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DATASHEET

High temperature stage for Environmental SEM (ESEM)

Enabling *in situ* experimentation to 1000°C or 1400°C

A total turnkey solution for dynamic *in situ* heating studies includes the combination of Thermo Scientific ESEM platforms with Thermo Scientific heating substages.

Important parameters such as specimen heating, sample outgassing/contamination, environmental gas delivery, detection and image/movie capture are all well supported.

With an easy to install substage-module design, heating stages allow the operator to heat bulk samples up to 1000 °C or 1400°C in a specific gas environment. Samples of up to 5 mm diameter can be investigated inside a replaceable magnesium oxide ceramic crucible. Oxidation reactions are easy to drive with the standard water vapor environment. Other gases, including inert gases, can also be used with the auxiliary gas input. Along with accurate control of the gas environment, heater feedback and control is fully integrated in software. Both pre-defined experimental cycles and manual control are standard; yielding flexibility for unknown discovery as well as use for repeatable cyclic material comparisons.

Key Thermo Fisher Scientific technologies make the system robust for demanding heating experiments linking sample morphology, environment and thermodynamics. These enable controlling temperature of bulk materials and maintain imaging even with bulk sample outgassing. Uniform temperature is achieved by the heater's "micro-furnace" design (heating from the bottom and sides with over-sample heat shields to reflect the heat back to the sample surface). Temperature control can be calibrated for the thickness and thermal properties of investigated materials. Outgassing is mitigated with low vacuum gas flow that dilutes and carries evolved gas through to the pumps so that imaging is consistent and reliable. Chamber gas participates in image detection through the Thermo Fisher revolutionary gaseous secondary electron detector. Detector design yields better resolution (low beam skirting, better beam coherence) and signal collection that is insensitive to light or heat.

Thermocouple readings are integrated with image data so each frame is time-stamped and temperature-logged in addition to the normal TIFF data. The Thermo Scientific™ ESEM™ platforms have the ability to save individual TIFF in series or direct AVI files with the built-in recording functionality. AVI time can be compressed and a databar data adjusted each time a movie is built from a TIFF series. This yields both publication-quality TIFF images and movies from a single experiment. With the total solution, everything can be done in one go with no data loss.

Integrated Thermo Fisher heating stages are flexible for many experimental needs, allowing researchers to easily see things never seen before at high resolution; ensuring the best results with the widest range of sample types.

Key benefits
Easy operation from room temperature to highest temperatures
Ability to easily drive and visualize oxidation reactions
Detection of signal is insensitive to light; utilizing patented gaseous detection technologies
Continuous imaging during heating of bulk samples from ambient to 1400°C
Flexible recording of *in situ* experimentation
Ability to do EDS analysis before and after an experiment with optional EDS

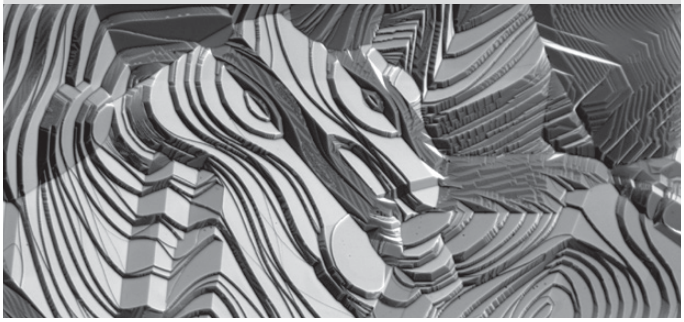

Figure 1. Image of a previously flat copper surface after heating in an oxidizing atmosphere illustrates grain re-arrangement. This data is possible to view live with the combination of ESEM™ and heating.

	1000°C	1400°C
		
Kit includes	1000°C module, mounting base and consumable starter kit	1000°C module, two 1400°C modules, heat shield swing arm, mounting base, manual user interface (for control of shield and sample bias) and consumable starter kit
Sample size	5 mm diameter x 5 mm height	5 mm diameter x 5 mm height
Temperature range	40 – 1000°C	200 – 1400°C for 1400°C modules 40 – 1000°C for 1000°C module
Temperature setting precision	1°C	1°C
Temperture stability	±5°C	±5°C
Heating rate	Standard software limit: 1 - 50°C/minute; Non-standard maximum possible: ~300°C/minute (service limit override required, decreases heater lifetime and eliminates any lifetime guarantee)	Standard software limit: 1 – 50°C/minute; Recommended: 10°C/minute above 1000°C. Non-standard maximum possible: ~300°C/minute (service limit override required, decreases heater life time and eliminates any lifetime guarantee)
Heater lifetime	100 hours guaranteed under standard conditions, virtually limitless for most use cases	1400°C modules: Lifetime is limited and depends strongly on the working temperatures. 4 hour guaranteed at maximum temperature for set of two modules
Cooling	Recirculating water flow from small chiller	Recirculating water flow from small chiller
Calibration accuracy at 400 Pa	±25°C @ 962°C (Ag melting point)	1400°C modules: ±25°C @ 1064°C (Au melting point) 1000°C module: ±25°C @ 962°C (Ag melting point)
Pressure range	Low vacuum & ESEM	Low vacuum & ESEM
Gas type	Water vapor, nitrogen, air, other gasses (restrictions apply, options may be needed)	Water vapor, nitrogen, air, other gasses (restrictions apply, options may be needed)
Standard detector	Ceramic GSED or LFD (large field detector)	Ceramic GSED or LFD (large field detector)
Thermocouple	K-type for 1000°C module	K-type for 1000°C module B-type for 1400°C module
Known sample incompatibilities	Corrosive substances when heated, free silicon	Corrosive substances when heated, free silicon
Usage	Ease of use, routine microscopist can master	Expert microscopist, user training and practice required
Heat shield type	Fixed; needed above 400°C	Swing-arm-design with integrated bias control

Essential specifications

Technical highlights

- Stage is available for 1000°C and 1400°C for use in ESEM systems
- Temperature ramp up to 50°C/minute are possible
- Temperature accuracy ±25°C
- Support of auxiliary gas environments is standard in FEG systems (optional in tungsten systems which require the Auxillary Gas Kit)
- 1400°C version includes control for sample bias and heat shield bias for advanced control at extreme temperatures

Software and control

- Software is integrated into the microscope user interface
- Multiple set-points, ramp and soak times can be programmed
- Capacity to hold and restart a sequence or to proceed to the next step
- Manual control of heater power is available
- Movie acquisition is built in to the Quanta user interface for both direct AVI or sequential TIFF acquisition
- Software for combining individual TIFF files into a movie is provided with user defined timing and databar composition

Hot stage detector

- Gas-ionization detection using proven Thermo Scientific technology
- Signal collection is insensitive to light or heat
- Integrated ceramic heat shield
- Beam protecting cone is integrated to extend the pumped intermediate vacuum zone, minimizing beam skirting
- Polymer coupling to the final lens disconnects, reducing/avoiding collision damage
- Detector is robust can be easily cleaned

Figure 2. Hot stage detector.

Installation requirements

The Thermo Scientific heating substages are compatible with all Thermo Scientific ESEM platforms.

Port requirements

- 1000°C: one port; hot/cold feedthrough
- 1400°C: two ports;
  - – hot/cold feedthrough
  - – swing arm heat shield

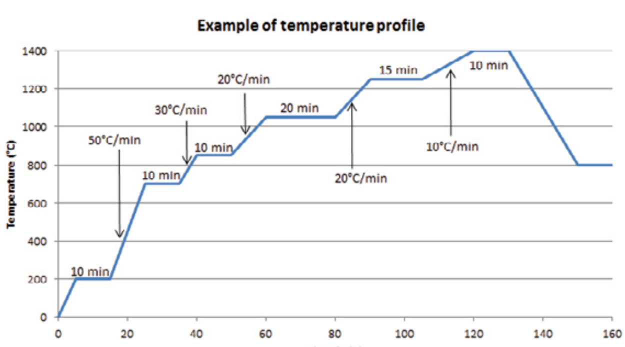

Figure 3. Example of temperature profile.

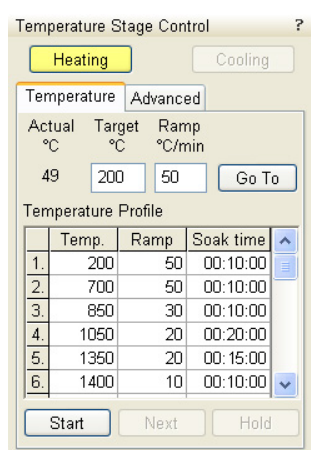

Figure 4. Software controls for heating program and manual heater control.

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Application images with the 1000°C module

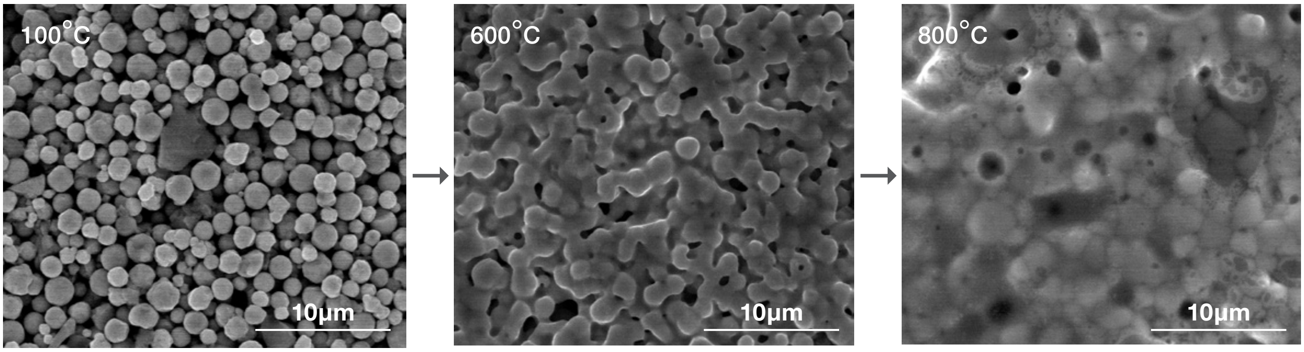

Figure 5. Silver paste used to make finger electrodes for solar cells. *Courtesy of Hiroshi Fujitani, Thermo Fisher Scientific NanoPort, Japan.*

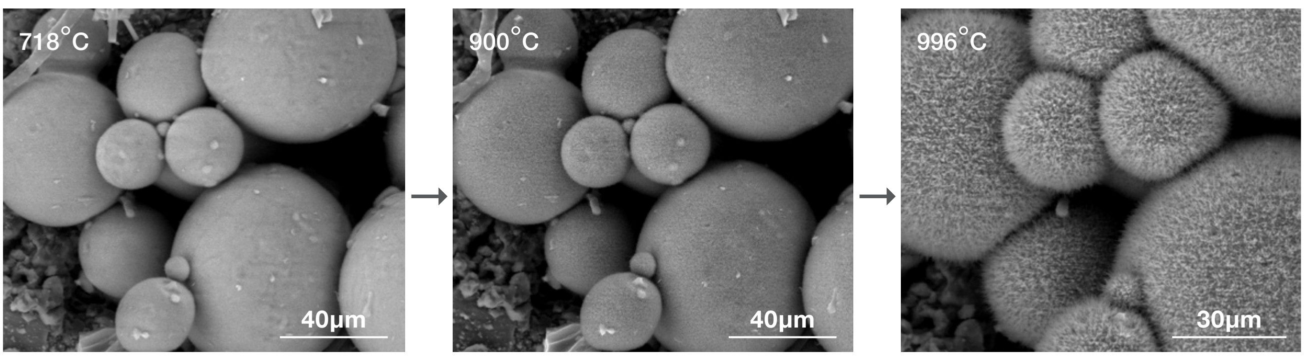

Figure 6. *In situ* heating of metal spheres allows monitoring of surface morphology. development. *Courtesy of Daniel Phifer, Thermo Fisher Scientific NanoPort, Hillsboro USA.*

Application images with the 1400°C module

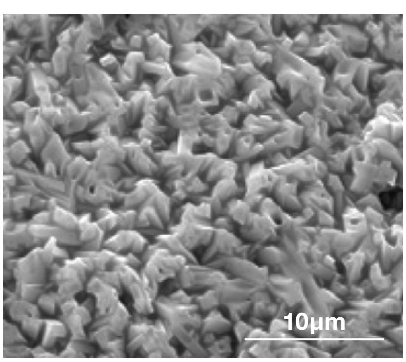

Figure 7. Superconducting alloy at 1100°C. *Courtesy of Professor Wei Li, Shanghai Jiaotong University and Wendy Chen, Thermo Fisher Scientific NanoPort, China.*

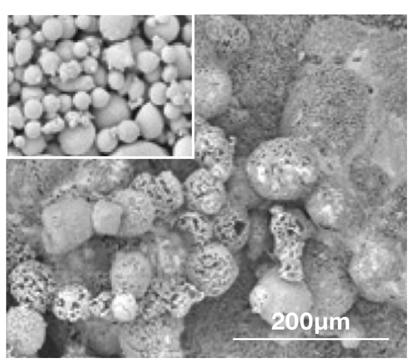

Figure 8. Metal-glass compound before (inset) and after heating to 1200°C in an inert imaging gas. *Courtesy of Ellen Baken, Thermo Fisher Scientific NanoPort, The Netherlands.*

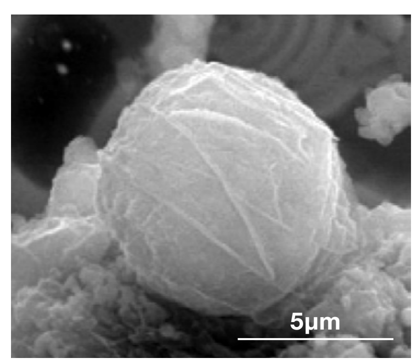

Figure 9. Bulk refractory spheres grown by heating *in situ* to 1400°C and cooling/holding at 800°C. *Courtesy of Professor Daoyuan Yang, Zhengzhou, University and Wendy Chen, Thermo Fisher Scientific NanoPort, China.*

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