod evaporation gun offers simple operation and reproducible results. A carbon fiber evaporation insert (3250) is available as an option (see Options and Accessories).

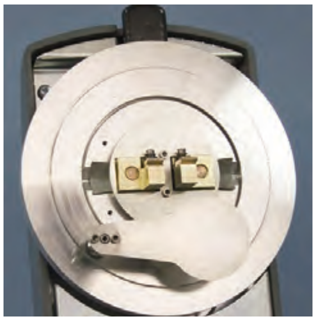

Carbon rod evaporation insert

Metal evaporation/ aperture cleaning option

A quick change insert (3260) allows metal evaporation from tungsten baskets or molybedenum boats - ideal for thin film applications. For ease of set up in a glove box, the metal charge can be loaded into the evaporation source away from the vacuum chamber.


Metal evaporation insert option

Each of the above configurations can be used with a range of optional accessories. See options and Accessories for details.


Carbon fiber evaporation insert option

Rapid data entry

At the operational heart of EMS150GB is a simple colour 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 provided.

Additional Information

Options and Accessories (including details the standard specimen stage)

Coating inserts included with the EMS150GB

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

- Sputtering head insert (3200) is suitable for oxidising and non-oxidising metals. Supplied with a 54mm Ø x 0.3mm thick chromium target (3417) as standard. For additional targets see "Ordering Information" section.

Note: changing sputtering targets is easy, but additional sputter head inserts can be purchased for even quicker coating material change - see (3210)

- Carbon rod evaporation head insert and accessories for 3.05mm Ø rods (3230)

Optional coating inserts

Carbon rod evaporation head insert for 6.15mm Ø rods (3240). Note that EMS recommends 3.05mm Ø rods as they offer greater process control and are more economical (less wastage)

continued »»»

EMS150GB Turbo-Pumped Sputter Coater/Carbon Coater — For Glove Box (continued)

- Carbon fiber evaporation head insert (3250)
- Metal evaporation and aperture cleaning head insert (3260), using tungsten wire baskets and molybdenum boats. Includes electrode extensions to allow upwards evaporation, if required. Note: when the electrodes are fitted some stage options cannot be used

Specimen stages

The EMS150GB has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage).

- 6800-S rotation stage, 50mm Ø (supplied as standard). This stage only rotates – no tilt or height adjustment.
- 6801 rotate-tilt stage, 50mm Ø with height adjustment (target to stage height variable between 37mm and 60mm). The tilt angle can be pre-set.
- 6803 variable angle 'Rota-Cota' rotary planetary specimen stage with 50mm Ø specimen platform with six stub positions for 15mm or 6.5mm or 1/8" pin stubs.
- 3360 flat rotation stage for 4"/100mm wafers. Includes gear box which needs to be fitted when the optional FTM is being used or for coating over the full area of the stage.
- 6804 rotation stage for glass microscope slides. Allows two 75 x 25mm slides to be coated.

4" wafer stage option

standard rotating specimen stage and optional film thickness monitor (FTM)

Note: all rotation stages have rotation speeds that can be variable between 8 and 20 rpm.

SPECIFICATIONS

Vacuum module size	267mm W x 490mm D x 494mm H (total height with coating head open: 767mm)
Power supply size	310mm W x 357mm D x 262mm H
User interface size & weight	160mm W x 157mm D x 42mm.
Weight:	40kg
Packed dimensions	725mm W x 660mm D x 787mm H (44kg)
Work chamber	Borosilicate glass 152mm Ø (inside) x 214mm 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 100 coatings carried out and reminders for when maintenance is due
Sputtering target	Disc style 57mm Ø x 0.3mm thick chromium target is fitted as standard
Specimen stage	60mm Ø rotation stage. Rotation speed 8 ~ 20 rpm
For alternative stages see Options and Acessories	
Specimen shutter	An automatic shutter is fitted as standard to shield specimens during pre-sputtering of oxidising metals and protection during evaporation outgassing procedures

Vacuum

Turbomolecular pump	Internally mounted, 70L/s air-cooled turbomolecular pump
Rotary pump	5m3/hr-1 two-stage rotary pump with oil mist filter (order separately: 91003)
Vacuum measurement	An active full-range gauge is fitted
Typical ultimate vacuum	$5x10^{-5}$mbar
Sputter vacuum range	Between $5x10^{-3}$ and $5x10^{-1}$mbar

Interlocks

The EMS150GB is interlocked to prevent continuous pumping of the glove box in the event of a vacuum leak

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)

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

Metal evaporation and aperture cleaning insert (option) For thermal evaporation of metals from filaments or molybdenum boat 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

Services and other information

Gases	Quick-connect inlet for: argon sputtering process gas, 99.999% ("zero grade")
Nitrogen venting gas (no quick-connect – uses the glove box atmosphere as the source	
Electrical supply	90-250V ~ 50/60 Hz 1400 VA including 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

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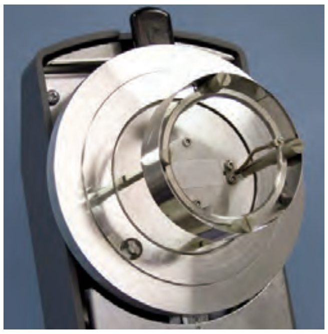
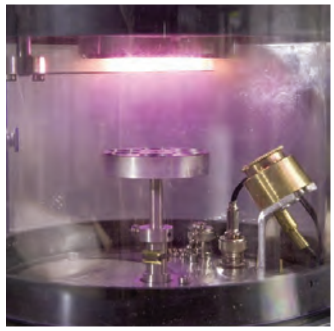
EMS150GB Turbo-Pumped Sputter Coater/Carbon Coater — For Glove Box (continued)

Other options, including FTM and glow discharge

- 3270 extended height chamber (87mm higher than the standard stage) For tall specimens.
- 3290 Film Thickness Monitor (FTM). Consists of a controller and quartz crystal oscillator built into the EMS150GB and a vacuum feed though, chamber mounted crystal holder and quartz crystal. As sputtered or evaporated material is deposited onto the crystal, so its frequency of oscillation is modified. This 'modification' is used to measure and control the thickness of material deposited.
- 4513 glow discharge insert. Used to modify surface properties (eg hydrophobic to hydrophilic conversion).



Glow discharge attachment option



During sputtering, optional film thickness monitor (FTM) shown

ORDERING INFORMATION

EMS150GB	A modular, high resolution turbomolecular-pumped sputter coater/carbon rod evaporator for glove boxes. **Includes:** sputtering insert, 54mm Ø x 0.3mm chromium target, carbon rod evaporation insert, carbon rods (3.05mm Ø x 300mm) and carbon rod shaper (manual operation). Fitted with a rotation stage	each

Rotary pump requirements (needs to be ordered separately)

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

Options and accessories

3230	Carbon rod evaporation head insert (for 3.05mm Ø rods	each
3240	Carbon rod evaporation head insert (for 6.15mm Ø rods).	each

Note that EMS recommends 3.05mm Ø rods as they offer greater process control and are more economical

3250	Carbon fiber evaporation head insert	each
3260	Metal evaporation and aperture cleaning head insert, including the ability to evaporate upwards or downwards. Supplied with a pack of ten tungsten filaments and a molybdenum boat.	each
3270	Extended height vacuum chamber (214mm high – the standard chamber is 127mm high). For increased source to sample distance and for coating large specimens	each
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	each
3300	Spare quartz crystal.	each
3320	Full range vacuum gauge for low and high vacuum measurement (a low vacuum Pirani gauge is fitted as standard)	each
4513	Glow discharge insert to modify surface properties (eg hydrophobic to hydrophilic conversion) or to clean surface residues	each
3600	Metal evaporation basket - pack of 10 (for use with metal evaporation head)	each
3630	Two-year spares kit for EMS150GB	each

Includes: chromium target, glass cylinder, carbon fiber cord, carbon fiber - fine, carbon rods 3.05mm, quartz crystals, O-rings — each

Specimen stages

6790-S	Swinging arm stage drive, a stage drive and positioning mechanism which positions the stage under the correct target. Also provides rotation drive to the stage. Rotation Speed Max 38 rpm Min 14 rpm	each
6800-S	Rotating specimen stage for 6" (152 mm) wafers, with rotation variable between preset limits.	each
6801	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 15 mm, 10 mm, 6.5 mm or 1/8" pin stubs. The stage rotation speed is variable between preset limits. The target to stage height is variable between 0 mm and 42 mm for the standard stage. When used with the extended height cylinder the target to stage height would be an additional 87 mm.	each
6802	50 mm Ø variable height specimen stage with six stub positions for 15 mm, 10 mm, 6.5 mm disc stubs or 1/8" pin stubs. Stage rotation speed variable between preset limits.	each

Note: *Target to stage height is variable between 10 mm and 53 mm for the standard stage. The stage is supplied with two mounting pillars; one provides 10 mm to 32 mm target to stage distance and the other 31 mm to 53 mm. An adjustable stop is used to set the height. When used with the extended height cylinder (optional accessory) the target to stage height would be an additional 87 mm.*

6803	50 mm Ø rotary tilting stage. A rotary planetary style stage with a variable tilt angle from horizontal to 30 degrees. The platform has six positions for either 6.5 mm, 10 mm and 15 mm disc stubs or 1/8" pin stubs. Rotation speed is variable between preset limits.	each

Note: *depending upon specimen height, this stage may require the optional extended height cylinder.*

6804	A 90 mm Ø specimen stage for glass microscope slides (up to two 75 mm x 25 mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pin stubs. The stage rotation speed is variable between preset limits. A gear box is included to allow the optional FTM to be used.	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

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

Innovative and versatile Sputter Coater and Carbon Evaporator for SEM Applications

QUICK OVERVIEW

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

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

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

KEY FEATURES

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

PRODUCT DESCRIPTION

Ideal for sputter coating large specimens for SEM

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

Triple sputtering head

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

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

Triple sputtering head (closed)

Triple sputtering head (open)

Molded case with color touch screen

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

The vacuum chamber has an internal diameter of 283 mm/12" and comes with an integral implosion guard. The EMS300R T also includes 'vacuum

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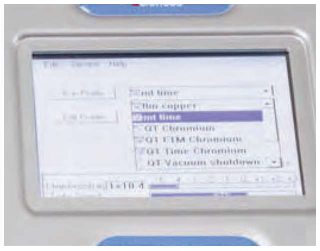
EMS300R *(continued)*

shutdown', which enhances vacuum performance by allowing the chamber vacuum to be maintained when the system is not in use.

A variable speed rotary specimen stage is fitted as standard for 8"/203 mm and 6"/152 mm wafers, with other stages available as options, please see the Options and Accessories section below.

Rapid data entry



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

Maintenance

The intuitive touch screen interface features maintenance prompts which highlight:

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

Improved Tolerance to Out Gassing Substrates

The pump time out has been moved to a new level defined by "safe outgas threshold" parameter, this is user definable between 5×10^{-2} and 2 mbar.

Until now the system would limit the time to achieve operational vac of 1×10^{-3} to 10 minutes. With the new modification once the "Safe outgas threshold" has been achieved the system will pump continuously. Implemented from software version 2-0042 onwards.

Controlled Evaporation Process

This process can automate a metal evaporation process when using low power evaporation sources. It can be used with the FTM.

The use of key properties about the source and required termination allow the process to:

- **Bring the source current up to the point of evaporation.**
- **Ramp the current up at a controlled rate until evaporation is seen.**
- **Close the shutter and shut the evaporation PSU down when the specified thickness is seen.**
- **Check the source for open or short circuit when starting the process.**

This process will be available on the EMS150T with software version 2-0048 or later. Consideration should be made to the thermal effects of the evaporation source when using the FTM to control the process.

Improvements to the Pulse Carbon Rod Evaporation

Continued work on this process has found that the thickness deposited can be variable if the power applied to the rods is either too high or too low. With the optimum current being a narrow margin between them. However, limiting the available voltage supplied to the rods increases this current window. This coupled with reducing the spigot diameter to work at a more optimum point in the PSU operation produces a more continuous spark which in turn produces a more repeatable thickness.

This improvement can be implemented by:

- **Software version 2-0046 or later implements the voltage limit. Note all the carbon rod profiles should be deleted if a unit is updated to this software version.**
- **The evaporation current should be set between 60 and 65A.**
- **The reduced spigot diamter of 1.2mm can be produced with the blue handle rod shaper.**

OPTIONS AND ACCESSORIES

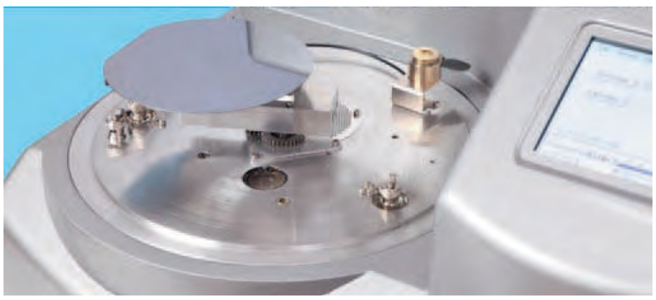
Specimen stages

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

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

Rotary planetary specimen stage

Glass microscope slide stage



Standard specimen stage

Lockable emergency stop (e-stop) switch option. Note: Does not inhibit or replace the normal on/off switch function

EMS300R *(continued)*

SPECIFICATIONS

Instrument Case	585mm W x 470mm D x 410mm H (total height with coating head open: 650mm)
Weight	36.4kg
Packed Dimensions	725mm W x 660mm D x 680mm H (44.8kg)
Work Chamber	Borosilicate glass 283mm ID x 127mm H
Safety Shield	Integral polyethylene terephthalate (PET) cylinder
Display	145mm x 320mm x 240mm color graphic thin film transistor (TFT) display
User Interface	Intuitive full graphical interface with touch screen buttons, includes features such as a log of the last 100 coatings carried out and reminders for when maintenance is due
Sputter Targets	Disc style 57mm Ø with thick depending upon the targets fitted. Three 57 mm Ø x 0.1 mm thick gold (Au) targets are fitted as standard.

Vacuum

Rotary Pump	50L/m two-stage rotary pump with oil mist filter.Vacuum measurement Pirani gauge
Typical Ultimate Vacuum	2 x 10^{-2} mbar in a clean system after pre-pumping with dry nitrogen gas
Specimen Stage	Flat rotation stage for 200mm/8" and 150mm/6" wafers fitted as standard. Rotation speed is variable between preset options. For alternative stages see Options and Accessories

Processes

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

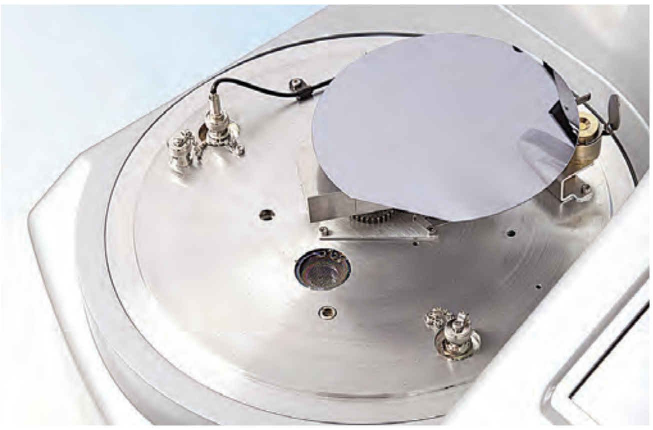
Services and other information

Gases	Argon sputtering process gas, 99.999% Nitrogen venting gas (optional)
Electrical Supply	90-250V 50/60Hz 1,400VA 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

Other options

Extended height chamber – for taller specimens

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



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

III EMS300R *(continued)*

ORDERING INFORMATION

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

Specimen stages

6400-S	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 6.5mm, 10 mm and 15 mm or 1/8" pin stubs. Stage rotation speed is variable between preset limits. No rotation is possible when in single target mode. Target to stage height is variable between 0 mm and 42 mm for the standard stage. When used with the extended height cylinder (optional accessory 10596) the target to stage height would be an additional 87 mm	each
6401	50 mm Ø variable height specimen stage with six stub positions for, 6.5 mm 10 mm and 15 mm disc stubs or 1/8" pin stubs. Stage rotation speed is variable between preset limits	each
6402	50 mm Ø rotary tilting stage. A rotary planetary style stage with variable tilt angle from horizontal to 30 degrees. The platform has six positions for either 6.5 mm 10 mm and 15 mm disc stubs or 1/8" pin stubs. Rotation speed is variable between preset limits. *Note: depending upon specimen height, this stage may require the optional extended height cylinder*	each
6403	A 90mm Ø specimen stage for glass microscope slides (up to two x 75mm x 25mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pins stub. Stage rotation speed variable between preset limits. Includes a gear box to allow the optional FTM to be used	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

Options and Accessories

6404	Film thickness monitor (FTM) attachment. Including oscillator, feed-through, quartz crystal holder and one C5460 quartz crystal	each
6405	Spare quartz crystal	each
6406	Extended height vacuum chamber (214 mm in height - the standard chamber is 127 mm high). For increased source to specimen distance and for coating larger specimens	each
6407	Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the Q300R T when bench depth is limited	each
6408	A lockable emergency stop (e-stop) switch which can be mounted on top of the system in a position easily accessible for the operator. It is provided with a key to release the knob after activation. Note: the addition of the e-stop does not inhibit or replace the normal On/Off switch function. The e-stop can be retrofitted to existing systems	each
6409	Coating shields. Shields can be fitted to protect large surfaces from coating deposition, easily removable for ease of cleaning	each
6410	Two-year spares kit for EMS300R T. Includes: 3 x 57mm Ø x 0.3mm gold (Au) target, standard glass chamber assembly, O-rings etc.	each
6411	6" Wafer Specimen Stage: A flat adjustable stage capable of accepting 6" or 101.6 mm wafers	each
6412	4" Wafer Stage : A flat 4 ½" 102mm flat, drop in wafer stage which accepts 2, 3, 4 " Wafers	each
6413	Additional Standard Glass Cylinder Assembly	each

Sputter targets

Note: The EMS 300T T Plus is fitted as standard with three 0.3 mm chromium (Cr) targets (3417). Other optional targets are available (three required):

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

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

Terms and Techniques

Rotary Pump

A type of vacuum pump where pumping is produced by moving air from one side of a rotating cylinder to another by means of an eccentric drum. Rotary pumps are used with all of our coaters, either to evacuate instruments directly, or to rough pump and back high-vacuum sputter coaters and vacuum evaporators.

Pirani Vacuum Gauge

A gauge for measuring vacuum that uses a heated sensor wire. Pressure is determined by measuring the current needed to keep the wire at a constant temperature. A Pirani gauge will only measure vacuum levels in the lower range (down to 10^{-3}mbar) and is typically used in low vacuum systems, such as rotary-pumped sputter coaters.

A Pirani gauge is sometimes used in conjunction with a high vacuum gauge, such as Penning, to measure vacuum in the low pressure range. Most of our high vacuum products now use full-range gauges — a single gauge which measures vacuum from atmosphere to high vacuum.

EMS 300T T Plus Triple Target, Large Chamber, Turbo-Pumped Sputter Coater

QUICK OVERVIEW

The EMS300T T Plus is a large chamber, turbo-pumped coating system, ideally suited for sputtering a single large diameter specimen up to 8"/200mm or multiple smaller specimens over a similar diameter. Ideal for thin-film applications and SEM/FE-SEM. It is fitted with three sputtering heads to ensure even deposition of individual large specimens or multiple specimens.

Please note it is not possible to sequentially sputter three different sputtering metals from each sputtering head. For sequential coating, see the EMS300T D Plus.

KEY FEATURES

- **Ultimate vacuum of $1x10^{-6}$mbar or less possible**
- **New touch and swipe capacitive screen**
- **USB port for upgrades and download of process log files**
- **Multiple-use profiles can be set up on one machine**
- **New software sorts recipes per user according to recent use**
- **16GB of memory can store more than 1000 recipes**
- **New multi-color LED visual status indicator**
- **Interchangeable stage options**
- **Three sputter heads for larger area deposition of different materials**

PRODUCT DESCRIPTION

Detachable chamber with built-in implosion guard

Removable glass chamber and easily accessible base and top plate allows for an easy cleaning process. Users can rapidly change the chamber, if necessary, to avoid cross contamination of sensitive samples. Tall chamber option is available for improved uniformity for sputtering and to hold larger substrates.

Triple Target Sputtering System

The EMS 300T T Plus is fitted with three individual sputtering heads to ensure even deposition of individual large specimens or multiple specimens. For economical coating of small specimens, 'single target' mode can be selected. They are ideal coaters for the preparation of large specimens for examination by SEM, FEG-SEM. To ensure even deposition, the EMS 300T T Plus coaters are fitted with a rotating specimen stage and three individual magnetron target assemblies, which enhance the efficiency of the process by using low voltages.

Triple sputtering head with automatic shutter open

Triple sputtering head with automatic shutter closed

Recommended applications for EMS300T T Plus:

- **Wafer Inspection**
- **Multiple sample preparation for SEM**

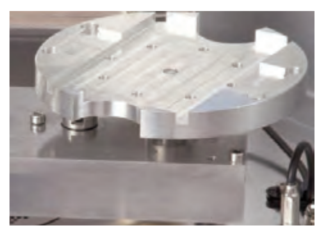
These products are for Research Use Only.

Multiple stage options

The EMS 300T T Plus has specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and the rotation speed is variable between pre-set limits. Flat rotation stage for 200 mm/8" and 150 mm/6" wafers (fitted as standard).

rotary planetary specimen stage (option)

Standard specimen stage



Specimen stage for glass microscope slides (option)

EMS300T T Plus *(continued)*

EMS300T T Plus has a 200 mm wafer capability

Safety

The EMS 300T T Plus meets key industry CE standards

- All electronic components are protected by covers
- Implosion guard prevents user injury in event of chamber failure
- Vacuum interlocks remove power from deposition sources to prevent user exposure to high voltage in event of chamber being opened
- Overheating protection shuts down power supply

Vacuum control

High vacuum turbo pumping allows sputtering of a wide range of oxidizing and non-oxidizing metals for thin film and electron microscopy applications. Automatic vacuum control which can be pre-programmed to suit the process and material, therefore removing the need for manual intervention or control.

Cool magnetron sputtering

Sputter coating is a technique widely used in various applications; it is possible to create a plasma and sputter metals with high voltage, poor vacuum and no automation. However, this is not suitable for electron microscopy applications because it can heat the sample and result in damage when the plasma interacts with the sample. The EMS 300T T Plus uses low temperature enhanced-plasma magnetrons optimized for the turbomolecular pump pressures, combined with low current and deposition control, which ensures your sample is protected and uniformly coated.

The EMS 300T T Plus uses easy-change, 57 mm diameter, disc-style targets which are designed to sputter oxidizing and noble metals. The EMS 300T T Plus is fitted as standard with a chromium (Cr) sputter target. Other targets options include; Au, Au/Pd, Pt/Pd, Pd, Pt, Cu, Ir, W, ITO and Al, etc.

Pulsed cleaning for aluminum sputtering

Aluminum (Al) rapidly forms an oxide layer which can be difficult to remove. The EMS 300T T Plus has a special recipe for Aluminum that reduces the oxide removal time and prevents excessive pre-sputtering of the target.

Film thickness monitor

The EMS 300T T Plus can be fitted with an optional film thickness monitor (FTM), which measures the coating thickness on a quartz crystal monitor within the chamber, in order to control the coating thickness of material deposited on to the sample.

Film thickness monitor option

New user interface has been thoroughly updated:

- Dual-core ARM processor for a fast, responsive display
- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Quick and easy creation of process sequences with a simple copy, drag and drop operation

Allows multiple users to input and store coating recipes.

New feature to sort recipes per user according to recent use.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

continued »»

EMS300T T Plus *(continued)*

SPECIFICATIONS

Instrument Case	590mm W x 535mm D x 420mm H (maximum height during the opening of the coating head: 772mm)
Weight	36 kg (packed: 59kg)
Packed Dimensions	730 mm W x 630 mm D x 690 mm H
Work Chamber	Borosilicate glass with integral PET implosion guard Size 300 mm outside diameter x 127 mm High
Display	115.5mm W x 86.4mm H (active area), 640 RGB x 480 (display format), capacitive touch color display
User Interface	Full graphical interface with touch screen buttons, includes features such as a log of the last 1000 coatings and reminders for when maintenance is due

Specimen Stage

A flat rotation stage for 6”(150mm) and 8” (200mm) wafers is fitted as standard.

A rotating/tilt stage and the ‘rota cota’ rotary tilt stage are also options

Vacuum

Rotary Pump	4 m^3/hr, two stage rotary pump with oil mist filter for the 300T T Plus
Turbo Pump	Internally mounted 70L/sec air cooled
Vacuum Measurement	Pirani gauge as standard, full range gauge available as an option
Ultimate Vacuum	5 x 10^{-5}mbar*
Sputter Vacuum Range	5x10^{-2} to 5x10^{-3} mbar*

*Typical ultimate vacuum of the pumping system in a clean instrument after pre-pumping and venting with dry nitrogen gas

Processes

Sputter Deposition Current	Single target: 1 - 140mA All targets: 60 - 420mA
Visual Status Indicator	A large status multi-color indicator light provides a visual indication of the state of the equipment, allowing users to easily identify the status of a process at distance.

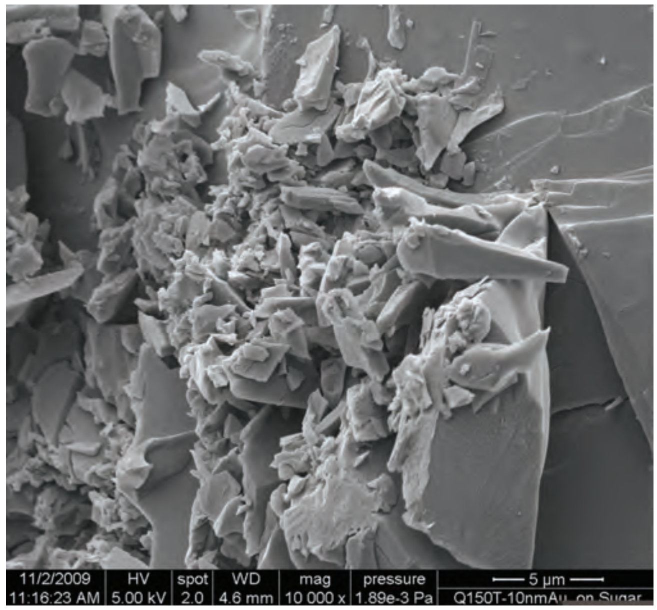
The indicator LED shows the following states:

- Initialization
- Process running
- Idle
- Coating in progress
- Process completed
- Process ended in fault condition

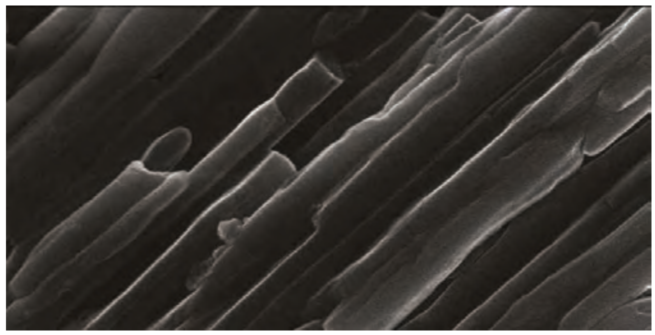
Audio indication also sounds on completion of the process.

Services

Gases	process gas argon, 99.999% Nominal 5 psi



10nm Au on Sugar



Sea mineral sample 3nm Cr, Mag x 25k

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EMS300T T Plus *(continued)*

ORDERING INFORMATION

EMS 300T T Plus	Triple Target, Large Chamber, Turbo-Pumped Sputter Coater, fitted with three sputtering heads to ensure even metal deposition. Includes three 57 mm Ø x 0.3 mm chromium (Cr) sputter targets. A flat rotation stage for 200 mm/8" and 150 mm/6" wafers is fitted as standard.	each

Rotary pump requirements (needs to be ordered separately)

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

Specimen stages

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

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

Options and Accessories

6555	Film thickness monitor (FTM) attachment. Including oscillator, feed-through, quartz crystal holder and one quartz crystal	each
6556	Spare quartz crystal	each
6557	Extended height vacuum chamber (214 mm in height – the standard chamber is 127 mm high). For increased source to specimen distance and for coating large specimens	each
6558	A lockable emergency stop (e-stop) switch which can be mounted on top of the system in a position easily accessible for the operator. It is provided with a key to release the knob after activation. Note: the addition of the e-stop does not inhibit or replace the normal On/Off switch function. The e-stop can be retrofitted to existing systems	each
6559	Coating shields. Shields can be fitted to protect large surfaces from coating deposition – easily removable for ease of cleaning	each
6560-A	Vacuum spigot allows more convenient connection of the vacuum hose to the rear of the EMS 300T T Plus when bench depth is limited	each
6561	Full range, active vacuum gauge capable of measurement over the range of 1000 mbar to 5×10^{-9} mbar. Typical ultimate vacuum of system is 5 x 10-5 mbar. Note: gauge must be factory fitted	each
6562	Spares kit, including: spare standard glass cylinder, three 3417 chromium (Cr) sputtering targets, vacuum tubing with coupling insert, argon gas tubing, three sputter head magnets, rotary pump oil mist filter and fuses	each

Sputter targets

Note: The EMS 300T T Plus is fitted as standard with three 0.3 mm chromium (Cr) targets (3417). Other optional targets are available (three required):

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

What is... Carbon Evaporation?

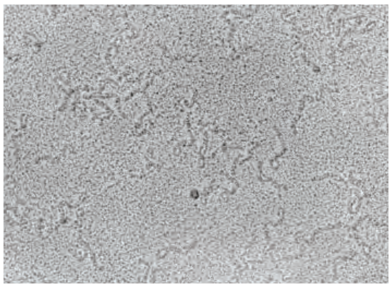
The use of Carbon Evaporation is well known in Electron Microscopy for support films and replicas in TEM, and X-Ray Microanalysis and conducting coatings in SEM.

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



Spectrin molecules

EMS 300T ES Large Chamber Turbo-Pumped Evaporator/Sputter Coater

QUICK OVERVIEW

The EMS 300T ES is a large chamber, turbomolecular-pumped coating system ideally suited to metal evaporation onto large diameter specimens up to 6"/152 mm (for example a wafer) or smaller multiple specimens. The EMS 300T ES also comes with interchangeable sputtering and carbon evaporation inserts to allow a coating radius of up to 4"/102 mm.

KEY FEATURES

- **Metal evaporation, carbon evaporation and metal sputtering – in one space saving design**
- **Larger area metal evaporation – up to 6"/152 mm**
- **Larger area sputter/carbon coating – up to 4"/102 mm diameter**
- **High vacuum sputtering – oxidizing and non-oxidizing (noble) metals: suitable for SEM, high resolution FE-SEM and many thin film applications**
- **High vacuum carbon coater – ideal for SEM and TEM carbon coating**
- **Controlled, ramped carbon rod evaporation – precise control of carbon thickness. Non-sparking process gives superior quality films**
- **Up to 60 minutes sputtering time – thick films capabilities**
- **Three-Year Warranty**

PRODUCT DESCRIPTION

The EMS 300T ES is a large chamber, turbomolecular-pumped coating system ideally suited to metal evaporation onto large diameter specimens up to 6"/152 mm (for example a wafer) or smaller multiple specimens. The EMS 300T ES also comes with interchangeable sputtering and carbon evaporation inserts to allow a coating radius of up to 4"/102 mm.

The sputter coating insert will deposit both oxidizing metals, e.g. chromium and Aluminum and non-oxidizing (noble) metals such as gold and platinum. A chromium (Cr) target is fitted as standard.

The EMS 300T ES has a full range of optional accessories, including specimen stages and film thickness measurement which means the system can be tailored to the precise requirements of the user.

Thermal evaporation of metals

The EMS 300T ES allows controlled thermal evaporation of metals onto large substrates (up to 6"/152 mm). For many evaporative processes, tungsten filaments supplied with the system are used. However, some metals require the use of a molybdenum boat, which can also be used for heat-cleaning SEM and TEM apertures.

The evaporation head is normally positioned for downwards evaporation, but for small specimens upward evaporation is possible using two terminal extensions supplied with the system.

Thermal evaporation insert

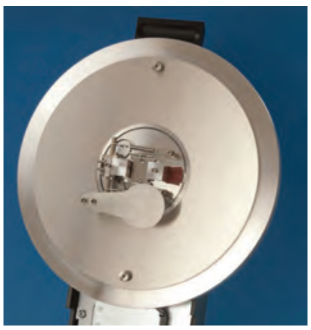
Sputter coating for high resolution FE-SEM and thin film applications

Sputtering insert

The advance design of sputtering head, power supply and system control allows sputtering of both oxidizing and non-oxidizing (noble) metals for thin film applications and for scanning electron microscopy (SEM) coating. The full range of target materials available is extensive and detailed in the Ordering Information section.

For sputtering applications where thick films are required, then the EMS 300T ES can operate for up to 60 minutes.

High vacuum carbon evaporation for SEM and TEM



Carbon rod evaporation insert

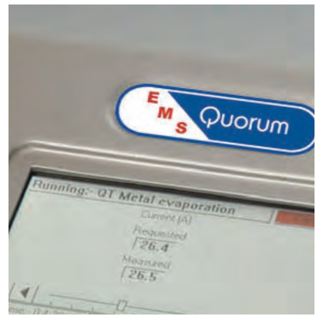
The carbon rod evaporation insert allows high quality carbon films to be deposited over a radius of up to 4"/102 mm.

The EMS 300T ES uses controlled ramped carbon rod evaporation to ensure optimum control of the process and quality of results (with or without the optional film thickness measurement system). In addition the quality of the resulting carbon films is enhanced by the eradication of "sparking" which is a common problem with less advanced coating systems.

High vacuum turbomolecular pumping and vacuum measurement

The EMS 300T ES is fitted with an internally mounted 70 L/s turbomolecular pump backed by a 5m3 hr two-stage rotary pump (order separately). A full range vacuum measurement gauge is included. Typically ultimate vacuums of around 5 x 10-5 mbar can be expected in a clean system after pre-pumping with dry nitrogen gas. For details of pumping options, see Ordering Information.

Touch-screen control and stored recipes



At the operational heart of the EMS 300T ES is a color touch screen which allows users to rapidly enter and store their own process data. A range of typical sputtering and evaporation profiles are pre-installed. The EMS 300T ES uses an 'Intelligent' recognition system that automatically detects the type of coating insert fitted and "becomes" either an evaporator, carbon coater or sputter coater.

Vacuum chamber and specimen stages

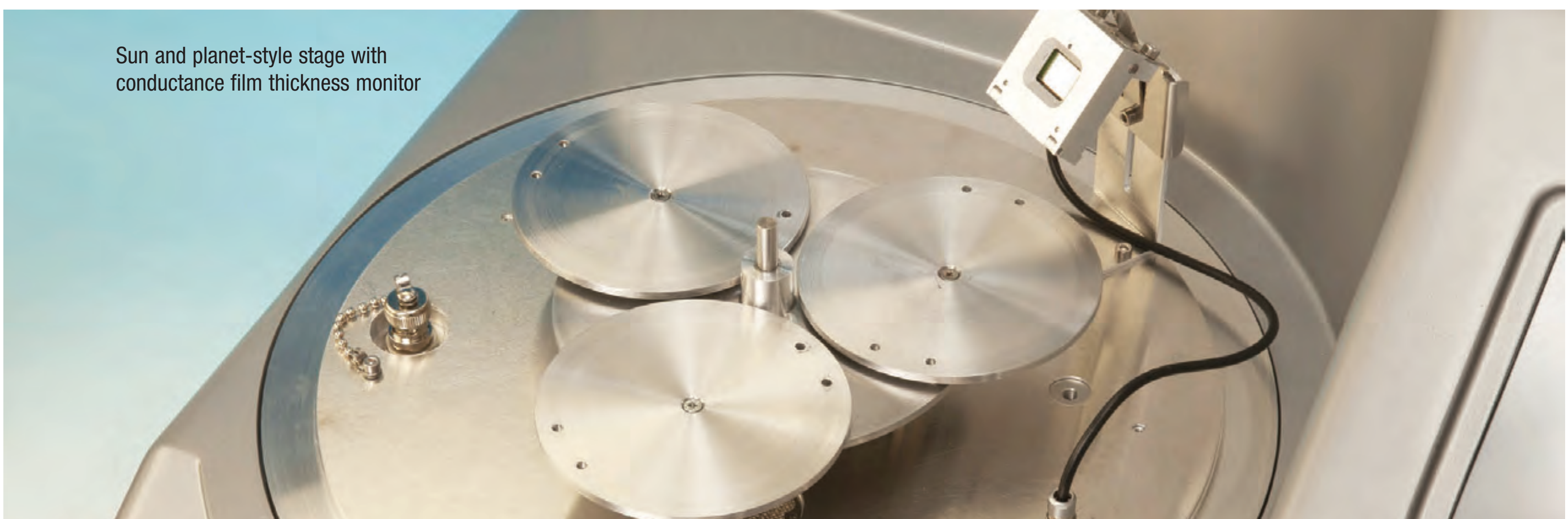
The EMS 300T ES is presented in a custom-molded, one-piece case allowing easy servicing access. The case houses all the working components and includes an automatic bleed control that ensures optimum vacuum conditions during sputtering.

The vacuum chamber has an internal diameter of 283 mm/11" and comes with an integral safety guard. The vacuum shutdown option enhances vacuum performance by allowing the chamber vacuum to be maintained when the coater is not in use.

A variable speed rotary specimen stage is fitted as standard and accommodates specimens up to 4"/100 mm in diameter. For details of other stages, see Ordering Information.

Pumping Requirement

A suitable rotary vacuum pump is required. The Pfeiffer DUO 6 5m3/hr two-stage rotary vacuum pump (91003) is ideal for this purpose. Dry pumping alternatives are also available. See Ordering Information for more details.



Sun and planet-style stage with conductance film thickness monitor

Terms and Techniques

Magnetron Sputtering

Magnetron sputtering using a crossed-field electromagnetic configuration keeps the ejected secondary electrons near the cathode (target) surface and in a closed path on the surface. This allows a dense plasma to be established near the sputter target surface. The ions that are accelerated from the plasma do not sustain energy loss by collision before they bombard the sputter target.

For electron microscopy (EM) specimen coating, the magnetron sputtering head design ensures that minimal heat energy (electrons) reach the specimen surface. This is important as it reduces heat damage to specimen and is a significant factor in ensuring the grain size within the sputtered film is optimally small essential for high resolution field emission scanning electron microscopy (FE-SEM).

Film Thickness Monitor (FTM)

A film thickness monitor can be used to monitor and control the thickness of sputtered and evaporated metal films. A gold-coated quartz crystal is mounted in the vacuum chamber of the coating system, ideally close to the specimen or substrate. The quartz crystal is made to oscillate at a defined frequency, using an externally-mounted oscillator. As metal is deposited on the quartz crystal, the frequency of oscillation alters and the change is converted to a digital (eg LED) display on the monitoring unit.

Film thickness monitors are available for use with most of our coating systems and cryo preparation systems.

Turbomolecular Pump

A turbomolecular (turbo) pump is a type of vacuum pump used to obtain and maintain high vacuum. The principle of operation is that gas molecules within a vacuum chamber can be given momentum in a desired direction by repeated collisions with a rapidly spinning turbine rotor. The rotor hits gas molecules from the inlet of the pump towards the exhaust in order to create or maintain a vacuum. A turbo pump normally works in tandem with a low-vacuum pump, such as a rotary vacuum pump, which is used to rough pump the vacuum system (eg sputter coater or vacuum evaporator) during initial pump-down period, and to back the turbo pump (ie remove gases from the back of the pump) during high-vacuum operation.

Iridium Sputter Coating

Sputtering with iridium (Ir) is increasingly popular for high resolution sputter coating of field emission scanning electron microscopy (FE-SEM) specimens, because iridium will produce films with very small grain structure and it is a non-oxidising metal. It is increasingly preferred to chromium (Cr) as a coating material for FE-SEM. Iridium-coated specimens can be stored at atmospheric pressure, compared to chromium - which readily oxidises on contact with air.

EMS300T ES *(continued)*

Options and Accessories

Specimen stages and holders

The EMS 300T ES has additional specimen stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except for the rotary planetary stage). Rotation speeds are variable between preset limits.

- Rotation stage for 4" wafers (supplied with system)
- Rotation stage for 6" wafers
- Flat rotation stage for SEM specimen stubs
- Rotation stage with preset tilt for SEM specimen stubs
- Rotate-tilt (rotary-planetary style) stage
- Rotation stage for glass microscope slides
- Eight-place stage for 25 mm or 30 mm embedded polished specimens
- “Sun and planets” style rotary stage; Three platforms, each 92 mm Ø
- Microscope coverslip stage for nine 20 mm x 20 mm coverslips
- TEM grid holder

Other options

- Extended height chamber (supplied with the EMS 300T ES)
- Standard height chamber
- Film thickness monitor (FTM)
- Conductance film monitor (CFM)

SPECIFICATIONS

Dimensions	585 mm W x 470 mm D x 410 mm H, total height with coating head open is 710 mm
Weight	37 kg
Packed dimensions	725 mm W x 660 mm D x 680 mm H (45 kg)
Work chamber	Borosilicate glass 283 mm ID x 215 mm H
User interface	Intuitive full graphical interface with touch-screen menus and buttons
Sputter target	Disc-style 57 mm Ø with thickness depending upon the target fitted. One 0.3 mm thick chromium (Cr) target (3417) is fitted as standard
Pumping	Internally-mounted 70 L/s turbomolecular pump
Rotary pump	5m^3 hr two-stage rotary pump with oil mist filter. (Order separately: see 91003). Dry pumping option available.
Typical ultimate vacuum	5 x 10^{-5} mbar in a clean system after pre-pumping with dry nitrogen gas. Measurement using a full range Penning gauge
Specimen stage	Stage for 4" wafer supplied as standard. For alternative stages, see Ordering Information
Services and other Information	
Gases	Argon sputtering process gas, 99.999%. Nitrogen venting gas (optional)
Electrical supply	90-250 V 50/60 Hz 1,400 VA including rotary pump, 110/240 V voltage selectable

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Fax (215) 412-8450 or 8452 • email: info@emsdiasum.com or stacie@ems-secure.com • www.emsdiasum.com

EMS300T ES *(continued)*

ORDERING INFORMATION

EMS 300T ES	Turbomolecular-pumped sputtering, metal evaporation and carbon coating system with 300 mm Ø x 215 H mm work chamber. Consists of high quick release sputter insert with a 3417 57 mm Ø x 0.3 mm chromium (Cr) target. High vacuum carbon rod evaporation coater and metal evaporation head. Coating inserts are interchangeable. A 4" wafer stage supplied as standard. Integrated full range gauge assembly for high vacuum measurement.	each

Rotary pump requirements – order separately

Pumps supplied with vacuum hose and coupling kit

91003	5m³ hr two-stage Pfeiffer Duo 6 rotary pump with oil mist filter	each
6550-A	Diaphragm pump. A "dry" alternative to the standard 91003 oil-based rotary pump	each
6548	Edwards nXDS6i scroll pump. Lubricant-free and hermetically sealed, giving totally clean and dry vacuum to prevent cross-contamination	each

Options and Accessories – specimen stages

Rotation stages rotation speed variable between preset limits

6551	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 15 mm, 10 mm, 6.5 mm or " pin stubs. Stage rotation speed is variable between preset limits. No rotation when in single target mode. Target to stage height is variable between zero and 42 mm for the standard stage. When used with the extended height cylinder (optional accessory 10596) the target to stage height would be an additional 87 mm	each
6552	50 mm Ø variable height specimen stage with six stub positions for 15 mm, 10 mm, 6.5 mm disc stubs or 1/8" pin stubs. Stage rotation speed variable between preset limits	each
6553	50 mm Ø rotary tilting stage. A rotary planetary style stage with variable tilt angle from horizontal to 30 degrees. The platform has six positions for either, 6.5 mm, 10 mm, 15 mm disc stubs, or " pin stubs. Rotation speed is variable between preset limits. Note: depending upon specimen height, this stage may require the optional extended height cylinder	each
6554	A 90 mm Ø specimen stage for glass microscope slides (up to two 75 mm x 25 mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pins stub. Stage rotation speed is variable between preset limits. Includes gear box to allow optional FTM to be used.	each
6547-SP	Rotation stage for 4" or 6" wafers	each
6513	Sun and planet-style rotation stage (each stage is 92 mm Ø)	each
6549	Eight-place stage for 25 or 30 mm embedded, polished specimens	each
6512	Nine-place TEM grid holder	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 >>>>

Other Options and Accessories

6555	Film thickness monitor (FTM) attachment. Including oscillator, feed-through, quartz crystal holder and one quartz crystal	each
6556	Spare quartz crystal for FTM	each
6555-SP	Conductance film monitor (CFM) attachment including housing, feedthrough, glass slides and cable. A factory fitted only option for monitoring sheet resistance of evaporated films allowing termination at a known resistance	each
6557	Extended height glass cylinder, 300 mm Ø x 215 mm high (supplied as standard)	each
6557-10	Standard height glass cylinder, 300 mm Ø x 127 mm high	each
6558	A lockable emergency stop (e-stop) switch mounted on top of the system for easy access	each
6559	Coating shields. Can be fitted to protect large surfaces in the chamber from coating deposition – easily removable for ease of cleaning	each
6560-A	Vacuum spigot allows a more convenient connection of the vacuum hose to the rear of the EMS 300T ES when bench depth is limited	each

Evaporation Supplies

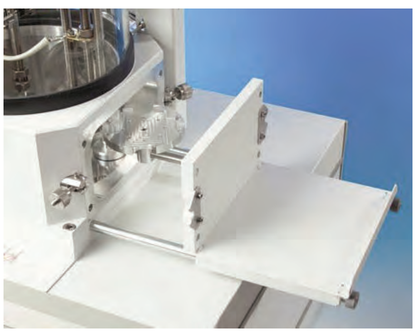
73830-SP	Tungsten wire baskets – pack of 10	each
73810-SP	Molybdenum boats – pack of 10	each

Sputter targets

The EMS 300T ES is fitted as standard with one 57 mm x 0.3 mm chromium (Cr) target. Other optional targets are available:

3410	57mm Ø x 0.1mm Gold	each
4524	57mm Ø x 0.2 mm Gold	each
3411	57mm Ø x 0.1mm Gold/Palladium (80/20)	each
4527	57mm Ø x 0.2mm Gold/palladium (80/20)	each
3412	57mm Ø x 0.1mm Platinum	each
4530	57mm Ø x 0.2mm Platinum	each
3413	57mm Ø x 0.1mm Nickel	each
3414	57mm Ø x 0.1mm Silver	each
3415	57mm Ø x 0.1mm Palladium	each
3416	57mm Ø x 0.1mm Copper	each
3538	57mm Ø x 1.0mm Aluminum	each
3421	57mm Ø x 1.5mm Carbon	each
3417	57mm Ø x 0.3mm Chromium	each
3418	57mm Ø x 0.5mm Tungsten	each
3419	57mm Ø x 1.5mm Chromium	each
3420	57mm Ø x 0.2mm Tungsten	each
3424	57mm Ø x 1.5mm Titanium	each
3425	57mm Ø x 0.3mm Platinum/Palladium (80/20)	each
3426	57mm Ø x 0.3mm Gold	each
3427	57mm Ø x 0.3mm Gold/Palladium (80/20)	each
3428	57mm Ø x 0.3mm Platinum	each
3429	57mm Ø x 0.5mm Titanium	each
3430	57mm Ø x 0.1mm Ironeach	each
3431	57mm Ø x 0.3mm Iridium	each
3432	57mm Ø x 0.1mm Cobalt	each
3433	57mm Ø x 0.1mm Tin	each
3434	57mm Ø x 0.1mm Molybdenum	each
3436	57mm Ø x 0.1mm Tantalum	each
3437	57mm Ø x 3mm Indium Tin Oxide (90/10)	each
3539	57mm Ø x 0.5mm Magnesium	each

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



EMS 975 specimen drawer with stage in specimen exchange position

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

OVERVIEW

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

The EMS 975 allows for carbon evaporation, metal evaporation from both baskets and crucibles and sputter coating option. A range of techniques can be practiced including carbon support films and replicas for TEM, carbon/metal evaporation, low angle shadowing and sequential layer coating using dual source evaporation and the sputter option can be used for a range of target materials.

The system flexibility is further enhanced by the use of a microcontroller, which readily allows the customer access to a range of options, but readily 'defaults' to optimum operating conditions, allowing both fully automatic and manual override as required. The unique loading rack out drawer system gives the user easy sample access with good sample size and the hinged lid assembly makes any other areas of the system readily accessible.

The unit has a turbo pump, externally mounted for convenience and easy exchange, and is backed up by a Rotary Vacuum Pump. The complete pumping sequence is under fully automatic control, achieving a high vacuum for evaporation.

The unit is bench mounted, with easy to use controls, and cannot be damaged by inadvertent use.

The EMS-975S is based on the EMS-975, however it has a special load lock door which allows the entry of 8" wafers with carbon coating, or other samples up to 140mm x 140mm square.

FEATURES

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

PRODUCT DESCRIPTION

Coating sources

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

Work chamber

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

Menu-driven control

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

Turbomolecular pumping and venting

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

EMS 975 *(Continued)*

control.

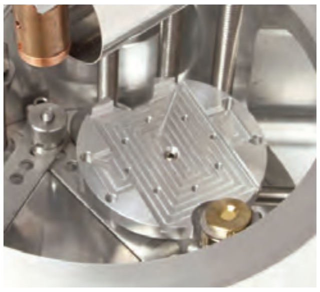
The vacuum pump-down sequence is automatically controlled by the system microprocessor. Vacuum measurement is by a combined pirani/penning gauge and is displayed digitally.

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

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

Specimen stages

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



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

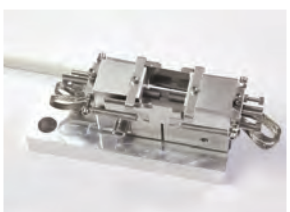
The rotary stage is mounted on a sliding access port on the side of the chamber. This allows the user to exchange specimens quickly without having to remove the glass chamber and disturb any coating set-up. The standard flat stage may be tilted. For rotary shadowing techniques, the standard stage can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

Chamber base plate and evaporation power supplies

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

EMS 975S Thermal Evaporator

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



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

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

SPECIFICATIONS

Dimensions and Weight	450mm (W) x 500mm (D) x 300mm (H). 65kg
Work Chamber	Borosilicate Glass 250mm (Dia.) x 300mm (H) with hinged top plate (can accommodate samples to 200mm) (8") in Dia.
Implosion Guard	Polycarbonate - readily removable for maintenance
Carbon Source	Adjustable height with tilt control of 0-200°. Uses 6.15mm Ø carbon rods (source for 3.05mm Ø carbon rods can be fitted - please specify at time of ordering)
Metal Source	Adjustable height with tilt control 0-200°. Supplied with pack of 10 B5230 tungsten specimen baskets
Specimen Stage	With tilt facility 0-45°
Vacuum Gauge Range	Atm to 1 x 10^{-7} mbar
Operating Vacuum	Towards 1 x10^{-5} mbar, typically achieved within 15 minutes
Low Voltage Evaporation Supply	Pulsed or variable control. Selectable: 0-5V-15V-25V, out-gas current: 0-25A
Services	Nitrogen gas (if used for venting). Argon gas (if the optional sputtering attachment is fitted)
Vacuum Pumping	100L/s turbomolecular pump. Requires a 50L/m 'backing' rotary pump with oil mist filter
Electrical Supply	230V/50Hz (8A max including pump), 115V/60Hz (16A max including pump)

Supplied with Accessory kit including: carbon rods (6.15mm x 100mm), evaporation filaments, manual rod shaper and operating manual

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

ORDERING INFORMATION

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

Replacement Source

91077	Carbon Rods (6.15mm Dia.)	each

Replacement Parts

91033	Glass Cylinder	each
91034	"L" Gaskets	each

Optional Accessories

91004-S	EMS 105 Aperture Cleaning Head	each
92060	EMS 190 Low Angle Shadowing	each
K-4175	Sputtering Module	each
410123	Manual Shutter Assembly	each

EMS 300T D Plus Dual Target Sequential Sputtering System

QUICK OVERVIEW

Suitable for multi-layer sequential sputtering of two materials, the EMS300T D Plus has two independent sputtering heads, which allows sequential sputtering of two metals without the need to break vacuum. The system is fully automated with user defined recipes controlling the pumping sequence, time, number of sputter cycles, and the current used during the process. Unlimited layers of varying thickness from two target materials can be sputtered sequentially by cycling between both targets. When not in use the targets are shuttered for protection from contamination.

KEY FEATURES

- **Capable of achieving vacuum of 5×10^{-5} mbar**
- **New touch and swipe capacitive screen**
- **USB port for upgrades and download of process log files**
- **Multiple-user profiles can be set up on one machine**
- **New software sorts recipes per user according to recent use**
- **16GB of memory can store more than 1000 recipes**
- **New multi-color LED visual status indicator**
- **Interchangeable stage options**
- **Three sputter heads for large area deposition of different materials**
- **Single head selection for small samples**

PRODUCT DESCRIPTION

Improved Interface

- Dual-core ARM processor for a fast, responsive display
- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Quick and easy creation of process sequences with a simple copy, drag and drop operation

Allows multiple users to input and store coating recipes. New feature to sort recipes per user according to recent use.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material.

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

Recommended applications for EMS300T D Plus:

- **Ideal for multi-layer coating**
- **Adhesion studies**

These products are for Research Use Only.

Detachable chamber with built-in implosion guard

Removable glass chamber and easily accessible base and top plate allows for an easy cleaning process. Users can rapidly change the chamber, if necessary, to avoid cross contamination of sensitive samples. Tall chamber option is available for improved uniformity for sputtering and to hold larger substrates.

Dual head sputtering – for sequential sputtering

The EMS 300T D Plus has two independent sputtering heads to allow sequential sputtering of two different metals without the need to 'break' vacuum, for example, a thin 'seeding' layer of chromium (Cr) followed by deposition of gold (Au). An automatic shutter mechanism enables cleaning of oxidizing sputter targets and protects the second target and substrate during coatings. For single metal applications one target can be selected.

Multiple stage options

The EMS 300T D Plus has substrate stages to meet most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except the rotary planetary stage). A swinging arm stage drive is supplied as standard, which is a stage drive and positioning mechanism that positions the stage under the correct target. Rotation speed is variable between 14-38 rpm.

In addition a flat, adjustable stage capable of accepting 4" (101.6 mm) wafers is supplied as standard with the EMS 300T D Plus.

As an accessory, a 6" wafer stage is available, which is a flat adjustable stage capable of accepting 6" or 150 mm wafers. The stage includes two masks for improving uniformity of coating.

continued >>>>

III EMS 300T D Plus *(continued)*

Rotation stage – 50 mm Ø. This stage only rotates and has no tilt or height adjustment.

Rotate-tilt stage – 50 mm Ø. With height adjustment (target to stage height variable between 30-80 mm). The tilt angle can be pre-set (horizontal to 30°).

Rotation stage for glass slides – 25 mm x 76 mm

Safety

The EMS 300T D Plus meets key industry CE standards

- All electronic components are protected by covers
- Implosion guard prevents user injury in event of chamber failure
- Vacuum interlocks remove power from deposition sources to prevent user exposure to high voltage in event of chamber being opened
- Overheating protection shuts down power supply

Vacuum control

High vacuum turbo pumping allows sputtering of a wide range of oxidizing and non-oxidizing metals for thin film and electron microscopy applications. Automatic vacuum control which can be pre-programmed to suit the process and material, therefore removing the need for manual intervention or control.

Cool magnetron sputtering

Sputter coating is a technique widely used in various applications; it is possible to create a plasma and sputter metals with high voltage, poor vacuum and no automation. However, this is not suitable for some applications because it can heat the substrate and result in damage when the plasma interacts with the substrate. The EMS 300T D Plus uses low temperature enhanced-plasma magnetrons optimized for the turbomolecular pump pressures, combined with low current and deposition control, which ensures your substrate is protected and uniformly coated.

The EMS 300T D Plus uses easy-change, 57 mm diameter, disc-style targets which are designed to sputter oxidizing and noble metals. It is fitted with gold (Au) and chromium (Cr) sputter targets as standard.

continued »»»

Dual sputtering heads and automatic shutter

Rotary planetary stage and dual-channel film thickness monitor - both Q300T D Plus option

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

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

New user interface has been thoroughly updated:

- Dual-core ARM processor for a fast, responsive display
- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes
- Quick and easy creation of process sequences with a simple copy, drag and drop operation

Allows multiple users to input and store coating recipes.

New feature to sort recipes per user according to recent use.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

EMS300T D Plus *(continued)*

SPECIFICATIONS

Instrument Case	590mm W x 535mm D x 420mm H (maximum height during the opening of the coating head: 772mm)
Weight	36 kg (packed: 59kg)
Packed Dimensions	730 mm W x 630 mm D x 690 mm H
Work Chamber	Borosilicate glass with integral PET implosion guard Size 300 mm outside diameter x 127 mm High
Display	115.5mm W x 86.4mm H (active area), 640 RGB x 480 (display format), capacitive touch color display
User Interface	Full graphical interface with touch screen buttons, includes features such as a log of the last 1000 coatings and reminders for when maintenance is due

Specimen Stage

A flat adjustable stage capable of accepting either 4" or 6" wafers is mounted on a swinging arm stage, which rotates the stage under the targets to optimise coating. Rotation speed is variable from 14rpm to 38rpm

Vacuum

Rotary Pump	50L/min two stage rotary pump with oil mist filter
Turbo Pump	Internally mounted 70L/sec air cooled
Vacuum Measurement	Pirani gauge as standard, full range gauge available as an option
Ultimate Vacuum	5×10^{-5}mbar*
Sputter Vacuum Range	5×10^{-3} to 5×10^{-2} mbar*

*Typical ultimate vacuum of the pumping system in a clean instrument after pre-pumping and venting with dry nitrogen gas

Processes

Sputter Deposition Current	150 mA
Visual Status Indicator	A large status multi-color indicator light provides a visual indication of the state of the equipment, allowing users to easily identify the status of a process at distance.

The indicator LED shows the following states:
• Initialization • Process running • Idle • Coating in progress
• Process completed • Process ended in fault condition
Audio indication also sounds on completion of the process.

Services

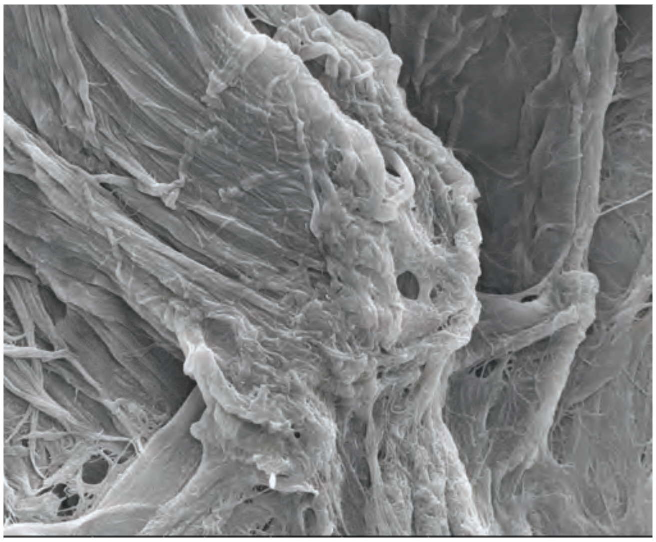
Gases	process gas argon, 99.999% Nominal 5psi
Vent Gas	Nitrogen (optional). Nominal 5psi

Pulsed cleaning for Aluminum sputtering

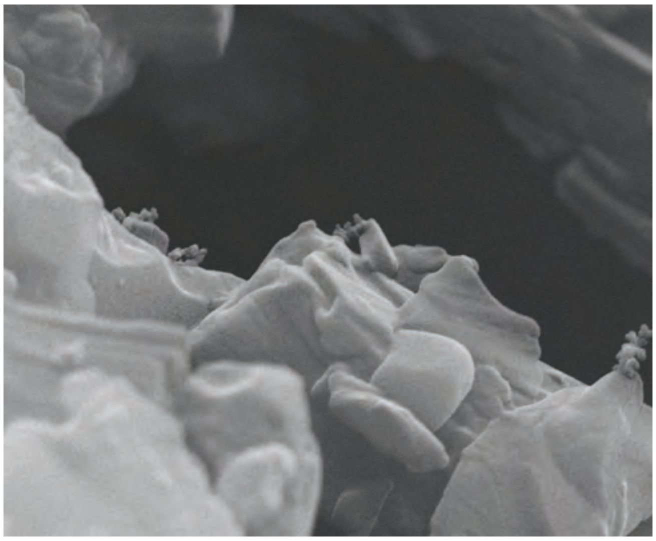
Aluminum (Al) rapidly forms an oxide layer which can be difficult to remove. The EMS 300T D Plus has a special recipe for Aluminum that reduces the oxide removal time and prevents excessive pre-sputtering of the target.

Film thickness monitor

The EMS 300T D Plus can be fitted with an optional dual film thickness monitor (FTM), which measures the coating thickness on two quartz crystal monitors located within the chamber. The thickness measured on the monitor can be correlated to the thickness on the substrate using a mathematical formula built into the software; this allows the user to control the thickness of material deposited on to the substrate. For example, the EMS 300T D Plus can automatically terminate a coating profile when the required thickness has been achieved. Alternatively, the process can be terminated by time.



10nm Au on Filter Paper



10nm Au on Salbutamol

EMS 300T D Plus *(continued)*

ORDERING INFORMATION

EMS300T D Plus	Dual Target Sequential Sputtering System; includes a 57 mm Ø x 0.3 mm chromium (Cr) target and a 57 mm Ø x 0.1 mm thick gold (Au) target . A flat rotation stage for 4"/100 mm wafers is included.	each

Rotary pump requirements (needs to be ordered separately)

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

Specimen stages

6790-S	Swinging arm stage drive, a stage drive and positioning mechanism which positions the stage under the correct target. Also provides rotation drive to the stage. Rotation Speed Max 38 rpm Min 14 rpm	each
6800-S	Rotating specimen stage for 6" (152 mm) wafers, with rotation variable between preset limits.	each
6801	Rotating 50 mm Ø specimen stage with adjustable tilt. The platform has six specimen stub positions for 15 mm, 10 mm, 6.5 mm or 1/8" pin stubs. The stage rotation speed is variable between preset limits. The target to stage height is variable between 0 mm and 42 mm for the standard stage. When used with the extended height cylinder the target to stage height would be an additional 87 mm.	each
6802	50 mm Ø variable height specimen stage with six stub positions for 15 mm, 10 mm, 6.5 mm disc stubs or 1/8" pin stubs. Stage rotation speed variable between preset limits. Note: Target to stage height is variable between 10 mm and 53 mm for the standard stage. The stage is supplied with two mounting pillars; one provides 10 mm to 32 mm target to stage distance and the other 31 mm to 53 mm. An adjustable stop is used to set the height. When used with the extended height cylinder (optional accessory) the target to stage height would be an additional 87 mm.	each
6803	50 mm Ø rotary tilting stage. A rotary planetary style with a variable tilt angle from horizontal to 30 degrees. The platform has six positions for either 6.5 mm, 10 mm and 15 mm disc stubs or 1/8" pin stubs. Rotation speed is variable between preset limits. Note: depending upon specimen height, this stage may require the optional extended height cylinder.	each
6804	A 90 mm Ø specimen stage for glass microscope slides (up to two 75 mm x 25 mm slides or a single 75 mm x 50 mm slide). The stage can alternatively accommodate up to six 1/8" SEM pin stubs. The stage rotation speed is variable between preset limits. A gear box is included to allow the optional FTM to be used.	each

For our complete selection of specimen stages, please see the **Specimen Stage Selection Guide** on page 84 »»

Options and Accessories

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

Sputter targets

Note: The EMS 300T D Plus is fitted with a 57 mm diameter 0.3 mm thick chromium (Cr) target and a 57 mm diameter 0.1 mm thick gold (Au) target as standard. Other optional targets are available:

3409	57mm Ø x 0.76mm Aluminum	each
3410	57mm Ø x 0.1mm Gold	each
3411	57mm Ø x 0.1mm Gold/Palladium (80/20)	each
3411-3	57mm Ø x 0.3mm Gold/Palladium (80/20)	each
3412	57mm Ø x 0.1mm Platinum	each
3413	57mm Ø x 0.1mm Nickel	each
3414	57mm Ø x 0.1mm Silver	each
3415	57mm Ø x 0.1mm Palladium	each
3416	57mm Ø x 0.1mm Copper	each
3417	57mm Ø x 0.3mm Chromium	each
3418	57mm Ø x 0.5mm Tungsten	each
3419	57mm Ø x 1.5mm Chromium	each
3420	57mm Ø x 0.2mm Tungsten	each
3421	54mm Ø x 1.5mm Carbon	each
3422	57mm Ø x 0.1mm Aluminium	each
3423	57mm Ø x 0.1mm Platinum/Palladium (80/20)	each
3424	57mm Ø x 1.5mm Titanium	each
3425	57mm Ø x 0.3mm Platinum/Palladium (80/20)	each
3426	57mm Ø x 0.3mm Gold	each
3427	57mm Ø x 0.3mm Gold/Palladium (80/20)	each
3428	57mm Ø x 0.3mm Platinum	each
3429	57mm Ø x 0.5mm Titanium	each
3430	57mm Ø x 0.1mm Ironeach	each
3431-2	57mm Ø x 0.2mm Iridium	each
3431	57mm Ø x 0.3mm Iridium	each
3432	57mm Ø x 0.1mm Cobalt	each
3433	57mm Ø x 0.1mm Tin	each
3434	57mm Ø x 0.1mm Molybdenum	each
3435	57mm Ø x 0.3mm Magnesium	each
3436	57mm Ø x 0.1mm Tantalum	each
3437	57mm Ø x 3mm Indium Tin Oxide (90/10)	each

Techniques and Applications

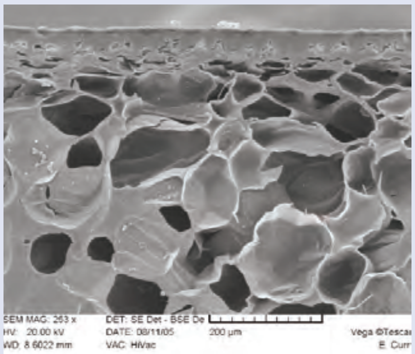
What is...
Freeze Drying?

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

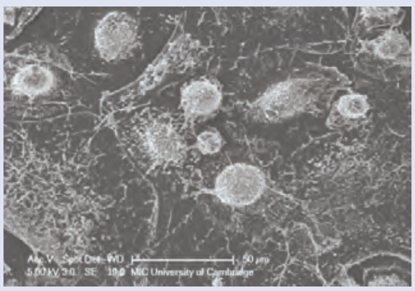
Its application is to reduce the distortion which occurs when a wet specimen dries by normal evaporation. This distortion is due to the forces of surface tension going from a liquid to a vapor phase such as water to water vapor, commonly the situation in a Biological Specimen. However, if we freeze the 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.

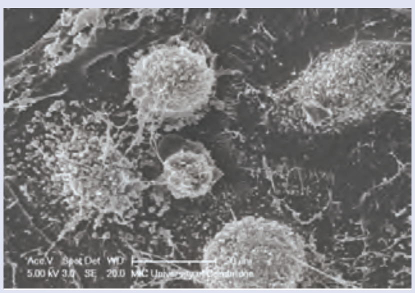
For particular applications, however, it can be necessary to freeze dry at temperatures below -80°C with lower sublimation rates for delicate specimens. This requires a better vacuum than can be obtained using a rotary vacuum pump.

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.



Freeze Dried Apple (Images courtesy of Dr. Eric Curry, USDA, ARS, Tree Fruit Research Laboratory, Wenatchee, WA US)





Human monocyte derived macrophages, freeze dried and coated with carbon. (Images courtesy of Dr. Jeremy Skepper of the Multi-Imaging Centre, University of Cambridge)

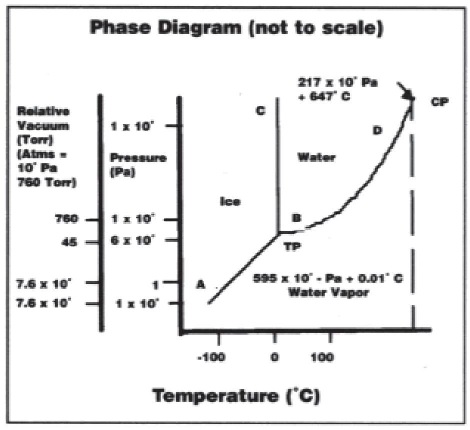
Freeze Drying Principles

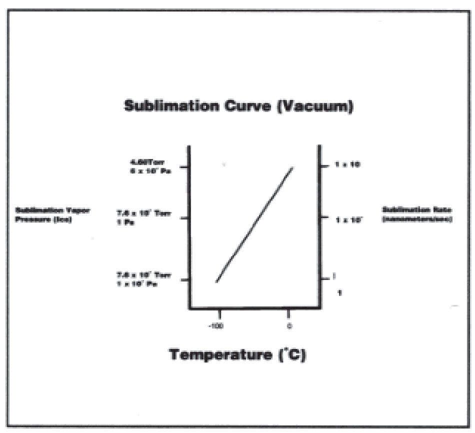
Sublimation consideration

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.





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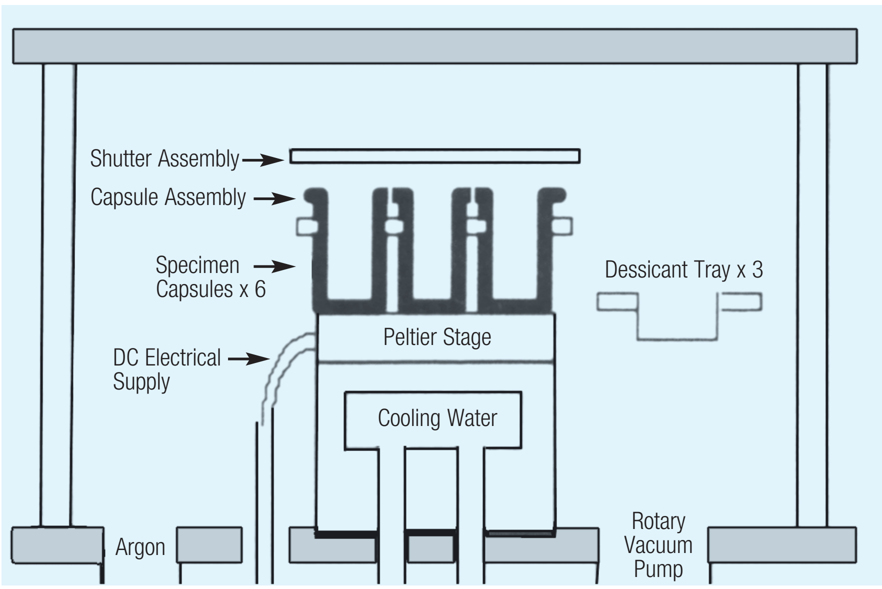
Techniques and Applications

In practice therefore, the system pressure (relative vacuum) used, is much lower than the Triple Point pressure. The Fig. gives an indication of the sublimation pressure (Saturated Vapor pressure at differing temperatures, and satisfactory freeze drying will be determined by this, and the partial pressure (Unsaturated Vapor pressure) of the water vapor in the vacuum system, which is required to be lower. The right hand scale gives an indication of Sublimation rates, (assuming partial pressure of H_20 is not limiting) which are totally temperature dependent.

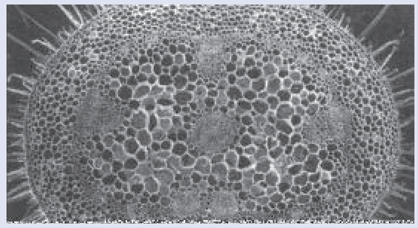
Freezing Considerations

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

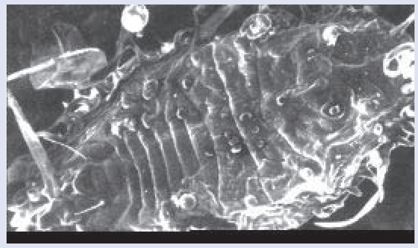
A range of cryogens and freezing techniques are being applied to the freezing of specimens, the understanding of the mechanisms of which, are still at an early stage. However, with exceptions, (Hyperbaric freezing containment of ice crystals is generally less than 20 micrometers from the surface, and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



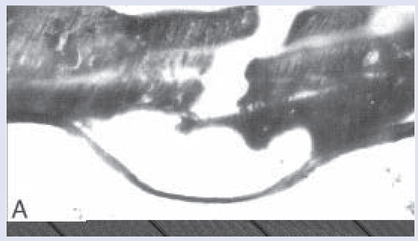
Cross-sectional View of Peltier Cooled Stage



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

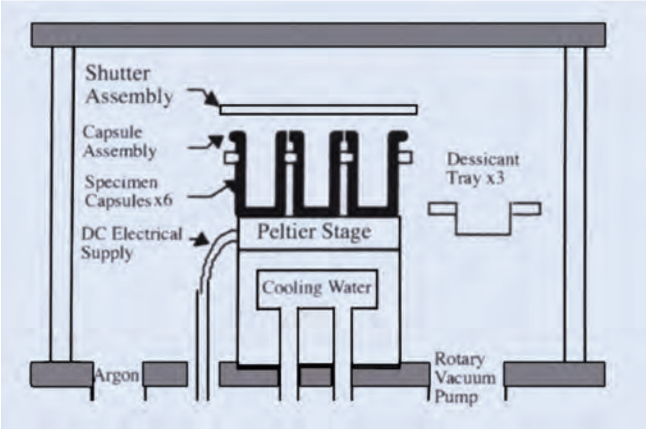
EMS 750 Freeze Dryer

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.



Cross-sectional View of Peltier Cooled Stage

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

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

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 **Plus** **Stainless Steel Base**	Borosilicate Glass 165mm(Dia.) x 125mm(H) 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

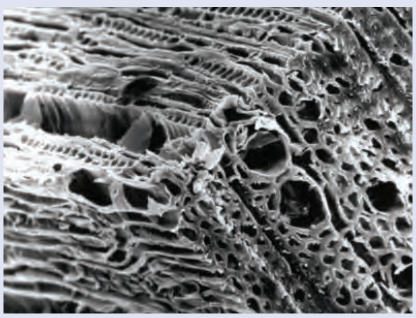
Optional Accessories

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

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.



Mature Spruce Wood

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

Techniques and Applications

A summary of the critical point drying method

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

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

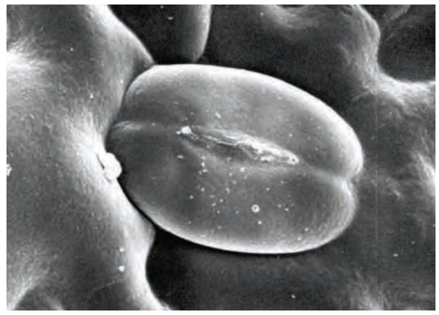
All three models have found general acceptance in many laboratories throughout the world. Together, these critical point dryers offer the user a choice most suited to the particular specimen preparation requirements.

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

Critical point drying relies on this physical principle. The water in biological tissue is replaced with a suitable inert fluid whose critical temperature for a realizable pressure is just above ambient. The choice of fluids is severely limited and CO_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 vapor 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.



Stomatal Pore on Xerophyte Leaf Surface

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

Barley Leaf

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

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

Techniques and Applications

Critical Point Drying Principles

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

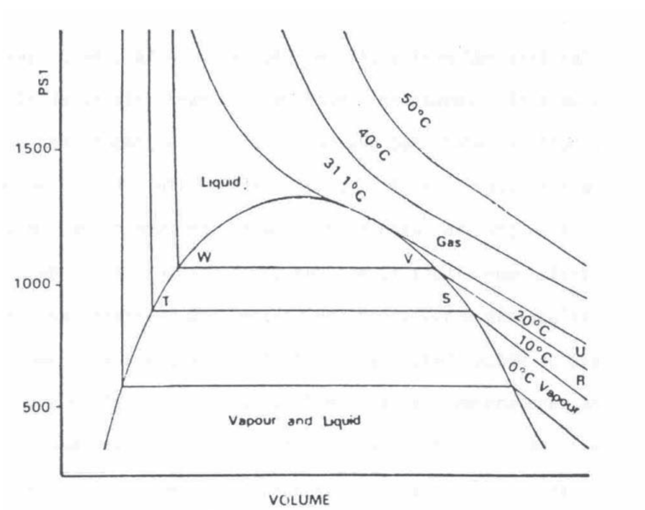
If a specimen had been in the liquid during this process it would have experienced a transition from a ‘wet’ to a 'dry' gas environment without being in contact with a surface, in this way avoiding the damaging effects of surface tension.

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

The Critical Phenomena

The principle of the experiments, which were initially carried out using carbon dioxide (CO_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.

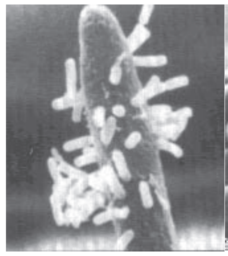
The results are best understood by considering the graph obtained from plotting pressure (P) against volume (V) for the series. This is shown in Figure 1; the curves obtained are termed ‘isothermals’



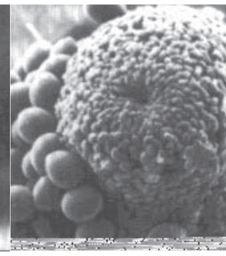
Consider first the 10° C isothermal at low applied pressure. The CO_2 is gaseous (vapor) and generally exhibits the characteristics of a gas (Boyle’s Law) over the range from 'r' to 's'. From point 's' a very slight increase in pressure results in a change from vapor state to the liquid state. This is 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.



Fruit body neck with spores adhering to the sides



Bacteria adhering to the tip of a fungus.

Powdery Mildew, hyphal filament on leaf surface.

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

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

However, it is not surprising that the initial investigations were on CO_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

continues

Techniques and Applications

Critical Point Drying Principles (continued)

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 vapor fixation) prior to the transitional stage is only mentioned in its most basic terms, procedures vary according to the type and nature of the specimens. Further references should be obtained.

a) Intermediate Stage

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

(Wet Specimen) H_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.)

Critical point drying stages

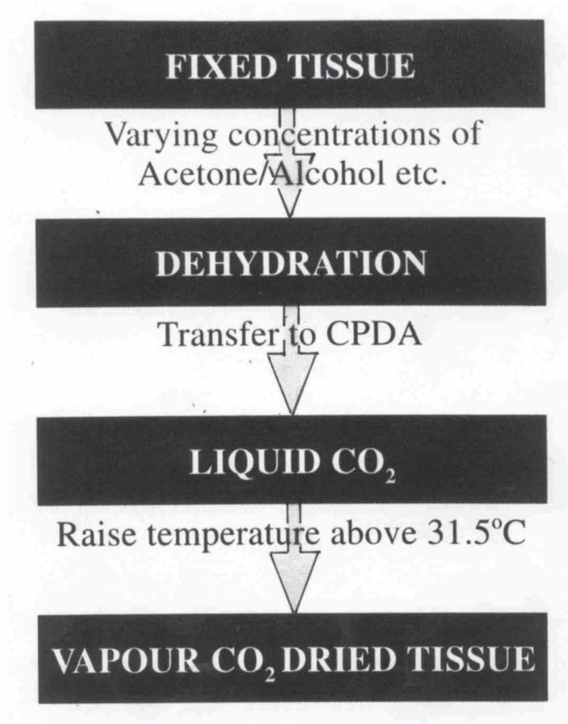


Figure 3: Dehydration Intermediate Fluids for CPD

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

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

CO_2 grades required for critical point drying

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

In most parts of the world, 'normal' grade of CO_2, from the suppliers, is specified as 'N4.5' or 99.995% minimum purity with a maximum level of 50ppm of impurities.

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

We are not aware of anyone who has ever reported either superior results using N5.5 purity vs N4.5, or inferior results using N4.5 vs N5.5. However, we do want our customers to have the benefit of such detail about liquid carbon CO_2 procurement in the event they should ever find reason to believe that their particular specimens might benefit from the higher purity product.

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

Techniques and Applications

Critical Point Drying Principles (continued)

(b) Transitional Stage

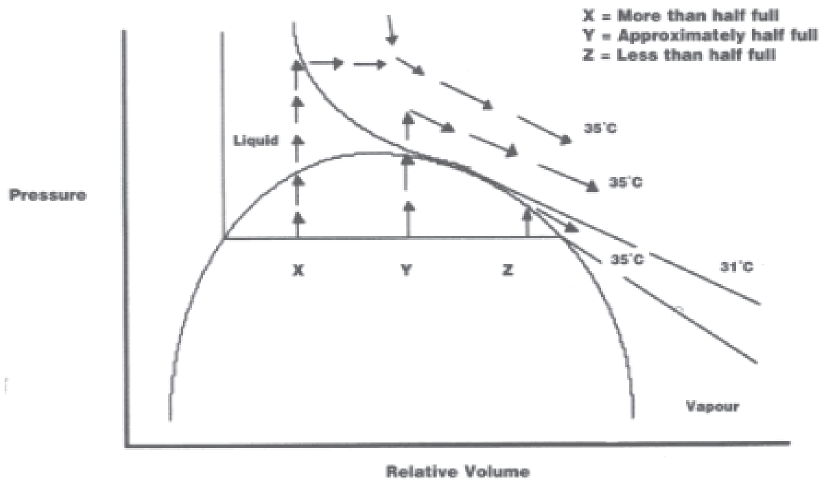
As discussed previously (see Figure 1) the conditions for which the critical point passage can be obtained for CO_2 are 31.1°C and 1072 psi. However, it must be remembered that these isothermals are obtained from a fixed mass of gas and an applied pressure for a series of constant temperatures.

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.)

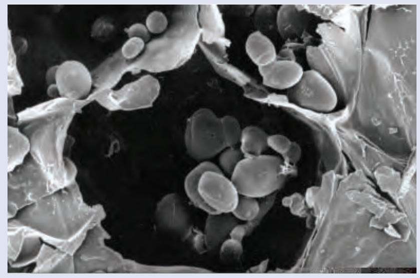
If we consider 'X' with the liquid CO_2 more than half filling the total available volume and we heat from 10°C to 35°C then we will make the transition from liquid to gas. The pressure rise will be rapid as the liquid will expand and the level will increase before the critical temperature is reached.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

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

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

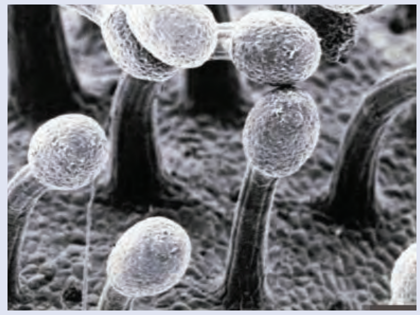
Ideally, we wish have a situation where the liquid fills the specimen chamber, while still only accounting for approximately 50% of the total volume available. This will ensure that specimens are not uncovered during initial flushing stages and in addition this should enable critical constants of temperature,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

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



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 3100 Critical Point Dryer

QUICK OVERVIEW

The E3100 large chamber critical point dryer has been an industry standard for over 35 years and is used in numerous scanning electron microscopy (SEM) laboratories around the world. Primarily used for critical drying of biological and geological specimens, the E3100 can also used for the controlled drying MEMs, aerogels and hydrogels.

The design of the E3100 features a large, horizontal pressure chamber measuring 63.5 mm internal diameter x 82 mm 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 25 mm diameter window which gives an unsurpassed view of the liquid level during the critical point drying process.

KEY 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 CO2 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 critical point drying process runs and/or in laboratories where the room temperature may be high.

The temperature of the E3100 chamber is raised with a hot water supply. Mains water can be used but a more elegant method involves the use of the optional E4860 Recirculating Heater/Chiller which can be used to pre-cool the chamber to below ambient temperature prior to loading specimens and then to heat the chamber to the critical temperature.

Safety

Operator safety is of course an important consideration with all pressure vessels. If during the critical point drying process the pressure and temperature are inadvertently exceeded, a safety bursting disc is incorporated in the chamber support. The design has been independently type-tested to proof pressures in excess of the working pressure and bursting disc rupture pressure.

Specimen holder (boat)

An important feature is the design of the E3100 transfer boat. This permits specimens in the intermediate fluid to be transferred to the chamber of the critical point dryer. On sealing the chamber, the intermediate fluid begins to drain and can be replaced with liquid CO2. In this way the specimens are never allowed to dry out during the loading and transfer stage of the critical point drying process.

EMS 3100 Specimen Boat

The E3100 is supplied with the E3100-01 tissue boat which has three slots each with three tissue baskets, making a total of nine. Optional holders are available and are listed under Ordering Information.

Bonded chamber seals – Nitrile or EPDM?

The EMS 3100 is fitted with a standard nitrile bonded window and door seals. Nitrile is a good general material due to its 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 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.

Electron Microscopy Sciences In PA: (215) 412-8400 • Toll-Free (800) 523-5874
Fax (215) 412-8450 or 8452 • email: info@emsdiasum.com or stacie@ems-secure.com • www.emsdiasum.com

EMS 3100 Critical Point Dryer *(continued)*

OPTIONS AND ACCESSORIES

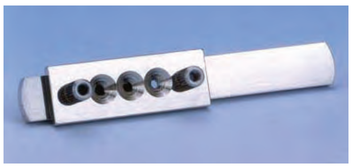
Glass microscope coverslip holder (option):

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

Service Kits:

Service kits are available; see the ordering table below. Please call our service department for specific questions.

Service Kit

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 4860 Recirculating Heater/Chiller to control heating and cooling cycle (please specify voltage)**

For more information on the EMS 4860 Recirculating Heater/Chiller, see page 4.

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 4860 Recirculating Heater/Chiller are not used)**
- **Mains power supply (for EMS 4860 only)**
- **Fume cupboard or window, or an area of good ventilation**
- **Space for CO_2 siphon cylinder**

CO_2 Cylinder: The EMS 3100 requires 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.

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 3100 requires 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 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 3100	Large Chamber Critical Point Dryer Chamber dimensions: 63.5mm Ø x 82mm length	each
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	
E3100-061	Service Kit for EMS 3100	Kit

EMS 850 Critical Point Dryer with Thermoelectronic Heating and Adiabatic Cooling

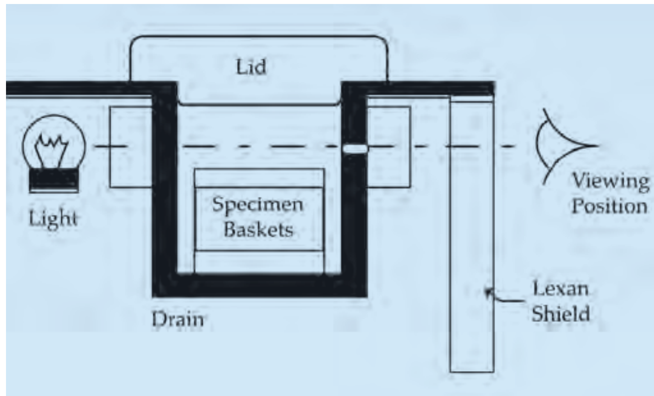
The EMS 850 Critical Point Dryer is designed for use with CO_2, having first replaced any water in the specimen by a series of dehydration, often in the same fluid such as Acetone, which will also be the intermediate fluid.

(Wet Specimen)–Water–Acetone–30%–100%–CO_2–C.P.D.–(Dry Specimen)

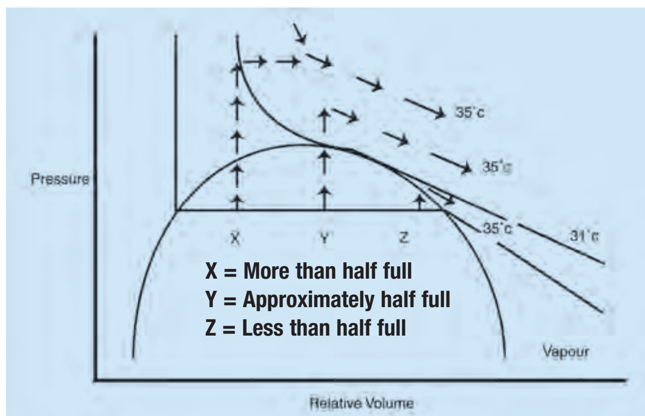
The specimens for critical point drying are located in the pressure chamber of the EMS 850. The chamber is pre-cooled to allow it to be readily filled with liquid CO_2 from a gas cylinder. The chamber is then heated to just above the critical temperature with subsequent critical pressure being achieved. 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.



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



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.)

ORDERING INFORMATION

91090	EMS 850 Critical Point Dryer complete	each

Replacement Parts

91091	Mesh Basket	each
91092	Porous Spec Pots	10/pack)

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

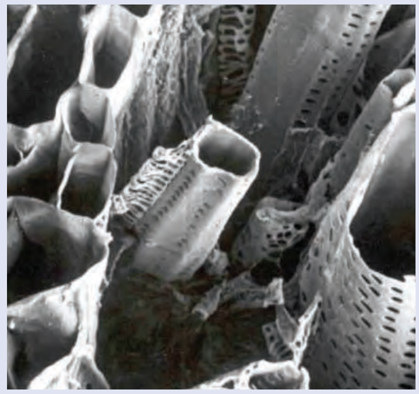
100mm or 150mm diameter wafers are held in a PTFE holding tray. The tray including wafer is immersed in acetone in order to remove all moisture from the specimen. After dehydration, the wafer and holder are transferred into the pre-cooled specimen chamber using the wafer transfer device. On completion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

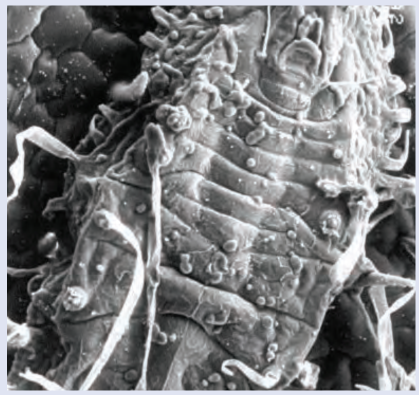
Requirements

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



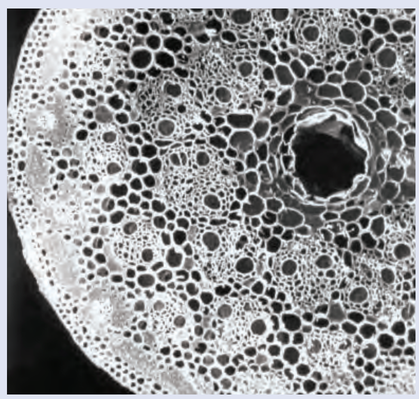
Mature Barley Root

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



Aphid

Critical point dried aphid on a leaf surface.



Bamboo Stem

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

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 + 0_2 -> 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 + CF_4 -> e + CF_3 + F (Dissociation) or e + CF_4 -> 2e + CF_3+ + F (Dissociation)

Summary

Exposing a gas to the RF potential at reduced pressure creates a plasma which contains active species - for example, in the case of oxygen, atomic oxygen. Oxygen atoms will oxidize organic molecules more readily than oxygen molecules. So typically a cellulose material can be converted to carbon dioxide, carbon monoxide and water at room temperature, rather than at elevated temperatures (eg burning) and furthermore the oxidation is more controllable.

Types of Reactor Systems

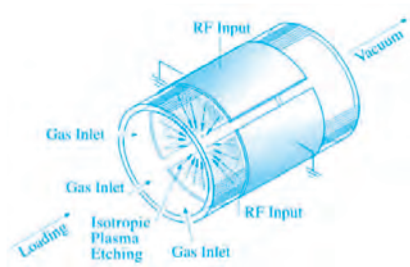
There are many types of reactors available. They are all glow discharge systems but vary considerably in terms of excitation frequency (5kHz - 5GHz), operating pressure (1mbar - atmospheric pressure) and electrode arrangement.

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

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

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

Barrel Reactor



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.

Frequently Asked Questions...

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

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

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

How automated is the EMS 1050?
After initial set up of the operating conditions operation is fully automatic (one-button operation).

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

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

Techniques and Applications

Plasma Applications (continued)

Plasma polymerisation

Plasma polymerization refers to the polymerisation of active species generated in a plasma. For example, the introduction of polysiloxanes onto hard contact lenses improves the hydrophilic nature of the surface. An application in the soft drinks industry using CF_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.

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

Techniques and Applications

Plasma Chemistry

Photo resist removal

The removal of photo resist by plasma etching from silicon or GaAs semiconductor slices is a very safe and effective process. The thickness of resist is normally 1-3 microns and has usually been baked on for about 1 hour at 100°C. Using O2 at full power, 1 micron should disappear in about 20-30 minutes. Ion implanted resist is very hard and in some cases will take longer to etch, up to two hours. One way of reducing time is by fitting a larger rotary pump, therefore allowing more gas flow for the same pressure. Normal operating pressure is between 0.4-0.2mbar.

Silicon etching

To etch silicon and silicon nitride from slices, a fluorinated gas is required, usually CF_4. Films are normally in the order of 100-500 microns and are quickly removed; 2-5 minutes normally being sufficient. Visual colour changes make complete removal obvious. CF_4 will also attack the chamber and window. Quartz, which does not contain boron, is offered as standard instead of glass to avoid boron contamination.

Asbestos detection

Plasma is now accepted as the standard process for SEM examination of asbestos fibers caught in acetate filter papers. Filters are surface etched in O_2 at approximately 100W for 2-8 minutes, this being sufficient to etch away the filter surface, leaving the asbestos fibres for SEM examination.

Atomic absorption spectroscopy preparation

Plasma is widely used in AAS as a convenient way of removing the organic binder from a host of specimens making trace metal determination easier. Specimens are presented dry and crushed and etched in O_2. Ashing times vary but are usually 1-3 hours.

General cleaning

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

Plastic treatment

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

These reactions take place in high purity O_2 at relatively low temperatures and power input (10-40W). Process times are relatively short; 15 seconds – 2 minutes usually being sufficient. Interest has been shown in putting down 'barrier layers' on plastic containers to increase shelf life by stopping the ingress of CO_2 and stopping the contents of the container reacting with the plastic. The films are normally of inert fluorine and are produced using CF_2.

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

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

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

EXAMPLES OF PLASMA GASES AND THEIR APPLICATIONS

O_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

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

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

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.

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.

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.

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.

References:

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

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

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

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

Detection of inorganic materials in biological specimens
Source unknown

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

Use of chemically reactive gas plasma in preparing specimens for SEM and Electron Probe Microanalysis
Richard S Thomas. SE.M/1974 part I proc 7th SEM Symph –April/1974

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

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

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

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

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

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

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

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

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.

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.

nfrared 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.

IAAS 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.

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.

References:

Glow discharge surface treatment for improved cellular adhesion
L Smith, Polymer Chem Div, 170th ACS meeting, Chicago 25.8.75.

Quantitative determination of Silicon in Organo Silicon compounds using low temperature RF-discharge Oxygen plasma
M Velodina, Analytical Letters 10 (14), 1189-1194 (1977).

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

Polarography and voltammetry in environmental research and surveillance of toxic metals
H W Nurnberg. International Symposium on Industrial Electro-Chemistry, Madras December 1976.

Trace chemistry of toxic metals in biometrics
P Valenta, Z Anal Chem. Band 285 (1977)

Studies on ecotoxicological base lines and speciation of heavy metals in natural waters and rain
H W Nurnberg, Institute of Chemistry, Nuclear Research Centre (KFA), PO Box 1913, D-5170, Julich, F.R.G.

Electronic low-temperature ashing of bituminous coal
H J Gluskoter, Illinois State Geological Survey, Urbana, Ill USA. March 1965.

Quantitative determination of the mineral matter content of coal by a radio frequency-oxidation technique
Frank W Frazer. Fuel 1973.

Quantitative infrared multicomponent determination of minerals occurring in coal
P A Estep, Anal. Chem. Vol 34. No 11. Oct 1968 Pp1454-1457

Use of electrically excited Oxygen for the low temperature decomposition of organic substrate
C F Gleit. Anal. Chem Vol 34. No 11. Oct 1968 pp 1454-1457

Low temperature Oxygen-Fluorine RF ashing of biological materials in PTFE dishes prior to determination of Tin, Iron, Lead and Chromium by AAS
E V Williams. Analyst. Sept 1982. Vol 107. pp1006-1013

Cadmium in blood analysis
Francois Claeys, Institute d'Hygiene and d'Epidemoloqie Bruxelles.

Recovery of tracers from ashed blood
Dr Piduttu, Catholic University, Rome, Dept of Industrial Hygiene.

Determination of Cadmium in blood after destruction of organic material by low-temperature ashing
G Carter and W Yeoman. Analyst. Vol 105. March 1980 Pp 295- 297

EMS 1050 Plasma Asher

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

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

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

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

FEATURES

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

Product Description

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

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

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

Chamber, specimen handling and gas control

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

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

"Rack Out" Specimen Stage

Application Example

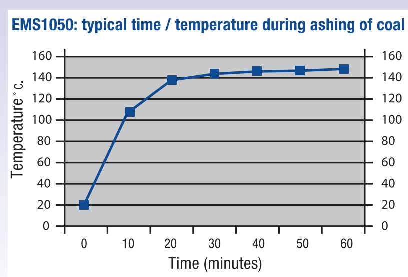
Low temperature plasma ashing of coal...

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

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

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

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



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Power, tuning and vacuum monitoring

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

Automated microprocessor control

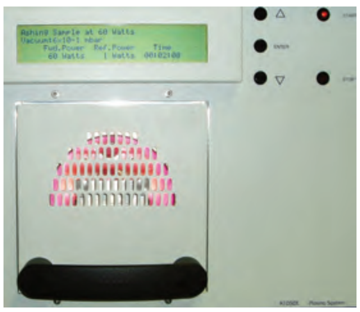
The EMS 1050 is fully automatic. Control parameters for time, power and vacuum are easy to preset and can be monitored and adjusted throughout the process run.

'Autotuning' of RF power for optimum control and reproducibility

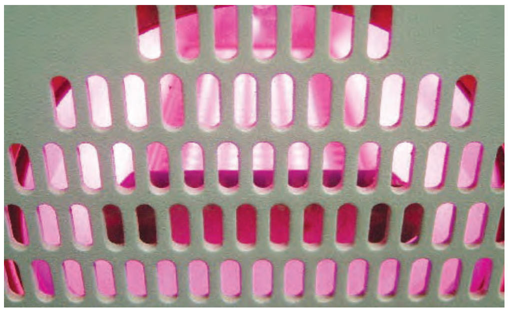
During the plasma process the 'autotune' facility ensures that the RF power is automatically impedance-matched to any variation in the system or loading. This means conditions in the chamber are maintained at their optimum - important as it gives faster reaction times, greater reproducibility of results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

Pumping options

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



EMS 1050 during operation



EMS 1050 during operation - close up

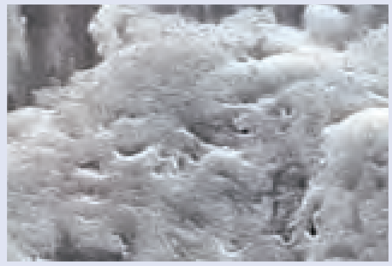
SPECIFICATIONS

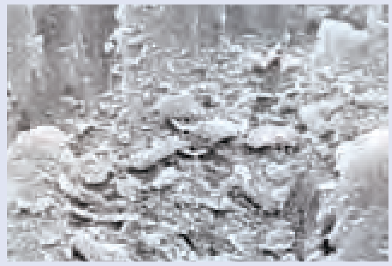
Instrument Case	450mm(W) x 350mm(D) x 300mm(H)
Barrel Work Chamber	'Pyrex' 160mm(L) x 110mm(Dia.) (Borosilicate Glass as standard)
Weight	25Kg
Plasma Output	Solid State Power Supply: 0-150 watts continuously variable at 13.56 MHz with Tuning Control of forward and reflected power to optimize RF power 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.

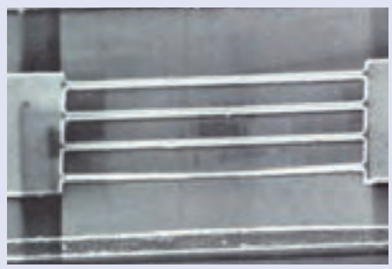
ORDERING INFORMATION

93000	EMS 1050 Plasma Asher	each
91005-F	Rotary Vacuum Pump (Fomblin)	each
6563	Capacitance Manometer	each
6564	Quartz Chamber and Door	each





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



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

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

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 a 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:

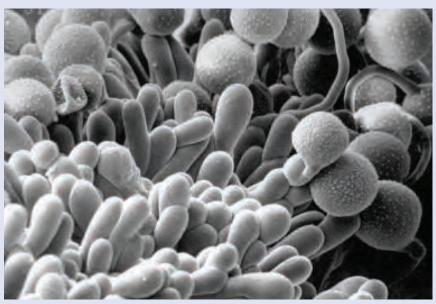
- Specimen viewed in fully hydrated state
- Soluble materials are retained
- Less relocation of highly diffusible elements
- Little or no mechanical damage
- Time lapse experiments and evaluating industrial processes at timed intervals
- Usually no exposure to toxic reagents
- Rapid process
- High resolution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

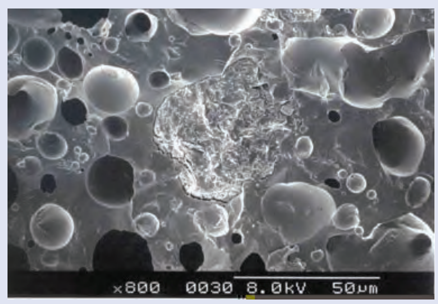
Botanical



Pollen of cactus Zygocactus truncatus

Germinating pollen grains of Zygocactus truncatus.

Foodstuffs



Chocolate Bar

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.

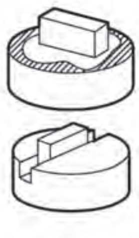
Botanical: Cryo-SEM is the perfect method for highly hydrated botanical material.

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



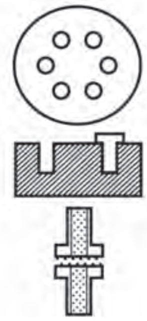
This technique is used for edge observation and fracture. Roughen surface of stub with fine emery paper. Specimen is placed on its edge in a machined slot and secured with mounting media.

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).

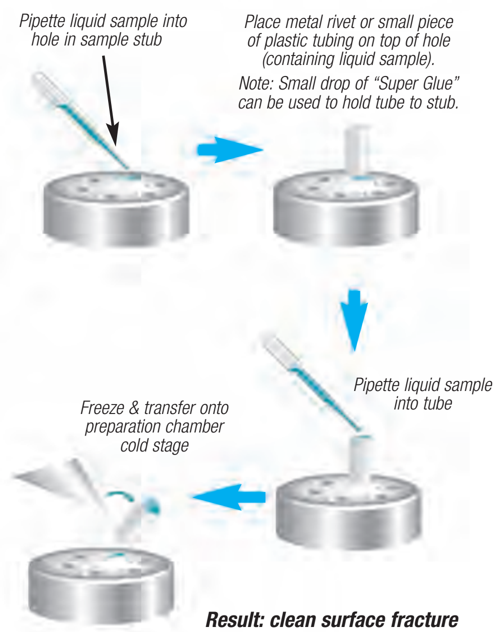
The specimen is laid on surface so that its dampness slightly dissolves the film emulsion allowing the specimen to adhere to the film surface. Exposed unused film with the emulsion side uppermost is secured to the stub with mounting media. It may be useful to scrape off the protective coating of the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

Rivet mounting

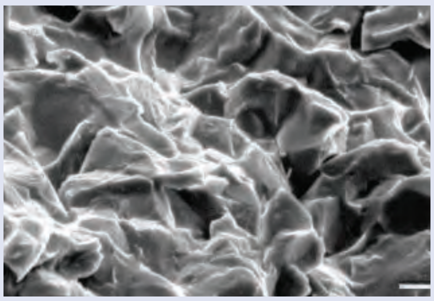


For liquids and for when specimens need to be frozen off the stub to achieve fast freezing rates. The rivet is placed in the hole and filled with liquid prior to freezing. If the specimen needs to be frozen away from the stub, two liquid-filled rivets are held together and then frozen prior to transfer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

Alternative rivet mounting method



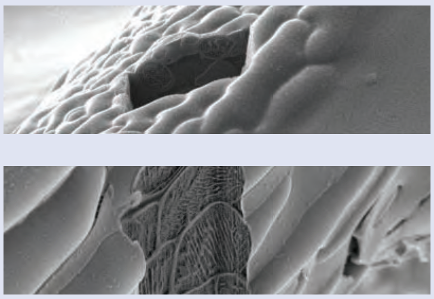
Geological



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.

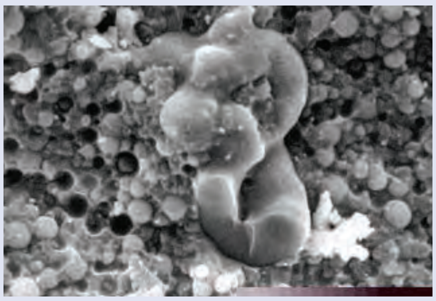
Cryo-DualBeam



Arabidopsis plant

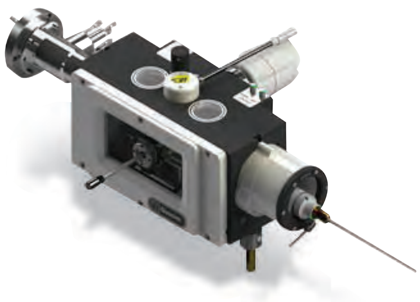
Cryo-FIB/SEM. Image courtesy of Hannah Edwards and Arabidopsis plants provided by Darren Wells, Centre for Plant Integrative Biology, School of Biosciences, University of Nottingham, UK.

Polymers



Stable emulsion of a hydrophobic polymer

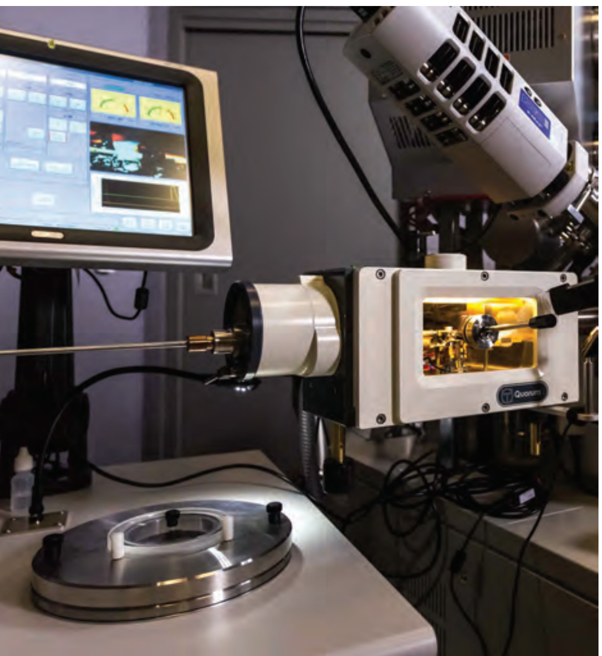
This image illustrates a stable emulsion of a synthetic liquid polymer dispersed in an aqueous continuous phase.



PP3010T Cryo-SEM/Cryo-FIB/SEM Preparation System

KEY FEATURES

- High resolution performance on SEM, FE-SEM and FIB/SEMs
- Totally gas cooled, including cryo preparation chamber – no boiling nitrogen on the SEM
- Efficient cooling (down to at least -190°C)
- 24 hours plus run times on one fill of LN2 are typical – allowing unattended overnight operation (at typical operating temperatures)
- Large recipe driven touch-screen interface
- Automated sublimation, coating and system start up
- Superb specimen visibility (including preparation chamber CCD camera)
- Fully compatible with SEM beam deceleration/stage bias modes up to 5kV
- Off column cooling and pumping systems – minimum mass on the SEM
- On-screen data logging and diagnostics
- Pumped storage of the cryo transfer device
- Prepdek™ workstation – self contained work area, extra bench space not required
- Cryo workflow options
- Specialist support and three-year warranty



PRODUCT DESCRIPTION

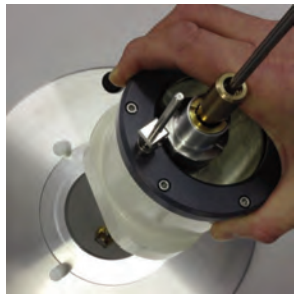
The PP3010T is a highly automated, easy-to-use, column-mounted, gas-cooled cryo preparation system suitable for most makes and models of SEM, FE-SEM and FIB/SEM.

The PP3010T has all the facilities needed to rapidly freeze, process and transfer specimens. The cryo preparation chamber is turbomolecular pumped and includes tools for cold fracturing, controlled automatic sublimation and sputter coating. After processing, the specimen is transferred from the cryo preparation chamber onto a highly stable SEM cold stage for observation. Cold trapping in the cryo preparation chamber and SEM chamber ensures the whole process is frost-free. Specimen process times are typically between five and ten minutes.

PRODUCT FEATURES

Mounting, Freezing and Transferring Specimens – Easy with the Prepdek™ Workstation

The PP3010T Prepdek™ workstation is fitted with a slushy nitrogen freezing station, connected to the pumping system. Rapid freezing reduces ice crystal damage and results in improved specimen preservation. For handling pre-frozen material the Prepdek™ freezing system allows specimens that have been frozen by alternative freezing methods (or stored field specimens) to be manipulated – in or just above liquid nitrogen – and then transferred under vacuum into the PP3010T preparation chamber for subsequent processing and observation.



Slushy nitrogen freezing station

Additionally the TEM Prep Slusher and Glove Box Interface/Airlock options allow workflow amongst a range of other platforms, including cryo-TEM, cryoultramicrome, XPS and glove box.

Cryo Transfer Device – Including Vacuum Storage

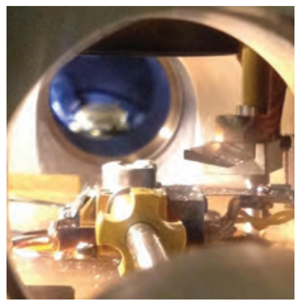
The vacuum transfer device is compact (fits easily into one hand), reliably vacuum-tight and has a bayonet connection to the specimen shuttle to ensure rapid pick up and transfer.

Set into the Prepdek™ work surface is a pumped storage tube for the cryo transfer device (see Prepdek™ workstation section below).

Specimen transfer device

Specimen Stubs, Shuttles

The PP3010T is supplied with universal 10 mm specimen stubs with surface slots, holes and a flat area – useful for most specimen types. Blank and slotted stubs are also included. In addition a range of optional holders is available, including shuttles for large specimens and top-loading holders for high pressure freezing, TEM Autogridstm (for cryo-FIB/SEM applications) and clamping shuttles for hard specimens.



View during specimen transfer

Cryo Preparation Chamber

The cryo preparation chamber is connected directly to the microscope and includes a highly efficient nitrogen gas cold stage, extensive cold trapping and facilities to fracture, sublimate and sputter coat specimens. The chamber is fitted with two fully integrated and interlocked gate valves. The outer load-lock valve includes a pumped airlock which accepts the cryo transfer device – the inner SEM valve ensures rapid high-vacuum to high-vacuum specimen exchange.

Highly Efficient Gas Cooled Stage and Cold Traps

At the heart of the cryo preparation chamber is a nitrogen gas cooled specimen stage. The stage has a dovetail fitting to accept a cryo shuttle and can be precisely controlled over a temperature range from 100°C to -190°C or lower. Large gas cooled cold traps located above, below and behind the specimen stage ensure clean, high vacuum conditions in the chamber.

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III PP3010T Cryo-SEM/Cryo-FIB/SEM Preparation System (continued)

Both the cold stage and cold traps are cooled with the fully integrated CHE3010 off-column cooling system (see below), which at normal operating temperatures give typical hold times of at up to 24 hours between fills (provided the nitrogen gas is dry).

High Visibility – Plus CCD Camera
There is superb visibility into the preparation chamber. In addition to the large front window (75 x 150 mm), there are two top viewing ports. The chamber is lit by three LEDS and a CCD camera allows the specimen cold stage area to be viewed on the control screen and the images saved.

An optional stereo microscope can also be fitted to the cryo preparation chamber

Cold Fracturing
Twin fracturing tools manipulators (actively cooled) are available and allow a range of specimen types to be cold fractured.

Front-mounted fracturing and specimen manipulation tool

The PP3010T is fitted as standard with a front mounted fracturing and manipulation device. The ball-jointed mount offers flexible movement of the blade which can be used both as a surface pick (probe) and a fracturing knife.

An optional micrometer-advanced fracturing tool with rigid blade is available, in addition to the standard front-mounted tool.

Fractured fragments are captured in the large cold trap located below the specimen stage.

Automatic Sublimation and Sputtering
Sublimation temperatures and times can be preset and stored for easy retrieval. The process is fully automatic and graphically displayed on the control screen, showing the actual verses the predicted temperature curves.

The high resolution sputter coater is based on the market leading series of bench top coaters. The coating system will give fine grain films essential for FE-SEM applications. A platinium target is fitted as standard – optional metals include gold, gold/palladium, chromium and iridium. An optional fully integrated carbon fiber evaporation head can also be fitted.

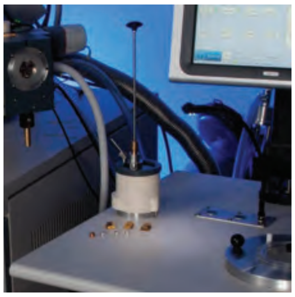
Remotely mounted turbomolecular pumping system

An optional terminating film thickness monitor is available.

Turbomolecular Pumping – High Vacuum Performance
The preparation chamber is pumped by a remotely-positioned 70 L/s turbomolecular pumping system. Typical preparation chamber vacuums when cold are in the region of 10^{-7}mbar or better. Positioning the turbomolecular pump away from the SEM ensures total elimination of mechanical vibration and has the advantage of significantly reducing the total cryo system mass connected to the SEM. A vacuum buffer tank (remotely located in the Prepdek™) is automatically pumped when required. The pumping system is connected to the preparation chamber by flexible stainless-steel bellows, which allows flexible positioning of the pumping system.

A 5 m^3/hr rotary vacuum pump is required to "back" the turbomolecular pump and for slushing and rough pumping operations. The rotary pump can be located up to five meters from the system, allowing remote location if required. Dry pumping alternatives are available – see Ordering Information.

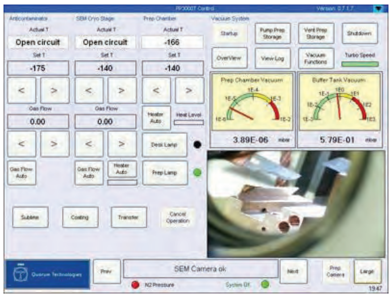
Prepdek™ Workstation
The Prepdek™ workstation has been designed to allow specimen mounting, freezing (plus pre-frozen specimen manipulation) and transfer device storage on one ergonomically designed work surface. The control electronics are mounted in a sealed but accessible cabinet beneath the Prepdek™.



When not in use, the cryo transfer device can be stored under vacuum in the pumped storage tube, located on the Prepdek™ work surface

Set into the work surface is a pumped storage tube which allows the cryo transfer device to be stored under vacuum conditions when not in use.

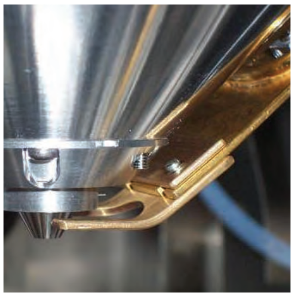
Panel PC Touch Screen User Interface
The PP3010T is controlled using a large touch screen panel PC, mounted on the Prepdek™ workstation. User-defined 'recipes' can be entered and stored for instant future access. The screen can be set to suit operator preferences; for example, vacuum measurement can be displayed in millibar, Pascal or Torr.



Typical screen view during operation (with camera image minimised)

Many of the key steps in the specimen preparation process are automated (system set up, gas flow control, sublimation and sputter coating).

SEM Cold Stage, Cold Trap and Cooling System
A highly stable, thermally isolated, nitrogen gas-cooled stage attaches to the SEM stage. The SEM stage and cold trap are cooled by separate cold gas circuits – both capable of reaching temperatures of -190°C or lower. This configuration allows the operator to select stage and cold trap temperatures that are optimized for specific specimens. For example, for some non-biological materials, it is useful to hold the specimen at very low temperatures – for example, a cold stage temperature of -175°C. This is possible with the PP3010T, as cold trap temperatures of -190°C or lower can be selected, but not possible with conduction cooled systems. The SEM cold stage has a temperature range of down to -190°C and a temperature stability of < 0.5°C.



Gas-cooled SEM cold trap (temperatures down to -190°C). Tailor-made to suit each SEM

III PP3010T Cryo-SEM/Cryo-FIB/SEM Preparation System (continued)

Compatibility with SEM Stage Bias Mode
The PP3010T cold stage is fully compatible with SEM stage bias/beam deceleration modes of up to 5kV.

CHE3010 Off-Column Cooling
The CHE3010 is a fully integrated, remotely mounted cooling system which comes as standard with every PP3010T. The CHE3010 is used to cool the SEM stage, SEM cold trap and cryo preparation chamber cold stage and cold traps and will typically reach temperatures down to -190°C or lower.

The CHE3010 is remotely positioned (typically on the floor behind the microscope) and at normal operating temperatures can run for up to 24 hours between fills. This greatly simplifies the cryo process (no more checking on dewar status and topping off), but also allows overnight, unattended operation – particularly useful for some automated FIB/SEM "slice and view" protocols.

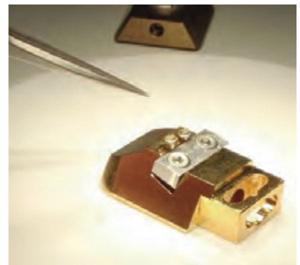
Single Port Interface to the SEM or FIB/SEM
Where SEM geometry allows, both the cryo preparation chamber and the SEM cooling system can be fitted to a single chamber port (the minimum port diameter is 38 mm). This gives a tidy installation and frees up a valuable chamber port.

OPTIONS AND ACCESSORIES

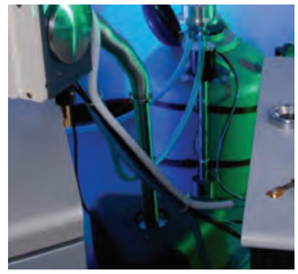
Specimen shuttles and stubs
The PP3010T is supplied with a selection of holders, and a range of additional specimen shuttles and stubs is also available. (See Ordering Information for details).

Carbon Evaporation and Film Thickness Monitor
A carbon evaporation attachment and a terminating film thickness monitor can be fitted. Both are fully integrated (no external control boxes required).

Pressurized LN_2 Dewar
The PP7450/60L is a highly recommended option that generates dry nitrogen gas used for cooling the SEM cold stage & cold trap and cryo preparation chamber and cold shields. In addition, LN_2 can be decanted for



Specimen shuttle



Gas cooling dewar and turbo pump

SPECIFICATIONS

Cryo Preparation Chamber (column-mounted)	Standard?
Gas cooled preparation chamber with a twenty-four hour run time between fills	Yes
Two integral gate valves (loading and SEM) with appropriate electrical interlocks	Yes
Variable temperature gas-cooled specimen stage	Yes
Large cold shield above, below, behind the cold stage	Yes
Robust micrometer-fed fracturing knife (actively cooled)	Option
Side-mounted surface knife/probe (actively cooled). A range of scalpel blades can be fitted to suit different specimen requirements	Yes
Automatic sublimation (controlled and viewed on the touch screen)	Yes
Fully automatic, high resolution sputter coater with platinum (Pt) target. (Other targets, including gold (Au), gold/palladium (Au/Pd), chromium (Cr) and iridium (Ir), are available as options.) Sputtering controlled and viewed on the user touch screen	Yes
Carbon fiber evaporation head and power supply	Option
Large front viewing window (150 x 78mm) plus top viewing ports	Yes
Preparation chamber camera (CCD)	Yes
Vacuum transfer device	Yes
Chamber illumination — three LEDs	Yes
Pumping System and Controls	
Remotely-mounted turbomolecular pumping system (70L/s). Includes: vacuum buffer tank, vacuum valves and stainless-steel bellows connection to the preparation chamber. Typical preparation chamber vacuum when cold: 10^{-7} mbar	Yes
Single 50L/m rotary pump required	Order separately
SEM Cooling Dewar, SEM Cold Stage and Cold Trap (anticontaminator)	
Gas-cooled nitrogen cold stage assembly (-190°C). Temperature stability of >0.5°C	Yes
Separate gas-cooling circuits for SEM stage and SEM anti-contaminator	Yes
21L capacity, off-column cooling dewar with run time between fills of up to 24 hours	Yes
SEM CCD camera-fitted when space allows	Yes
LED lighting (interlocked)	Yes

System Control and Specimen Handling	Standard?
Control via a color user touch screen monitor (15") mounted on the Prepdek™	Yes
• Multi-ability user interface screen • Quick, easy overview of system status • User-definable "recipes" can be stored • Quick access to videos outlining preparation techniques and system maintenance • Fully automatic sputtering • Automatic sublimation • Quick, easy overview of system status • CCD camera image of preparation chamber	
Twin liquid nitrogen slushing and specimen handling system — ideal for handling pre-frozen specimens. Mounted on the Prepdek™	Yes
System electronics stored in a ventilated, sealed unit under the Prepdek™	Yes
Specimen Shuttles and Stubs (Others available — see Ordering Information)	
• (2) AL200077B specimen shuttles (to hold 10mm diameter cryo stubs) • E7402 blank 10mm stubs — pack of 10 • E7449-5 multi-stubs 7mm high (with holes and slots) — pack of 5 • 11541 multi-stubs 5mm high (with holes and slots) — pack of 5 • 20529 Dovetail holder shuttle • 328116510 Brass rivets for fracturing liquids — pack of 100 • E7406 Copper (Cu) stub with 3mm x 3mm slot — pack of 5 • E7407 Copper (Cu) stub with 1mm x 3mm slot — pack of 5	Yes
Installation and Training	
Installation and training at the customer site	Contact EMS
Support and Other Information	
Comprehensive start-up kit with key spares	Yes
Three-year warranty	Yes
SEM column interfaces and SEM stage adaptor (tailored to each microscope)	Yes
Some Options and Accessories (see Ordering Information for full list)	
Terminating film thickness monitor (FTM)	Option
Self-pressurizing LN_2 dewar and regulator (for storage and venting)	Option
Carbon fiber evaporation head	Option
Wide range of specimen holders and specimen stubs	Option

Electron Microscopy Sciences In PA: (215) 412-8400 • Toll-Free (800) 523-5874
Fax (215) 412-8450 or 8452 • email: info@emsdiasum.com or stacie@ems-secure.com • www.emsdiasum.com

PP3010T Cryo-SEM/Cryo-FIB/SEM Preparation System (continued)

slushing (freezing). During normal operation the PP7450/60L will generate dry nitrogen gas for up to eight days usage.

If the PP7450/60L is not included, appropriate, locally sourced, nitrogen gas cylinders can be used. It is important to ensure that it has low moisture content – if in doubt, please contact us.

Cryoflow Work Options

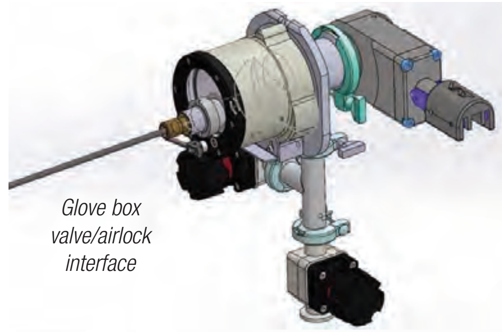
Glove Box Valve/Airlock InterfaceGlove Box Valve/Airlock Interface
The airlock is connected to a glove box using a generic NW fitting and for most applications requires a suitable pumping system (rotary pump or turbomolecular plus diaphragm pumping system).

The airlock is designed to accept the PP3010T vacuum transfer device.

For full details of this and other accessories please see Ordering Information.

TEM Prep Slusher OptionTEM Prep Slusher
Offers an easy, convenient way of transporting pre-frozen specimens to and from the PP3010T.

- Conveniently locates into the Prepdek™ workstation of the PP3010T
- Freely transportable, e.g. between high pressure freezer, cryoultramicrotome and the PP3010T
- Ideal for loading/unloading TEM grids and grid holders
- Tilting holder for cryo shuttles (holders). Allows the easy transfer of specimens from external freezers (e.g. high pressure, jet, slam etc)
- Option for PP3010T and previous PP3000T and PP2000/PP2000T models



Cryo preparation chamber with cryo transfer device fitted

ORDERING INFORMATION

For a full quotation, including on-site installation and customer training, please contact us.

PP3010T	**Cryo-SEM Preparation System for SEM, FE-SEM and FIB/SEM applications.**	each

Includes: *column-mounted cryo-preparation chamber with off-column turbo pumping system. SEM cold stage and cold trap, Prepdek™ workstation with dual freezing and specimen manipulation facilities, automatic sputtering and sublimation. Touch screen user interface mounted on the Prepdek™ workstation. Transfer device, (2) specimen shuttles, (10) blank 10 mm stubs, (5) multi-stubs 7 mm high, (5) multi-stubs 5 mm high, Dovetail holder shuttle, (100) Brass rivets for fracturing liquids, (5) Copper (Cu) stub with 3 mm x 3 mm slot, (5) Copper (Cu) stub with 1 mm x 3 mm slot. Microscope interfaces, start-up kit, mounting media and operation manual.*

Pumping

The PP3010T requires one 50L/m rotary pump (dry pumps available on request).

91005	50L/m 115/230V 50/60Hz rotary vacuum pump with oil mist filter	each

Options and Accessories

PP7450	Pressurized dewar (60L) for LN_2 storage and venting gas supply	each
10996	Glove Box Interface/Airlock – for vacuum or inert gas transfer	each
10997	TEM Prep Slusher option for transporting pre-frozen specimens to and from the PP3010T	each
10998	Carbon fiber evaporation head including 1m high purity carbon fiber	each
10996	Film thickness monitor (FTM)	each
12145	Micrometer controlled fracturing device with tool steel blade. Note: the standard ball-joint mounted fracturing tool is fitted as standard. The 12145 can be fitted in addition.	each
13060	Two-years spare kit for PP3010T	each

Specimen Shuttles

AL200077B	Standard specimen shuttle with hole for 10mm stub, two included as standard	each
12434	Specimen shuttle without 10mm hole (flat surface 22mm x 13mm) for large specimens	each
13524	Shuttle for clamping hard, flat specimens. Suitable for flat specimens (front of shuttle with clamp lever) and cross-fracturing (sprung-loaded vice at rear of shuttle).	each
10245	Top loading freeze-facture “Balzers” planchette holder shuttle	each
10246	Top loading specimen holder shuttle (similar to AL200077B but stub clamping mechanism is located on the top – for handling pre-frozen specimens mounted on a stub), one included as standard	each
10247	Top loading rivet holder shuttle (vice style). Holds two rivets	each
E7433	Rivet holder stub, screw down style (for use with 10246).	each
12406	Special shuttle for cryo-FIB/SEM of TEM Autogrid™, accepts two TEM Autogrid™ holders. Includes cryo shield	each

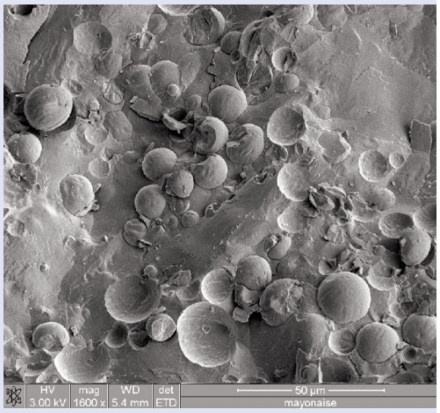
Specimen Stubs (10mm diameter)

E7449-5	Universal specimen stub with holes and slots (pack of 5) (10mm dia. X 7mm high), two packs included as standard	each
E7401	Specimen stub shuttle (spare)	each
E7402	Aluminium (Al) stubs (pack of 10), one pack included as standard	each
E7403	Copper (Cu) stubs (pack of 10)	each
E7405	Screw down stub for thin hard specimens (x1)	each
E7406	Copper (Cu) stub with one 1mm wide x 3mm deep slot (pack of 5), one packet included as standard	each
E7407	Copper (Cu) stub with one 3mm wide x 3mm deep slot (pack of 5), one packet included as standard	each
32816510	Brass rivets for fracturing liquids (pack of 100), one pack included as standard	each

Sputter Targets and Carbon Fiber (all targets 24.5mm diameter)

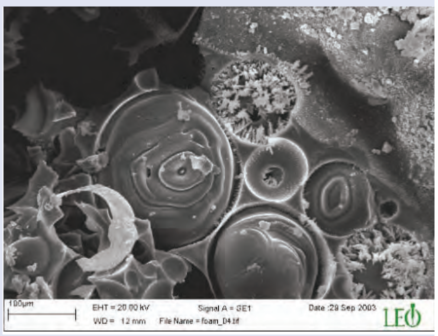
E7400-314A	Gold (Au) target 0.008” thick	each
E7400-314B	Gold/palladium (Au/Pd 80:20) target 0.2mm thick	each
E7400-314C	Platinum (Pt) target 0.2mm thick	each
E7400-314IR	Iridium (Ir) target 0.008” thick	each
E7400-314Cr	Chromium (Cr) target 0.3mm thick	each
91047-1	Carbon fiber cord, high purity — 100cm	each
91047-5	Carbon fiber cord, high purity — 10m	each

Building on the success of the PP3010T cryo-SEM/FIB/SEM preparation system, we are pleased to announce three new related products for ambient and cryo temperature transfer.



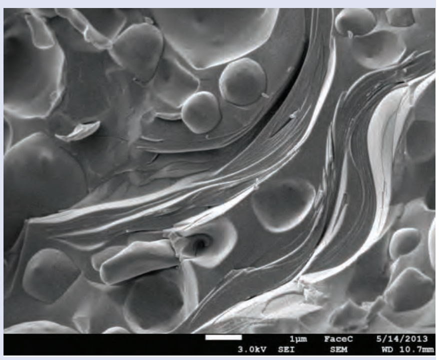
Mayonnaise

Image courtesy of FEI Company.



Shaving Cream

Fractured at -140ºC, sublimated at -90ºC for 3 minutes and coated with 5nm of platinum.



Face Cream

Anti-aging face cream. Specimen rapidly frozen in slushy nitrogen, fractured at -140°C and sputter coated with 5nm of platinum

Cryo Transfer Systems

PP3004 QuickLok

Ambient temperature airlock for SEM, FIB/SEM, beamline and vacuum platforms

Quick Overview

The QuickLok provides a rapid way of transferring ambient temperature specimens into SEM, FIB/SEM or other suitable vacuum systems. A key feature of the QuickLok is the ability to vacuum transfer specimens that are sensitive to normal environmental conditions. The transfer device uses a sealed vacuum chamber which can be interfaced to a glove box for inert gas transfer or allow vacuum transfer from a wide range of platforms.

Key Features

- **Rapid specimen exchange**
- **Vacuum and inert gas transfer**
- **Field-retrofittable to most systems**
- **Upgrade path to CoolLok**
- **Custom designed holders available**
- **3 year warranty**

Components

Mounted onto a suitable vacuum chamber port, the QuickLok consists of a loading chamber body with integrated controls for pumping, venting and transfer. A custom-designed interface flange and connections to the pumping system are included (see Pumping below).

The compact vacuum transfer device has an easy-release bayonet fitting to a dovetail-profile specimen holder (shuttle). Standard shuttles are included, but optional holders allow a range of specimen types to be handled.

Inside the microscope is a stage to accept the specimen shuttle. To aid specimen exchange an interlocked LED chamber light is mounted to the inside of the QuickLok interface.

Use

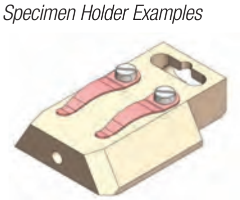
The specimen is mounted on a suitable holder and the transfer device fitted onto the QuickLok. The airlock and transfer device are then evacuated to a pre-set vacuum and the gate valve opened. The specimen is then guided onto the microscope stage.

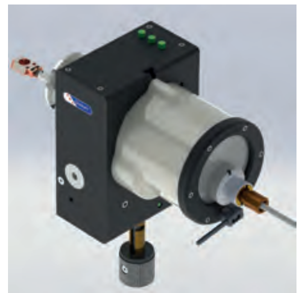
For transfer from other vacuum systems, or a glove box, additional interface flanges are available on request.

Pumping

The QuickLok requires either a rotary pump or oil-free vacuum turbomolecular pumping station (see Options).

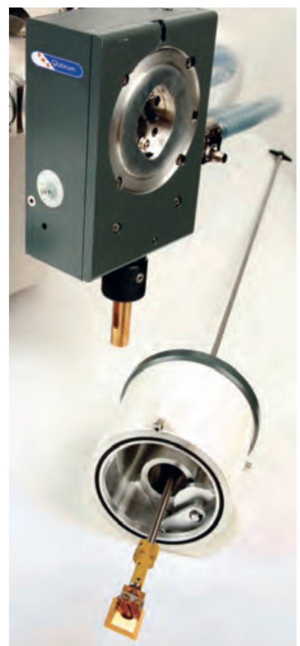
Specimen Holder Examples



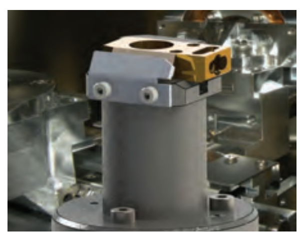


PP3004 QuickLok

Simple controls for specimen exchange



QuickLok and specimen transfer device

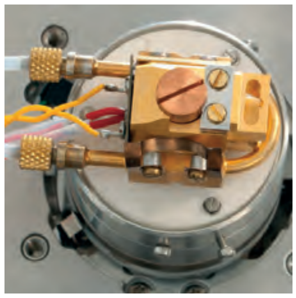


QuickLok specimen stage and adaptor to SEM

III Cryo Transfer Systems (continued)

PP3005 SEMCool

Non-airlock cryo cooling for SEM, FIB/SEM, beamline and vacuum platforms



Quick Overview

The SEMCool is based on the PP3006 CoolLok but without the PP3004 QuickLok components. It is designed for cryogenic applications where airlock exchange of specimens into the microscope is not required.

Key Features

- Temperature range down to -190°C, with stability better than 0.5°C
- Off-column cooling with all-day runtime between fills
- Independent cooling of cold stage and cold trap
- Upgrade path to CoolLok
- 3 year warranty

Components

Specimen holders and transfer device: The compact vacuum transfer device has an easy-release bayonet fitting to a dovetail-profile specimen holder (shuttle). Standard shuttles are included, but optional holders allow a range of different specimen types to be handled.

Cold trap - adapted to installation

Cold stage and cold trap: A highly stable, thermally isolated, nitrogen gas-cooled cold stage attaches to the microscope stage. The location and shape of the cold trap is tailored to suit the internal geometry of the microscope. Both cold stage and cold trap are capable of reaching temperatures down to -190°C with a stability of <0.5°C. For easy specimen exchange an LED chamber light is fitted.

The cold stage connects to the microscope stage using an adaptor and has a dovetail fitting to accept a specimen holder. When not in use the cold stage is uncoupled and stored within the chamber with the gas and electrical fittings connected.

Temperature controller

Controller and cooling system

Cooling dewar, trolley and controller: The cold stage and cold trap are cooled by a remotely-positioned, vacuum isolated 21 L dewar and heat exchanger assembly which at normal operating temperatures can run for up to 24 hours between fills. The gas lines between the dewar and the microscope interface are vacuum isolated for maximum thermal efficiency.

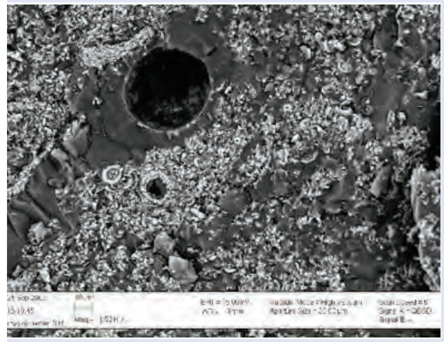
The cooling dewar sits on a floor-mounted trolley which also houses the monitor/controller for cold stage and monitor for cold trap, plus nitrogen gas flow controllers.

Use

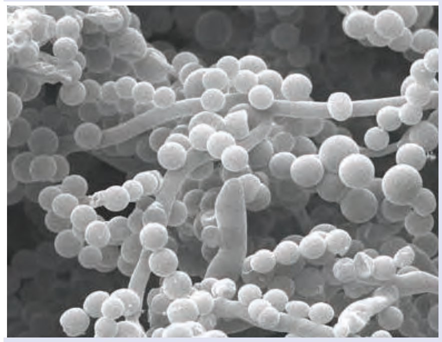
Vent the SEM, locate specimen holder on the cold stage, re-pump the SEM and then cool down to the required temperature. To exchange specimen, warm to above 0°C and vent the SEM.

Pumping

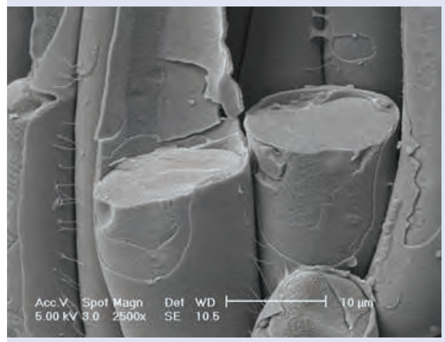
The SEMCool requires a rotary pump to periodically evacuate the vacuum isolated lines (see Ordering Information).



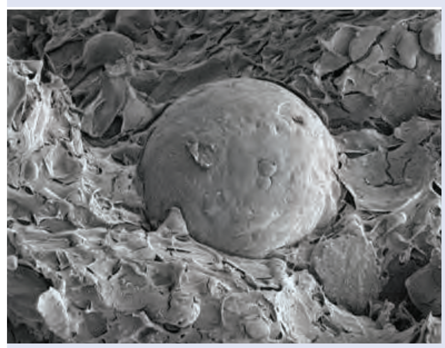
Cross-section of oil/water/rock.



Cryo prepared image of blue stilton cheese (Penicillium roqueforti).



Cross-section through plant palisade cells.

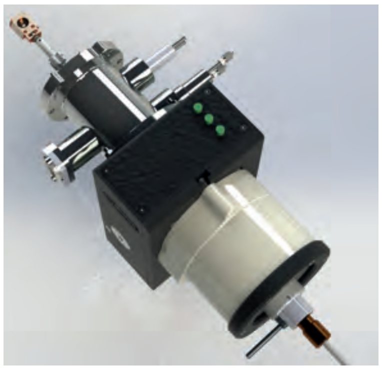


Cross-section image through sunscreen

III Cryo Transfer Systems (continued)

PP3006 CoolLok

Cryo transfer systems for SEM, FIB/SEM, beamline and vacuum platforms



PP3006 CoolLok

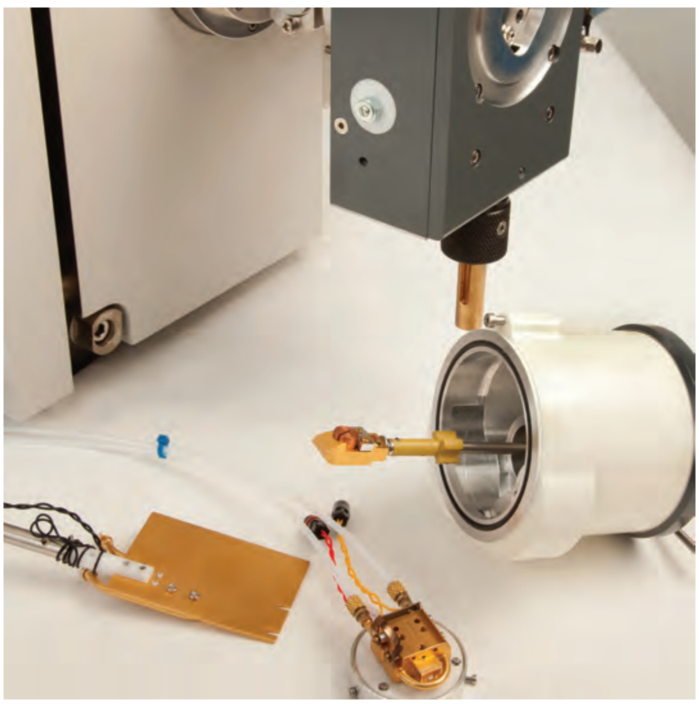
Quick Overview

The CoolLok offers rapid transfer and cryo temperature observation of specimens for SEM, FIB/SEM, beamline or other vacuum systems. Applications include thermal protection of beam-sensitive specimens and low temperature observation of materials such as plastics, polymers low-K dielectrics and hard-soft mixtures. The system can also be used for inert gas transfer of ambient temperature specimens from a glove box.

Please Note: The PP3006 is not a replacement for the PP3010T, which is a full cryo preparation system. The PP3006 does not have a cryo preparation chamber and is designed for materials applications where cold fracturing and sputtering are not required.

Key Features

- Rapid specimen exchange
- Temperature range down to -190°C with stability better than 0.5°C
- Off-column cooling with all-day runtime between fills
- Independent cooling of cold stage and cold trap
- Vacuum or inert gas transfer
- Rapid specimen freezing option
- 3 year warranty

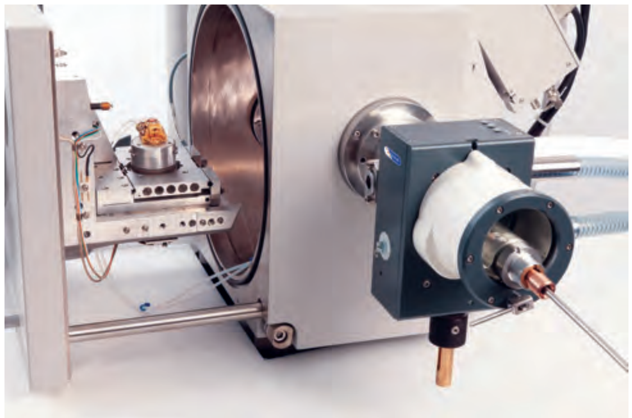


On-microscope components: airlock, cold stage, cold trap plus cryo transfer device

Components

Vacuum airlock cold gas feedthrough Mounted onto a suitable vacuum chamber port, the CoolLok consists of a loading chamber body with built-in controls for pumping, venting and transfer. A custom-designed interface flange to the vacuum chamber and connections and fittings to the pumping system are included (see Pumping below). The interface has cold nitrogen gas feeds to and from the microscope cold stage and cold trap.

Load lock with vacuum isolated gas cooling lines

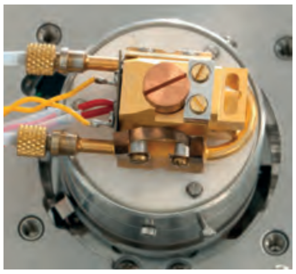


PP3006 installation example

Specimen holders and transfer device The compact vacuum transfer device has an easy-release bayonet fitting to a dovetail-profile specimen holder (shuttle). Standard shuttles are included, but optional holders allow a range of different specimen types to be handled.

III Cryo Transfer Systems (continued)

PP3006 CoolLok (continued)


Cold stage


Cold trap - adapted to installation

Cold stage and cold trap A highly stable, thermally isolated, nitrogen gas-cooled cold stage attaches to the microscope stage. The location and shape of the cold trap is tailored to suit the internal geometry of the microscope. Both cold stage and cold trap are capable of reaching temperatures down to -190°C with a stability of <0.5°C. For easy specimen exchange an LED chamber light is fitted.

The cold stage connects to the microscope stage using an adaptor and has a dovetail fitting to accept a specimen holder. When not in use the cold stage is uncoupled and stored within the chamber with the gas and electrical fittings connected.


Dewar and Controller

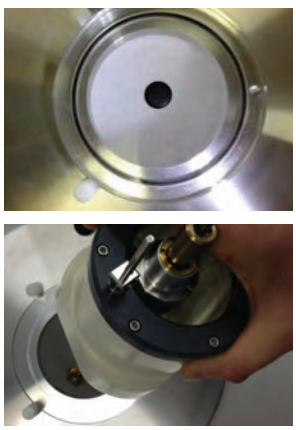

Heat exchanger

Cooling dewar, trolley and controller The cold stage and cold trap are cooled by a remotely-positioned, vacuum isolated 21L dewar and heat exchanger assembly which at normal operating temperatures can run for up to 24 hours between fills. The gas lines between the dewar and the microscope interface are vacuum isolated for maximum thermal efficiency.

The cooling dewar sits on a floor-mounted trolley which also houses the monitor/controller for cold stage and monitor for cold trap, plus nitrogen gas flow controllers.

Rapid freezing station (24429) With the standard CoolLok, specimen freezing is by contact with the microscope cold stage following transfer and therefore freezing rates are relatively slowly. This is suitable for hard, non-hydrated specimens, but for liquid-based material rapid freezing is essential to reduce the detrimental effects of ice crystal growth and to allow through-vacuum transfer onto the cold stage.

For these applications the optional nitrogen slush freezing station is required. However, for many applications (especially lifesciences) cold fracturing and sputter coating are essential process steps and require the advanced capabilities of the EMS PP3010T – a full cryo preparation system.


Plunge freezing in slushy nitrogen

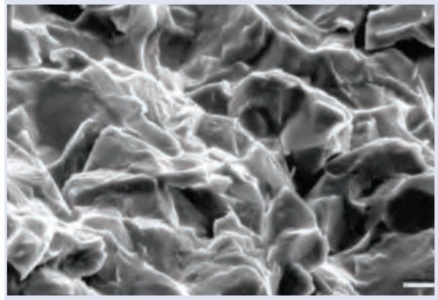
Use

The specimen is mounted on a suitable holder (shuttle) and the transfer device fitted onto the airlock and the dead space evacuated to a pre-set vacuum level. The gate valve is opened and the specimen guided onto the SEM stage.

For transfer from other vacuum systems, or a glove box, additional interface flanges are available on request. Vacuum transfers can be made from the optional 24429 trolley-mounted nitrogen slush freezing station, if fitted.

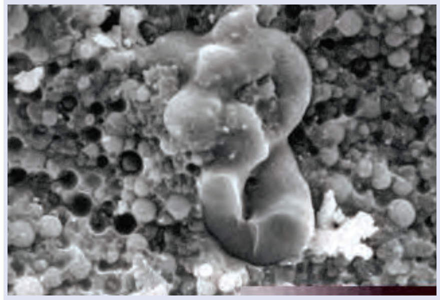
Pumping

The QuickLok requires either a rotary pump or oil-free turbomolecular pumping station (see Options).



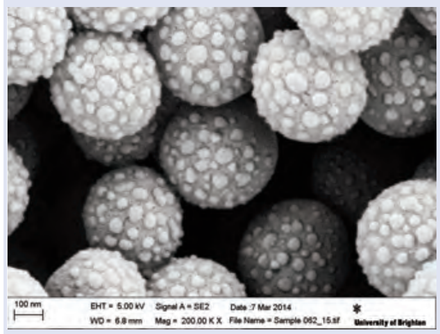
Wax crystals in gas oil

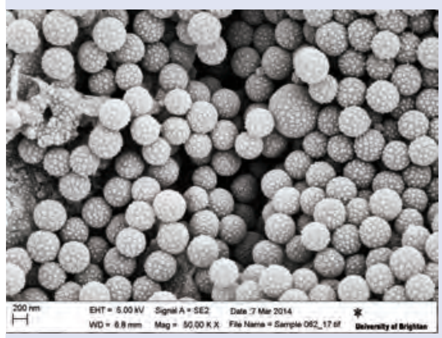
When cooled to a temperature below about 2°C, the waxes in fuel oils such as this tend to crystallize out. Wax crystal size and shape can be varied by altering the rate at which the oil is cooled.



Stable emulsion of a hydrophobic polymer

This image illustrates a stable emulsion of a synthetic liquid polymer dispersed in an aqueous continuous phase**.**





Latex

Latex particles are very electron beam sensitive, so cryo-SEM is an ideal method for there observation

III Cryo Transfer Systems (continued)

SPECIFICATIONS for PP3004, PP3005, PP3006

	PP3004	PP3005	PP3006
Temperature	Ambient	RT to -190°C	RT to -190°C
Cooling Runtime	N/A	Up to 24 hours	Up to 24 hours
LN_2 Dewar Capacity	N/A	21 liters	21 liters
Cool-Down Time to -190°C	N/A	Typically <15 minutes	Typically <15 minutes
Rapid Freezing (slushy LN_2)	N/A	Optional (24429)	Optional (24429)
Dewar Trolley Footprint	N/A	50 x 50 cm	50 x 50 cm
Airlock Weight	2.5 kg	2.5 kg	2.5 kg
Pumping Requirements	Rotary pump or dry pump	Rotary pump or dry pump	Rotary pump or dry pump
Nitrogen Gas	For venting and valve operation	Venting and cooling	Venting and cooling
Power Requirements (excluding pump)	300 W	300 W	300 W
Maximum Specimen Size	Flat specimens up to 23 x 26 mm. For taller specimens the maximum height will reduce from a mid-point of 9mm. Please contact us for more details.		

ORDERING INFORMATION *For a full quotation, including on-site installation and customer training, please contact us.*

PP3004	**QuickLok Ambient Temperature Transfer System** **Includes:**	
	Airlock assembly. Pump and vent and transfer controls, valve and fittings to the pumping system (see: Pumping below). Custom designed interface flange to the microscope vacuum chamber	
	Microscope dovetail stage to accept specimen shuttle. LED chamber light (interlocked)	
	Specimen transfer device for vacuum or inert gas transfer	
	Specimen holders. Specimen shuttle with holding clips, specimen shuttle blank, specimen shuttle (to hold a 10mm dia. specimen stub), blank 10 mm stubs – packet of 10	each
PP3005	**SEMCool Non-Airlock Low Temperature System** **Includes:**	
	Nitrogen gas cooled cold stage with heater and sensor and cold trap with temperature sensor. Temperature controllable with a range down to -190°C, 21 L liquid nitrogen dewar with trolley, heat exchanger and LED chamber light. Pump fittings (see: Pumping below).	
	Temperature and nitrogen gas flow controller mounted on the dewar trolley.	
	Specimen holders. 3 specimen shuttles (to hold 10 mm Ø cryo stubs), blank specimen shuttle, specimen shuttle with holding clips, blank 10 mm Ø stubs (packet of 10), 5 multi-purpose specimen stubs. Note: other holders available	
	Specimen mounting compounds (colloidal graphite and Tissue-Tek®)	each
PP3006	**CoolLok Cryo Transfer System** **Includes:**	
	Airlock assembly. Pump and vent and transfer controls, gate valve and fittings to the pumping system (see: Pumping below). Custom designed interface flange to the microscope vacuum chamber.	
	Cooling system. Nitrogen gas cooled cold stage with heater and sensor and cold trap with temperature sensor. Temperature controllable with a range down to -190°C, 21 L liquid nitrogen dewar with trolley, heat exchanger and LED chamber light.	
	Specimen transfer device	
	Specimen holders. 3 specimen shuttles (to hold 10 mm Ø cryo stubs), blank specimen shuttle, specimen shuttle with holding clips,blank 10 mm Ø stubs (packet of 10), 5 multi-purpose specimen stubs. Note: other holders available	
	Specimen mounting compounds (colloidal graphite and Tissue-Tek®), interlock cable and pump fittings	each

Pumping

The PP3004 QuickLok and PP3006 CoolLok require either a rotary pump or high vacuum turbomolecular pumping station (recommended). The PP3005 requires a rotary pump for evacuating the vacuum isolated gas lines.

13034	Pfeiffer Duo 6 — 5 m^3/hr rotary vacuum pump with oil mist filter	each
24426	Pfeiffer HiCube 80 turbomolecular and diaphragm pumping system	each

Options and Accessories

24429	Rapid cooling station (for PP3006 only) Consists of a floor-mounted trolley, liquid nitrogen freezing chamber mounted into the work surface which interfaces to the cryo transfer device, connections to vacuum pump (order separately)	each
PP7450	Pressurized (60 L) LN_2 dewar. Boil-off nitrogen gas is used for cooling the stage and cold trap (PP3005 and PP3006 only)	each
13296	Sircal in-line gas dryer. Helps to reduce water content of nitrogen gas supply	each

Specimen Holders

10245	Top-loading specimen shuttle for planchettes	each
10246	Top-loading specimen shuttle, to take a 10mm stub	each
10247	Top-loading specimen shuttle for rivets (vice style)	each
E7433	Rivet holder specimen stub, screw-down style (for use with 10246)	each
E7449-5	Universal specimen stub with surface holes and slots (5 pack)	each
E7401	Specimen stub shuttle (spare)	each
E7402	Aluminum (Al) stubs (10 pack)	each
E7403	Copper (Cu) stubs (10 pack)	each
E7405	Screw down stub for thin, hard specimens	each
E7406	Copper (Cu) stubs with 3 x 3mm slots (5 pack)	each
E7407	Copper (Cu) stubs with 1 x 3mm slot (5 pack)	each
32816510	Brass rivets for fracturing liquids (100 pack)	each

Sputter Targets and Carbon Fiber

E7400-314A	Gold (Au) target 0.008" thick	each
E7400-314B	Gold/palladium (Au/Pd) (80:20) target 0.2mm thick	each
E7400-314C	Platinum (Pt) target 0.008" thick	each
E7400-314IR	Iridium (Ir) target 0.008" thick	each
E7400-314CR	Chromium (Cr) target 0.3mm thick	each
91047-1	Carbon fiber cord — high purity — 1m	each
91047-5	Carbon fiber cord — high purity — 5m	each

Coaters and Coater Targets Replacement Parts

EMS 450 Carbon Coater

The EMS 400 and 450 Carbon coaters have been replaced by the EMS 150R E Plus.

Parts and accessories for the EMS 450 are listed below.

Carbon

91045	Carbon String, 1 meter	meter
91046	Carbon Cord, 1 meter	meter

Replacement Parts

91013	Glass Cylinder 165mm (6")	EMS 450, 500, 550	
91014	"L" Gaskets to suit 165mm (6") cylinder EMS 450, 500, 550		1 pair

EMS 500, 550, 575, 650 and 675 Sputter Coaters and EMS 975 and 975S Large Chamber Turbo Evaporator

EMS is pleased to offer the 150 and 300 series sputter coater and combined carbon and sputter coater.

- **EMS150R ES Plus – a combined system with sputtering and carbon fibre coating replaces the EMS 500 and 550.**
- **EMS 150T S Plus replaces the EMS 575X**
- **EMS 300T D Plus replaces the EMS 575XD**
- **EMS 300R T Plus and EMS 300T T Plus replace the EMS 600, EMS 650, and the EMS 675X**

Parts and accessories for the EMS 575, 650, 675 and 975 are listed below.

Accessories

92045	EMS 50 Water Chiller

Replacement Targets

EMS strives to maintain accurate pricing. However, due to fluctuations in precious metal prices, pricing on products containing precious metals is not guaranteed. We will contact you if there is a discrepancy.

91010	Gold Target	EMS 500, 550, 575T	each
91011	Gold/Palladium Target	EMS 500, 550, 575T	each
91012	Platinum Target	EMS 500, 550, 575T	each
91013	Chromium Target	EMS 575T	each
91014	Tungsten Target	EMS 575T	each
91030	Gold Targets*	EMS 650, 675X	x3
91031	Gold/Palladium Targets*	EMS 650, 675X	x3
91032	Platinum Targets*	EMS 650, 675X	x3
91013	Chromium Targets*	EMS 675X	x3
91014	Tungsten Targets*	EMS 675X	x3

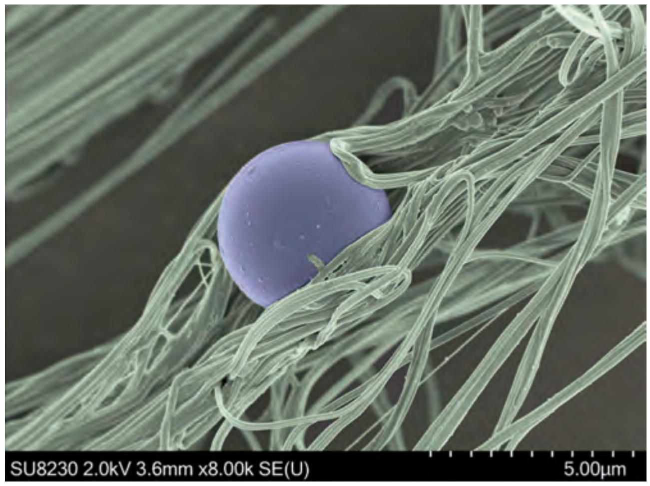
(*Recommended change as set of three)

Replacement Parts

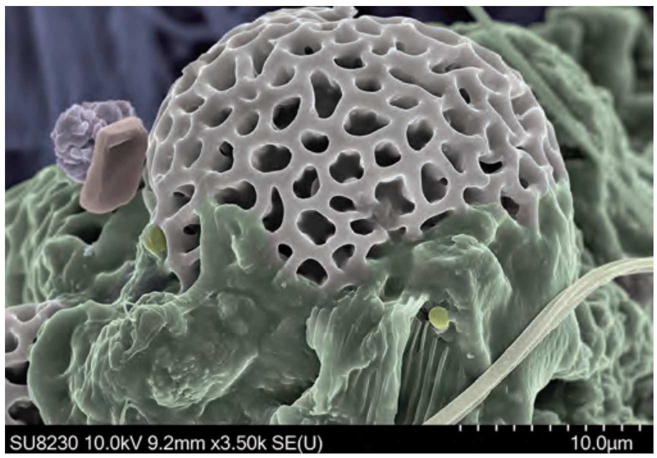
91033	Glass Cylinder 225mm (8")	EMS 650, 675X, 975, 975S	
91034	"L" Gaskets to suit 225mm (8") cylinder EMS 650, 675X, 975, 975S		1 pair

Replacement Source

91077	Carbon Rods (6.15mm Dia.),	EMS 975, 975S	12/pack



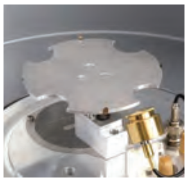
Algae spore in spider web, spore size 5 μm coated with 2nm Cr followed by 1nm Au mag x 8k



Radiolata, colorized

Maize root starch granules

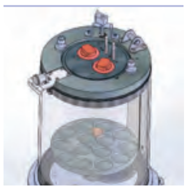
4500-2 Tilt Angle



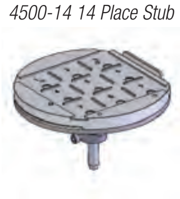
4500-3 4" Wafer

4500-12 Rotary Planetary

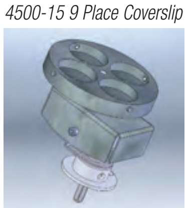
4500-13 8 Place Stub



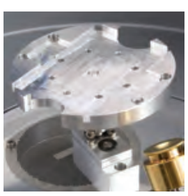
4500-14 14 Place Stub



4500-15 9 Place Coverslip



4500-16 4 Place 25mm Stub



4500-17 Glass Microscope

Selection Guide: Specimen Stages for EMS Equipment

The EMS line of specimen stages meets most requirements. All are easy-change, drop-in style (no screws) and are height adjustable (except rotary planetary stage). Examples:

Cat #	Stage Type	Description	Compatible Equipment
4500-1	50mm	Standard stage with six stub positions for 15 mm or 6.5 mm or 1/8" pin stubs (same as #3330, 6401, 6552)	All EMS Series
4500-2	Tilt	Rotate-tilt stage with six stub positions for 15 mm or 6.5 mm or 1/8" pin stubs. Tilts Up to 90° from horizontal (same as #3340, 4519, 6400-S, 6551)	All EMS Series
4500-3	4" Wafer	Adjusts to accept 2", 3", 4" wafers comes with **4500-6**, a 4" stub holder to accept up to 18 1/8 pin stubs (same as #6549)	All EMS Series
4500-4	6" Wafer	Adjusts to accept 4" & 6" wafers comes with **4500-7**, a 6" stub holder to accept up to 27 1/8 pin stubs (same as #6547)	EMS 300
4500-5	8" Wafer	Adjusts to accept 6" & 8" wafers comes with **4500-8**, an 8" stub holder to accept up to 54 1/8 pin stubs	EMS 300T T Plus EMS 300R T
4500-6	4" Stub Holder	A 4" stub holder to accept up to 18 1/8 pin stubs	All EMS Series
4500-7	6" Stub Holder	A 6" stub holder to accept up to 27 1/8 pin stubs	EMS 300T T Plus EMS 300R T EMS 300T D Plus
4500-8	8" Stub Holder	An 8" stub holder to accept up to 54 1/8 pin stubs	EMS 300T T Plus EMS 300R T
4500-9	4" Wafer & Offset Gearbox	A combination of **4500-3** and a small gearbox to offset the sample position Enable even coating of up to a 4" sample size. (same as #3360, 4522)	EMS 150T Plus EMS 150R Plus
4500-10	Fiber Stage	A stage accept single fibers or pins up to 1 mm dia. rotating horizontally to achieve all round coating	EMS 150T Plus EMS 150R Plus
4500-11	6" Square Wafer	Stage to accept 6" square wafer or Masks	EMS 300T T Plus EMS 300R T
4500-12	Rota Cota	"Rota Cota" planetary stage with six stub positions for 15 mm or 6.5 mm or 1/8" pin stubs. Tilts up to 30° from horizontal (same as #4521, 6402, 6553)	EMS 150T Plus EMS 150R Plus
4500-13	8 Place Stub	8 places for 25 or 30mm Polished embedded samples. Includes a polished Brass Tally	All EMS Series
4500-14	14 Place Stub	14 places for 25 or 30mm Polished embedded samples. Includes a polished Brass Tally	All EMS Series
4500-15	9 Place Coverslip	A Stage to accept 9 20X20 cover slips. The top part of stage lifts off and has a mechanism to lift the cover slips for easy removal	All EMS Series
4500-16	4 Place 25mm Stub	4 Place 25mm Stub Stage with locking screws. May be fitted to 4500-12 rota cota stage	All EMS Series
4500-17	Slide Stage	Microscope slide stage for up to two 75 mm x 25 mm slides or eight stub positions for pin stubs. (same as #3370, 4520, 6403, 6554)	All EMS Series

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