Technique Development on the Scanning Transmission X-ray Microscope at Canadian Light Source

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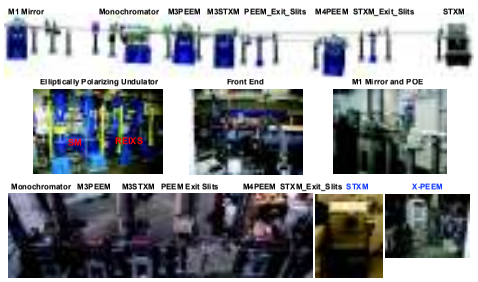
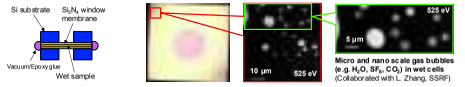
CLS 10ID-1 SM Beamline



In situ STXM

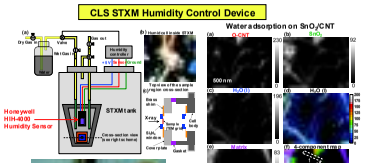
Wet Cells



Micro and nano scale gas bubbles (e.g. H_2O, SF_6, CO_2) in wet cells (Collaborated with L. Zhang, SSRF)

Humidity-Control Device

CLS STXM Humidity Control Device



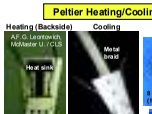
E-Cell (A. Hitchcock, McMaster U.)

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

- Enclosed device sealed by two Si_3N_4 windows, 0-100% Relative Humidity control
- Nebulizer water vapour carried by air-free helium gas, Peltier heating/cooling: -20°C – 100°C (E-cell)
- Application: water adsorption in nanomaterials (e.g. SnO_2/CNT), water effect in fuel cell operation

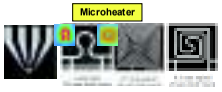
Sample Heating/Cooling

Peltier Heating/Cooling



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

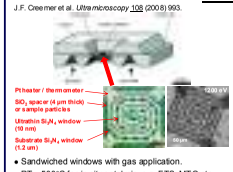
Microheater



- **Silson Microheater**: gold heater, RT – 500°C
- Single Si_3N_4 window
- STXM: 1.5 V, 25 mA, T = ~130°C (helium environment) (H.W. Nho, Chemistry, Hanyang University)

Nanoreactor

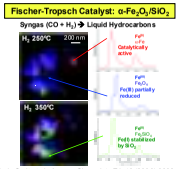
J.F. Creemer et al. *Ultramicroscopy* 108 (2008) 993.



- Sandwiched windows with gas application.
- RT – 500°C for *in situ* catalysis, e.g. FTS, MTO etc.

F.M.F. de Groot and B.M. Weckhuysen, Utrecht University

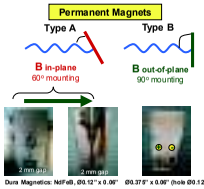
Fischer-Tropsch Catalyst: α-Fe_2O_3/SiO_2



E. de Smit et al. *Angew. Chem. Int. Ed.* 48 (2009) 3632.

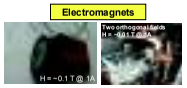
In situ Magnets

Permanent Magnets

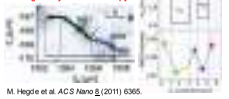


- **Type A:** 0.3 to 0.5 T in-plane magnetic field.
- **Type B:** 0.15 T out-of-plane magnetic field.
- **Electromagnets:** up to 0.1 T magnetic field.

Electromagnets

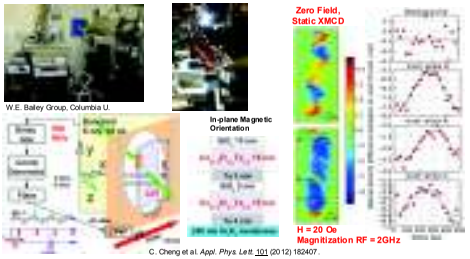


STXM-XMCD of a Single Mn:GaN Nanowire: Magnetocrystalline Anisotropy



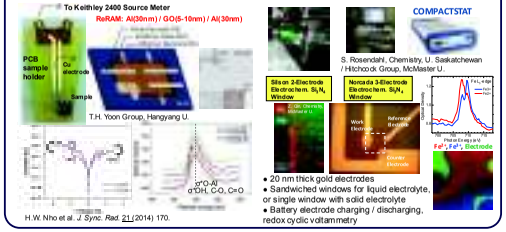
M. Hegde et al. *ACS Nano* 6 (2011) 6365.

XMCD Magnetization Dynamics



C. Cheng et al. *Appl. Phys. Lett.* 101 (2012) 182407.

Electric/Electronic and Electrochemical

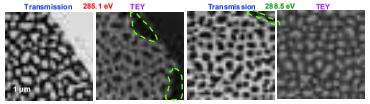


H.W. Nho et al. *J. Sync. Rad.* 21 (2014) 170.

- 20 nm thick gold electrodes
- Sandwiched windows for liquid electrolyte, or single window with solid electrolyte
- Battery electrode charging / discharging, redox cyclic voltammetry

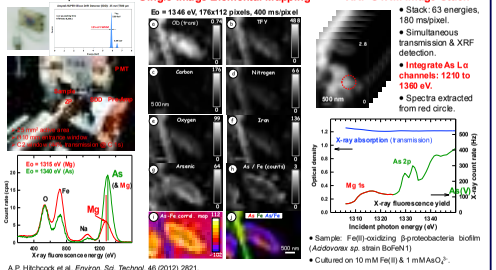
TEY-STXM

S.G. Urquhart Group, Chemistry, University of Saskatchewan.



S. Behyan et al. *AIP Conf. Proc.* 1365 (2011) 184.

LEXRF-STXM



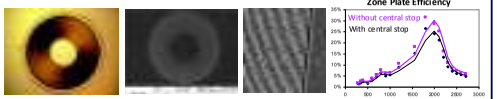
- Stack: 63 energies, 180 ms/pixel.
- Simultaneous transmission & XRF detection.
- **Integrate As Lα channels: 1210 to 1360 eV.**
- Spectra extracted from red circle.

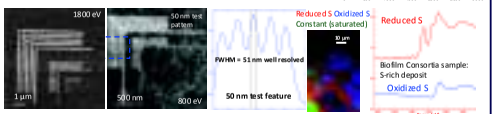
- Sample: Fe(II)-oxidizing β-proteobacteria biofilm (Acidovorax sp. strain BoFeN1)
- Cultured on 10 mM Fe(II) & 1 mM AsO_4^{3-}.

A.P. Hitchcock et al. *Environ. Sci. Technol.* 46 (2012) 2821.

- XRF detection limit about 0.001%, lower by about two orders of magnitude than transmission
- **K-shell XRF**: C*, N*, O, F, Ne, Na, Mg, Al, Si, P*, S*, etc. (*marginally detectable)
- **L-shell XRF**: Ca*, Sc*, Ti*, V*, Cr*, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Kr, Rb, Sr, Y*, Zr*, etc.

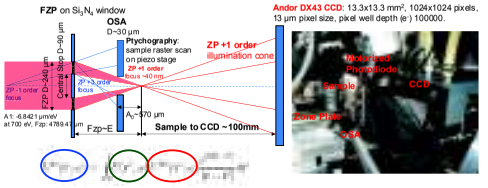
High Energy Zone Plate



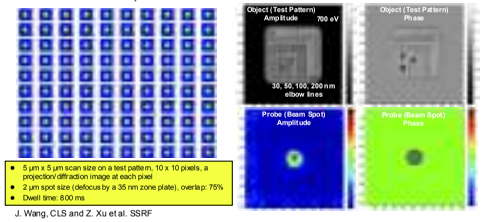


- 50 nm high energy zone plate developed by Applied NanoTools Inc., collaborated with CLS SM Beamline staff (J. Wang et al.) and the SM Beamteam (A.P. Hitchcock et al.)
- Close to theoretical efficiency at nearly all photon energies, flux ~2 MHz at the S 1s edge (2470 eV) with 60x60 μm slits

STXM-Ptychography



- **Diffraction (Scattering) Pattern: Fourier Transform** of **Object** and **Probe** function. **Inverse Fourier Transform: Phase Retrieval**.
- **STXM-Ptychography:** CCD image acquisition synchronized with image scan pixel clock, a robust diffractive microscopic technique aims to achieve highest spatial resolution (i.e. sub-5 nm).
- CCD x- and z-direction travel distance: 10 mm.
- CCD water cooling down to -50 ~ -45 °C.
- Motorized photodiode between sample and CCD.
- Beamline shutter controlled by CCD.

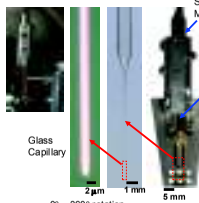


- 5 μm x 5 μm scan size on a test pattern, 10 x 10 pixels, a projection/diffraction image at each pixel
- 2 μm spot size (defocus by a 35 nm zone plate), overlap: 75%
- Dwell time: 800 ms

J. Wang, CLS and Z. Xu et al. SSRF

STXM Spectro-tomography

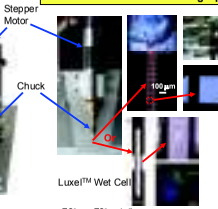
Glass Capillary Tomography



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

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

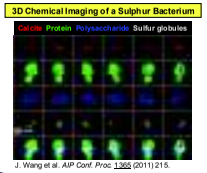
TEM Grid and Wet Cell Tomography



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

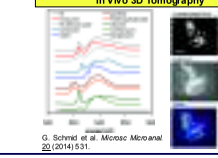
M. Obst, J. Wang, A.P. Hitchcock, *Geobiology* 7 (2009) 577.

3D Chemical Imaging of a Sulphur Bacterium



J. Wang et al. *AIP Conf. Proc.* 1365 (2011) 215.

Hydrated BoFeN1 Bacteria in LuxeI™ Wet Cell: In Vivo 3D Tomography



G. Schmid et al. *Microsc. Microanal.* 20 (2014) 531.

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