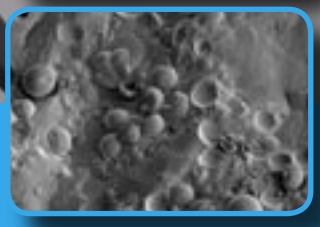
PP3000T

Cryo-SEM comes of age



- Recipe driven touch-screen interface
- Fully automated
- Highest performance on a wide range of specimens
- Superb specimen visibility
- Efficient cooling with all-day hold times
- Specialist support and three year warranty

Quorum Technologies

+44 (0)1273 815340
www.quorumtech.com

Why cryo-SEM?

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

Limitations of conventional 'wet' processing

- Shrinkage and distortion
- Relocation and extraction of soluble materials
- Mechanical damage (fragile specimens are easily damaged during critical point drying)
- For biological material toxic reagents are required (fixatives, buffers etc)
- Long processing times

Advantages of cryo-SEM

- Specimen viewed in its fully hydrated state
- Soluble materials are retained
- Little or no mechanical damage
- Ideal for time resolved experiments (biological and industrial)
- High resolution capability (compared to low-vacuum techniques)
- Extra information obtained by low-temperature fracturing
- Excellent for liquids, semi-liquids and beam sensitive specimens
- Rapid process – typically 5 - 10 minutes

PP3000T overview

The PP3000T is a "great leap forward" in cryo-SEM technology – combining the highest quality results with unparalleled ease of use.

The PP3000T is a column-mounted, gas-cooled cryo preparation system suitable for SEM, FE-SEM and FIB/SEM. 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 operating parameters.

Visibility is a key feature throughout the whole system. Chamberscope images from the preparation chamber and the SEM are displayed on the control screen. Five preparation chamber viewing windows give unsurpassed visibility of the specimen and chamber interior. The chamberscope window is protected by an automatic shutter to prevent build-up of sputtered material.

The PP3000T includes facilities needed to rapidly freeze and transfer specimens. The cryo preparation chamber has tools for cold fracturing and fully automatic controlled sublimation and specimen coating. Once prepared, the specimen can be transferred onto a highly stable SEM cold stage for observation. Efficient cold trapping in the cryo preparation chamber and SEM chamber ensures the whole process is frost free.

Specimen holders

The standard universal specimen stubs supplied with the PP3000T include surface holes, slots and a flat area – ideal for most applications. A range of additional holders is also available, including special top loading clamping holders for high pressure freezing rivets and planchettes.

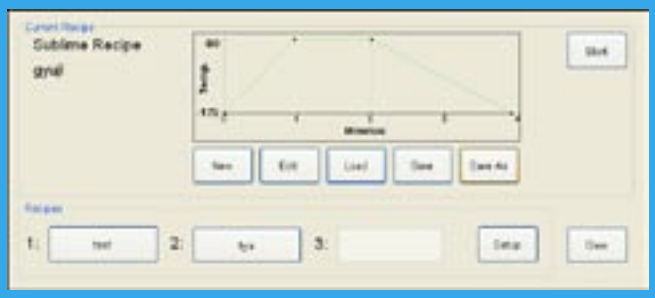
Prepdek™ workstation and touch-screen user interface

The PP3000T is controlled using an intuitive colour touch-screen, mounted on the user-friendly Prepdek™ workstation. The touch-screen allows user defined recipes to be rapidly entered and stored for instant access. The screen can be set to suit different operator levels and preferences (e.g. analogue or digital vacuum measurements). Chamberscope images of both the preparation chamber and SEM cold stages are displayed and at-a-touch can be expanded to fill the screen.

Although many of the key steps in the specimen preparation process are automated (airlock pumping, sublimation, sputter coating etc), further help is instantly available through user-friendly videos. These guide the operator through the system set up and then each specimen processing step in a concise and logical way.

Main display screen with all operational parameters visible to the user at all times

Password protected service screen gives the operator instant access to the system diagnostics



Sublimation parameters for temperature and time can be pre-set and stored

Sputtering parameters (time and current) can be pre-set and stored

Handling and transferring specimens

The PP3000T Prepdek™ workstation is fitted with a slushy nitrogen freezing station, connected to the pumping system. Rapid freezing reduces ice crystal damage which results in enhanced ultra-structural preservation.

For handling pre-frozen material, the Prepdek™ is also fitted with the Advanced Specimen Handling System. This allows specimens that have been frozen by alternative freezing methods (or stored field specimens) to be manipulated in liquid nitrogen and then transferred under vacuum into the PP3000T preparation chamber for subsequent processing and observation.

The vacuum transfer device is compact, vacuum tight and has a convenient bayonet connection to the specimen shuttle to ensure rapid transfer. In line with the automatic design of the PP3000T, when the vacuum transfer device is located on to the preparation chamber, the airlock is automatically pumped.

Cryo transfer device with specimen shuttle and stub insert

Cryo preparation chamber

The PP3000T preparation chamber is connected directly to the SEM and includes facilities for preparing a wide range of specimens.

Specimen stage cooling is by an integral liquid nitrogen dewar which has an all-day run time on a single fill (0.75L) of liquid nitrogen. The cold stage is connected directly to the dewar and includes a heater and sensor to precisely control the temperature from >+50°C to -190°C. Comprehensive cold traps surround the cold stage.

Cryo preparation chamber

High visibility

The PP3000T has superb chamber visibility. In addition to the large front window there are additional top and side windows. The specimen stage is lit by four LEDS.

A chamberscope allows the specimen stage to be viewed on the control touch-screen.

Twin fracturing manipulators (actively cooled) allow a range of specimen types to be fractured.

Preparation chamber front window

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 target is fitted as standard; other metals include Au, Au/Pd, Cr and Ir. An optional carbon fibre evaporation head can be fitted.

Off-column turbo-pumping system

Cryo preparation chamber pumping

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

The pumping system is connected to the preparation chamber by a stainless steel bellows.

SEM cold stage

SEM cold stage and 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 two separate cold gas circuits – both capable of reaching temperatures down to -192°C. This configuration allows the operator to select stage and cold trap temperatures that are optimized for specific specimens.

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.

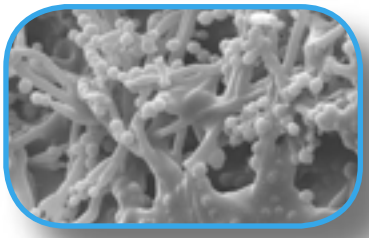
Off-column cooling system with 24 hour hold time

PP3000T Specification

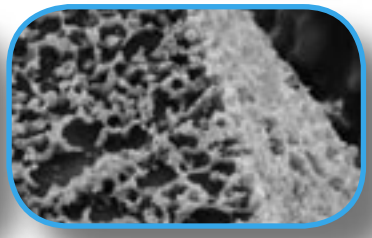
Inside the SEM:	Nitrogen gas cooling – rapid specimen stage cooling after sublimation, possible with conduction cooling Independent cooling of the SEM cold stage and cold trap – flexible, efficient cold trapping Stage and cold trap cooling down to -192°C – not possible by conduction cooling SEM chamberscope and LED illumination – excellent visibility for specimen exchange
SEM cooling:	Remote, off-column 12 litre cooling system with over 24 hour hold time – fill and forget, overnight operation possible
Column mounted preparation chamber:	Cold stage -190°C to +60°C Cold stage parking space for a second specimen shuttle – faster specimen processing Comprehensive cold trapping – clean contamination-free operation Multiple LED illumination – clear view of specimen with no shadows during fracturing Actively cooled, micrometer driven fracturing knife and probe – side mounted probe can be fitted with a range of scalpel blades to suit different specimen needs Chamberscope – large view of specimen on the display screen, cumbersome binocular not needed Non-boiling cooling dewar with all-day run time – fill and forget Large front window (138 x 73mm) plus top and side viewing ports – excellent specimen visibility Automated sublimation via user recipes – optimum control of this important process Automated, low energy sputter coater – fine grain, high resolution coating Shuttered window – chamberscope port is automatically shielded during coating to ensure the window remains free of sputtered metal Fully interlocked valve system with status lamps – safe operation Film thickness monitor (option) Carbon fibre evaporation attachment (option)
Preparation chamber pumping system	Floor-mounted turbo pumping with anti-vibration block and stainless steel vacuum connection to the preparation chamber – reduced mass on the SEM Base vacuum 10^{-6}mbar or better – high vacuum compatibility with SEM chamber Integral buffer tank – allows rotary pump to be automatically switched off for most of the time Single 90 L/m rotary pump required, dry pump available
Touch-screen control – user definable recipes and operator levels	Multi-ability user interface screen – ideal for beginners and experienced users Quick overview of system status – all the important operating data can be seen "at a glance" User defined "recipes" can be stored – reproducible results from a range of specimen types Quick access to videos outlining preparation techniques and system maintenance – instant help at every step
Dual slusher PrepDek® specimen preparation station:	Twin liquid nitrogen slushing and specimen handling system – ideal for handling pre-frozen specimens

Food science

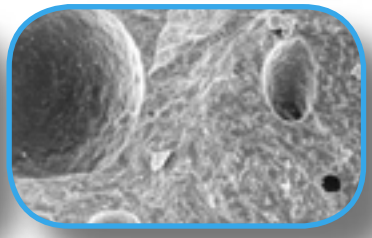
Cryo-SEM has for many years been an important technique in food science. Microstructure has a direct influence on the taste, texture and consumer preference.



Penicillium roqueforti fungi *in Blue Stilton cheese*

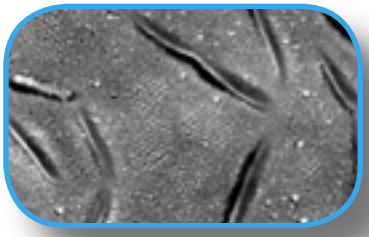


Yoghurt. *Fractured, sublimated and platinum coated*



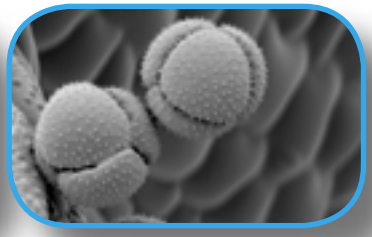
Ice cream. *The PP3000T Prepdek™ is fitted with a specimen handling system which allows pre-frozen material, such as ice cream, to be manipulated and then transferred under vacuum into the cryo preparation chamber*

Biology

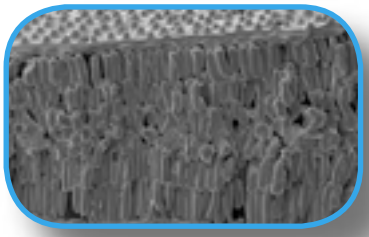


Yeast. *Intramembranous particles are around 9nm across and arranged in characteristic hexangonal arrays. Specimen cold fractured and sputtered with 4nm of platinium*

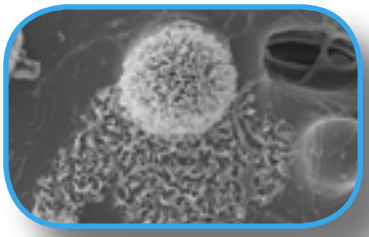
Mycorrhiza *(plant root fungi). In a mycorrhizal association, the fungus colonizes the host plant root system. Mycorrhiza are an important component of soil life and soil chemistry*



Sundew pollen *(Drosera adelae)*



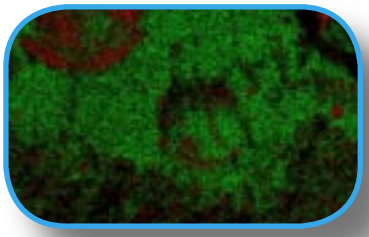
Cross-section through a leaf of ***Euphorbia myrsinites*** *(donkey-tail spurge)*



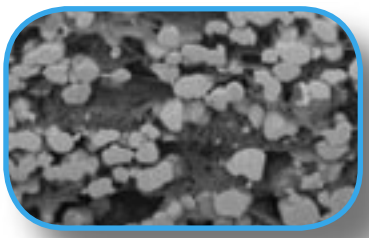
Wax-producing cells and wax on the Euphorbia leaf surface (wax is often extracted during critical point drying processing)

Materials

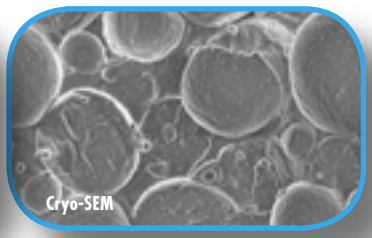
Oils and rocks. *Chalk rock saturated with brine and oil. Dark grey bubbles are dried oil droplets in brine-filled cracks*

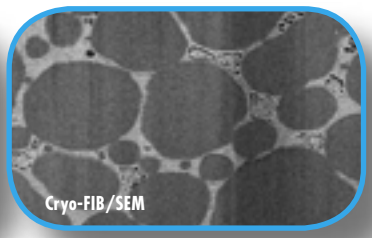


X-ray microanalysis overlay of the same specimen is used to further differentiate oil from water content (green: oxygen, red: carbon)



Mixed hardness material
Cryo-FIB/SEM section of a ceramic-filled, laminated polymer. A combination of hard and soft materials FIB-cut without damage or distortion. 2kV secondary electron image.





Cosmetic foundation cream *(oil-water emulsion)*
Comparing a cryo-SEM prepared specimen with cryo-FIB/SEM. Both images show large oil droplets, with the cryo-FIB/SEM image clearly demarcating the water phase (light areas) and additives (dark areas within the ice)

Images courtesy of Liverpool John Moores University, the Centre for Advanced Microscopy at the University of Reading, the University of York, the University of Stavanger, FEI Company and Nitto Analytical Techno-Center

Ordering Information

NB: For a full quotation, including on-site installation and customer training, please contact us or our local distributor

PP3000T		Cryo-SEM preparation system for SEM, FE-SEM and DualBeam applications. Including: column-mounted cryo-preparation chamber with off-column turbo-pumping system. SEM cold stage and cold trap, Prepdek™ workstation with dual freezing and specimen manipulation facilities, touch-screen user interface mounted on the Prepdek™ workstation. Transfer device, 2 x AL200077B and 1 x 10246 specimen shuttles, E7449-5 and E7402 specimen stubs. Microscope interfaces, start up kit and operation manual.
Pumping		The PP3000T requires one 90L/m rotary pump (dry pumps available on request)
	EK3180	RV5 90 L/m 115/230V 50/60Hz vacuum pump with oil mist filter
Options and accessories	**PP7450**	Pressurised dewar (30L) for LN_2 storage and venting gas supply
	PP10998	Carbon fibre evaporation head including 1m high purity carbon fibre
	PP10999	Film thickness monitor (FTM)
Specimen holders	**10245**	Top loading specimen shuttle for planchettes
	10246	Top loading specimen shuttle, to take a 10mm stub
	10247	Top loading specimen shuttle for rivets (vice style)
	E7433	Rivet holder specimen stub, screw-down style (for use with 10246)
	E7449-5	Universal specimen stub with surface holes and slots (pack of 5)
	AL200077B	Specimen stub shuttle (spare)
	E7402	Aluminium (Al) stubs (pack of 10)
	E7403	Copper (Cu) stubs (pack of 10)
	E7405	Screw down stub for thin hard specimens
	E7406	Copper (Cu) stubs with 3 x 3mm slots (pack of 5)
	E7407	Copper (Cu) stubs with 1 x 3mm slot (pack of 5)
	32816510	Brass rivets for fracturing liquids (pack of 100)
Sputtering targets	**E7400-314A**	Gold (Au) target 0.2mm thick
	E7400-314B	Gold/palladium (80:20%) target 0.2mm thick
	E7400-314C	Platinum (Pt) target 0.2mm thick
	E7400-314IR	Iridium (Ir) target 0.3mm thick
	E7400-314CR	Chromium (Cr) target 0.3mm thick

Distributed by:

Quorum Technologies Ltd
2 Acorn House, The Broyle, Ringmer, East Sussex BN8 5NN
T: +44(0)1273 815340 F: +44(0)1273 813439
E: sales@quorumtech.com www.quorumtech.com