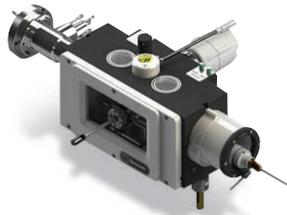


Cryo-SEM & Cryo-FIB/SEM Preparation

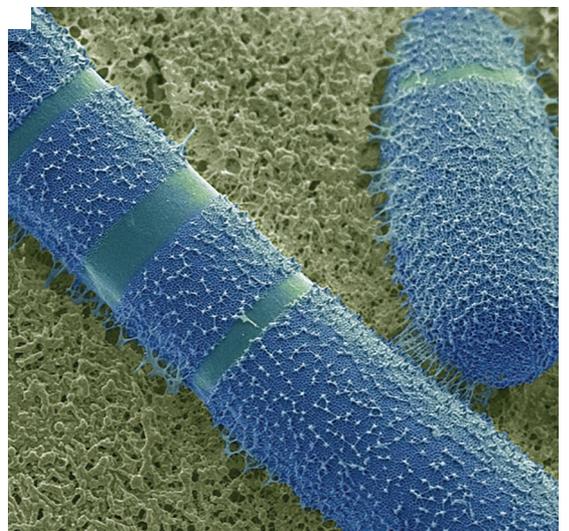
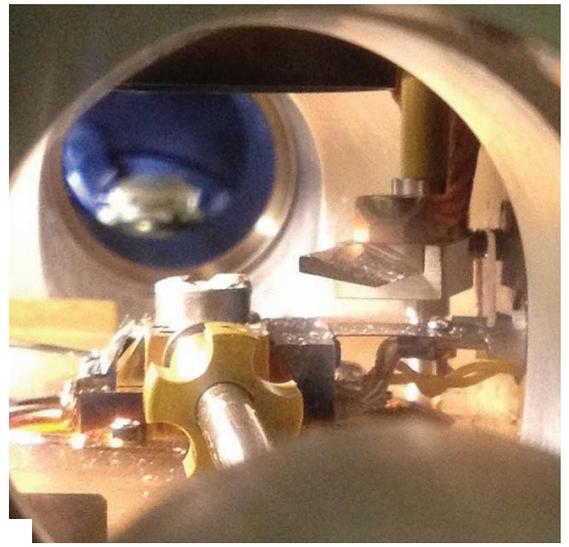


TECHNIQUES & APPLICATIONS

featuring the PP3010T Cryo-SEM Preparation System

www.emsdiasum.com

**Electron
Microscopy
Sciences**



What is...

Cryogenic Specimen Preparation?

In this instance we are referring to frozen hydrated bulk specimens for Scanning Electron Microscopy (SEM), commonly termed Cryo-SEM. When biological specimens are prepared by alternative methods, such as critical point drying, they may collapse and distort due to the removal of their water content. In addition diffusible elements are often removed or relocated, affecting the validity of subsequent X-ray microanalysis.

Cryo-SEM offers the best solution to this and in addition allows observation and analysis of liquid, semi-liquid and beam-sensitive specimens, such as emulsions, suspensions and foams. Cryo preparation is increasingly being used with FIB/SEM instruments for a wide range of specimen types, including some materials where low temperature milling conditions are desirable.

For biological and other “wet” specimens rapid freezing is essential in order to reduce morphological distortion, a key consideration for structural observation.

The aim of fast freezing is to reduce the size of ice crystals within specimens by reaching as quickly as possible the point at which recrystallization takes place (for pure water this is in the order of -130°C) and maintaining the specimen below this temperature during transfer, preparation and observation.

For larger specimens commonly used in SEM and FIB/SEM rapid freezing is normally done by plunging into liquid nitrogen in its ‘slushy’ form at -210°C. This is the standard method supplied with the PP3010T, but it is also fitted with an “advanced specimen handling” system which allows specimens that have been frozen by alternative (faster) freezing methods to be manipulated and loaded under liquid nitrogen and then transferred under vacuum into the PP3010T for subsequent processing and observation.

Cover Photos:



Soya Bean Leaf *Predatory Mite,* *Algae*
courtesy of
USDA Beltsville,
Dr Gary Buchan

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 90% of some animal and plant tissues it represents a most formidable specimen problem to most Microscopists.

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

Naturally the advent of various “high 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 has a number of additional advantages, including the ability to fracture and selectively remove surface water (ice) by controlled specimen sublimation.

Why choose cryo-SEM?

The limitations of conventional “wet processing” include:

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

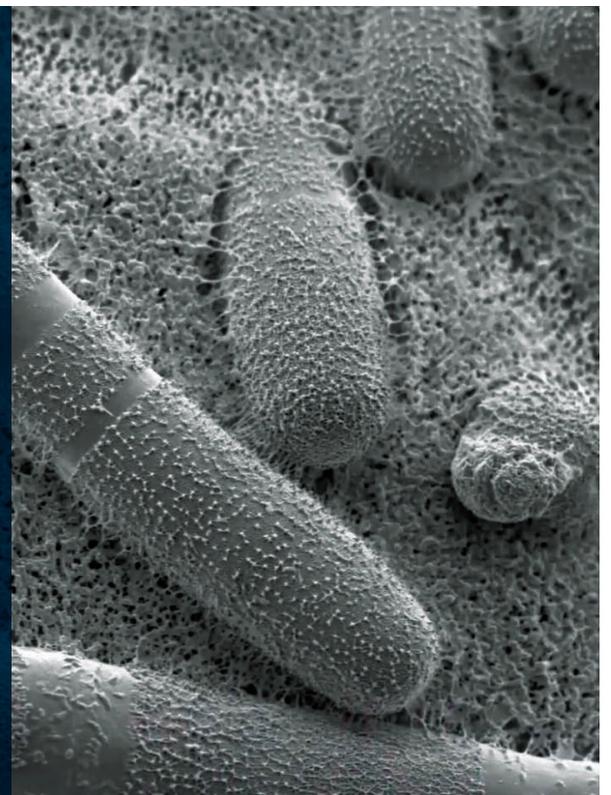
Advantages of cryo-SEM:

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

See how it works... Learn how to do it...

We've added video content to our website to help you get to know our latest products even better!

Stop by and see what it's all about.



PP3010T Cryo-SEM Preparation System

Quick Overview

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 sublimation and specimen coating. The specimen can then be transferred onto a highly stable SEM cold stage for observation. Cold trapping in the cryo preparation chamber and SEM chamber ensures the whole process is frost free. Specimen process times are typically between five and ten minutes.

Key Features

- High resolution performance
- Large “recipe” driven touch screen interface
- Easy to use — extensive automation, on-screen help, videos, data logging and diagnostics
- Column-mounted preparation chamber — essential for frost-free transfer and ease of use
- Cold stage temperature down to -190°C , plus comprehensive cold trapping (not possible with conduction cooling)
- Turbo pumping system mounted off-column — less mass on the SEM
- Unsurpassed specimen visibility — large front window, top viewing ports, multiple LED chamber lighting
- Cameras in the preparation chamber and SEM — cumbersome binocular not needed
- Automated start up, sublimation, and coating
- Fully compatible with SEM beam deceleration/stage bias modes up to 5kV
- Vacuum storage of the cryo transfer device
- Typical vacuum when cold: 10^{-6} mbar or better — specimen transfer into the SEM always high vacuum to vacuum
- Twin liquid nitrogen slushing and specimen handling system for pre-frozen specimens
- Fracturing/specimen manipulation device
- Prepdek™ workstation — self contained work area, extra bench space not required
- Specialized support backed up with a three-year warranty



Product Description

The PP3010T is a great leap forward in cryo-SEM technology. It combines the highest quality results with unparalleled ease of use.

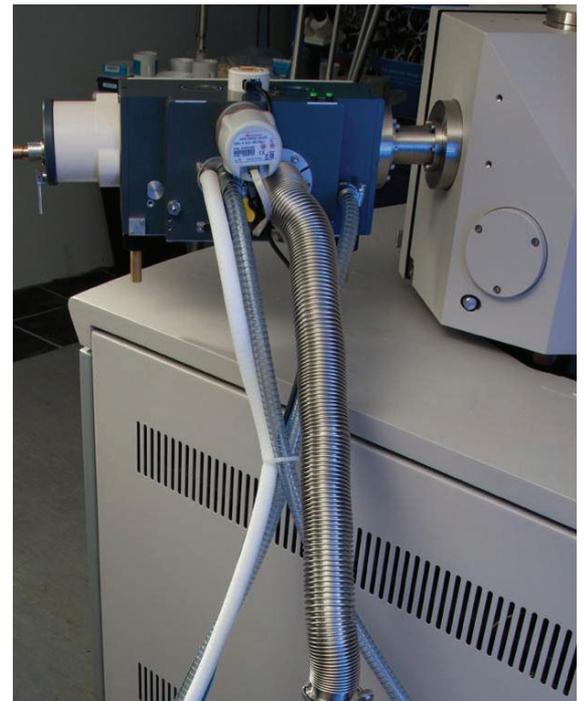
The PP3010T is a column-mounted, gas-cooled cryo preparation system suitable for use with SEM, FE-SEM and FIB/SEM instruments. Control is via a large and intuitive touch screen mounted on the spacious Prepdek™ workstation, giving the operator instant access to, and control of, all the key operating parameters.

Visibility is a key feature throughout the whole system. CCD camera images from the preparation chamber and the SEM are displayed on the control screen — the image can be expanded to full screen when required. Five preparation chamber viewing windows give unsurpassed visibility of the specimen and chamber interior.

On-column preparation chamber with off-column cooling and pumping

The PP3010T conveniently combines the advantages of what are often referred to as “on-column” and “off-column” cryo preparation systems. The preparation chamber is directly attached to the SEM, but with the turbomolecular pumping and advanced SEM cooling system mounted remotely from the SEM. In this way, the mass and volume attached directly to SEM is kept to a minimum.

There are significant advantages of having the preparation chamber attached directly to the SEM. In particular, specimen transfer is always from high vacuum to high vacuum, which greatly reduces the risk of specimen contamination (frosting). In addition, it makes the system easier to use and allows the operator a more flexible approach to specimen preparation and observation. This is because during a single processing run it may be useful to move the specimen between the preparation chamber and the SEM cold stage — and vice versa — on a number of occasions.



Remotely mounted turbomolecular pumping system

PP3010T Cryo-SEM Preparation System

Product Description (continued)

Prepdek™ workstation and touch screen user interface

The Prepdek™ workstation has been designed to allow specimen mounting, freezing (and 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™. Flexible LED lights give the user an excellent view of the preparation process.

Conveniently set into the Prepdek™ work surface is a pumped storage tube which allows the cryo transfer device to be stored under clean, dry vacuum conditions when not in use.

The control PC is mounted on a flexible arm and can be positioned to suit the user (eg angled towards the SEM operator during specimen observation — allowing key system parameters to be viewed at a glance).

The PP3010T is controlled using a 15" intuitive color touch screen, mounted on the user-friendly Prepdek™ workstation. The touch screen allows user-defined "recipes" to be rapidly entered and stored for instant future access. The screen can be set to suit different

operator levels and preferences — eg analog or digital vacuum measurements.

CCD camera images of both the preparation chamber and SEM cold stages are displayed and can be expanded to full-screen. Although many of the key steps in the specimen preparation process are automated (airlock pumping, sublimation, sputter coating, etc), further help is instantly available through user-friendly videos.

These guide the operator through the system set-up and then each specimen processing step in a concise and logical way.



Typical screen view during operation (with camera image minimised)

Handling and transferring specimens

The PP3010T Prepdek™ workstation is fitted with a slushy nitrogen freezing station, connected to the pumping system. Rapid freezing reduces ice crystal damage, which results in improved ultra-structural preservation.

For handling pre-frozen material, the Prepdek™ is also fitted with the Advanced Specimen Handling System, which allows specimens that have been frozen by alternative freezing methods (or stored field specimens) to be manipulated in liquid nitrogen and then transferred under vacuum into the PP3010T preparation chamber for subsequent processing and observation.

The vacuum transfer device is compact, vacuum-tight and has a convenient bayonet connection to the specimen shuttle to ensure rapid transfer. In line with the automatic design of the PP3010T, when the vacuum transfer device is located on the preparation chamber, the airlock is automatically pumped.

The PP3010T is supplied with universal 10mm specimen stubs with surface slots, holes and a flat area — useful for most specimen types, because the holes and slots can be used for liquids and to hold solid material for cross-section fracturing. Blank stubs are also included. A range of optional holders are available, including shuttles for large specimens and top-loading holders for high pressure freezing rivets and planchettes.

Cryo preparation chamber

The PP3010T preparation chamber is connected directly to the SEM and includes facilities for preparing all types of 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.

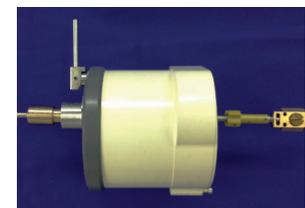
The stage has a dovetail fitting to accept a cryo shuttle and specimen and can be precisely controlled over a temperature range down to -190°C or lower. Large gas cooled cold traps located above and below the specimen stage ensure clean, high vacuum conditions in the chamber.



Specimen transfer device



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



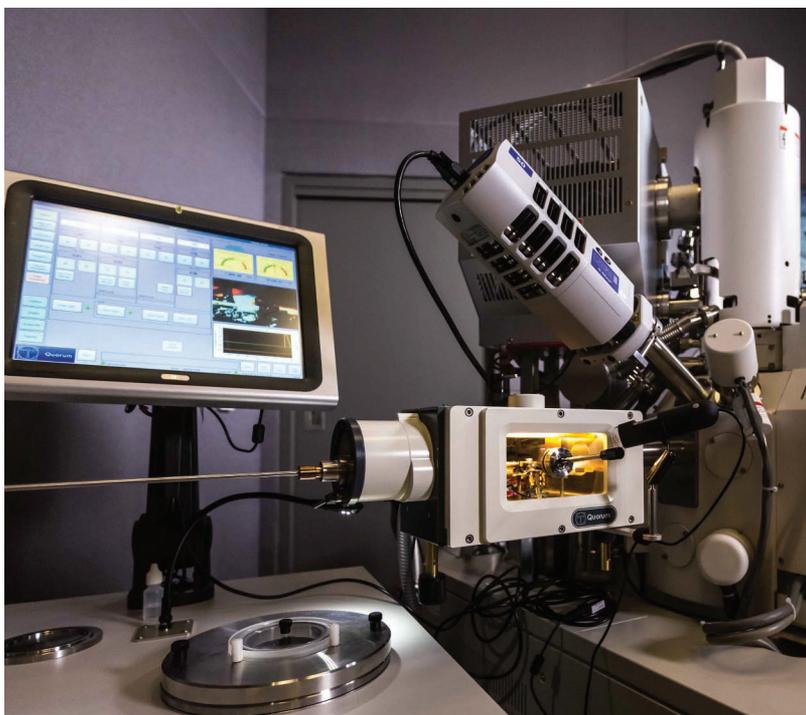
Transfer device, shuttle and universal specimen stub



Advanced specimen handling system



Transfer device



PP3010T Cryo-SEM Preparation System

Product Description (continued)

High visibility

The PP3010T has superb chamber visibility. In addition to the large front window there are additional top windows. The specimen stage is lit by three LEDs.

A CCD camera allows the specimen stage to be viewed on the control touch screen. Twin manipulators (actively cooled) are available and allow a range of specimen types to be fractured.

The PP33010T is fitted as standard with a front-mounted fracturing/manipulation device. The ball-jointed mount offers flexible movement of the blade and allows the scalpel to be used both as a surface pick (probe) and a fracturing knife.

An optional micrometer advanced fracturing tool (12145) is available (in addition to the standard side-mounted tool).

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

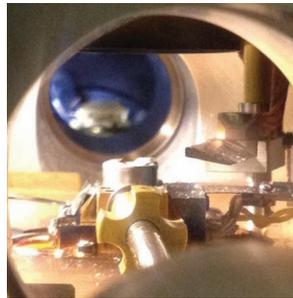
Automatic sublimation and sputtering

Sublimation and sputtering are fully automatic. The high resolution sputter coater is specifically designed for cryo applications and will give fine grain films that are essential for FE-SEM applications. A platinum (Pt) target is fitted as standard; other metals include gold (Au), gold/palladium (Au/Pd), chromium (Cr) and iridium (Ir). An optional carbon fiber evaporation head can be fitted.

An optional terminating film thickness monitor (FTM) is available. The system is fully integrated – no external control boxes.

Cryo preparation chamber pumping

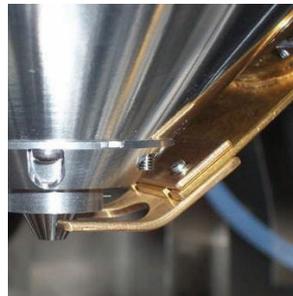
The preparation chamber is pumped by a remotely-positioned 70L/s turbomolecular pumping system. Typical preparation chamber vacuums during operation are in the region of 10^{-6} mbar or better. Positioning the turbomolecular pump away from the SEM ensures total elimination of mechanical vibration and significantly reduces the cryo system mass that is connected to the SEM. A vacuum buffer tank allows the rotary pump to be automatically switched off



View during specimen transfer



Front-mounted fracturing and specimen manipulation tool



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



Cryo preparation chamber with cryo transfer device fitted

for most of the time. The pumping system is connected to the preparation chamber by flexible stainless-steel bellows.

A 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.

SEM cold stage, cold trap and cooling system

A highly stable, thermally isolated, liquid nitrogen gas-cooled stage attaches to the SEM stage. The SEM stage and cold trap are cooled by separate cold gas circuits — both capable of reaching temperatures down to -190°C . 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 — a cold stage temperature of -175°C and a cold trap temperature of -190°C . The SEM cold stage has a temperature range down to -190°C and a temperature stability of $<0.5^{\circ}\text{C}$.

Off-column cooling

The cold nitrogen gas-cooling dewar for the SEM stage and cold trap is remotely positioned (typically on the floor behind the SEM). The system will run for up to 24 hours between fills.



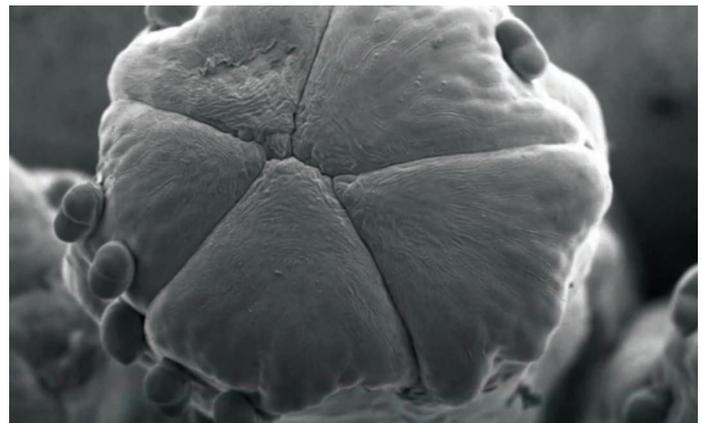
Nitrogen gas cooled cold stage – temperature range down to -190°C



Plunge freezing in slushy nitrogen.



Gas cooling dewar and turbo pump



Stamen from Yellow Mum

PP3010T Cryo-SEM Preparation System

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.	Yes
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

Control via a color user touch screen monitor (15") mounted on the Prepdek™	Yes
<ul style="list-style-type: none"> 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)

<ul style="list-style-type: none"> (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
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Installation and Training

Installation and training at the customer site	Contact EMS
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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

Ordering Information

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

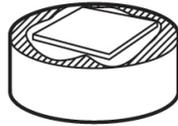
PP3010T	Cryo-SEM Preparation System for SEM, FE-SEM and FIB/SEM applications.	each
<i>Including: column-mounted cryo-preparation chamber with off-column turbo pumping system. SEM cold stage and cold trap, Prepdek™ workstation with dual freezing and specimen manipulation facilities, automatic sputtering and sublimation. Touch screen user interface mounted on the Prepdek™ workstation. Transfer device, (2) AL200077B specimen shuttles, (10) E7402 blank 10mm stubs, (5) E7449-5 multi-stubs 7mm high, (5) 11541 multi-stubs 5mm high, 20529 Dovetail holder shuttle, (100) 328116510 Brass rivets for fracturing liquids, (5) E7406 Copper (Cu) stub with 3mm x 3mm slot, (5) E7407 Copper (Cu) stub with 1mm x 3mm slot. Microscope interfaces, start-up kit, mounting media and operation manual</i>		
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		
PP7450/60L	Pressurized dewar (60L) for LN ₂ storage and venting gas supply	each
10920	Carbon fiber evaporation head including 1m high purity carbon fiber	each
12147	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-fracture "Balzers" planchette holder shuttle	each
20529	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	useful each
10247	Top loading rivet holder shuttle (vice style). Holds two rivets (see: 328116510)	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
11541	Universal specimen stub with holes and slots (pack of 5) (10mm dia. X 5mm high)	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
328116510	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 0.2mm thick	each
E7400-314B	Gold/palladium target 0.2mm thick	each
E7400-314C	Platinum (Pt) target 0.2mm thick (one included as standard)	each
E7400-314IR	Iridium (Ir) target 0.3 thick	each
E7400-314Cr	Chromium (Cr) target 0.3mm thick	each
C5421	Carbon fiber cord for use with optional 11920 carbon attachment (100cm)	each
C5421-10	Carbon fiber cord for use with optional 11920 carbon attachment (10m)	each

Techniques and Applications

Examples of specimen mounting techniques for cryo-SEM

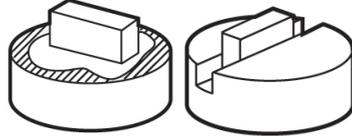
Surface mounting

This technique is used for leaf specimens etc. Roughen stub surface with fine emery paper. Specimen is laid on top of mounting media.



Edge mounting

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



Film emulsion mounting

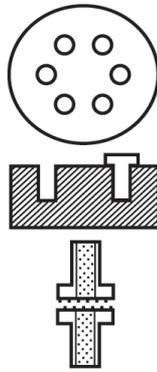
This technique is useful when a small specimen would be obscured by the Tissue-Tek mounting media, or when specimens need to be recovered. Specimens need to be slightly damp to use this method (good for nematode worms).



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

Rivet mounting

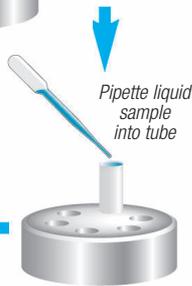
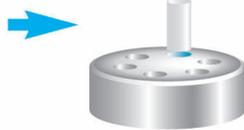
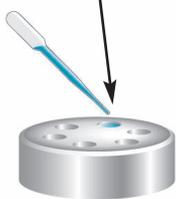
For liquids and for when specimens need to be frozen off the stub to achieve fast freezing rates. The rivet is placed in the hole and filled with liquid prior to freezing. If the specimen needs to be frozen away from the stub, two liquid-filled rivets are held together and then frozen prior to transfer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



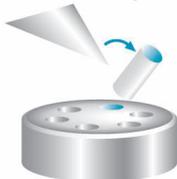
Alternative rivet mounting method

Pipette liquid sample into hole in sample stub

Place metal rivet or small piece of plastic tubing on top of hole (containing liquid sample).
Note: Small drop of "Super Glue" can be used to hold tube to stub.



Freeze & transfer onto preparation chamber cold stage

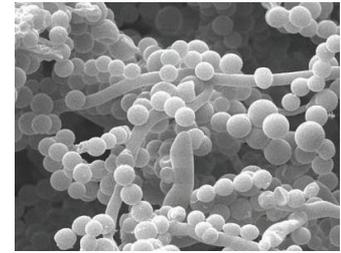


Result: clean surface fracture

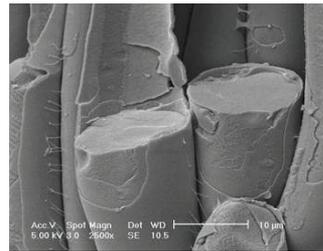
Cryo-SEM Micrographs



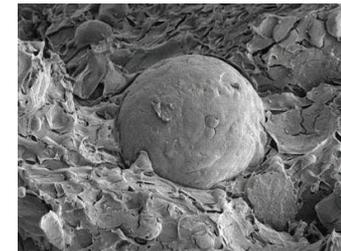
Cross-section of oil/water/rock.



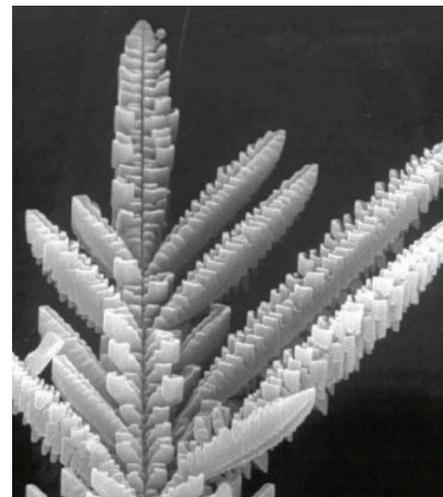
Cryo prepared image of blue stilton cheese (*Penicillium roqueforti*).



Cross-section through plant palisade cells.

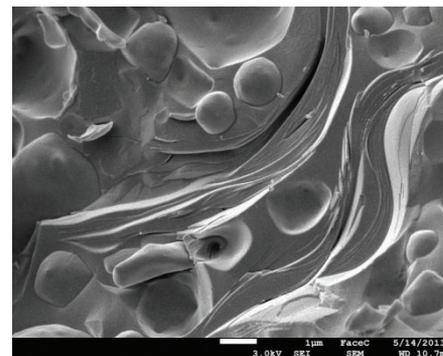


Cross-section image through sunscreen



Dendritic Ice Crystals

If it is cooled slowly, water forms dendritic ice crystals. These can have a variety of branching patterns — the complexity of which depends upon cooling rate. Arms extend from the main body of the crystal at an angle of 60°. Some, such as the one illustrated, resemble the arms of a snowflake. Bar: 2um



Face Cream

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

Specimen Transfer Systems

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...

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-retrofitable 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.



PP3004 QuickLok



Simple controls for specimen exchange

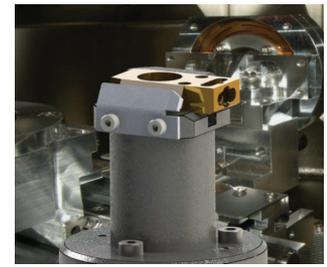


QuickLok and specimen transfer device

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

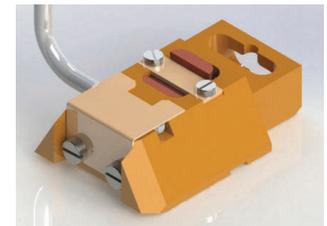
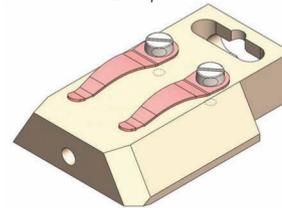
Pumping

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



QuickLok specimen stage and adaptor to SEM

Specimen Holder Examples



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

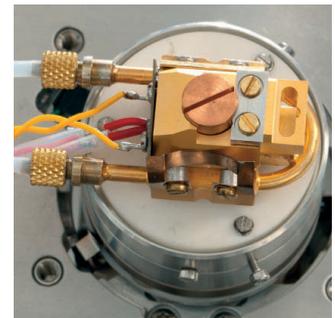
- Temperature range down to -190°C , with stability better than 0.5°C
- Off-column cooling with all-day runtime between fills
- Independent cooling of cold stage and cold trap
- Upgrade path to CoolLok
- 3 year warranty

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 stage and cold trap: A highly stable, thermally isolated, nitrogen gas-cooled cold stage attaches to the



Gas cooled stage



Controller and cooling system

Specimen Transfer Systems

PP3005 SEMCool (continued)

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 $<0.5^{\circ}\text{C}$. 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.

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

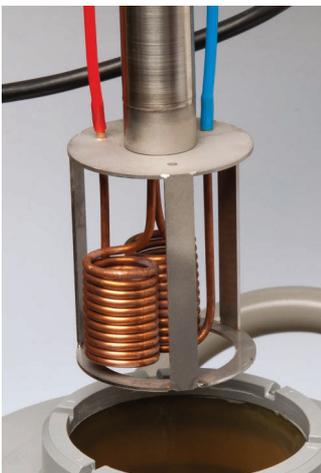
Vent the SEM, locate specimen holder on the cold stage, re-pump the SEM and then cool down to the required temperature. To exchange specimen, warm to above 0°C and vent the SEM.

Pumping

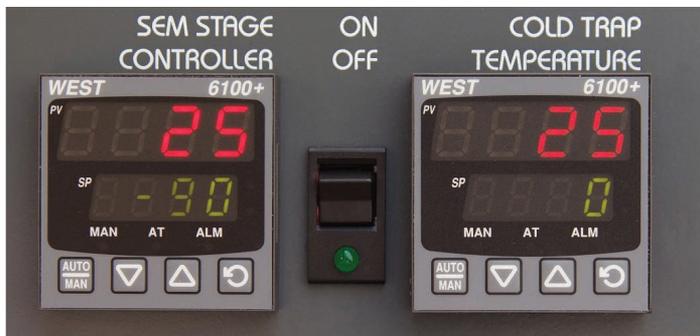
The SEMCool requires a rotary pump to periodically evacuate the vacuum isolated lines (see Ordering Information).



Cold trap - adapted to installation



Heat exchanger and dewar



Temperature controller

PP3006 CoolLok

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

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.

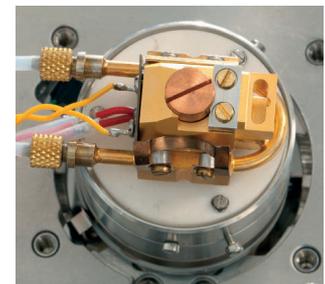
Key Features

- 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

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.



PP3006 CoolLok



Cold stage

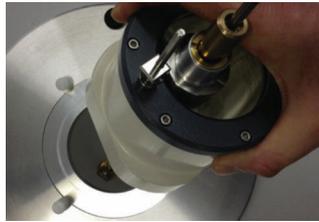


PP3006 installation example

Specimen Transfer Systems

PP3006 CoolLok (continued)

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.



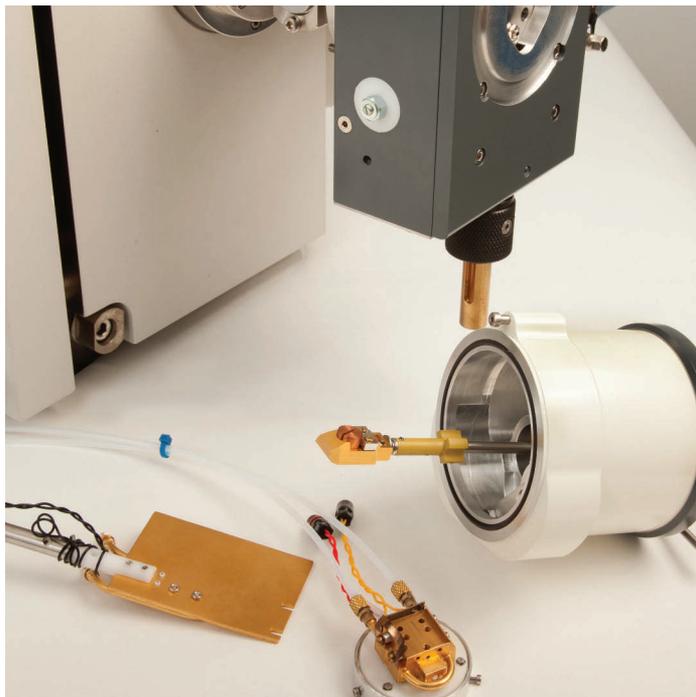
Plunge freezing in slushy nitrogen

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



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

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 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 $<0.5^{\circ}\text{C}$. For easy specimen exchange an LED chamber light is fitted.

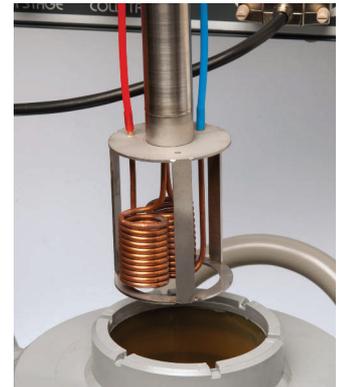


Cold trap - adapted to installation

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.

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.



Heat exchanger and dewar

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).

Specimen Transfer Systems

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
LN₂ 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 LN₂)	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 9 mm. Please contact us for more details.		

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

PP3004	<p>QuickLok Ambient Temperature 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 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 10 mm diameter specimen stub), blank 10 mm stubs – packet of 10</p>	each
PP3005	<p>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®)</p>	each
PP3006	<p>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</p>	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

Cryo-SEM Applications

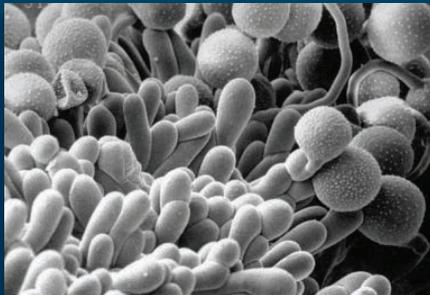
Zoological



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



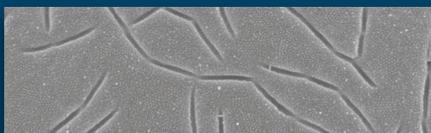
Pollen of cactus *Zygodactylus truncatus*

Germinating pollen grains of *Zygodactylus truncatus*.



Maize root starch granules

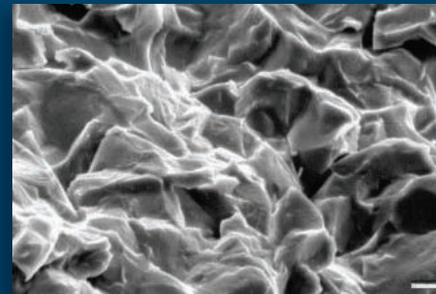
Fungi



Baker's yeast (*Saccharomyces cerevisiae*)

The specimen was rapidly frozen in nitrogen slush, fractured and coated with 4nm of platinum (Pt). 10nm yeast cell transmembrane particles (in hexagonal arrays) can be observed.

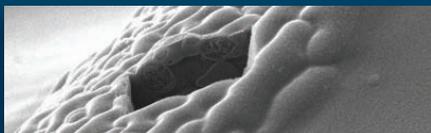
Geological



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.

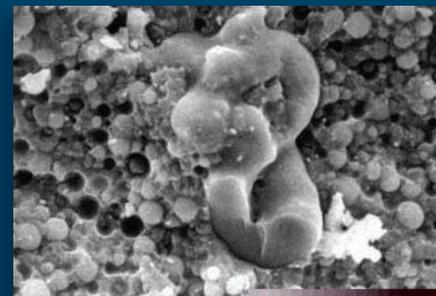
Cryo-DualBeam



Arabidopsis plant

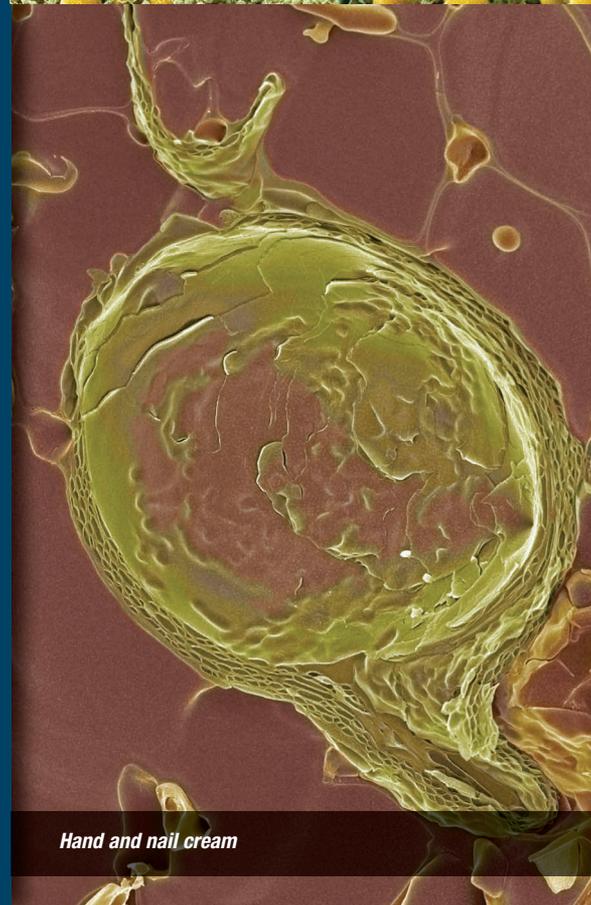
Cryo-FIB/SEM. Image courtesy of Hannah Edwards and Arabidopsis plants provided by Darren Wells, Centre for Plant Integrative Biology, School of Biosciences, University of Nottingham, UK.

Polymers



Stable emulsion of a hydrophobic polymer

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



Hand and nail cream

CONTACT US FOR MORE INFORMATION...

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look for us...

