

Advanced In Situ and Operando TEM at the NUANCE Center: Capabilities and Opportunities

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Outline

➤ In-situ gas cell TEM

(e.g., thermal catalysis, gas-solid interactions, gas sensor, optical-solid-gas interactions)

➤ In situ liquid cell TEM

(e.g., most liquid involved chemical process)

➤ In-situ biasing and/or heating

(e.g., electric field and/or temperature enabled phenomena)

➤ Cryogenic TEM

(e.g., beam damage remediation, low temperature transformation)

Temperature controlling holder at NUANCE-EPIC

Environmental :



Protochips/Hummingbird Gas



Protochips/Hummingbird Liquid



6 K

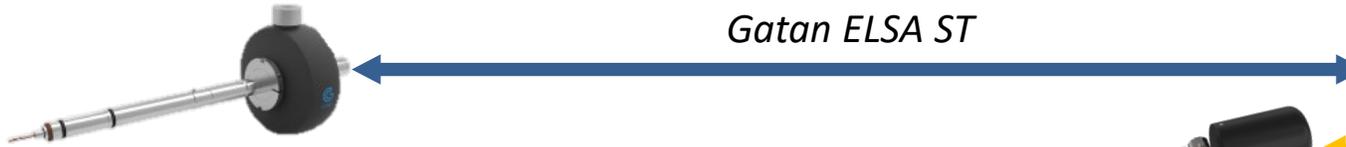
100 K

RT

373K

1273 K

Gatan ELSA ST

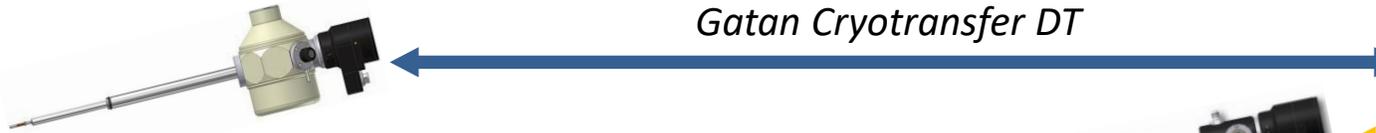


DENS MEMS Heater DT



Non-environmental :

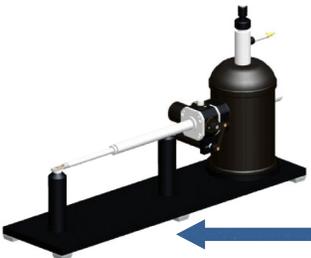
Gatan Cryotransfer DT



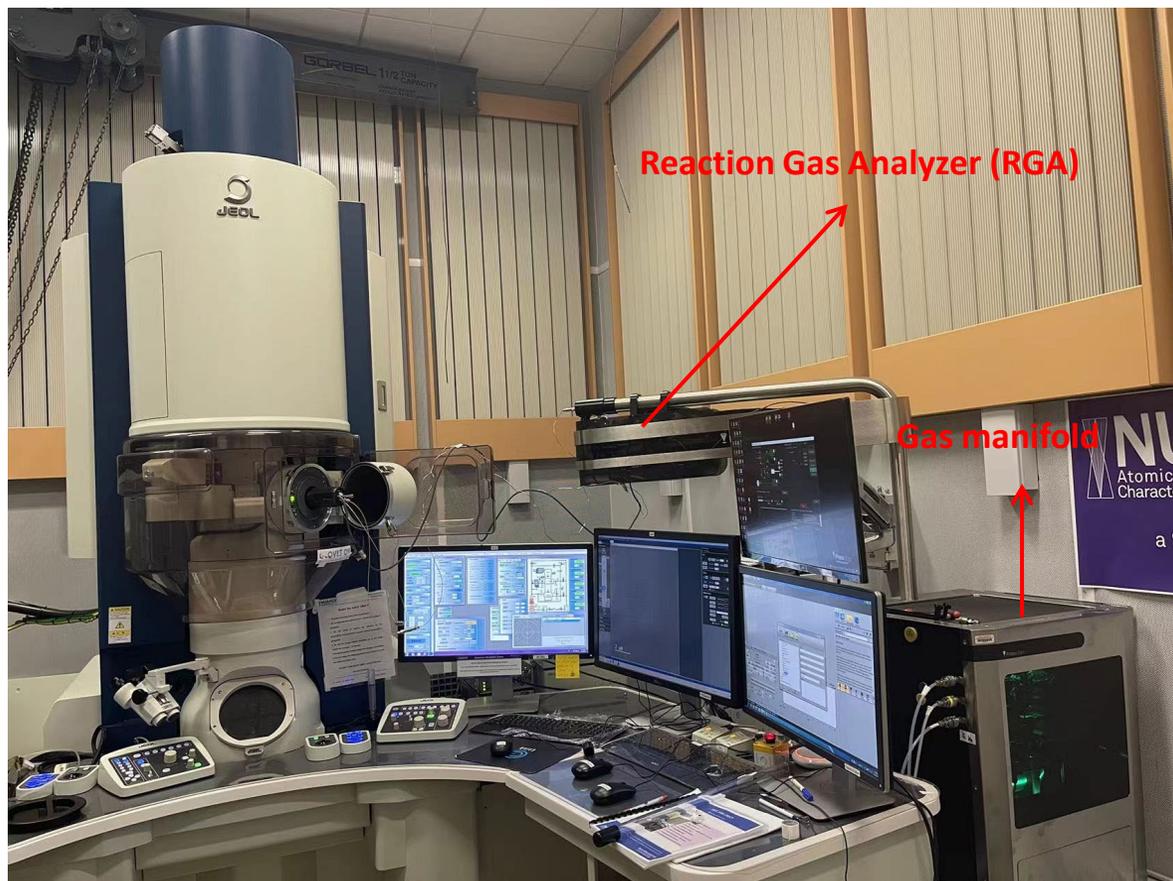
Gatan Furnace heater DT



Gatan L-He holder DT (ULTDT)



➤ In-situ gas cell TEM



- Protochips Atmosphere 210 System attached on ARM 200CF
- Pressure range: 760 Torr (101.3 kPa) to 1 Torr (0.13 kPa);
- Flow range: 0.005 sccm to 1 sccm (ml/min);
- Temperature range: RT to 1000 °C with max 10 °C/s heating rate;
- Gas mixing: 0.01%-99.99% mixture of up to 3 non-corrosive gases;
- Vapor allowable: water, methanol, ethanol, hexane, naphtha, etc;
- Reaction gas Analyzer (RGA) attached



Figure 1: Atmosphere 210 system.



Atmosphere Gas Delivery System

Assembled tip

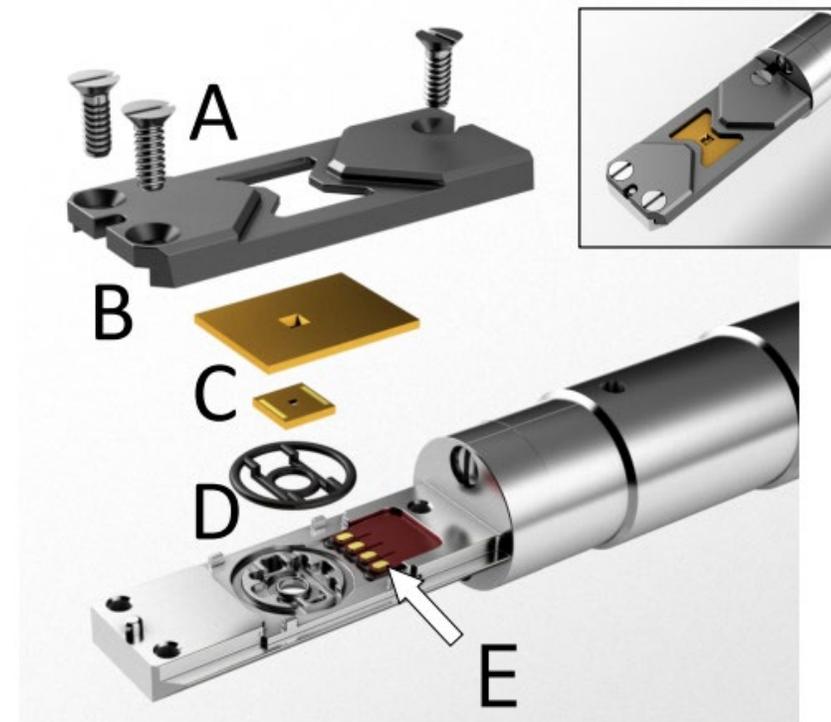
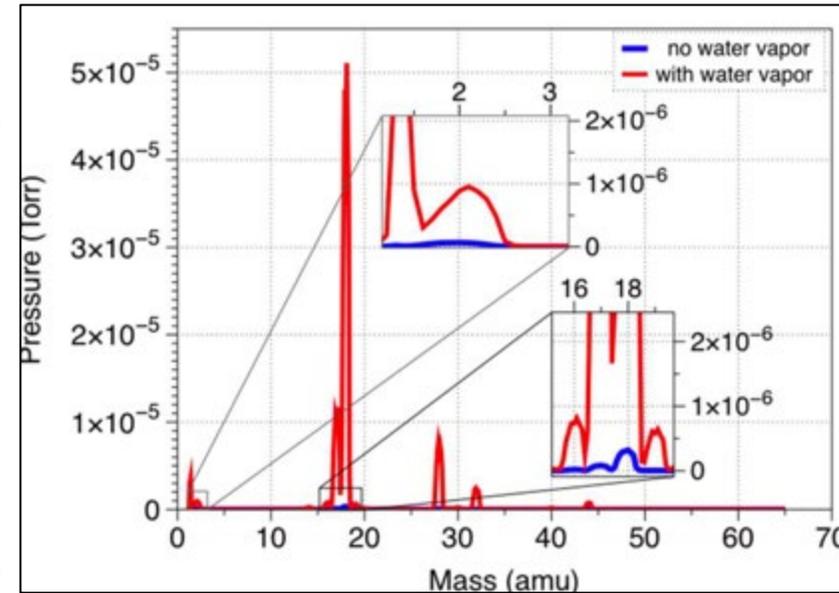


Figure 6: Atmosphere holder tip (EDS compatible). A) lid screws b) lid c) small/large e-chip pair d) gasket e) flex-circuit. Top right inset shows assembled tip.

- A: Lid screws
- B: Lid
- C: Small/large e-chip pair
- D: Gasket
- E: Flex-circuit

Be confident the system is clean before starting experiments

- Especially important for re-dox experiments –water and oxygen need to be absent
- Software-controlled protection of RGA during cleaning cycles – great for new mass spec users



Microsc. Microanal. 24 (Suppl 1), 2018

Software interface of Atmosphere gas system

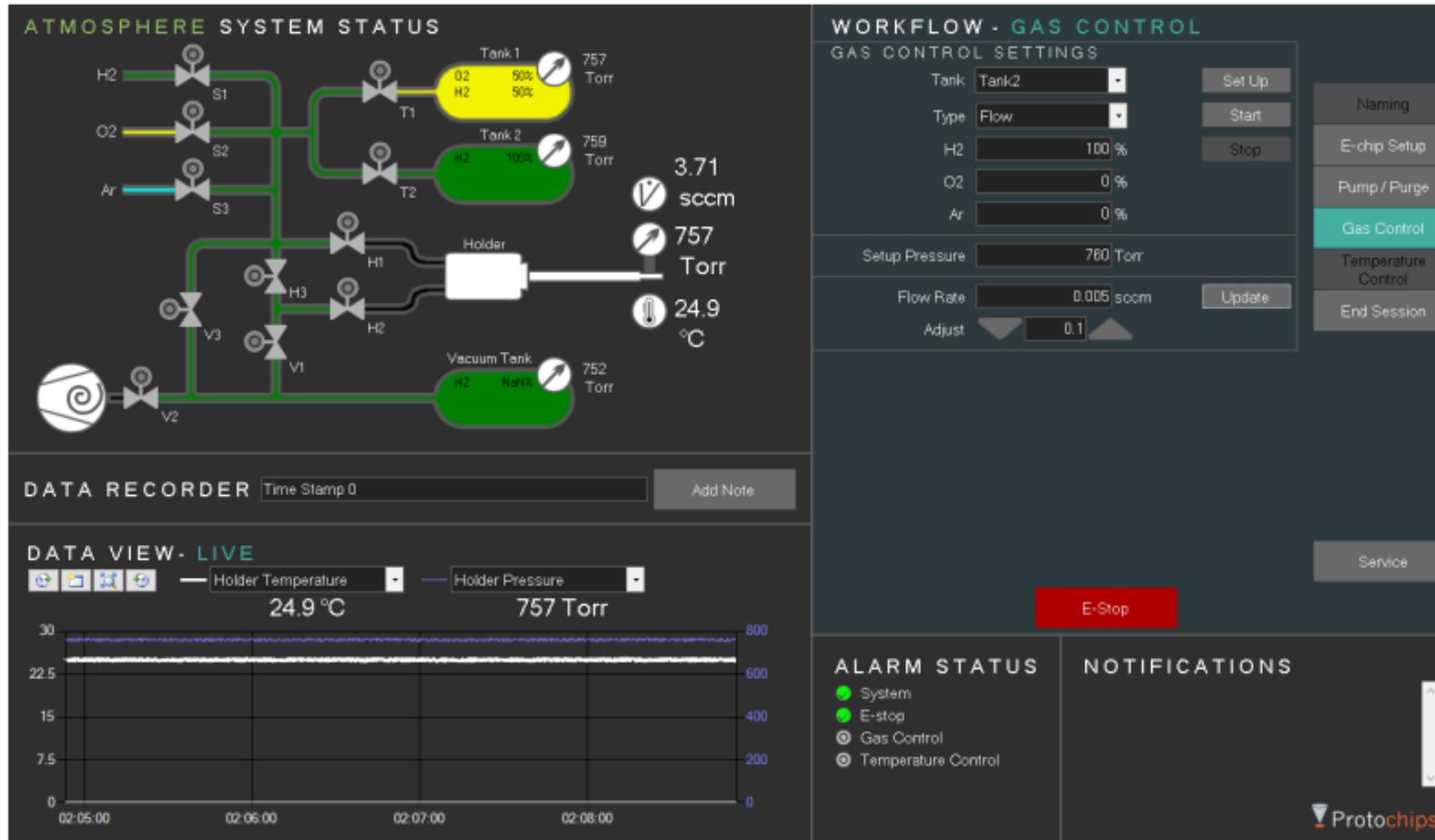


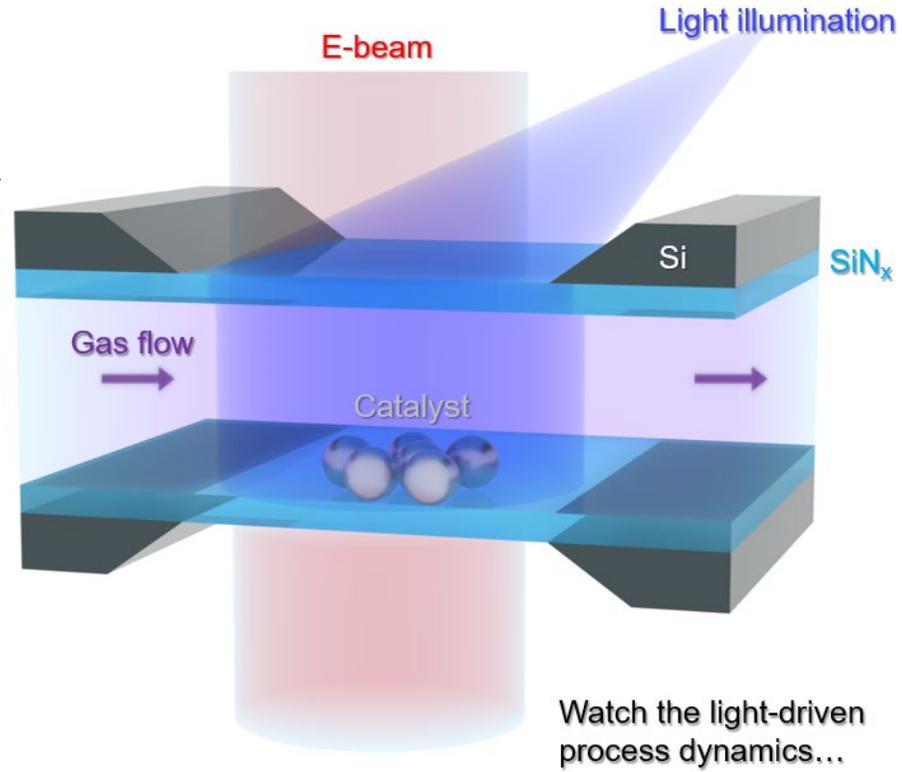
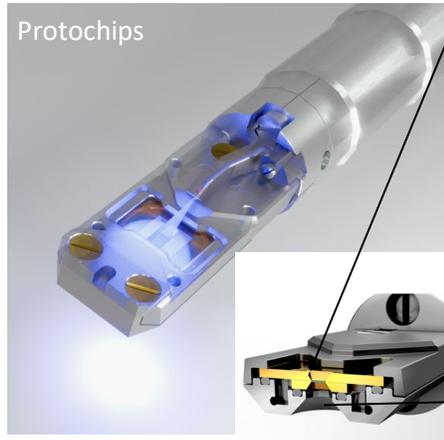
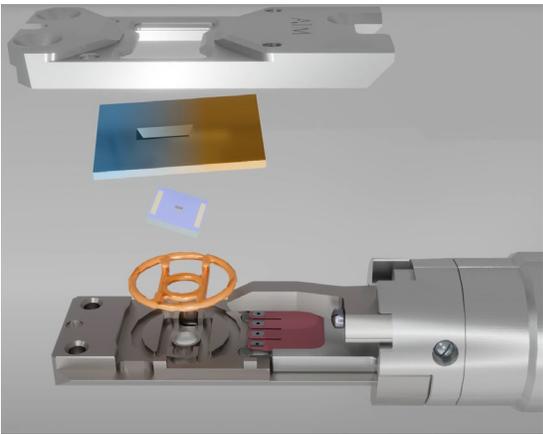
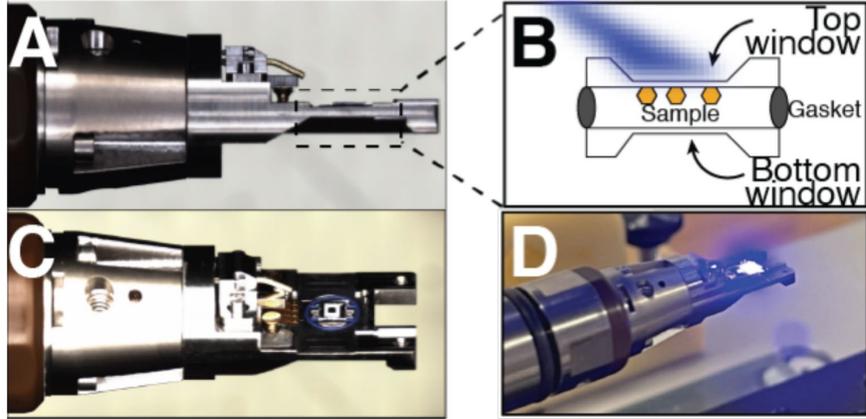
Figure 2: Screenshot of the atmosphere software. Panes include system status, workflow, data recorder, data view, alarm status, and notifications.

Gas compatibility

Chemical	System Compatible		In-Column Approved
	Viton Gasket	Perfluoroelastomer Gasket	
Air	Yes	Yes	Yes
Alkanes	Yes	Yes	NA
Alkenes	Yes	Yes	N/A
Alkynes	Yes	Yes	N/A
Ammonia (<10%)	No	Yes	N/A
Argon	Yes	Yes	Yes
Chlorine (Cl ₂)	No	No	No
Helium	Yes	Yes	Yes
Hydrochloric Acid (HCl)	No	No	No
Hydrogen	Yes	Yes	Yes
Hydrogen Sulfide (H ₂ S)	No	No	No
Nitrogen	Yes	Yes	Yes
Nitrogen Oxides (NO _x)	No	Yes	N/A
Oxygen	Yes	Yes	Yes
Water Vapor*	Yes	Yes	N/A
Xenon	Yes	Yes	N/A

- Any gas compatible with listed material can be used:
 - Ti (Grade V)
 - 316 SS
 - Silica
 - PEEK
 - Perfluoroelastomer
 - Si, SiNx, SU-8, gold

A novel light-coupled in-situ gas cell platform



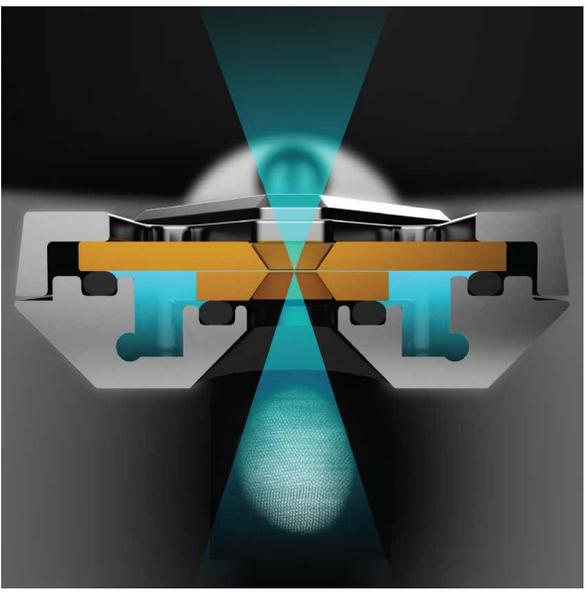
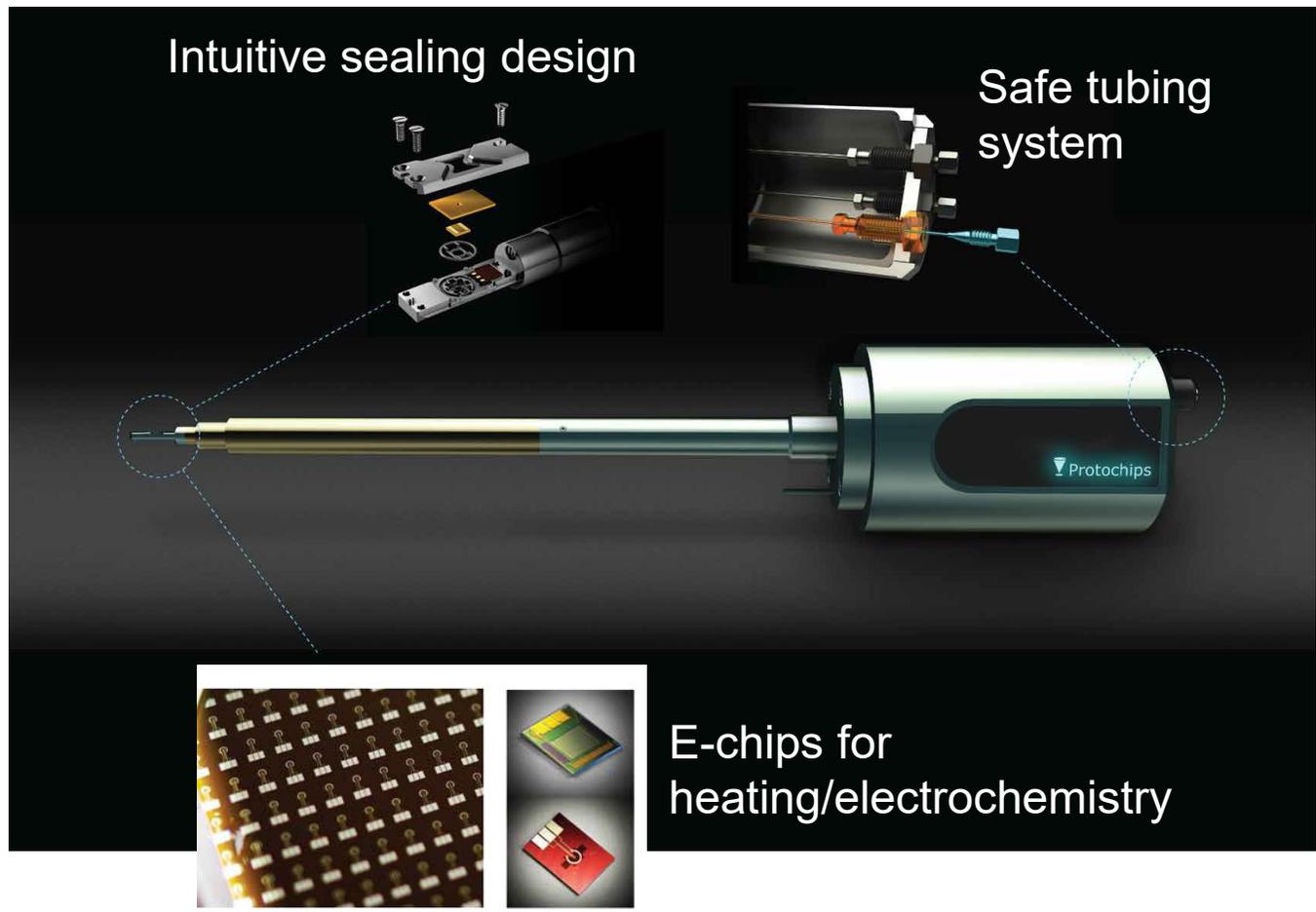
- Light source flexibility
- 350 nm - 2000 nm wavelength
- 0.0025 mm² Illuminated area
- ≥ 300 mW/cm² Power density
- Integration of photoillumination metadata with AXON
- Closed-loop temperature control

APPLICATIONS

- Photocatalysis
- Photochemistry
- Photovoltaics
- Optoelectronics
- Plasmonics

➤ In-situ liquid cell TEM

Poseidon Select holder

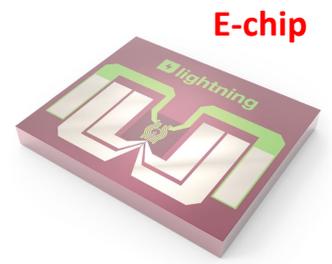


- Tilt only along X direction with around $\pm 20^\circ$
- Temperature range: RT to 100 °C with max 10 °C/s heating rate;
- Liquid flow controlled via external flow system (up to $\sim 8 \mu\text{L}/\text{min}$)
- Spacer-controlled liquid thickness ($\sim 150 \text{ nm}$ nominal spacers commonly used)
- EDS-optimized chip designs

➤ In-situ biasing and/or heating

1. Lightning heating & biasing TEM specimen holder
2. Nano-Chips starter pack
3. Heating control unit
4. Laptop with Impulse software
5. FIB stub 3.0
6. Keithley 2450 source measuring unit (optional)

Including:
Supporting tools



- Localized heating/biasing via E-chips
- Double tilt ($-30^\circ < \alpha < 30^\circ$, $-22^\circ < \beta < 22^\circ$)
- Heating and/or biasing



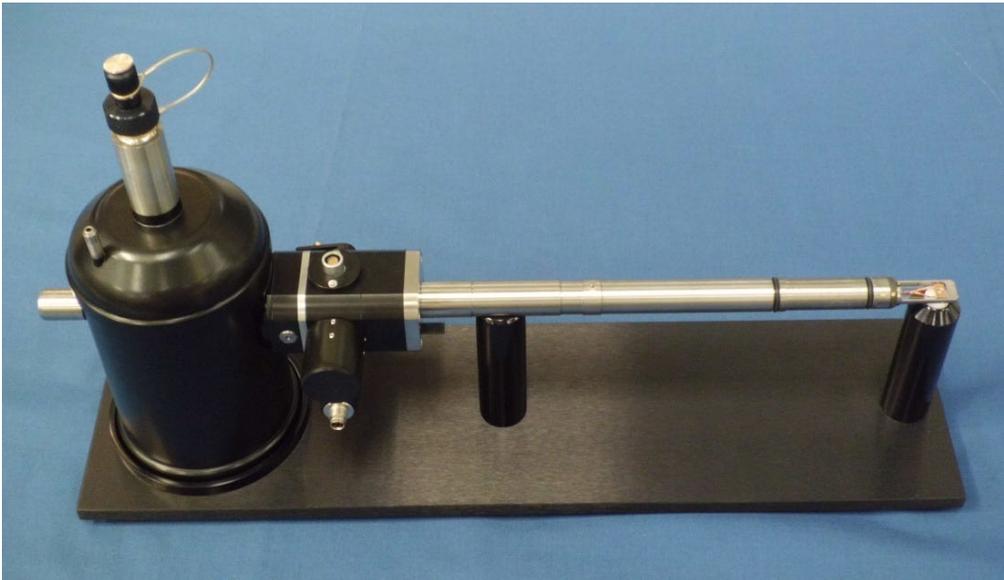
Keithley 2450 Source measurement unit

Nano-Chip	Lightning HB+
Heater material	Chemically inert encapsulated metal
Heating range	RT - 1,300 °C
Heating & Biasing range	RT - 900 °C
Temperature control mode	Closed 4-point probe feedback loop
Temperature stability	± 0,005 °C
Temperature accuracy	≥ 95 %
Temperature uniformity	≥ 99.5 %
Z-displacement T ≤ 500°C	≤ 200 nm
Sample preparation methods	Drop casting, thin film deposition, 2D materials transfer and FIB
Sample displacement	≤ 50 nm for ΔT = 600 °C
Detectable current range*	1 pA to 100 mA
Membrane Breakdown voltage @ RT (@ 900 °C)	≥ 150 V (≥ 150 V)
Attainable E-fields @ RT (@ 900 °C)	≥ 300 kV/cm (≥ 300 kV/cm)
Transparent area	up to 100 Hz

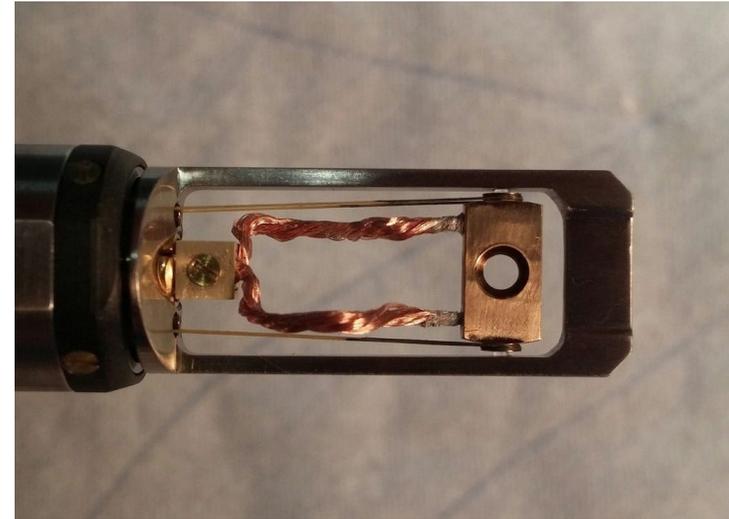
Model 2450
V-Ranges: 20mV – 200V
I-Ranges: 10nA – 1A
0.012% Basic Accuracy
Wideband Noise: 2mV _{rms} Typ.
Sweep Types: Linear, Log, Dual Linear, Dual Log, Custom, Source-Memory (SCPI 2400 Mode)
>250,000 Point Reading Buffer
>3000 Readings/Sec.
SCPI (2400 + 2450) + TSP Programming
GPIB, USB, Ethernet (LXI)
Front: Banana Jacks, Rear: Triax

➤ Cryogenic TEM

- **Double Tilt Liquid Helium Cooling Holder, Model ULTDT**



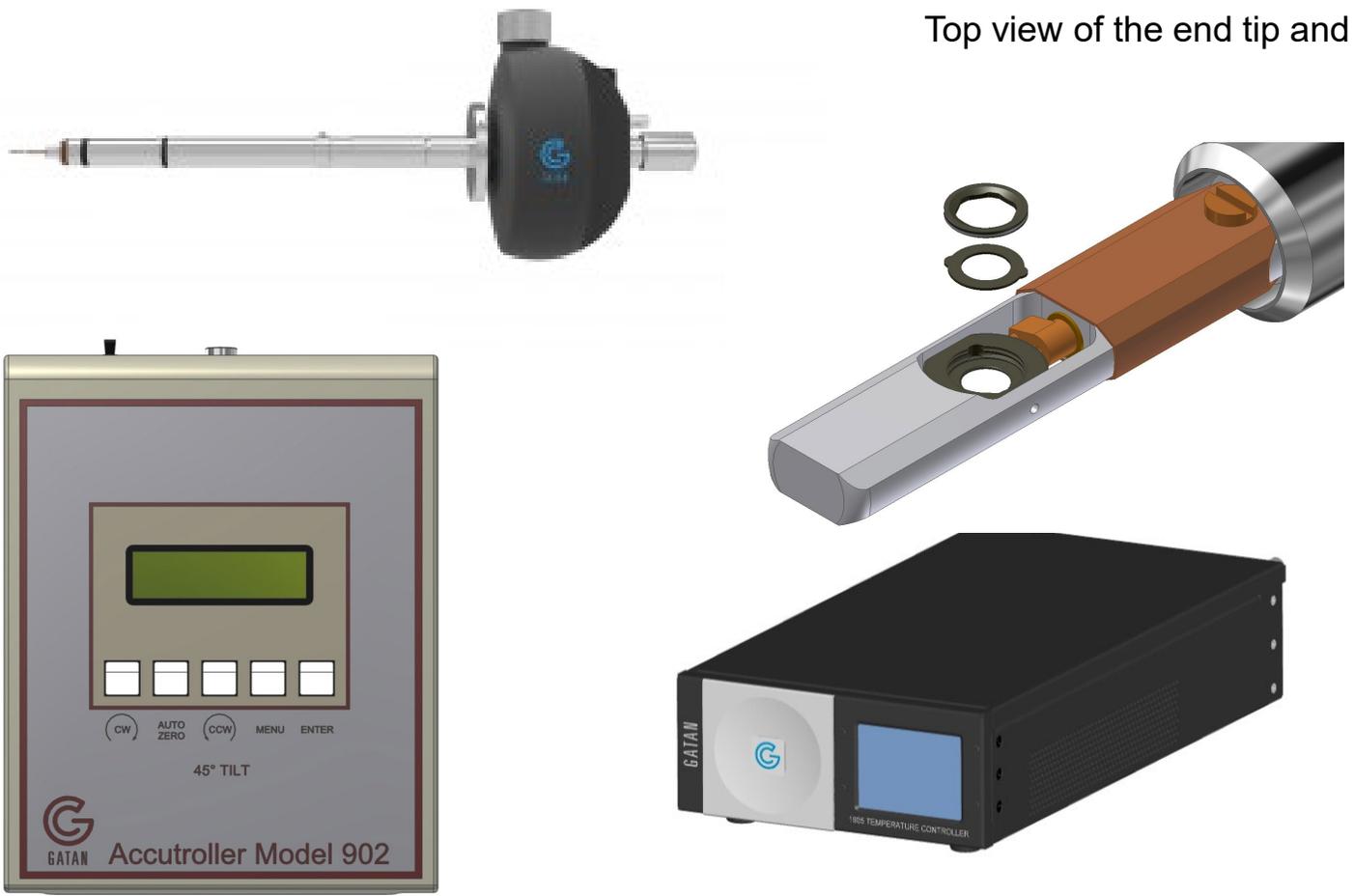
Top side view of the double tilt end tip and cooled cradle



- **Whole sample cooled**
- **Temperature ranging from 6K to 20K**
- **Double tilt ($-30^\circ < \alpha < 30^\circ$, $-20^\circ < \beta < 20^\circ$)**
- **Cooled using liquid He**
- **1hr hold time at desired temperature**
- **1.5nm/min drift rate**

- **Double Tilt Liquid N2 Cryo-Transfer Holder, Model 915**

Top view of the end tip and cooled cradle



- Whole sample cooled
- Temperature ranging from -170 °C to 80 °C
- Double tilt ($-30^\circ < \alpha < 30^\circ$, $-30^\circ < \beta < 30^\circ$)
- Cooled using liquid N2
- ~30 min hold time at desired temperature
- 1.5nm/min drift rate

Accutroller system for double tilt

Temperature controller, Model 1905

Thank you for your attention!

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