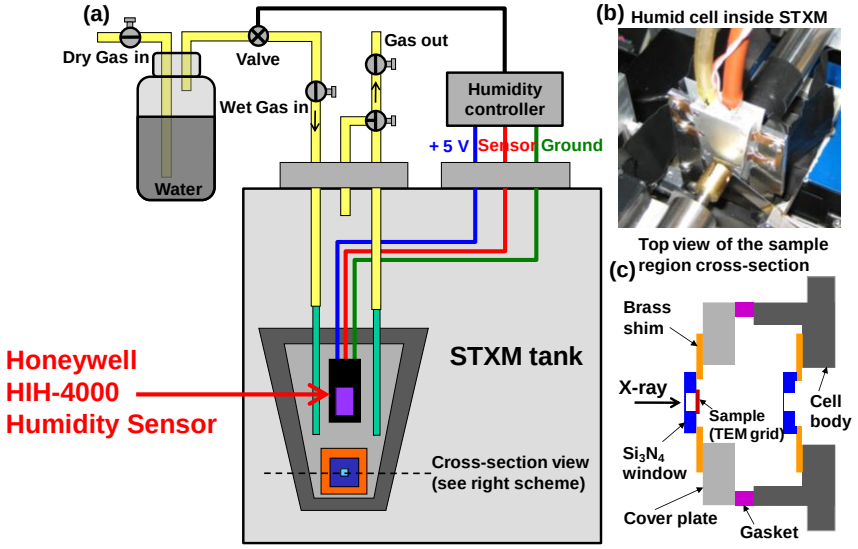
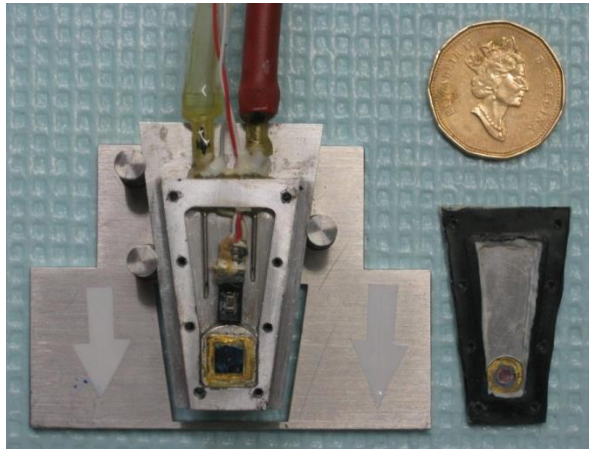
CLS-SM *In Situ* STXM – Humid Cell

In situ humidity control in STXM is achieved through an enclosed cell device with humidity monitoring and control. The cell is sealed by two Si_3N_4 windows to allow soft X-ray transmission though the cell. The humidity controller adjusts an electrically controlled proportional flow valve to control the flow rate of a wet helium gas stream to achieve a RH set point (RH = 0 - 1.0).



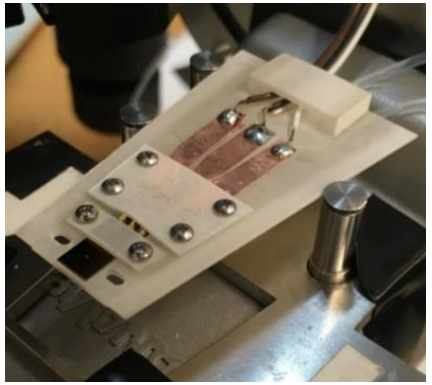


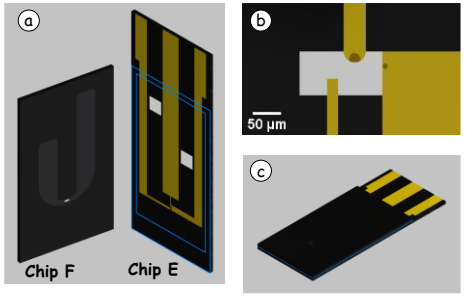
J. Wang et al. *J. Electron Spectrosc. Relat. Phenom.* 184 (2011) 296.

CLS-SM *In Situ* STXM – Electrochemical Flow Cell

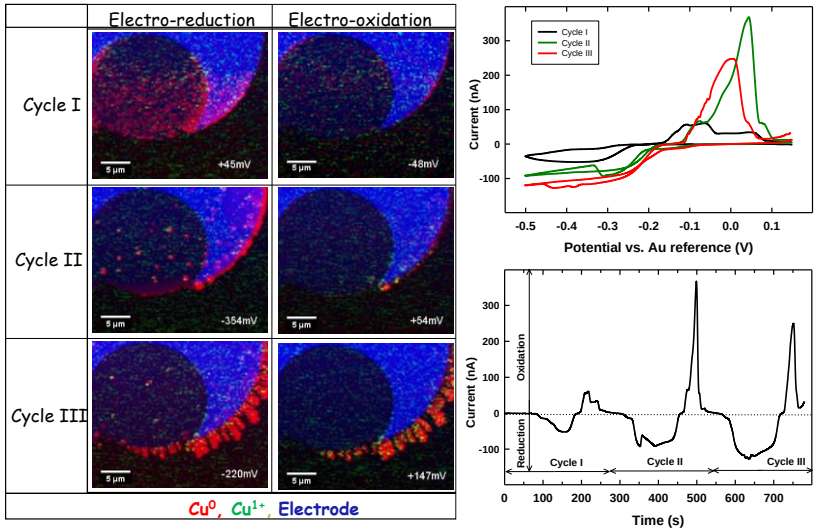
It is a 3-electrode device for real time *in situ* STXM studies of electrochemical processes under both static (sealed, non-flow) conditions and with a continuous flow of electrolytes. The device was made using a combination of silicon microfabrication and 3D printing technologies.

NORCADA STXM Electrochemical Flow Cell





- 20 nm thick gold electrodes
- Sandwiched windows
- Window surface special coating
- Liquid thickness up to 1.5 µm
- Redox cyclic voltammetry

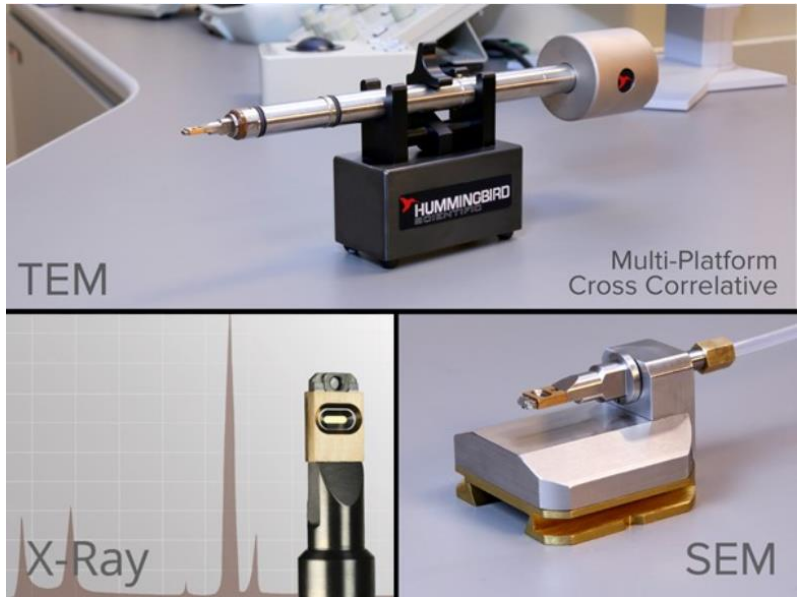


V. Prabu et al., Rev. Sci. Instrum. 89 (2018) 063702.

CLS-SM *In Situ* STXM – Electrochemical Flow Cell

It is a 6-electrode device for real time *in situ* STXM studies of electrochemical processes under a continuous flow of electrolytes. The device was made using *in situ* TEM technology with custom designed holder for STXM.

Hummingbird Scientific *In Situ* Electrochemical Device

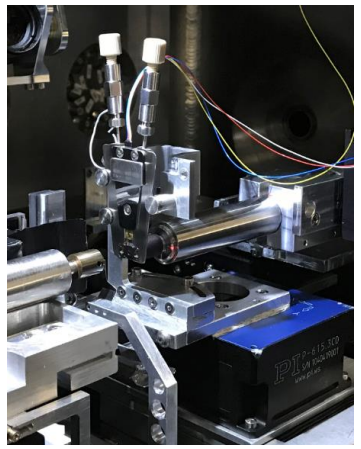


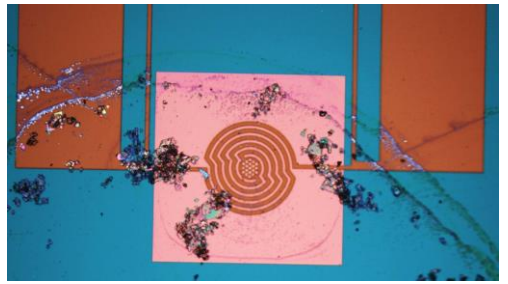
J. Lim et al., *Science* 353 (2016) 566-571.

CLS-SM *In Situ* STXM – Sample Heating

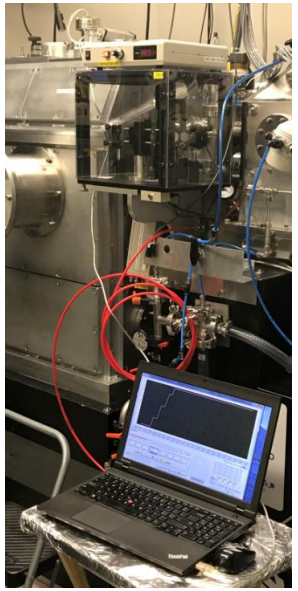
The NORCADA STXM sample heating device integrates a spiral micro-heater and temperature monitoring for RT to over 1000 °C. The device will be lent to CLS SM beamline for two years for free use and evaluation.

NORCADA Micro-Heater

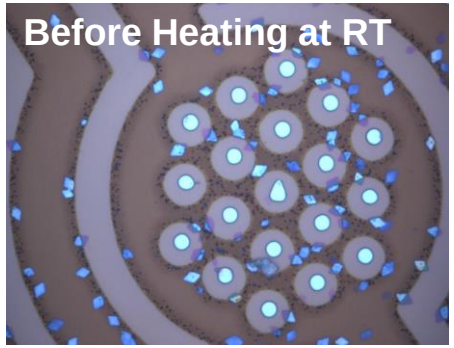


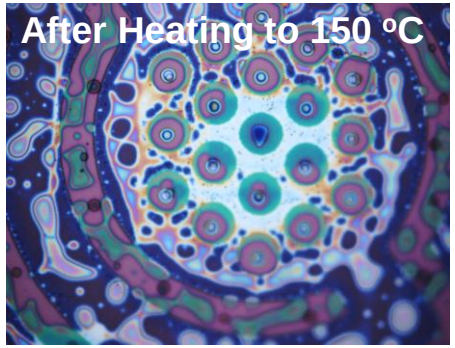


- Pt heater, RT – 1100°C
- Single Si_3N_4 window
- Heating controller, PC, software
- Gas flow and handling system (optional)

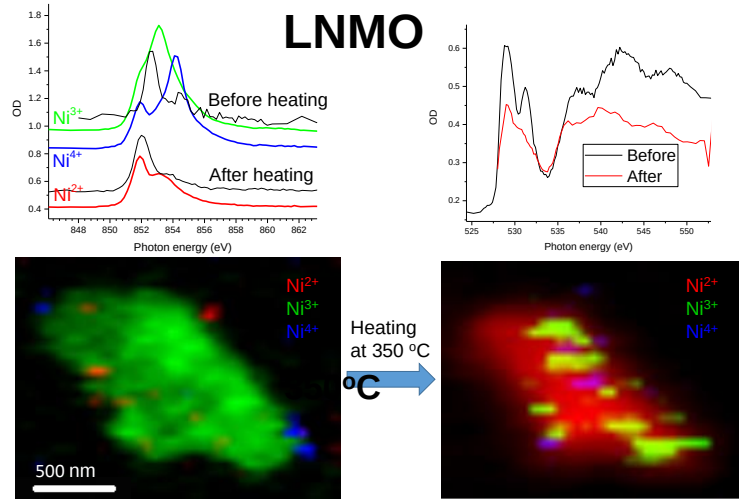


C32 Alkane Heating Experiment





LNMO

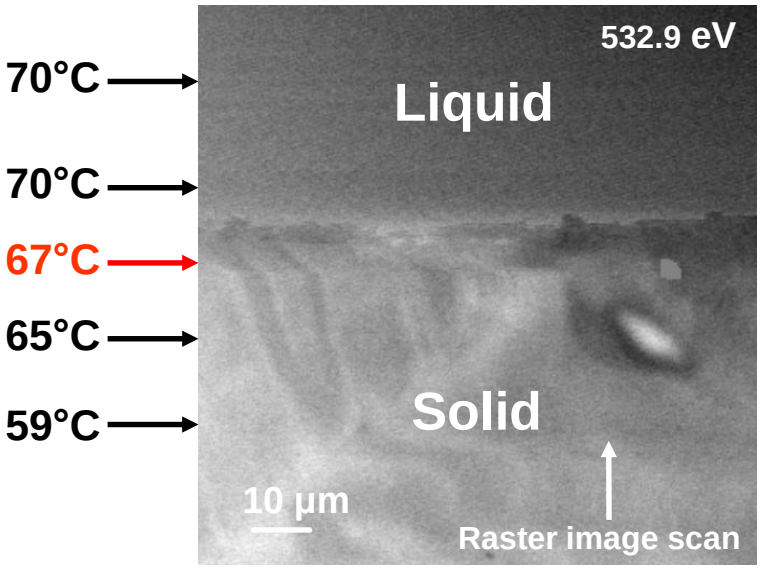


J. Zhou and J. Wang et al., CLS, Norcada and U. of Sask.

CLS-SM *In Situ* STXM – Sample Heating/Cooling

The custom STXM sample heating/cooling device utilizes a Peltier cooler for mild sample heating and cooling from about 0 °C to about 80 °C.

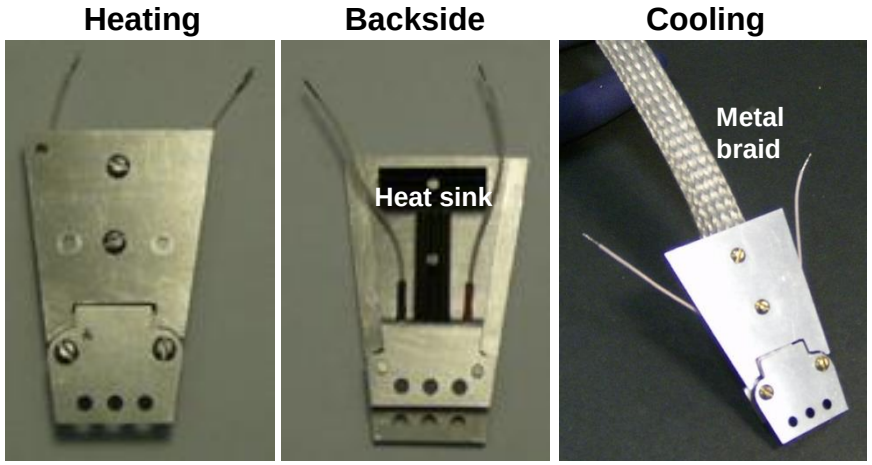
Stearic acid $CH_3(CH_2)_{16}COOH$, m.p.: 69°C



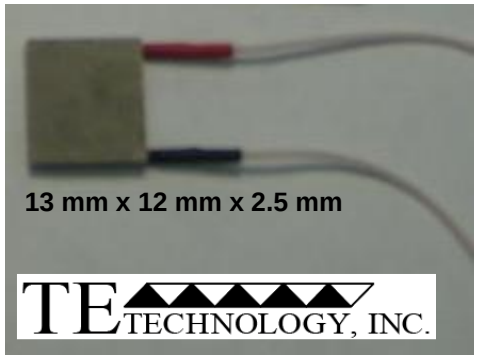
A.F.G. Leontowich et al. *Analyst* 137 (2012) 370.

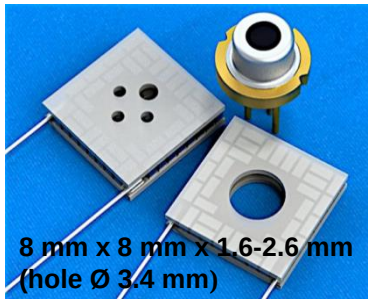
Peltier Heating/Cooling

Variable Temperature STXM Sample Plate



A.F.G. Leontowich, Chemistry, McMaster University.

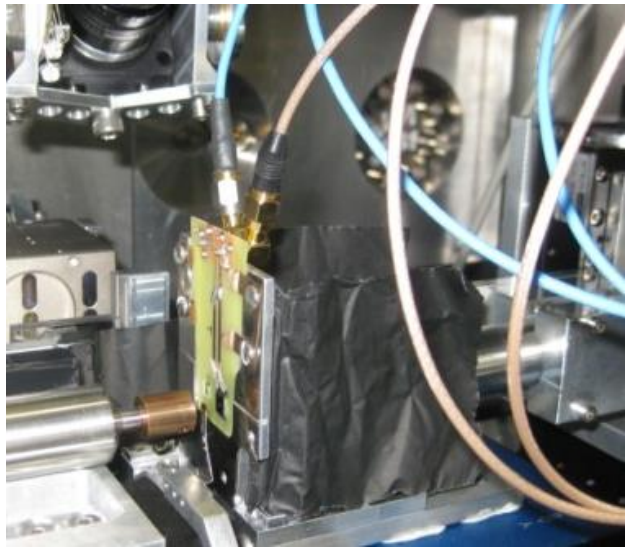


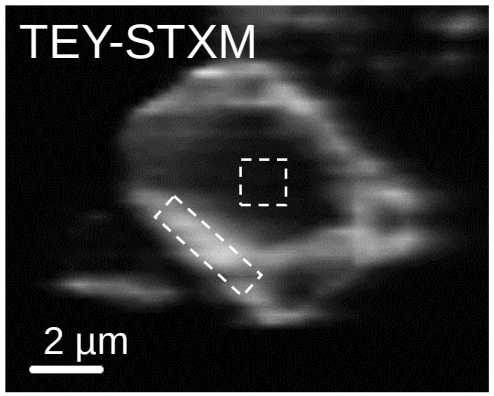


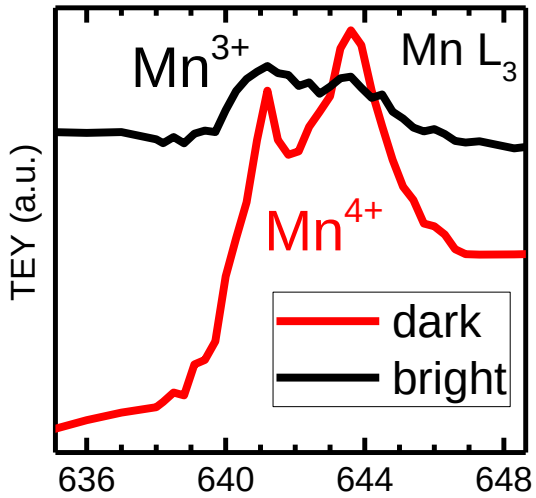
- -5°C – 230°C (Solder: SnSb) or 280°C (AuSn)
- ΔTmax = ~70°C
- Cooling efficiency relies on heat sink.

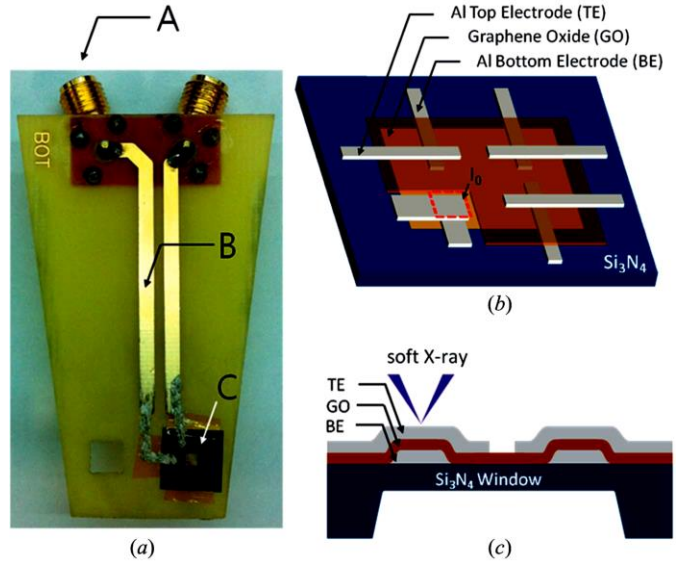
CLS-SM *In Situ* STXM – Electric/Electronic Device

The custom STXM sample electric/electronic device can apply DC bias voltage on samples or allow TEY drain current measurement from isolated samples.









J. Zhou, J. Wang et al., *PCCP* 18 (2016) 22789.

H.W. Nho et al. *J. Synchrotron Rad.* 21 (2014) 170.

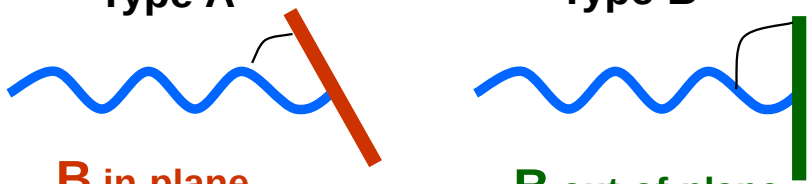
CLS-SM *In Situ* STXM – Permanent and Electro-magnets

These custom permanent and electro-magnets provide a wide range of in-plane and out-of-plane magnetic fields on STXM samples.

Permanent Magnets

Type A

Type B



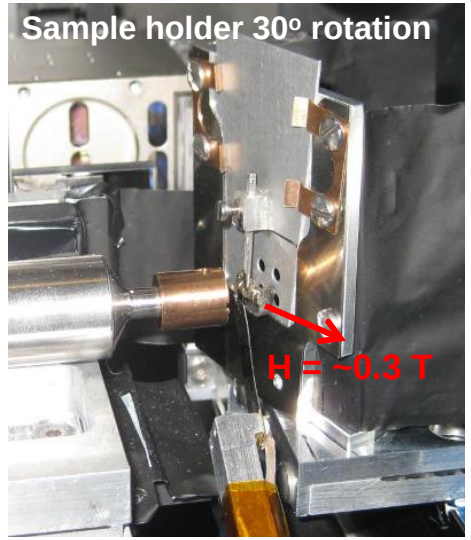
B in-plane
60º mounting

B out-of-plane
90º mounting

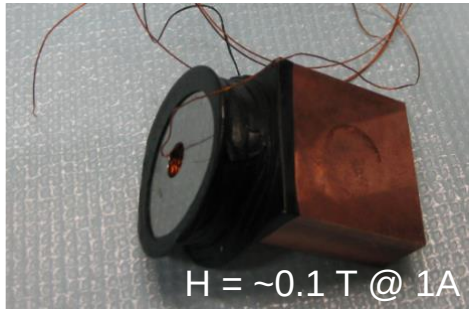
Dura Magnetics: NdFeB, Ø0.12” x 0.06”
Ø0.375” x 0.06” (hole Ø0.125”)

- **Type A:** 0.3 to 0.5 T in-plane magnetic field.
- **Type B:** 0.15 T out-of-plane magnetic field.

STXM-XMCD for In-Plane Magnetization



Electromagnets

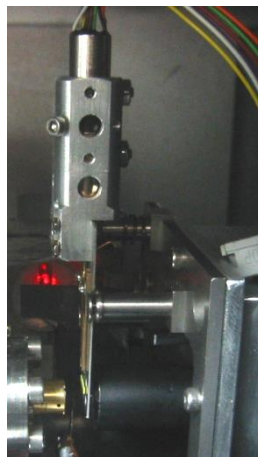


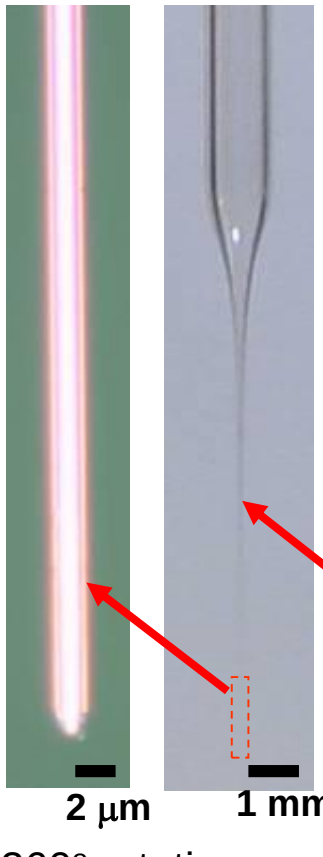
- **Electromagnets:** up to 0.1 T out-of-plane magnetic field.

CLS-SM *In Situ* STXM – Tomography

The glass capillaries and Luxel™ wet cells allow *in situ* STXM tomography for hydrate or liquid samples.

Glass Capillary Tomography

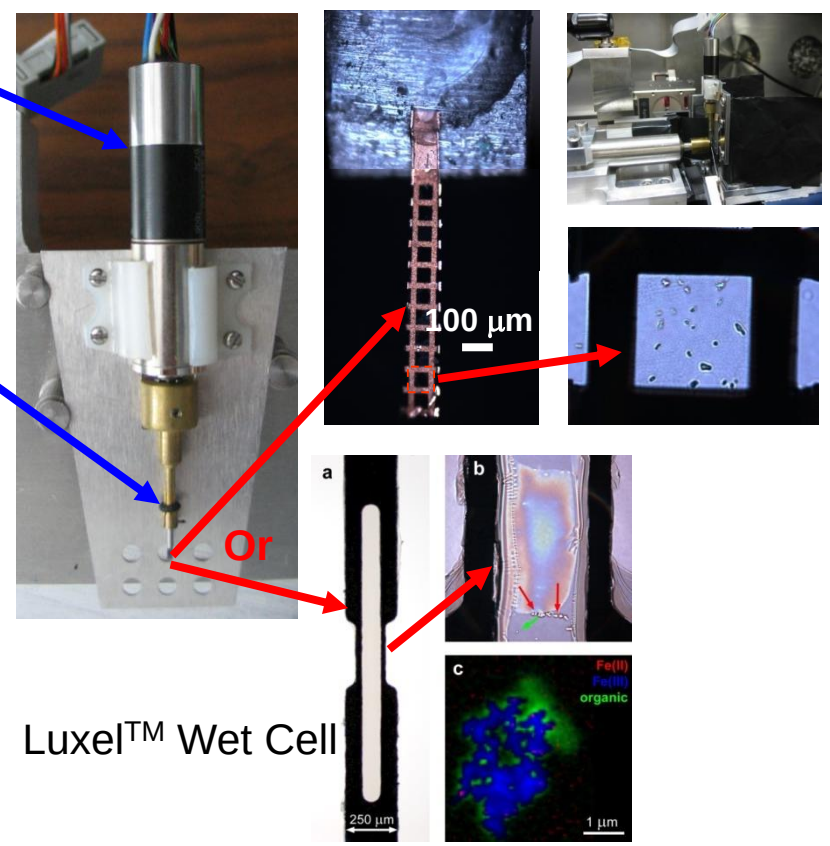




- 0º – 360º rotation
- Rotation wobble: < 300 μm
- Used for O 1s edge and above.

G.A. Johansson et al. *J. Sync. Rad.* 14 (2007) 395.

TEM Grid and Wet Cell Tomography



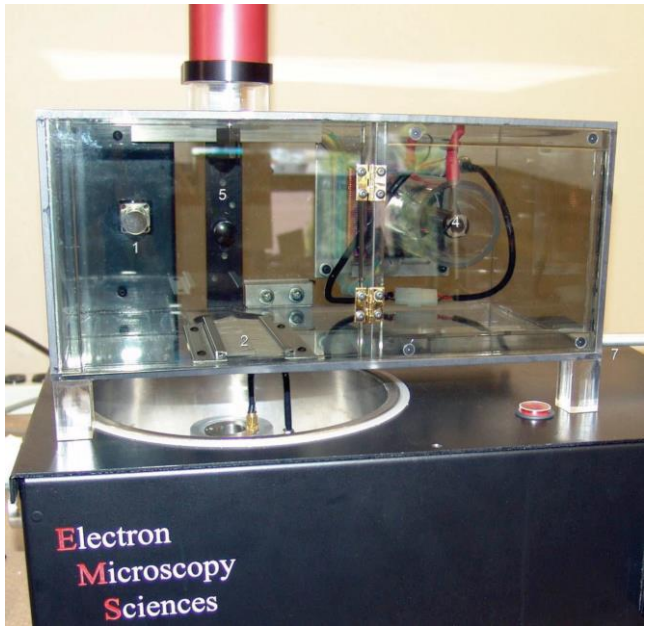
- -70º – +70º rotation
- Rotation wobble: < 200 μm
- Used for S 2p and C 1s edges and above.

M. Obst, J. Wang, A.P. Hitchcock, *Geobiology* 7 (2009) 577.

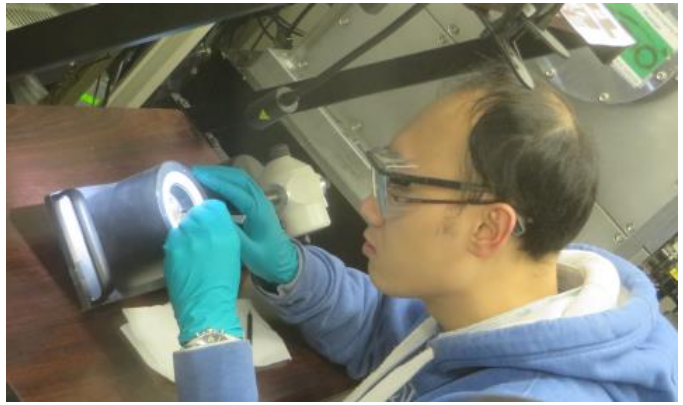
CLS-SM *In Situ* Cryo-STXM

Cryogenic sample preparation, handling, and measurement by Cryo-STXM allow in vivo hydrate samples to be analyzed.

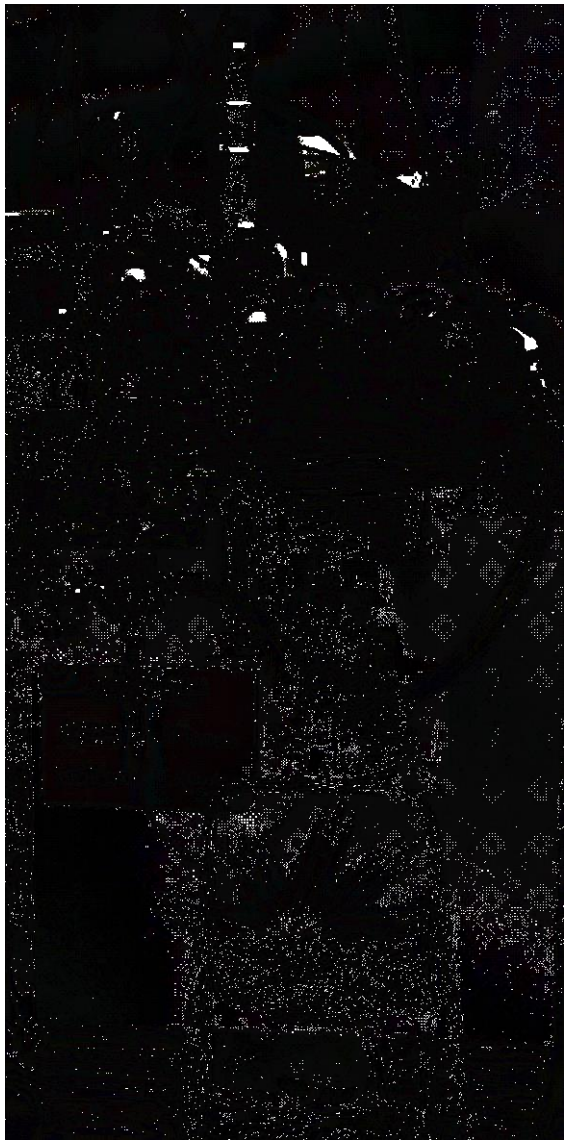
EMS-002 Cryo Workstation



Gatan Model 630.DH Workstation



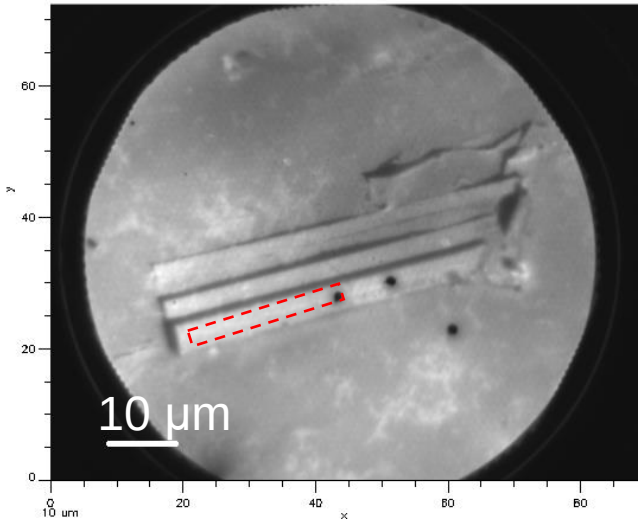
SM Cryo-STXM

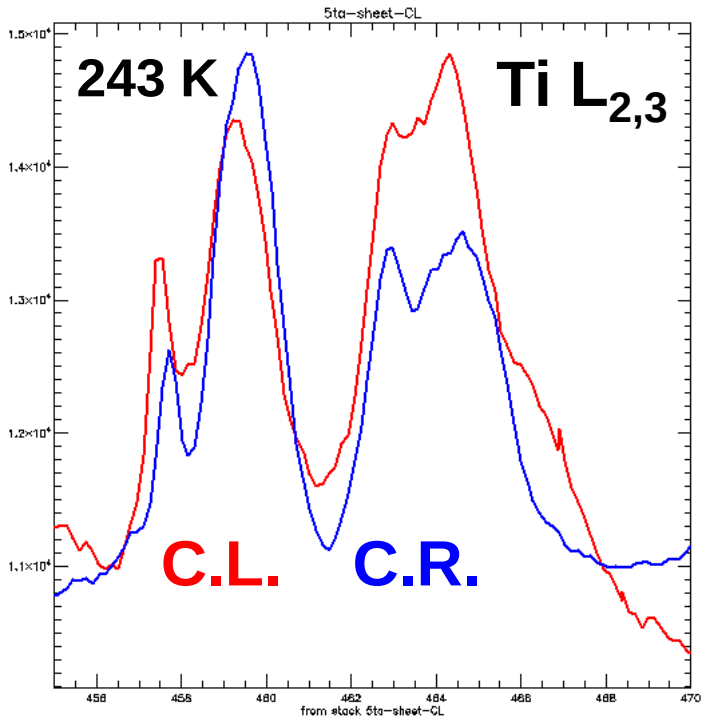


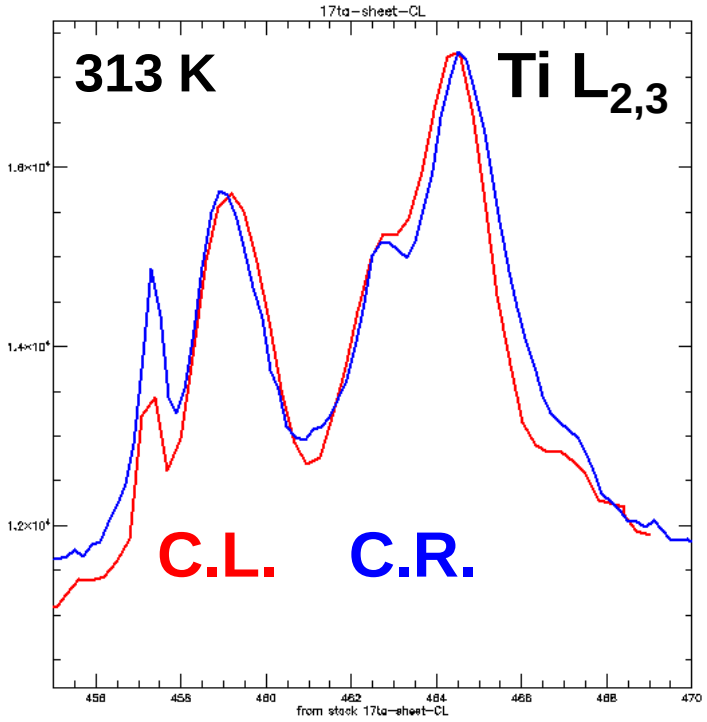
CLS-SM *In Situ* X-PEEM - Cooling

X-PEEM samples can be cooled to 120K for magnetic studies or other phase transition studies.

TiS_3 Nanosheets on Cr_2O_3(0001) Surface







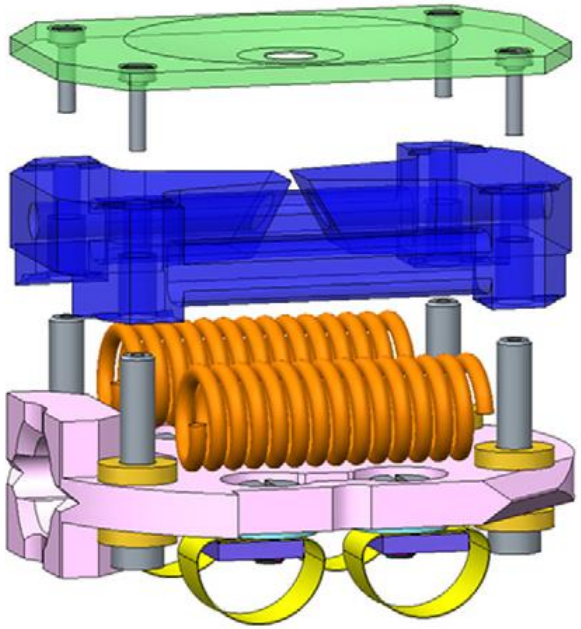
- *In situ* X-PEEM cooling and heating study of XMCD effect of TiS_3 nanosheets
- Magnetic moment flipping when sample was cooled below critical point temperature at 307 K

Canadian Light Source | Centre canadien de rayonnement synchrotron

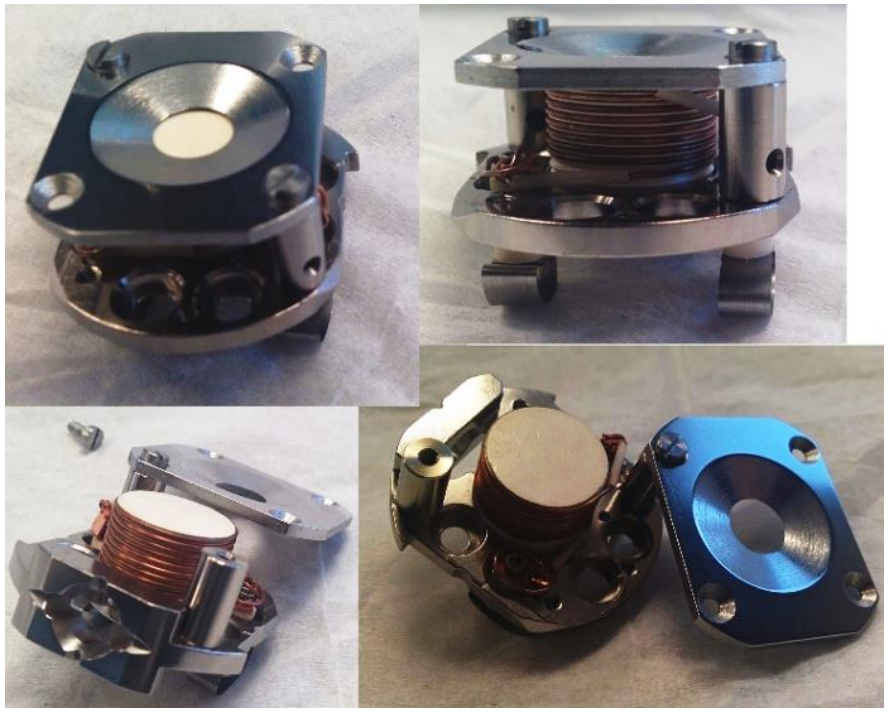
CLS-SM *In Situ* X-PEEM – Electromagnets

The PEEM magnetic sample holder with modifications can provide both in-plane and out-of-plane weak magnetic fields on the sample.

In situ Magnetic Sample Cartridge In-Plane Magnetic Field



In situ Magnetic Sample Cartridge Out-of-Plane Magnetic Field

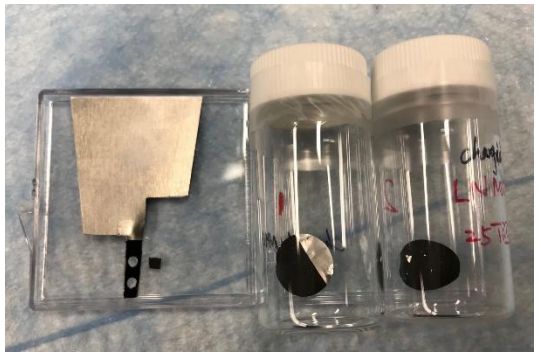


- UHV compatible
- Ceramic core with soft iron inner core, magnetic field up to 100 Gauss
- Use heater electrical leads for electric current application

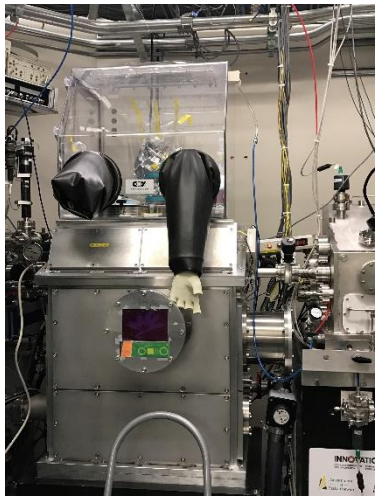
CLS-SM Portable Glovebox and Nano-coater

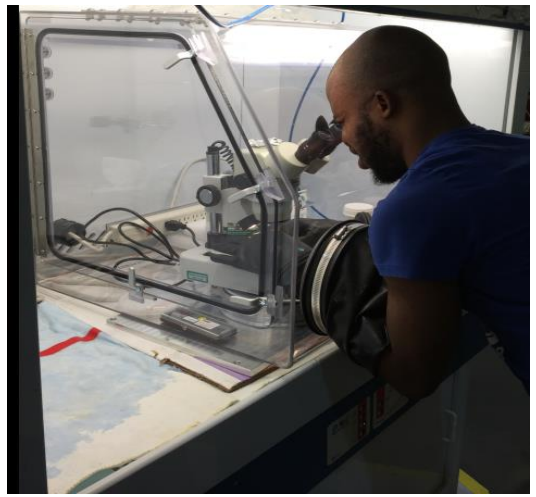
The SM portable glove box can be mounted on Ambient-STXM or used as a standing alone equipment for air sensitive sample handling, mounting or transferring for both STXM and X-PEEM. A nano-coater is available for X-PEEM sample surface modification or protection.

Glove Box for Ambient-STXM

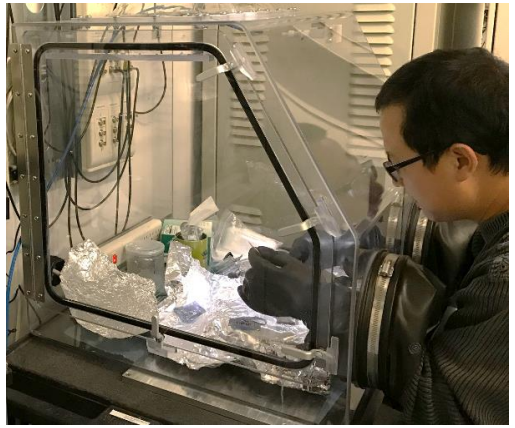


Portable glove box for air sensitive STXM sample mounting





Glove Box for X-PEEM



Portable glove box for air sensitive X-PEEM sample mounting

Nano-coater