# HUMMINGBIRD

## **TEM Gas Flow**



	1300 Series - Single Channel	1300 Series - Multi-Channel
Pressure Range at Sample	1 to 2 bar	10- <sup>6</sup> mbar up to 2 bar
Experimental Gas Inlets	1	3 - 8*
Purge Capability	Yes	Yes
Gas Analysis Capability	No	Yes
Tubing System	All Metal	All Metal
Biasing Contacts	4 contacts	4 contacts
Heating Temperature	> 1000°C	> 1000°C
Holder Cleaning	Bakeable to 160° C	Bakeable to 160° C
EELS / EDS Compatible	Yes	Yes
TEM Compatibility	TFS, JEOL, Hitachi, Zeiss	TFS, JEOL, Hitachi, Zeiss

\* Contact us for Custom Configurations



#### **Overview**

Our in-situ TEM gas cell specimen holder allows researchers to study material behavior in gases and at elevated temperatures (>1000°C), obtaining atomic resolution images of gas-solid interactions at real-world reaction temperatures and pressures. Gases are introduced to the microfabricated environmental cell via one of our dedicated gas delivery systems. Cell pressure is fully user-controlled and can be adjusted from high vacuum to above atmospheric pressure. Local specimen heating is provided via an integrated MEMS heater with a temperature sensor calibrated for accurate readings. To ensure clean gas delivery, the entire holder can be baked at up to 160°C. Now also with custom data/image integration options.

Sample research applications for which realistic reaction conditions can be created in the gas environmental cell are:

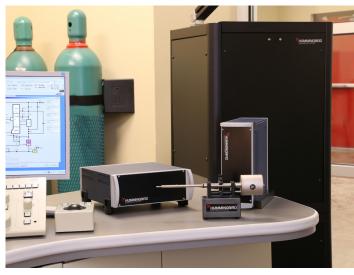
- ) Gas catalysis
- > Growth of nano-structures
- > Fuel-cell research
- > Thin film deposition

#### Single-channel gas delivery system

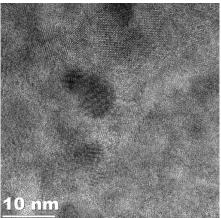
Hummingbird's single-channel atmospheric gas delivery system delivers a single pressure-controlled experimental gas to the sample holder. This system, which comes with a built-in purging line for safe cleaning, operates at or just above atmospheric pressure inside the gas cell. The gas-control software allows users to operate in a pressure-controlled, closed-loop feedback mode, and it gives the user full electronic control of the system. The software is also capable of logging gas delivery parameters.

#### Multi-channel gas delivery system

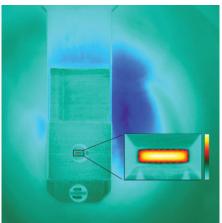
Hummingbird Scientific's multi-channel option is a fully configurable and scalable gas-delivery system designed to deliver multiple pressure-controlled gases to an environmental cell. It features up to eight gas channels that can be run independently or simultaneously in user-defined ratios. Like the single-channel delivery system, the multi-channel system has a built-in purging line for safe cleaning of the gas-flow path. It operates at a full range of sample pressures, from high vacuum ( $10^{-7}$  Torr) to above atmospheric pressure. In addition to the mixing capability, the system includes gas-control software & a built-in gas analyzer for post reaction analysis. When used in combination with Hummingbird Scientific's optional heating system and heating chips, the system also supports real-time observation of catalysis reactions in the TEM.



Hummingbird Scientific's gas-flow TEM holder comes with either a single-channel or multi-channel delivery systems.



TiN film on SiN substrate at 700C in ~1 atm nitrogen inside the Gas Cell TEM holder.



Heating

*IR image of Gas Flow Tip during hightemperature heating cycle.* 

#### **TEM Safety**

Our thin-film heating system for the gas holder discretely heats samples in the gas cell to > 1000°C. Low-drift, high image stability and long lifetimes > 160 hours make the heating system not only robust but allows tracking of the area of interest while imaging at highmagnification.

Heating is controlled via a custom-designed control box and software featuring closedloop temperature control and four-point probe temperature sensing from an on-chip sensor, which is accurate enough to not only measure the sample temperature but to detect power changes small enough to be able to perform nano-calorimetry.

Careful preparation of your samples and system are essential for effective use of environmental holders. A critical component of any holder system is a high-vacuum leak check station.

Our high-vacuum pumping station is a compact, all-in-one vacuum storage and seal-checking mechanism for TEM specimen holders. The station features short pumping and venting times, a low base pressure (<1e-6 mbar), and a glass viewing port for the holder tip.



#### Accessories



### *In-situ* TEM high-temperature reduction of high-entropy alloy nanocatalyst

Atomistic interaction of gas-solid phase is important to understand the working mechanism of various catalyst materials. This is specifically the case for high-entropy alloy (HEA) atomistically mixed with more than five elements. A research team from the University of Illinois, Chicago (UIC), Argonne National Laboratory (ANL), University of Pittsburgh, University of California, Riverside, and Northwestern University used Hummingbird Gas TEM holder to study the oxidation and reduction behavior of FeCoNiCuPt HEA in air and hydrogen gas at 400 °C, respectively. As the particles are heated in air at 400 °C, there is a growth of the oxide layer around the particles. Upon introduction of hydrogen gas, there is a further expansion of the oxide layer which transforms into porous structures and there is an outward diffusion of all transition metals (Fe, Co, Ni and Cu). The work presented here provides fundamental insights into the new class of alloy NPs for catalytic applications.

**Reference:** Song et al. Nano Lett. 2021, 21, 4, 1742–1748. DOI: 10.1021/acs.nanolett.0c04572

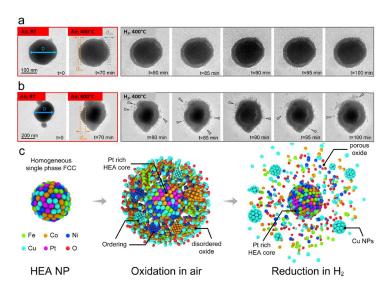


Figure: In-situ TEM oxidation and reduction of CoNiCuPt nanoparticles upon heating at 400 °C, in air and hydrogen, respectively. A shown in the schematic, all transition metals diffuse out. Pt remains intact in the core.

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#### **Selected Publications**

Boao Song, Yong Yang, Timothy T. Yang, Kun He, Xiaobing Hu, Yifei Yuan, Vinayak P. Dravid, Michael R. Zachariah, Wissam A. Saidi, Yuzi Liu, and Reza Shahbazian-Yassar. **"Revealing High-Temperature Reduction Dynamics of High-Entropy Alloy Nanoparticles via In Situ Transmission Electron Microscopy,"** *Nano Letters* (2021)

Boao Song, Yong Yang, Muztoba Rabbani, Timothy T. Yang, Kun He, Xiaobing Hu, Yifei Yuan, Pankaj Ghildiyal, Vinayak P. Dravid, Michael R. Zachariah, Wissam A. Saidi, Yuzi Liu, and Reza Shahbazian-Yassar. **"In Situ Oxidation Studies of High-Entropy Alloy Nanoparticles,"** ACS Nano (2020)

Mathilde Luneau, Erjia Guan, Wei Chen, Alexandre C. Foucher, Nicholas Marcella, Tanya Shirman, David M. A. Verbart, Joanna Aizenberg, Michael Aizenberg, Eric A. Stach, Robert J. Madix, Anatoly I. Frenkel & Cynthia M. Friend. **"Enhancing catalytic performance of dilute metal alloy nanomaterials,"** *Communications Chemistry* (2020)

Boao Song, Timothy T. Yang, Yifei Yuan, Soroosh Sharifi-Asl, Meng Cheng, Wissam A. Saidi, Yuzi Liu, and Reza Shahbazian-Yassar, **"Revealing** Sintering Kinetics of MoS2-Supported Metal Nanocatalysts in Atmospheric Gas Environments via Operando Transmission Electron Microscopy," ACS Nano (2020)



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