

Introduction and Applications of the Atmosphere System

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Outline

- ❖ What is in-situ?
- ❖ What is atmosphere system?
- ❖ Instruments in EPIC
- ❖ Instruction of atmosphere system
- ❖ Application of atmosphere system
- ❖ Questions

What is in-situ

In situ is a Latin phrase that translates literally to "on site" or "in position." It can mean "locally", "on site", "on the premises", or "in place" to describe where an event takes place and is used in many different contexts. [/Wikipedia](#)

In-situ (S)TEM

In situ transmission electron microscopy is a technique that allows researchers to study samples in **real-time, under real-world conditions**. ... For example, in situ TEM holders can produce heat conditions, apply atmospheric pressure, and allow for liquid microscopy study.

What is atmosphere system

In-situ gas TEM system

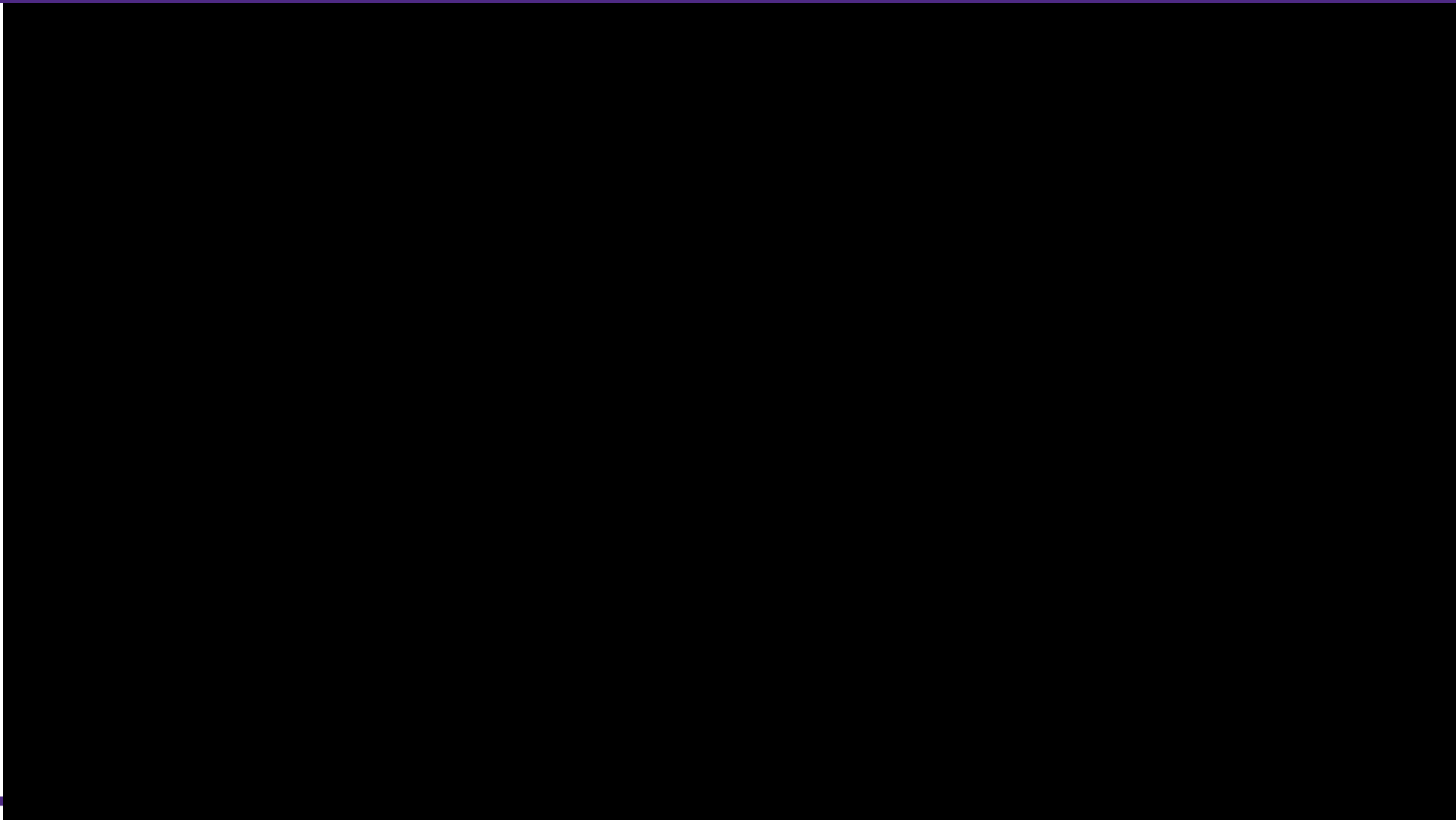
SPECIFICATIONS

Resolution	<1.5 Å (TEM dependent)
Operating Pressure Range	1.0 Torr - 760.0 Torr
Temperature	25° C - 1000°C
Flow Rate	0.005 mL/min - 1.000 mL/min
Gas Mixing	0.01% - 99.99% mixtures of up to 3 gases via volumetric blending
Vapor Introduction	Software-controlled introduction of water, methanol, ethanol, hexane, naphtha, etc.
System Cleaning	Automated pump and purge cycles with bake-out
Holder Base Pressure	3.0×10^{-2} Torr
Holder Base Pressure (w/RGA)	$<9.0 \times 10^{-7}$ Torr
EDS Capability	>3,000 CPS (Detector dependent)
Software Control	Independent control of gas composition, flow rate, pressure, and temperature
Data Synchronization	Pressure, temperature, time, gas composition, and flow rate with Clarity Echo for Gatan Microscopy Suite (GMS) version 1, 2, and 3*
STEM Compatibility	Top chip heater available for STEM imaging
Holder Assembly	Self-aligning using 1 gasket to complete in under 15 minutes



From Protochips Co.

What is atmosphere system



Prothcips Co.

Instruments in EPIC

ARM 200



ARM 300



Liquid flow holder /Hummingbird



Poseidon selection liquid heating holder /Protochips



Wildfire heating holder/ DENS Solution



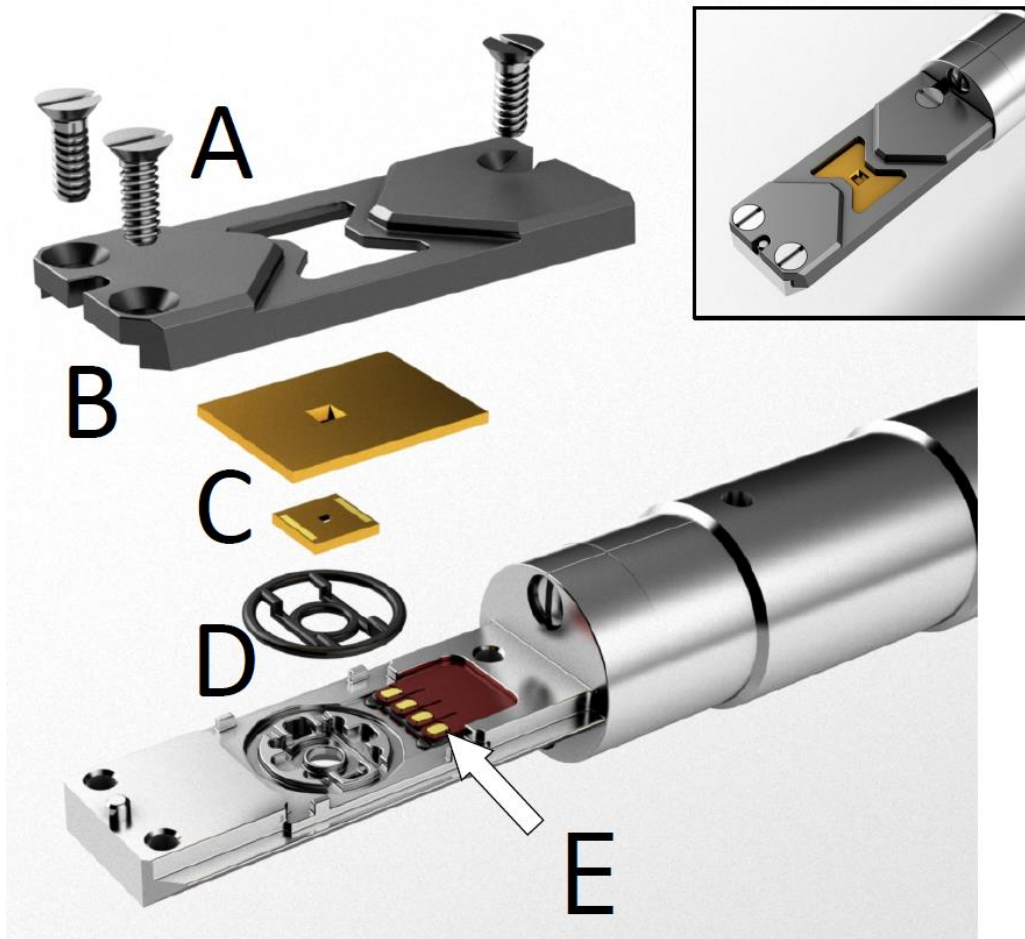
Instruction of atmosphere system



Figure 1: Atmosphere 210 system.

- Holder
- E-chips
- Gas delivery system
- Controller

Atmosphere holder



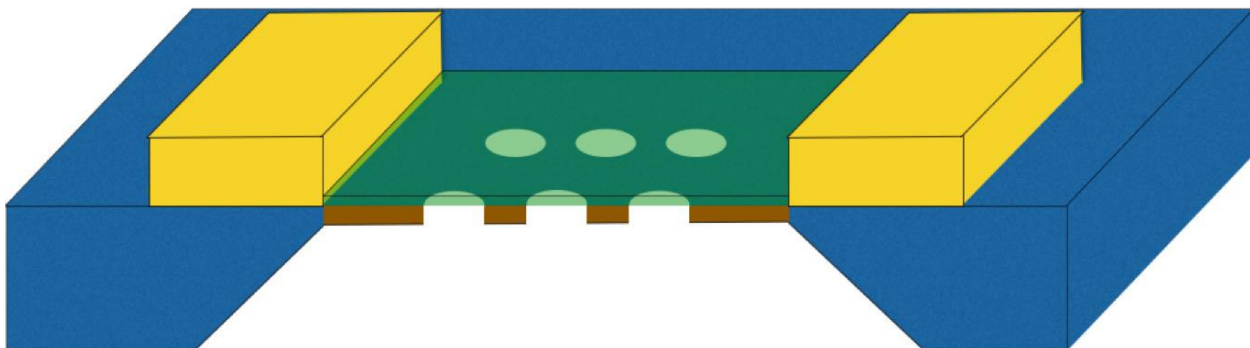
Assembled tip





- A. lid screws
- B. Lid
- C. small/large e-chip pair
- D. Gasket
- E. flex-circuit.



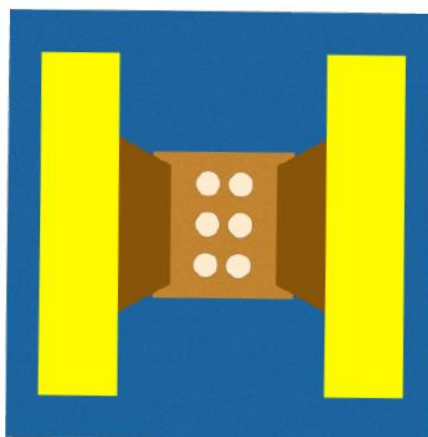
Back of the atmosphere holder showing the manual open/close valve.

Atmosphere E chips

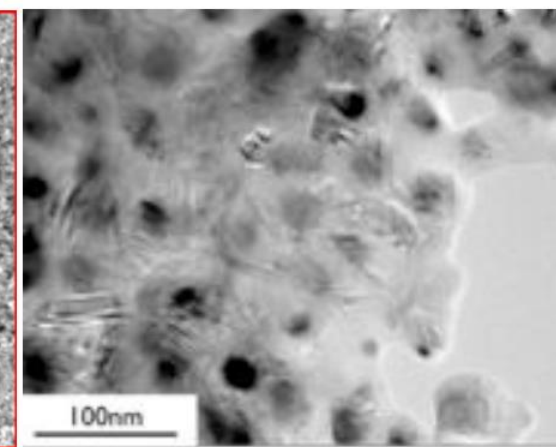
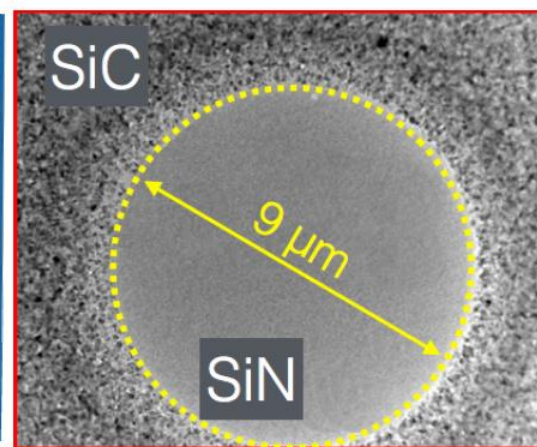


-  Spacer ~5 microns thick (Gold or SU-8 Polymer)
-  Amorphous Silicon Nitride, ~30nm thick
-  Ceramic heating membrane, ~120nm thick
-  Si wafer, 300 um thick

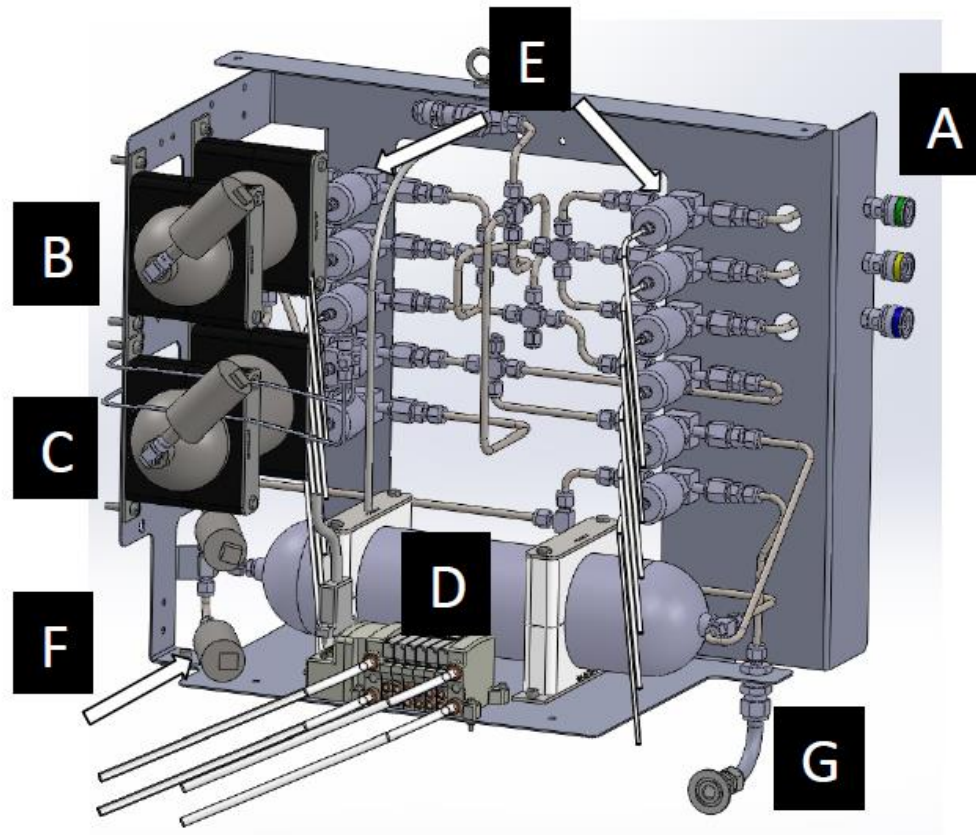
Cross section of the Heating chip



Top viewing of the heating chip



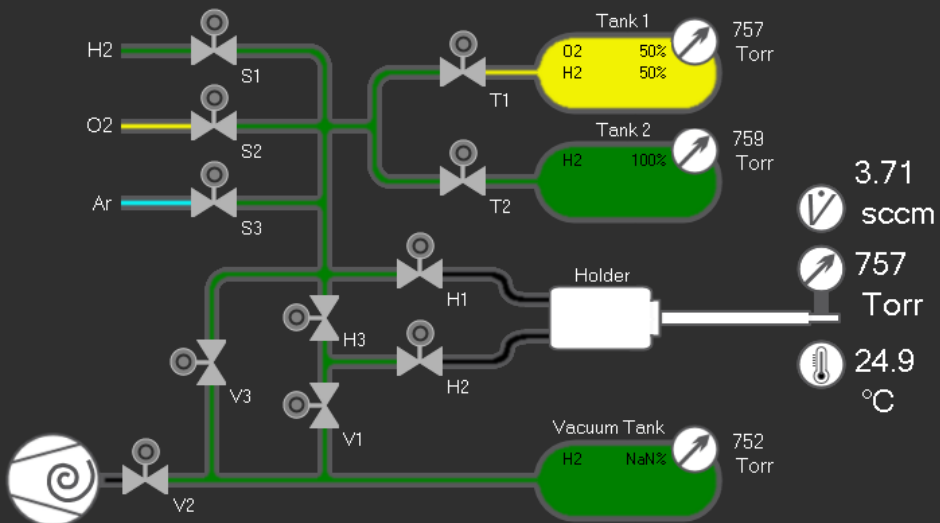
Gas delivery system



- A) 3 gas input ports
- B,C) experiment tank 1 and experiment tank 2
- D) vacuum tank
- E) pressure control valves (11)
- F) pressure sensor
- G) gas entry module for control valves

Controller

ATMOSPHERE SYSTEM STATUS



WORKFLOW - GAS CONTROL

GAS CONTROL SETTINGS

Tank: Tank2 [Set Up]

Type: Flow [Start] [Stop]

H2: 100 %

O2: 0 %

Ar: 0 %

Setup Pressure: 760 Torr

Flow Rate: 0.005 sccm [Update]

Adjust: 0.1

- Naming
- E-chip Setup
- Pump / Purge
- Gas Control**
- Temperature Control
- End Session
- Service

WORKFLOW - PUMP / PURGE

PUMP / PURGE SETTINGS

Backfill With Purge Gas

Perform Leak Check

Bake Out

Target: System

Pressure: [Torr]

Pump Down Max Duration: [min]

Backfill Gas: Manifold

Backfill Pressure: [Torr]

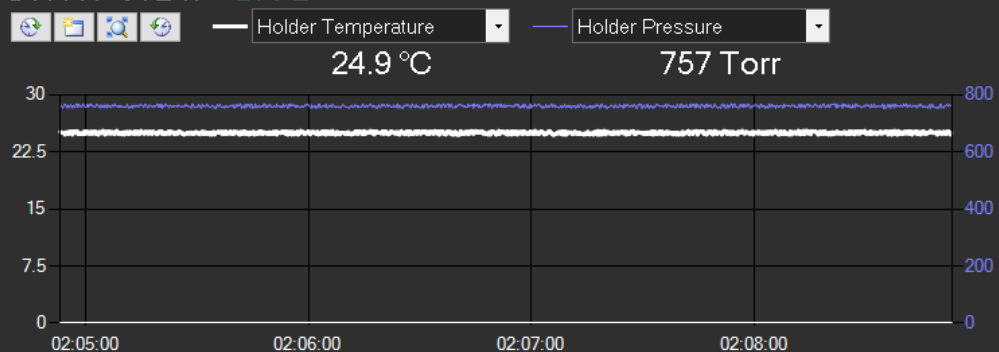
Backfill Cycles: 3

[Stop] [Start] [E-Stop] [Service]

DATA RECORDER

Time Stamp 0 [Add Note]

DATA VIEW - LIVE



ALARM STATUS

- System
- E-stop
- Gas Control
- Temperature Control

NOTIFICATIONS

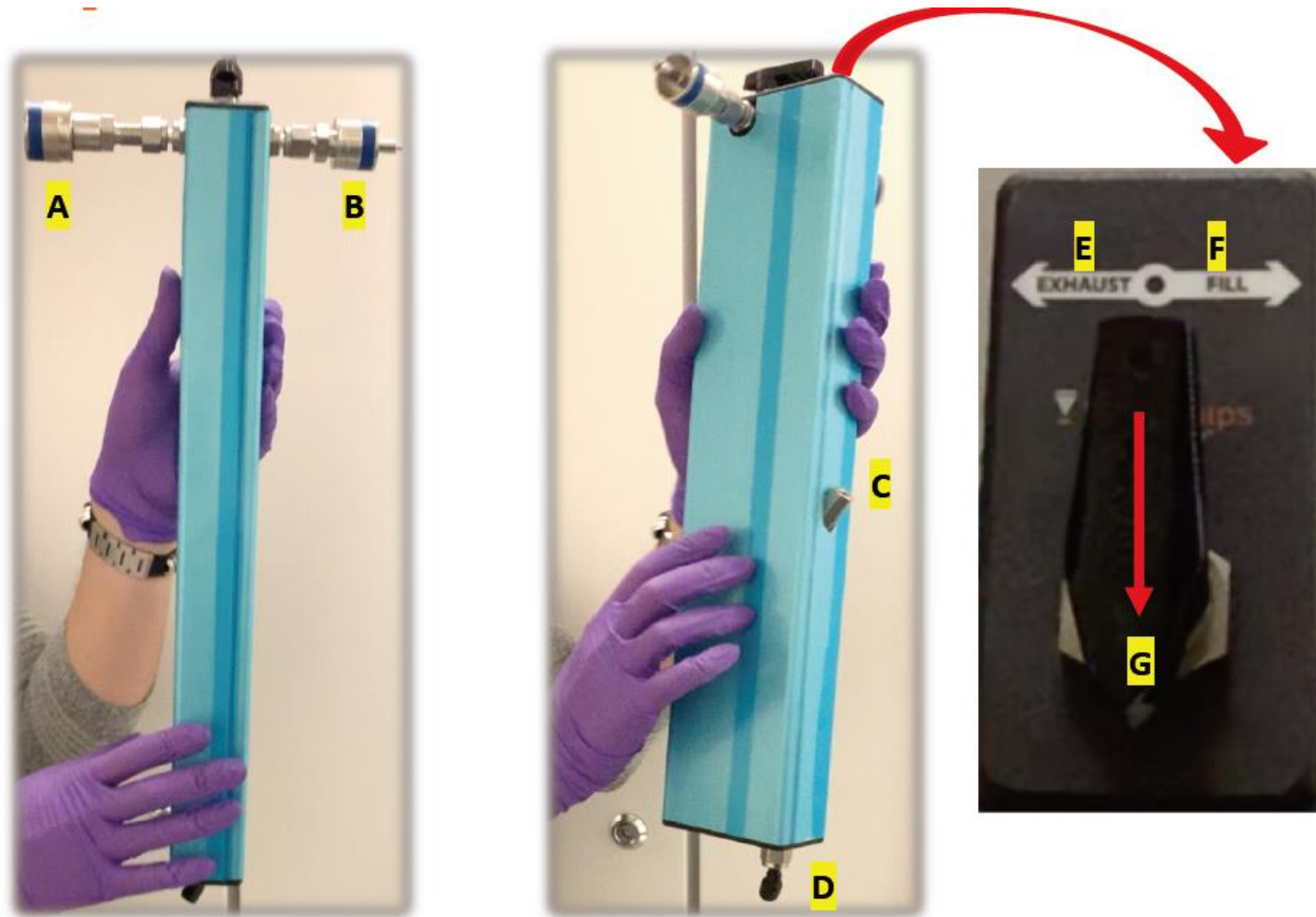
E-Stop



Each line can be cleaned independently.

Other accessories of gas system

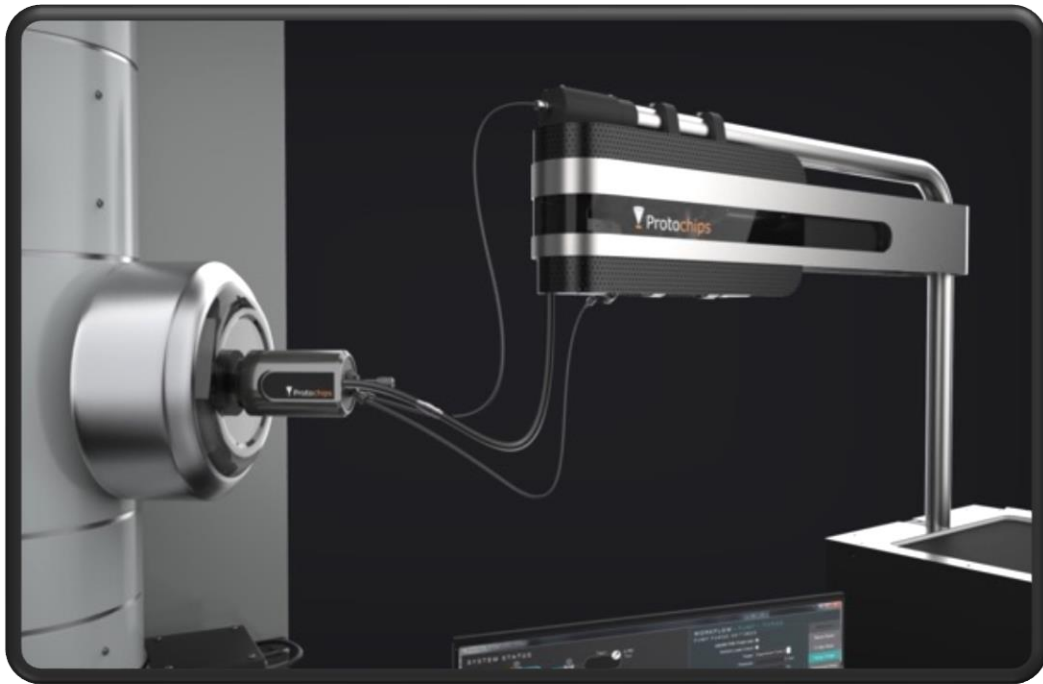
- Vapor kit



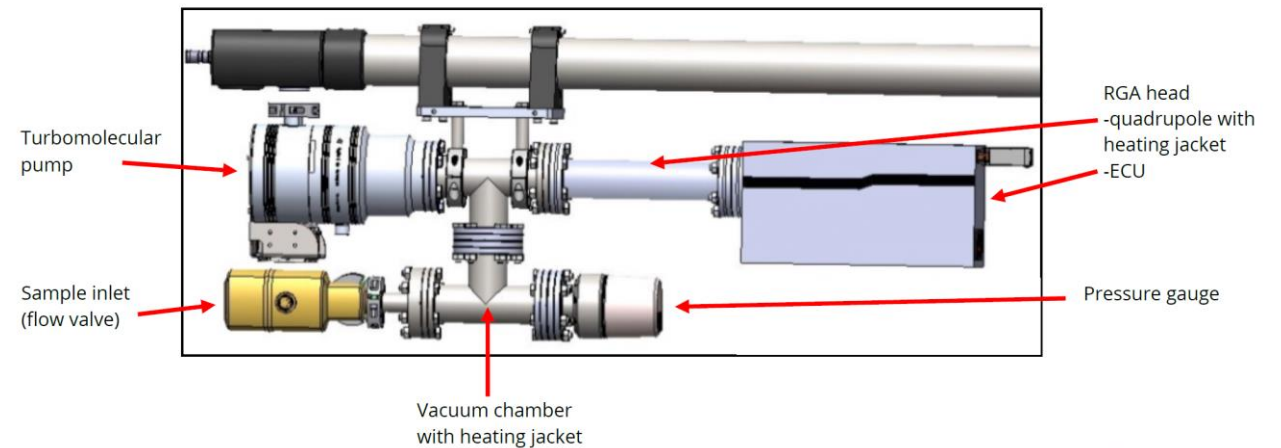
- A – Purge gas connection
- B – Manifold connection
- C – Syringe assembly port
- D – Waste tubing port
- E – “EXHAUST” used for cleaning vapor kit (open to A)
- F – “FILL” used when filling manifold with vapor (open to B)
- G – Lever facing down is “neutral” position (closed to A and B)

Other accessories of gas system

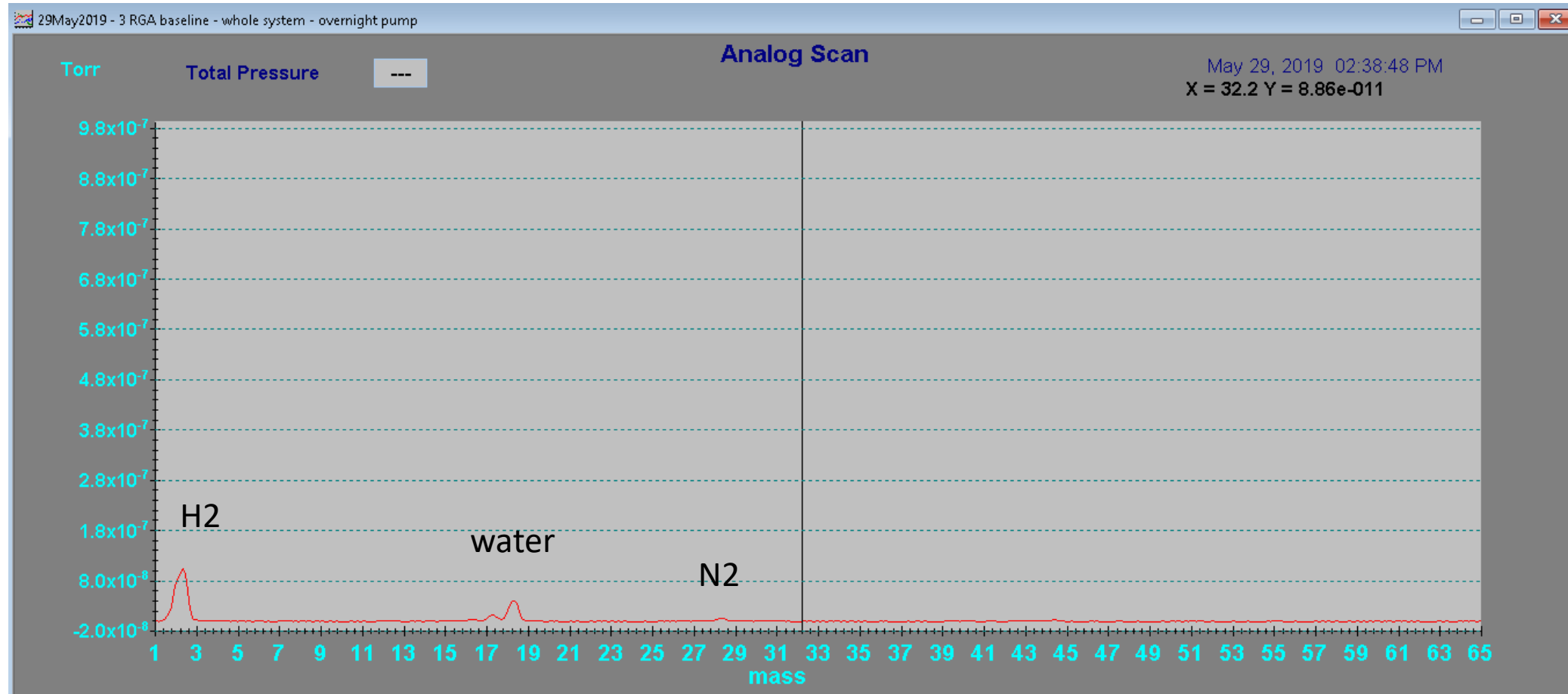
- RGA (Residual Gas Analysis)



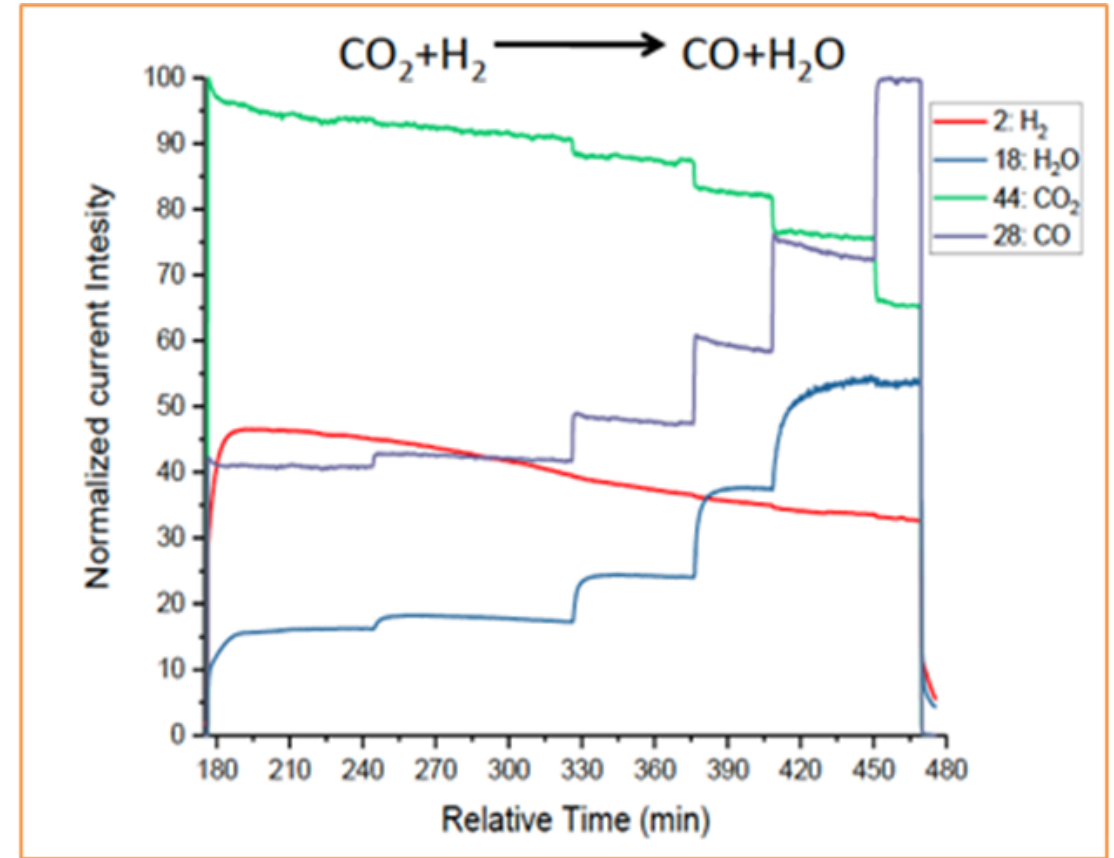
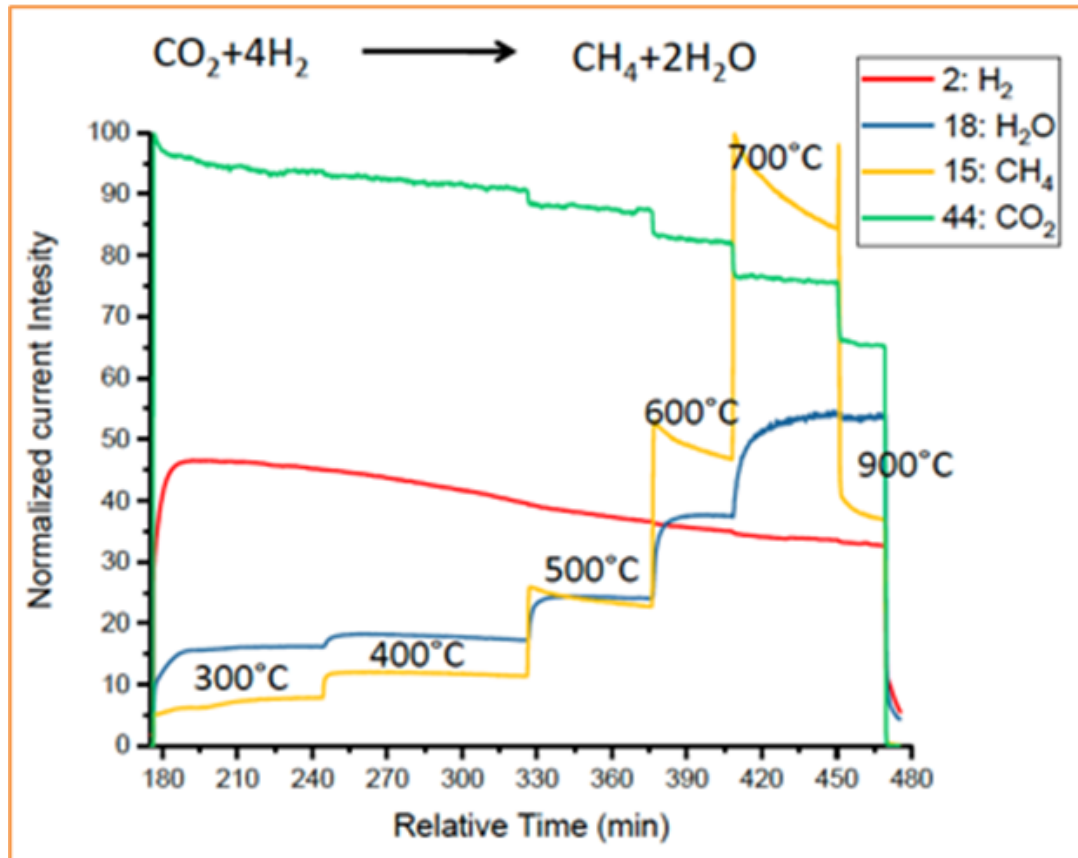
The RGA assembly:



RGA



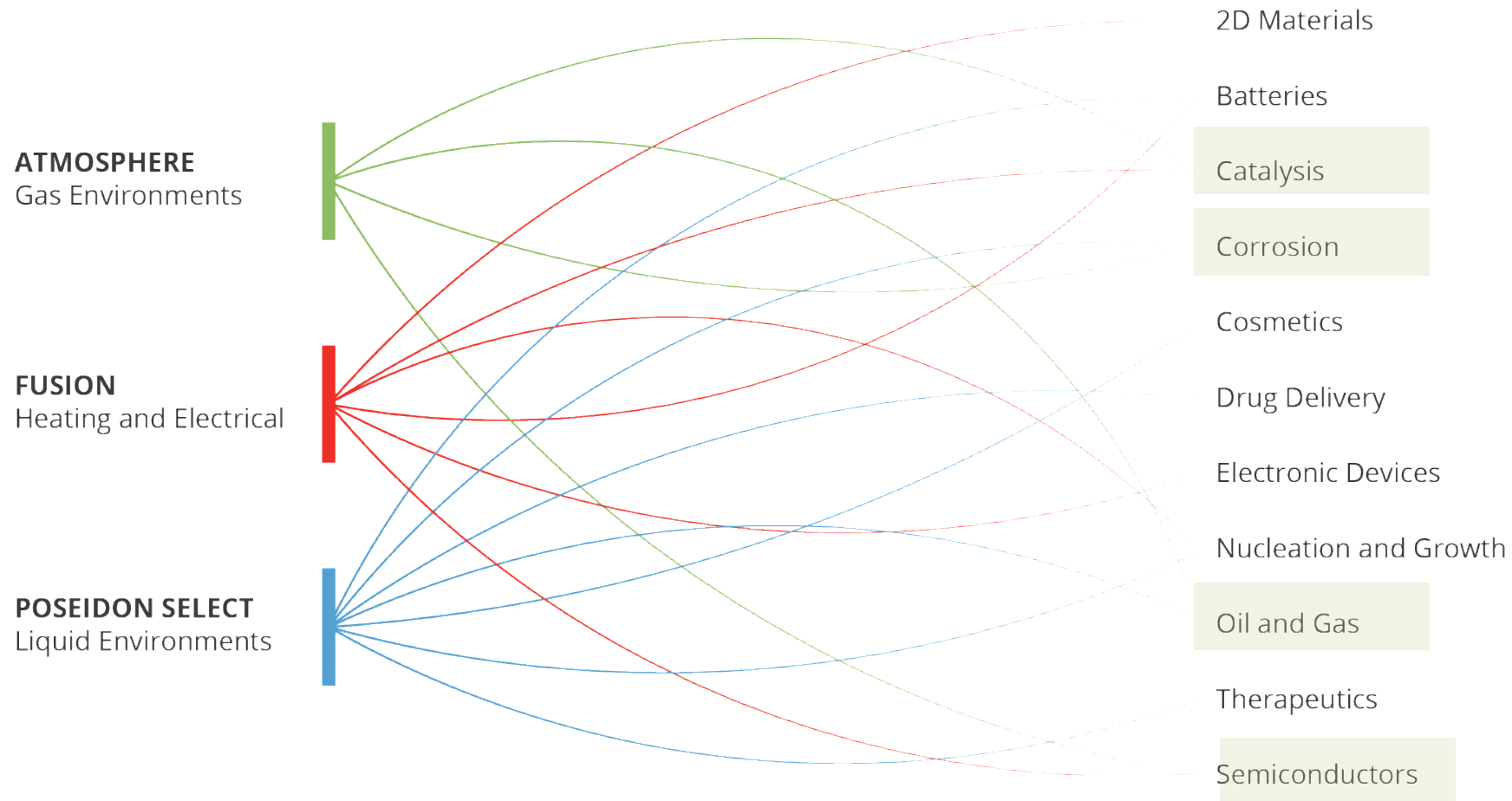
RGA



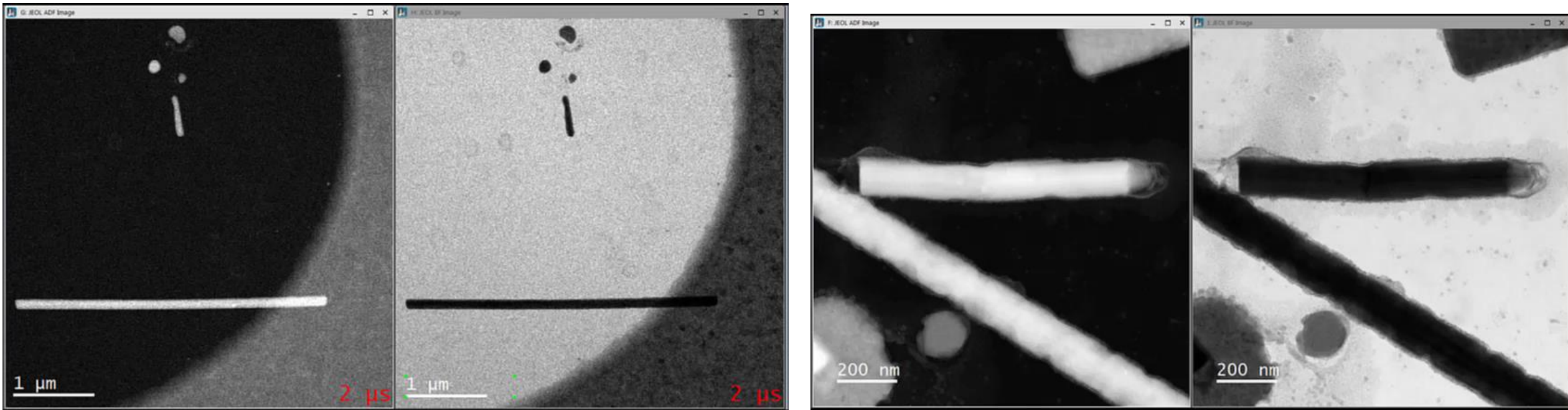
Prof. Ovidiu Ersen group



Application of atmosphere system



Application of atmosphere system

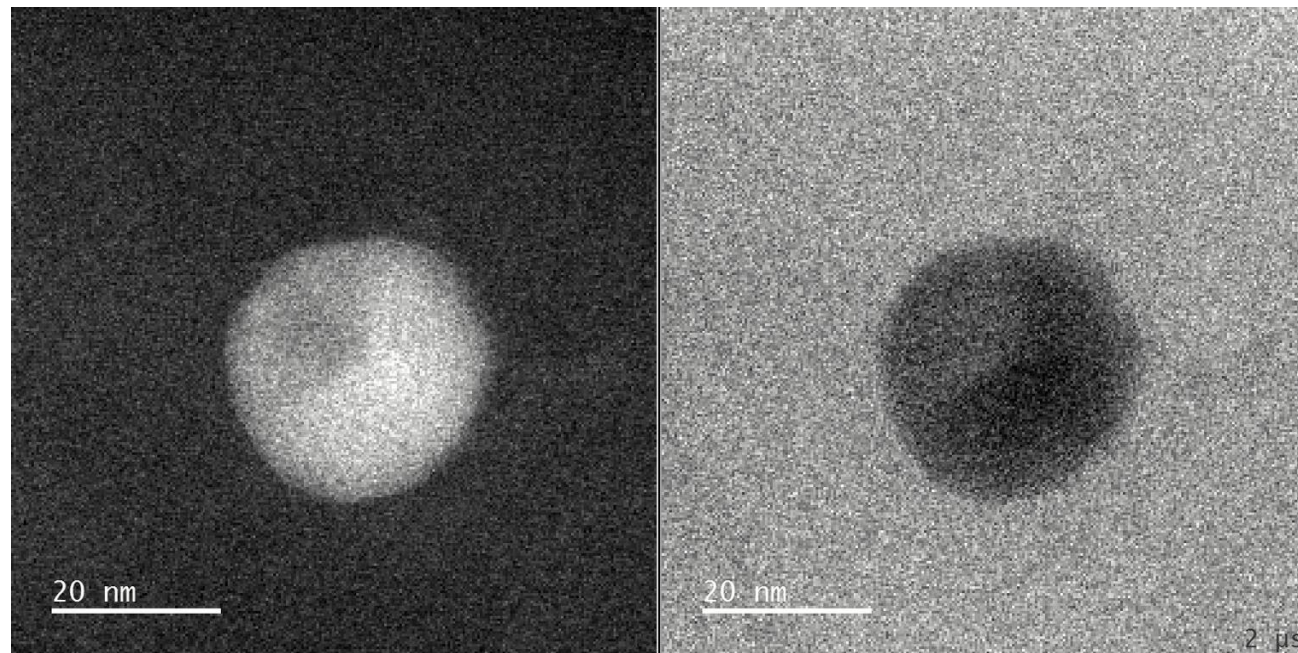


Cu NW in CO₂ gas at 200 °C.

Application of atmosphere system

Corrosion of AgCu in water vapor

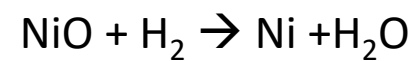
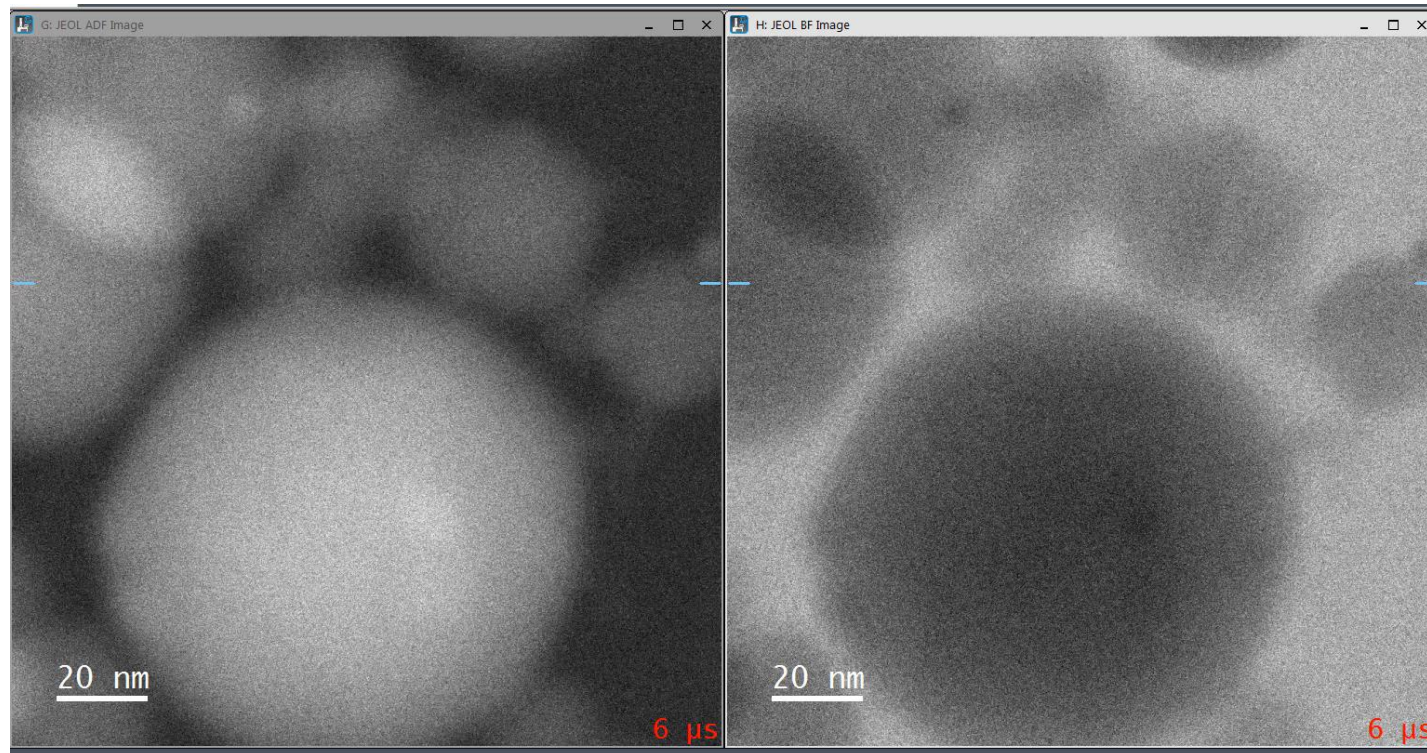
Vapor On, Real Time HAADF | ABF



Collaborate with Jingshan Du from Chad Mirkin's group

Application of atmosphere system

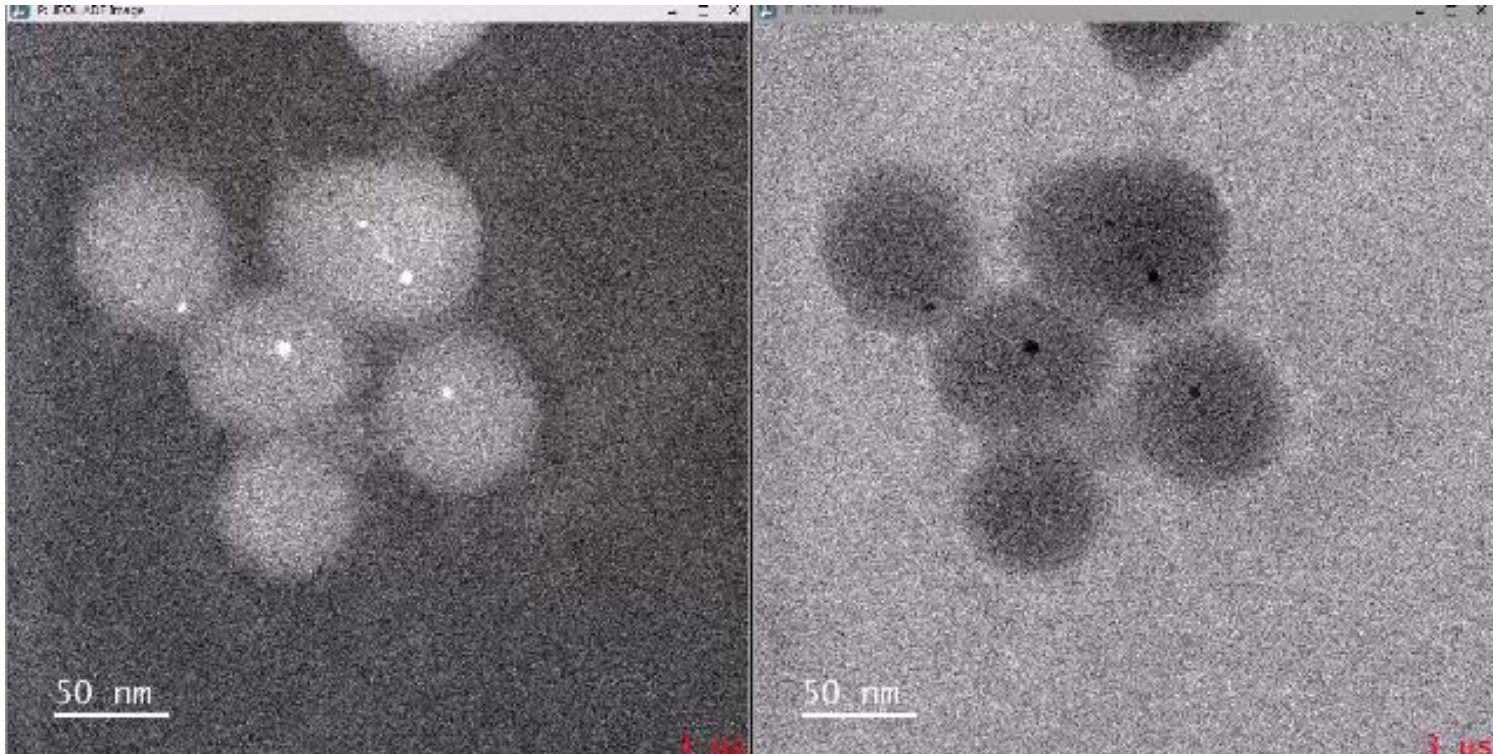
Reduction of NiO on Al₂O₃ in H₂



Collaborate with Dingdi Wang from Peter Stair group

Application of atmosphere system

Reduction of Au^{3+} @ Silica shell in H_2

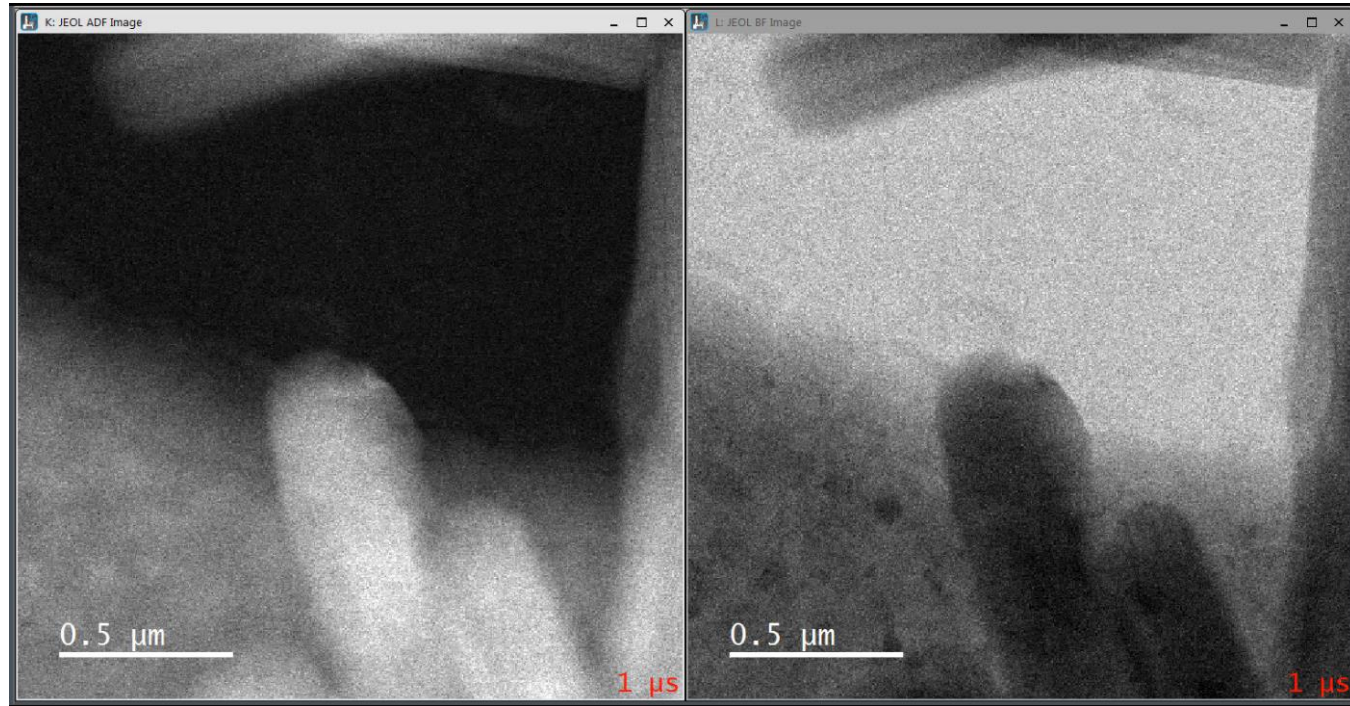


The Au ions are distributed homogeneous initially, during the reduction process, it nucleated first, then aggregated to a large one finally. But because of the preparation process, after a long-time observation, there always has some heavy carbon contamination. The expansion of carbon can be observed obviously.

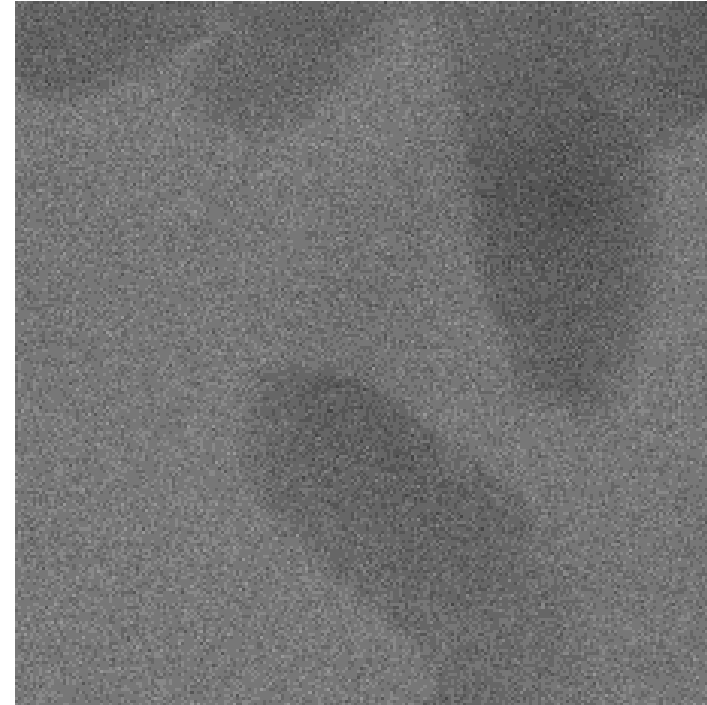
X64

Collabate with Liban from Chad Mirkin group

Application of atmosphere system



100x



Using K2 camera
at low dose
condition.

In-situ steps:

1. Drop cast sample on chips
2. Flow Ar overnight to dry the sample and remove contaminations.
3. Increase Temp to 150-200C (150c),
4. Flow 4%H2 (balanced with Ar) for 1h. Pressure 1atm, temp ramps rate is 5C/min

Collabate with from Timur from Omar's group

Thank you!

Any questions?