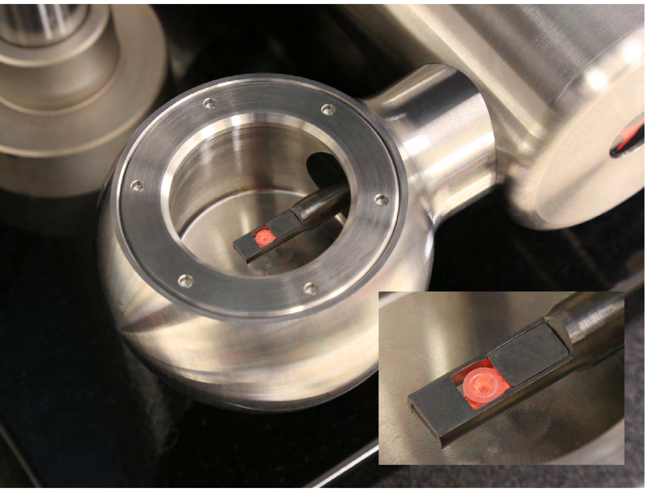
TEM Bulk Sample Heating

Technical Specs

1500 Series	ETEM	TEM
Max Operating Temperature	800°C	1000°C
Settled resolution at 800°C	Up to TEM resolution	Up to TEM resolution
Temperature Measurement	Direct Thermocouple	Direct Thermocouple
Tilt Range	Up to ±45° depending on objective pole	Up to ±45° depending on objective pole
Cooling at High Temperatures	Passive Conduction (No Liquid Cooling)	Passive Conduction (No Liquid Cooling)
Furnace Material	Non-Magnetic and Chemically Inert	Non-Magnetic and Chemically Inert
TEM Compatibility	FEI, JEOL	FEI

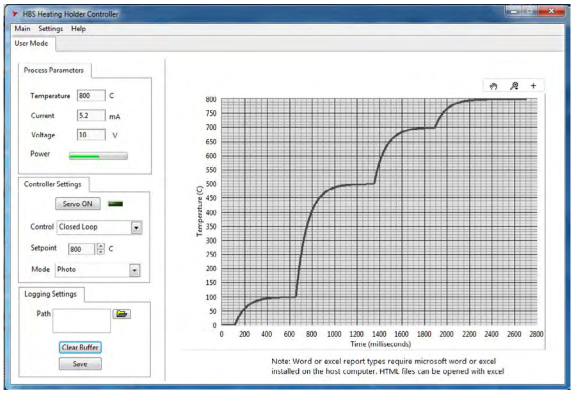
How It Works



Hummingbird Scientific's in-situ heating holder uses materials and geometries chosen for the best possible mechanical and thermal performance. The system is designed to minimize thermal drift. Its lower power consumption ensures a quick heater response and long heater lifetime. Our holder does not require water cooling, which allows for shorter image stabilization rates and better imaging performance above 500℃. In addition to a traditional TEM environment, our heating holder is designed to stand up to challenging ETEM experiments. The system features:

- Non-magnetic furnace material (critical for aberration-corrected experiments)
- Direct thermocouple measurements of sample temperatures
- Low thermal drift after stabilization
- Closed-loop control software

Software



- Sample temperature control in both closed loop and open loop mode
- Full data-logging and display capabilities
- High-resolution photo mode

Our heating holder uses a stand-alone heating control box which, when hooked up to a computer, provides both heating control and data collection. The control box interfaces with a custom-built Hummingbird software package.

In closed-loop mode, the system automatically ramps at a user-defined rate to the temperature set point and maintains the designated temperature. The built-in thermocouple guarantees an accurate temperature reading. This mode ensures a quick ramp-up. In open-loop mode, researchers can set the power to the heating element directly, while reading the temperature independently. This mode ensures highly accurate manual control. Photo mode temporarily minimizes changes to the element for maximum image stability.

Accessories

Accessories available for your heating holder:

- High-temperature Moly grid, mesh, with carbon film
- Moly grid, mesh, with SiO film
- Moly grid, Slotted
- 100/200 line, mesh, copper or gold
- Moly Washer (different geometries available)
- Slotted grid, copper

Maintenance Plan

Our heating holder was developed for accuracy and high reliability, but as with all resistive heating elements, occasional service is required after extended use of the heating element. In order to ensure that your holder is available for your research whenever you need it, we offer a optional maintenance plan. By purchasing this plan, you can prevent unexpected service costs and extended downtime. You will also receive free updates to the control software for the duration of the service contract.

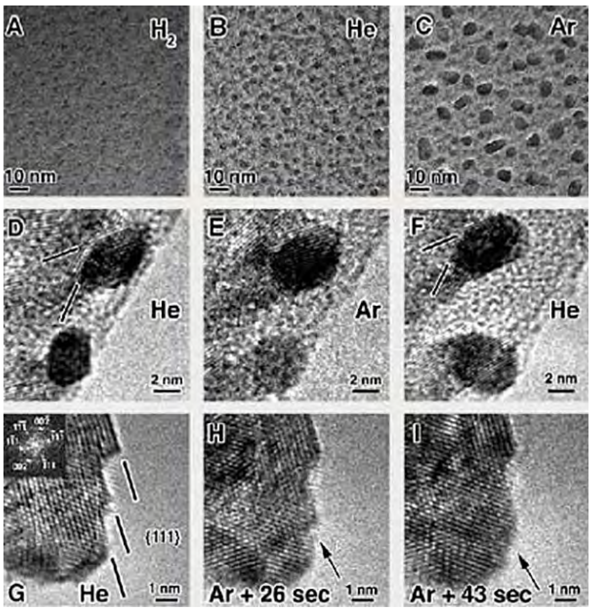
- All service included
- Guaranteed quick repair times
- Free software updates

Application Example

In-situ TEM Catalysis

One of the most exciting and promising areas of TEM-based research is the in-situ observation of materials interacting with gaseous environments. This research includes studies into the effect of gaseous over-pressures on the shape, structure, defects, and electronic activity of a material. It is perhaps most critical to the study of catalysis because it allows researchers to observe on the atomic scale how catalysts respond to their environment while they are active.

Below: Microstructural changes as a function of the gas environment of an Fe catalyst, shown in sequential high-resolution transmission electron micrographs:



(**A** to **C**) Size evolution of Fe catalysts after 60 min under H2 (A), He (B), and Ar (C) at 500°C and 500 mtorr.

(**D** to **F)** Series of images from the same two Fe catalyst particles held at 500°C, as the gas overpressure changes from (D) 500 mtorr He to (E) 500 mtorr Ar to (F) 500 mtorr He.

G to **I**) Series of images from a larger Fe catalyst particle along a 110 zone axis:

(**G**) Image taken in 500 mtorr He at 500°C, showing very strong {111} facets. The inset diffractogram confirms the zone axis orientation.
(**H**) After the introduction of Ar, local degradation of the facets begins.

(**I**) With further time at 500°C in the Ar environment, the facet has been completely removed.

For all cases, the H2O with base pressure of 10-2 mtorr is present. Arrows in **(H)** and **(I)** indicate the gradual defaceting features over time.

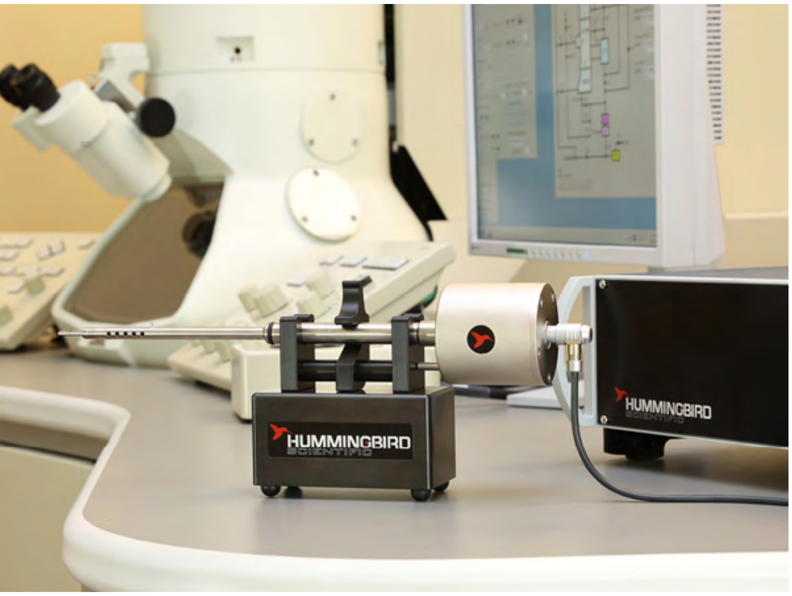
Reference: A.R. Harutyunyan, G. Chen,T.M. Paronyan, E.M. Pigos, O.A. Kuznetsov, K. Hewaparakrama, S.M. Kim, D. Zakharov, E.A. Stach, G.U. Sumanasekera. **“Preferential Growth of Single-Walled Carbon Nanotubes with Metallic Conductivity,”** Science 326 (2009) pp. 116–120.

Available For

FEI TECNAI/TITAN/CMX00 SUPER TWIN, X-TWIN, ULTRA-TWIN

JEOL 2010/2100/ARM, HR/ARP POLE, GRAND ARM

Product Summary



Hummingbird Scientific's in-situ bulk heating holder (1500 series) for the transmission electron microscope allows researchers to observe and characterize material behaviors and microstructures at elevated temperatures. The holder is designed to ensure high-resolution imaging across its entire temperature range, and it can support both traditional TEM and environmental TEM (ETEM) applications. The system has been thermally optimized for low power input and minimal sample drift and settling time in order to minimize test durations.

The holder's electronic interface, which controls all holder functions and data collection, features both closed-loop and open-loop temperature control for maximum flexibility and accuracy.

Sample Applications:

- Phase transformations
- High-temperature microstructural changes
- In-situ studies of gas-solid reactions (with ETEM)
- Catalysis materials studies

Selected Publications

L. He, J.P. Chu, C.L. Li, C.M. Lee, Y.C. Chen, P.K. Liaw, P.M. Voyles. **"Effects of Annealing on the Compositional Heterogeneity and Structure in Zirconium-Based Bulk Metallic Glass Thin Films,"** Thin Solid Films 561 (2014) pp. 87-92

S.M. Kim, S. Jeong, and H.C. Kim. **"Investigation of Carbon Nanotube Growth Termination Mechanism by In-situ Transmission Electron Microscopy Approaches,"** Carbon Letters 14:4 (2013) pp. 228-33

S.M. Kim, C.L. Pint, P.B. Amama, R.H. Hauge, B. Maruyama, E.A. Stach. **"Catalyst and catalyst support morphology evolution in single-walled carbon nanotube supergrowth: Growth deceleration and termination,"** Journal of Material Resolution 25 (2010) pp. 1875

A.R. Harutyunyan, G. Chen, T.M. Paronyan, E.M. Pigos, O.A. Kuznetsov, K. Hewaparakrama, S.M. Kim, D. Zakharov, E.A. Stach, G.U. Sumanasekera. **"Preferential Growth of Single-Walled Carbon Nanotubes with Metallic Conductivity,"** Science 326:5949 (2009) pp. 116-120

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