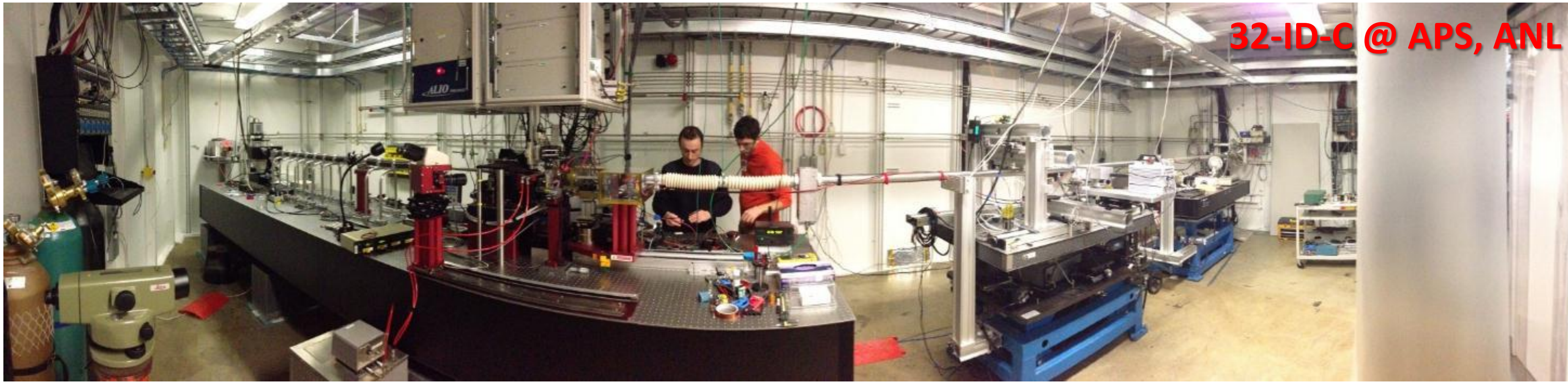
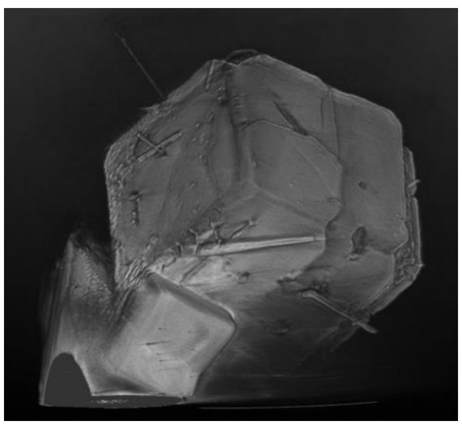
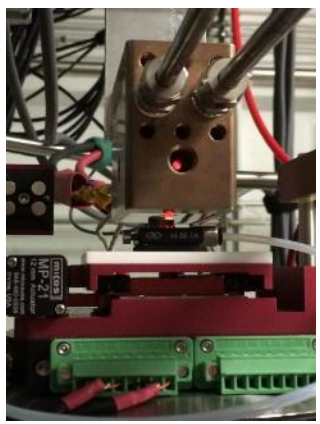
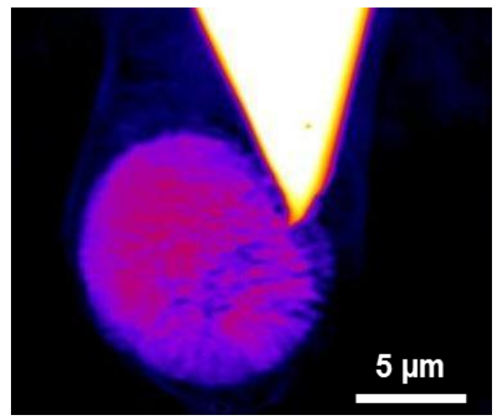
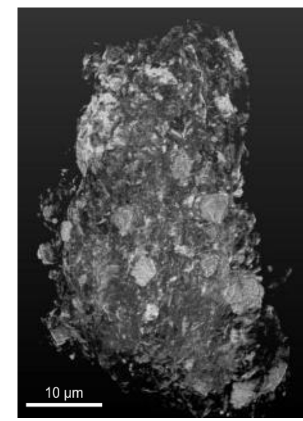
Nano-imaging with TXM at 32-ID: First year of operation











V. De Andrade, A. Deriy, M. Wojcik, D. Gürsoy, F. De Carlo

TWG talk, September 17th, 2015

U.S. DEPARTMENT OF ENERGY

Outline

I. TXM overview: specs and capabilities

II. Instrument upgrade

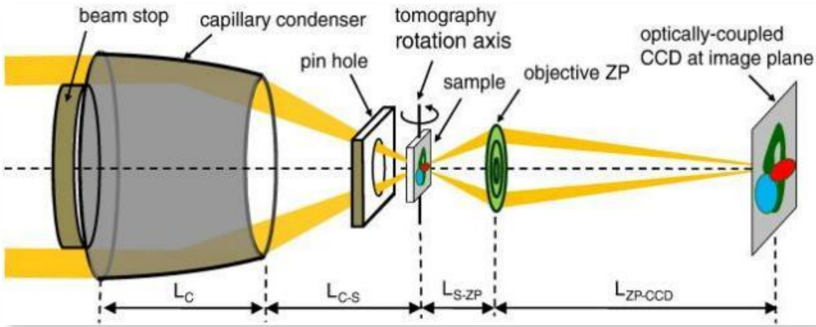
1. Temperature controlled area
2. Vibrations measurement
3. Improvement plan
4. Increasing efficiency with optics
5. Increasing detection efficiency
6. Increasing efficiency with smart reconstruction algorithms

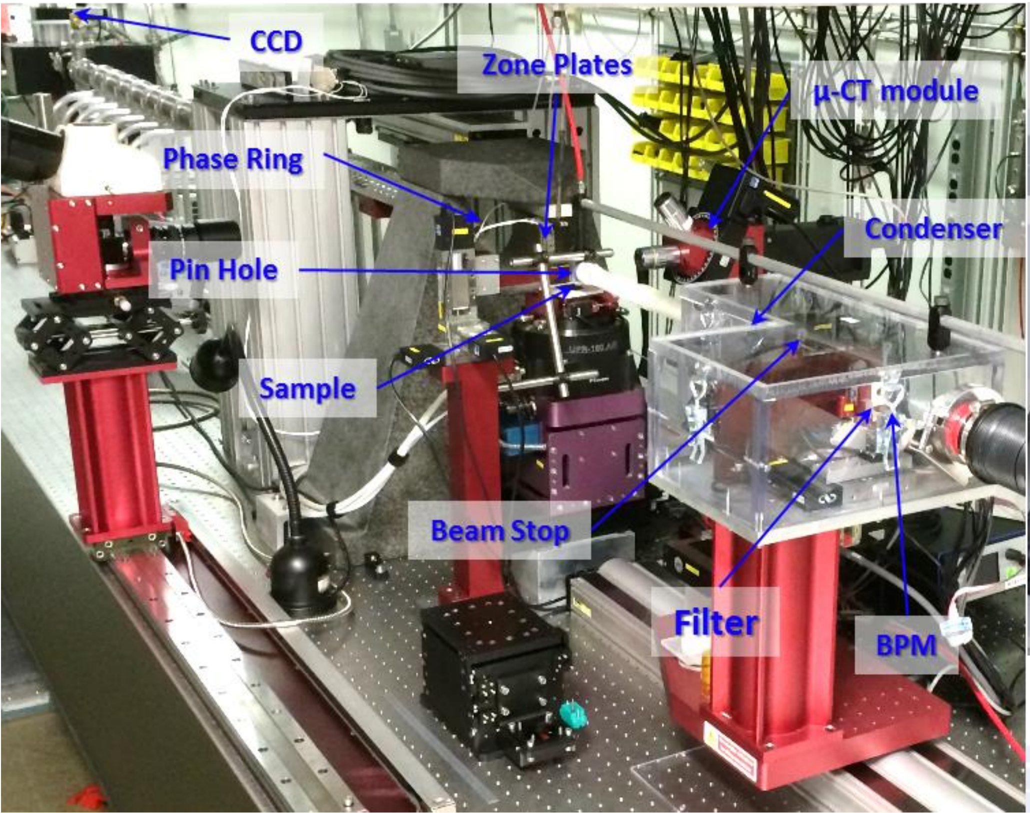
III. Quick overview of ongoing scientific experiments

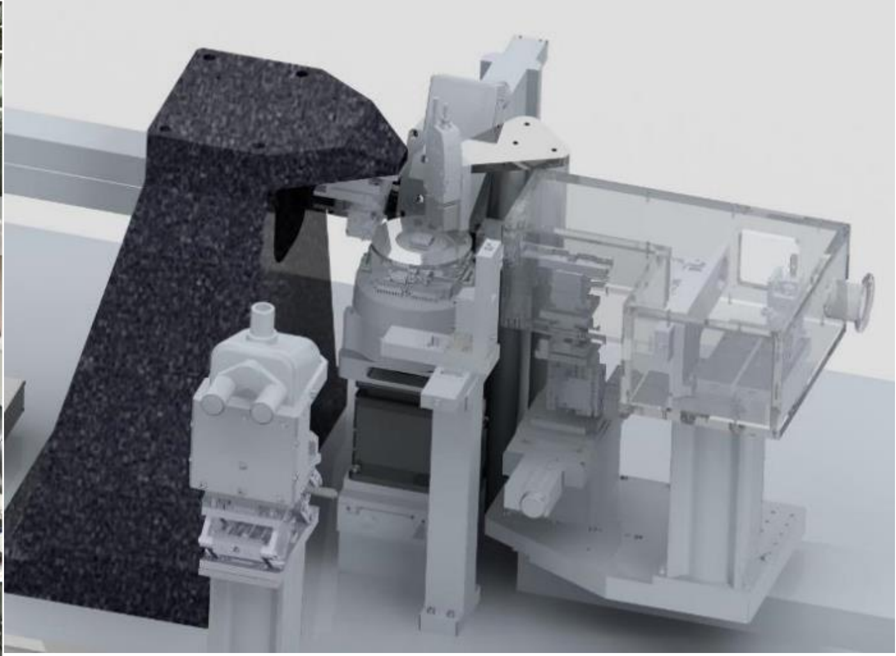
I. TXM overview: specs and capabilities

A new Transmission X-ray Microscope (TXM) has been developed and installed at 32-ID-C:

- Resolution: aiming to be the highest resolution full-field imaging system at APS (<20 nm)
- Energy range: 6 to 14 keV, $\Delta E/E = 10^{-4}$
- Techniques: combination of nano-scale imaging with absorption, phase contrast & spectroscopy
- *Multi-scale approach with an integrated μ-CT module*
- Operation in a wide range of samples environments (*-160ºC* < T < 1500ºC, P up to 100 GPa), chemical bath







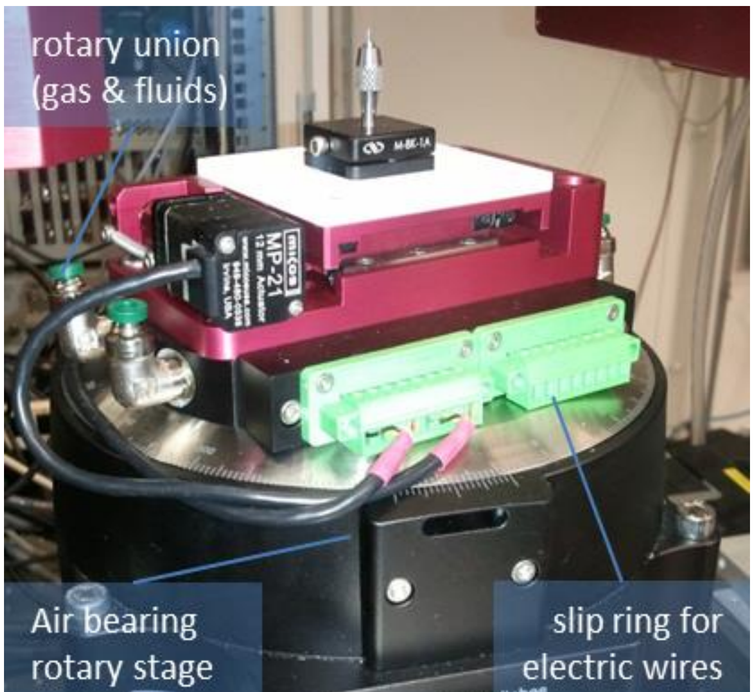
U.S. DEPARTMENT OF ENERGY

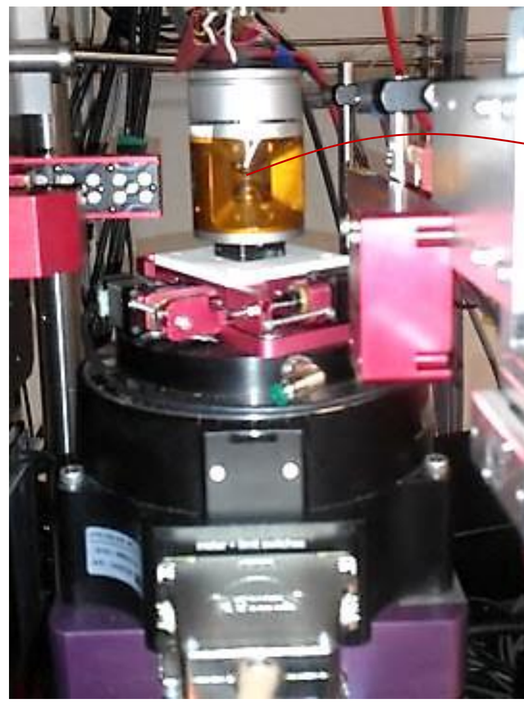
I. TXM overview: specs and capabilities

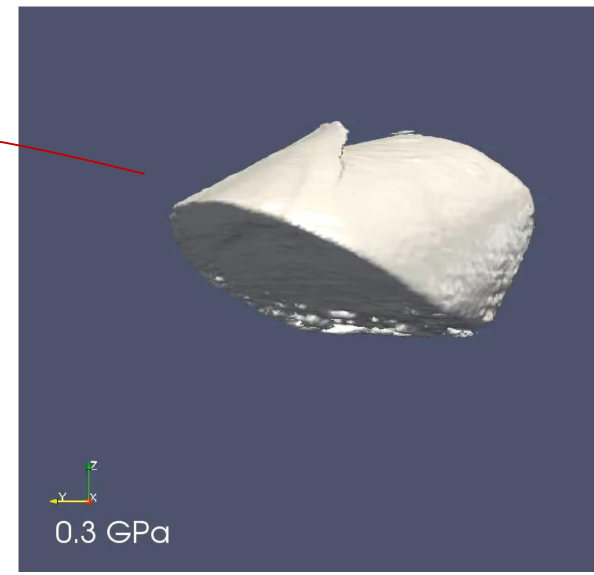
In-situ capabilities

in situ CT imaging with:

- ✓ *Electric and gas feed through allowing for a full rotation range*
- ✓ *mixed gas capability*
- ✓ *from -160 ºC to 1500 ºC*
- ✓ *High load capacity (e.g., 1 kg Diamond Anvil Cells can be accomodated)*







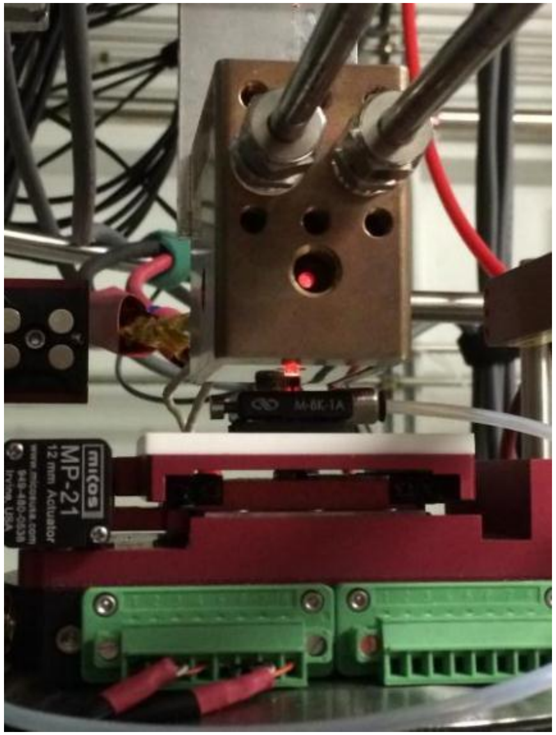
Diamond **A**nvil **C**ell (DAC)

Experiment performed by H. Liu and his team (HPSTAR, China) on metallic glass. Reconstruction with ASTRA toolbox by D Pelt.

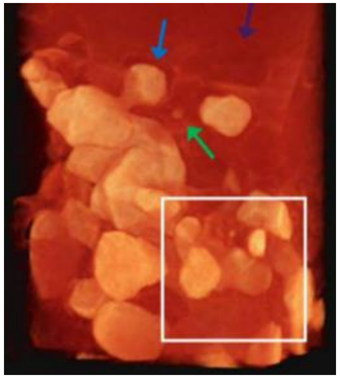
I. TXM overview: specs and capabilities

In-situ capabilities

In situ study of a solid oxide fuel cell anode

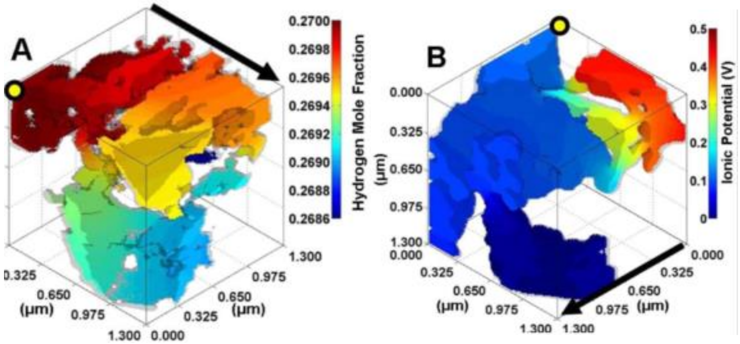


Experiment conducted by W. Chiu, A. Cocco and M. Degostin (Univ. of Connecticut) on Ni oxide fuel cell anode



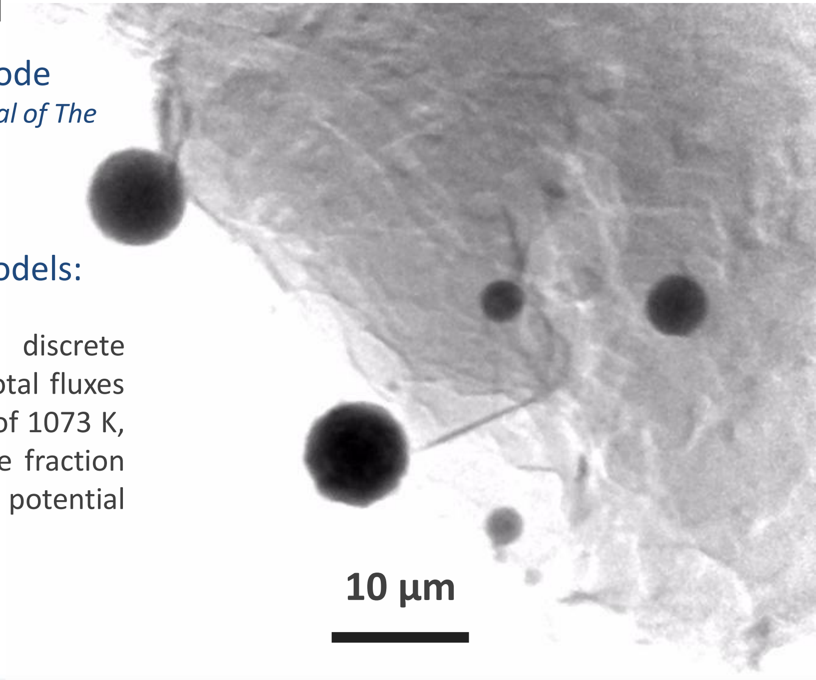
Ni distribution in a fuel cell anode

After K. N. Grew, Y. S. Chu *et al.*, *Journal of The Electrochemical Society*, 2010.



The anode reconstructed volume feeds 3D numerical models:

3D scalar distributions of the discrete transport. Results correspond to total fluxes at a current density of 3 A/cm^2, T^o of 1073 K, and include the [A] hydrogen mole fraction in the pore phase and [B] ionic potential differences in the YSZ phase.



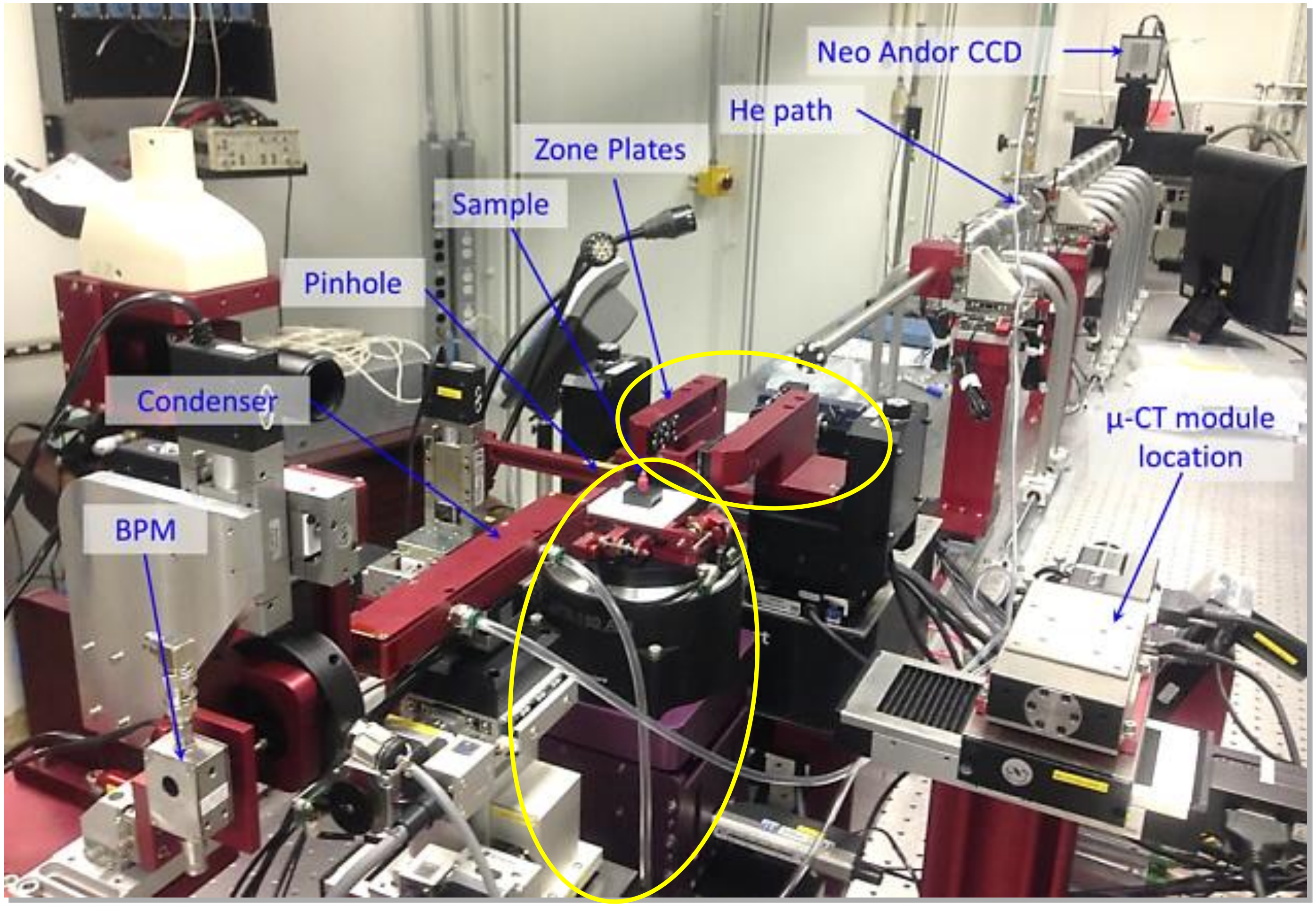
II. Instrument upgrade

Remarks about stability

1) ideally: small and stiff → high resonant frequency
   minimization of degree of freedom

2) minimize thermal expansion effects:
   - → choice of materials
   - → symmetric geometry
   - → T° controlled area

3) Diagnostic systems
   BPM



II. Instrument upgrade

1. Temperature controlled area

→ routing of the Hall AC system inside the hutch

→ installation of a closed loop system between an oven and sensor above the sample

→ laminar flow

→ air vestibule

<u>Issue:</u>

Historic choice of motors imposes to keep controllers inside the hutch

→ stages being replaced as fast as possible

II. Instrument upgrade

2. Vibrations measurement: from the floor to the sample

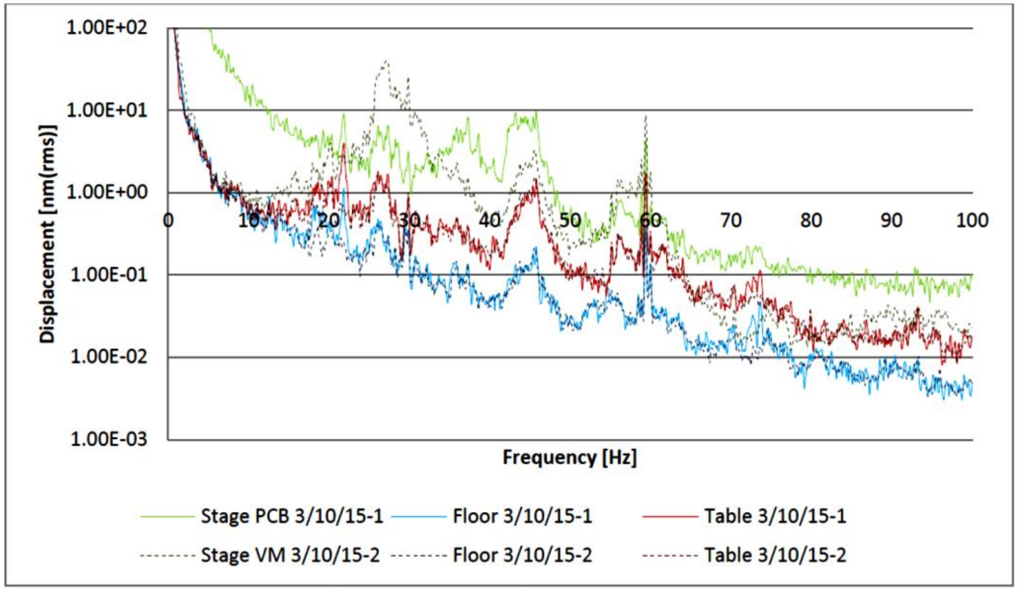


Figure 4: PCB Vs VibraMetric Accelerometers Perpendicular to Beam-Line Displacement Spectra 0-100Hz

Table 7: Peak Perpendicular Displacements Taken on Feb. 17, 2015

	Mag. [nm]	Freq. [Hz]	Mag. [nm]	Freq. [Hz]	Mag. [nm]	Freq. [Hz]	Mag. [nm]	Freq. [Hz]	Mag. [nm]	Freq. [Hz]
Floor	0.73	26.25	0.45	29.88	0.19	36.00	0.17	43.25	0.50	59.50
Table	2.33	26.25	4.52	29.88	0.91	36.00	0.78	43.25	3.46	59.50
Stage	7.28	26.25	7.81	29.88	12.58	36.00	11.51	43.25	6.93	59.50

Sample stage:

Horizontal motion: 13 nm (rms) → amplitude: 52 nm

Vertical motion: 4.4 nm rms → amplitude: 18 nm

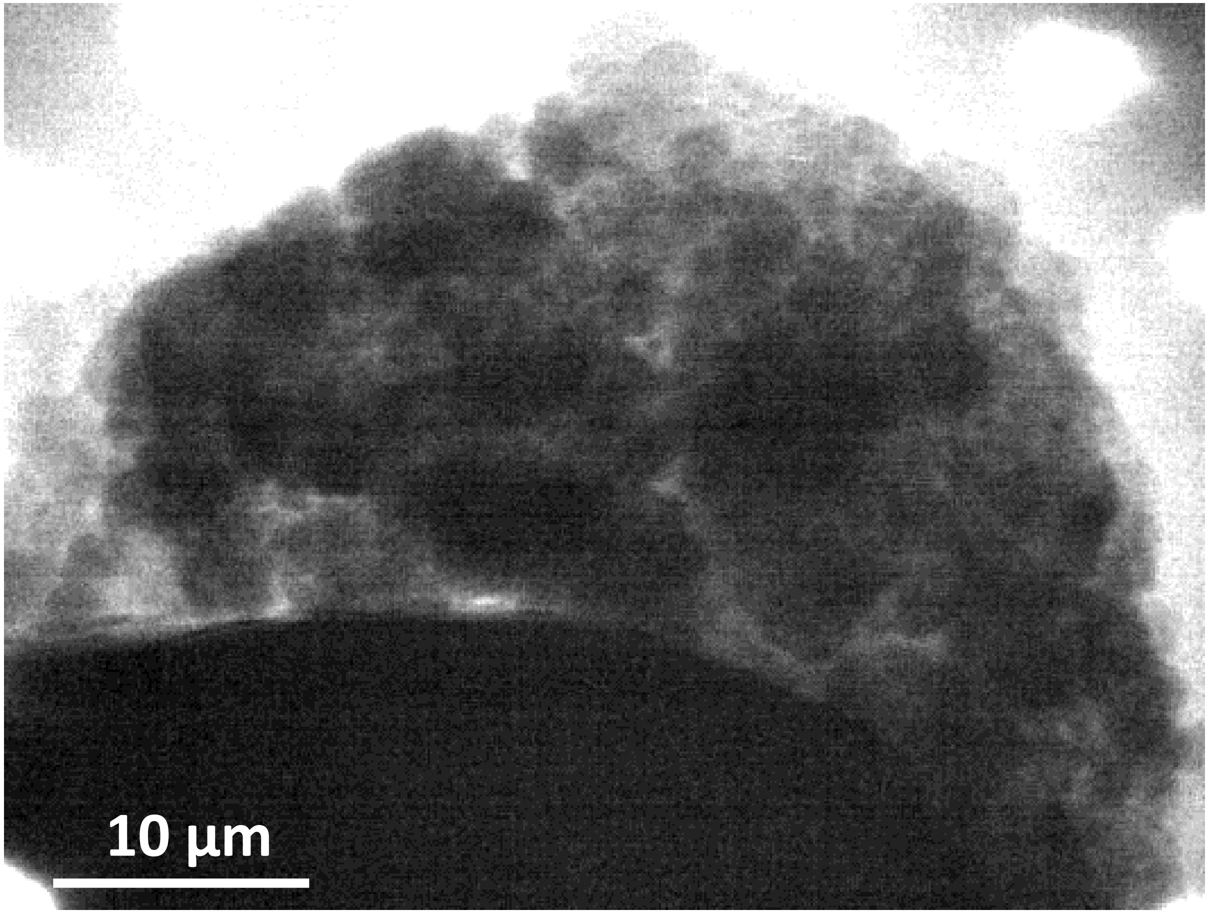
Measurements performed by Jayson Anton & Deming Shu

II. Instrument upgrade

2. Vibrations measurement: from the floor to the sample

20 ms exposure time

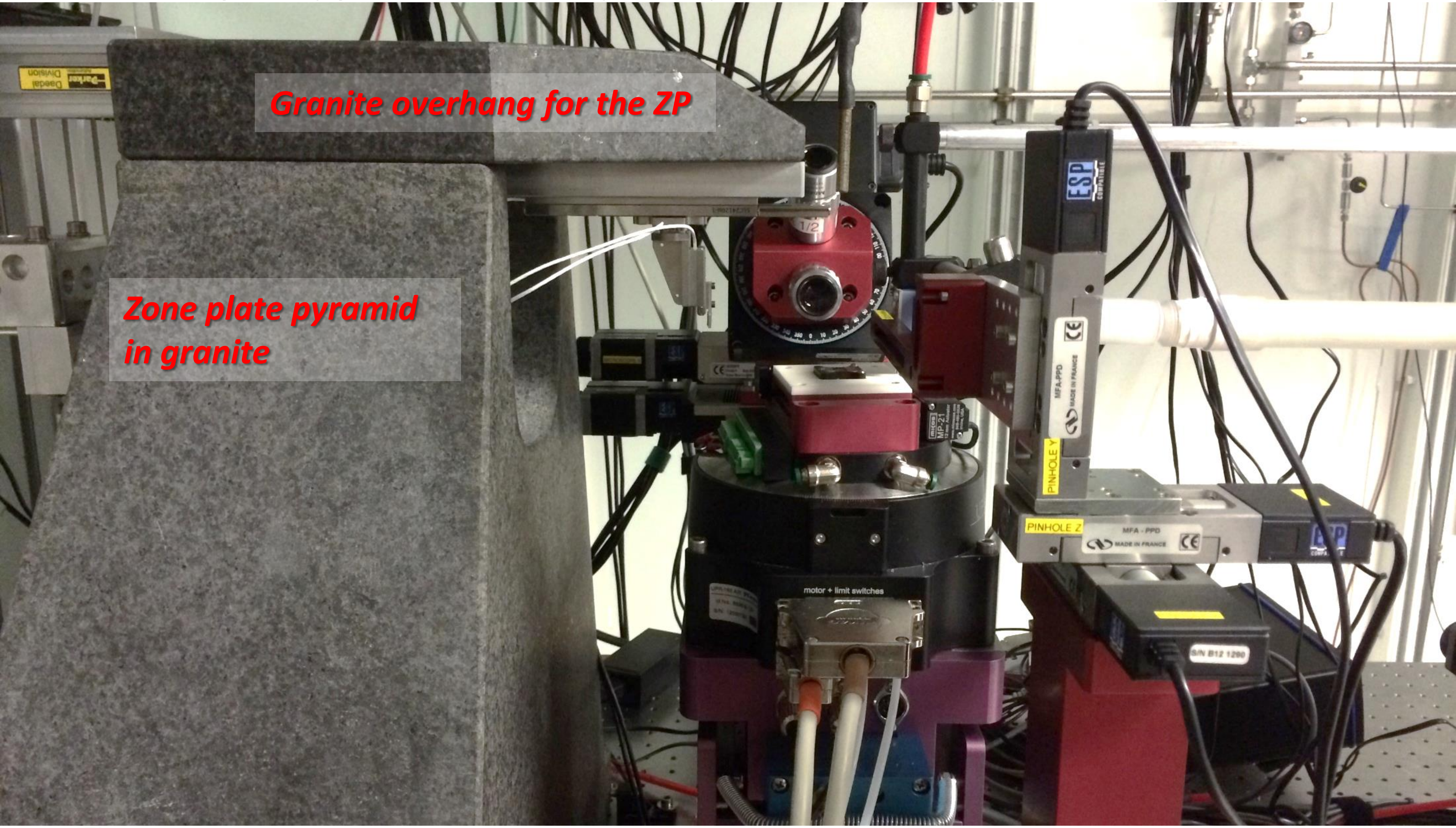
Battery sample



II. Instrument upgrade

3. Improvement plan

→ Redesign & upgrade of the zone pate & phase ring holder *(granite designed by D. Shu)*



II. Instrument upgrade

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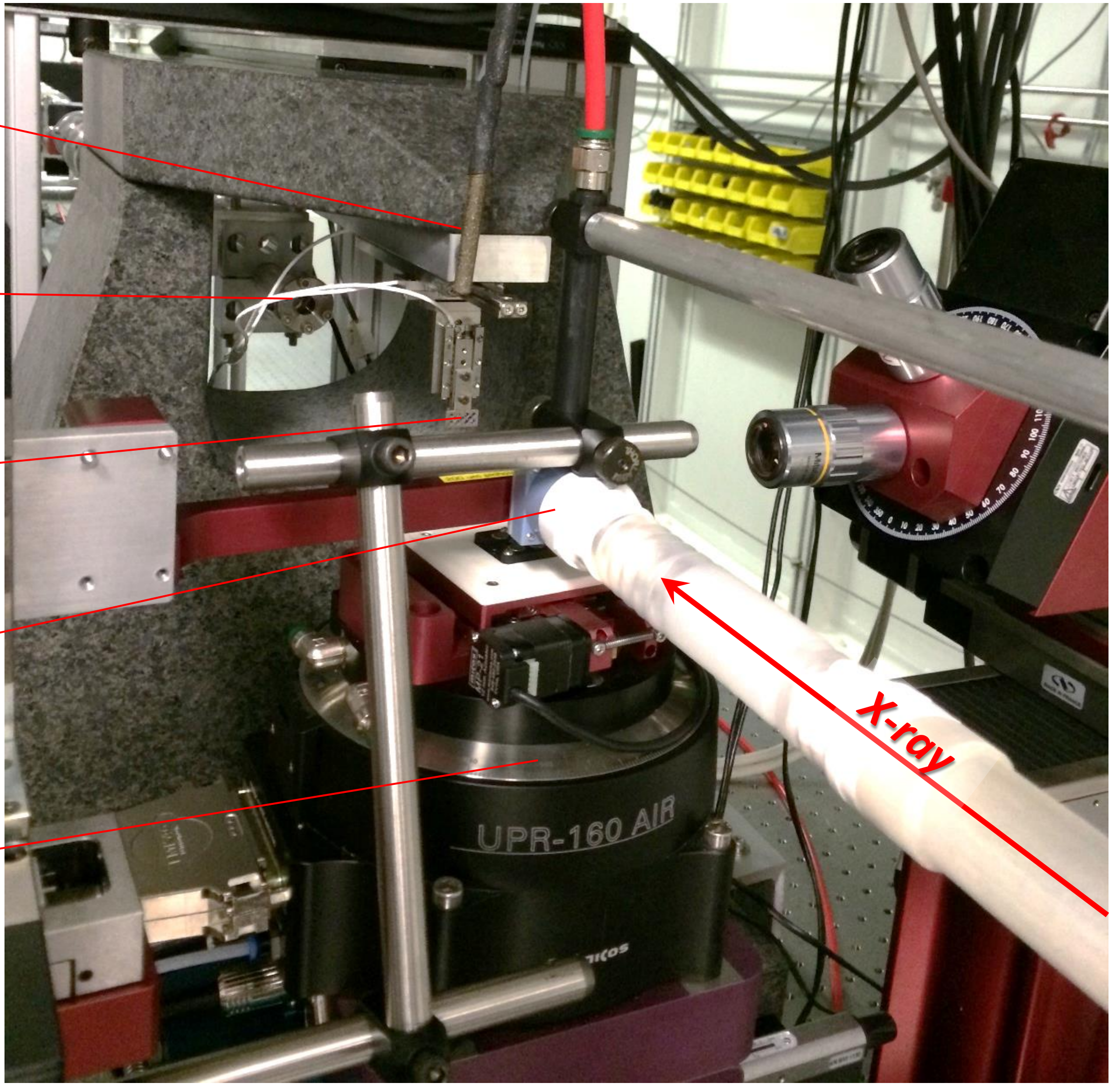
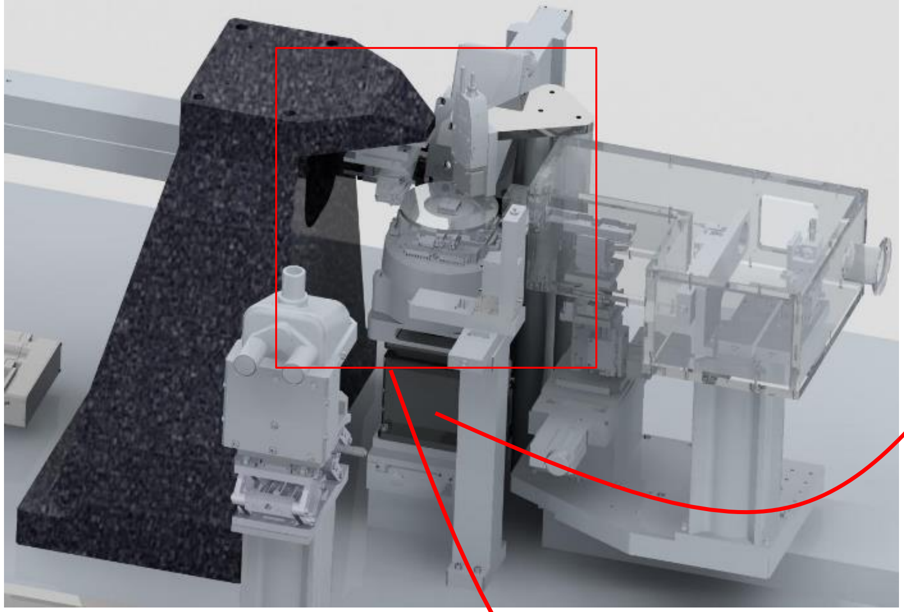
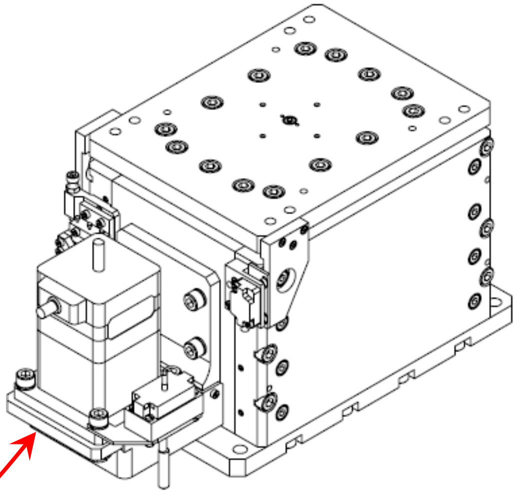


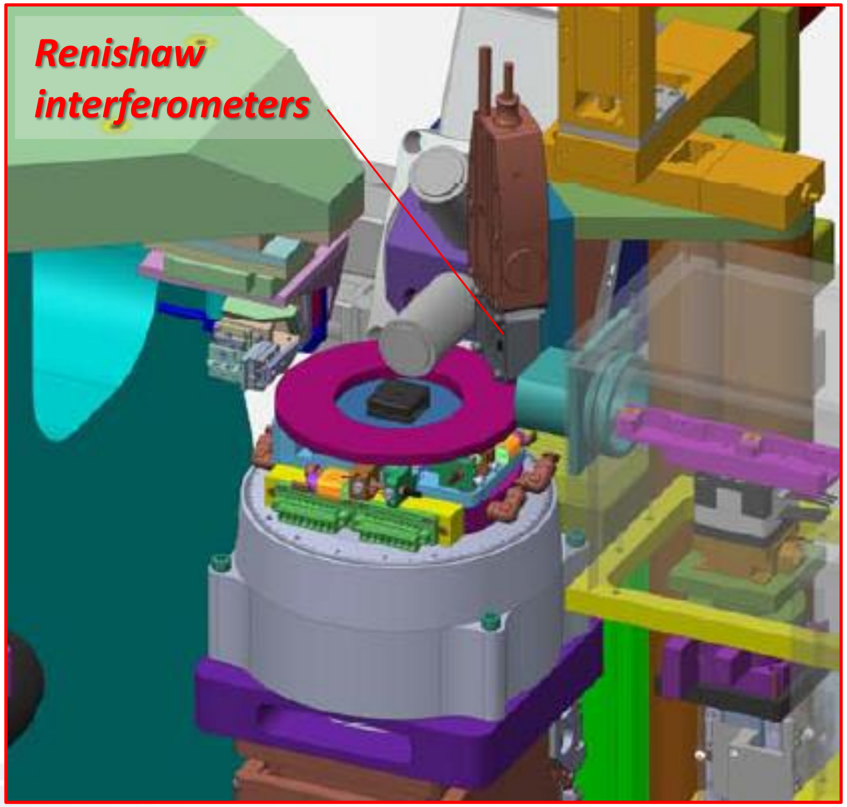
II. Instrument upgrade

3. Improvement plan





Modified T2-24 vertical stage
(Deming Shu design)
Almost 1:1 vibration transmission from the table



Remove 2 redundant degrees of freedom:
X Z below the rotation

ESRF characterized outstanding spindle stages:
Wobble: <0.1 µrad
Radial error: 33 nm @ 102 mm
Axial error: 14 nm @ 102 mm

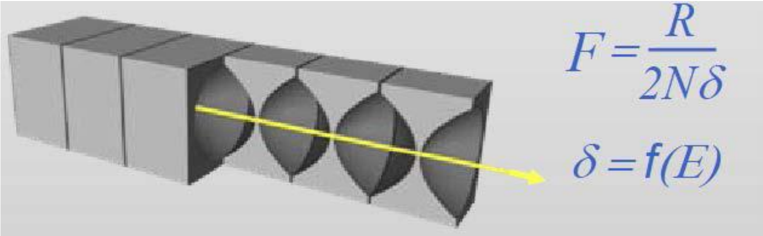
→ no need of runout correction

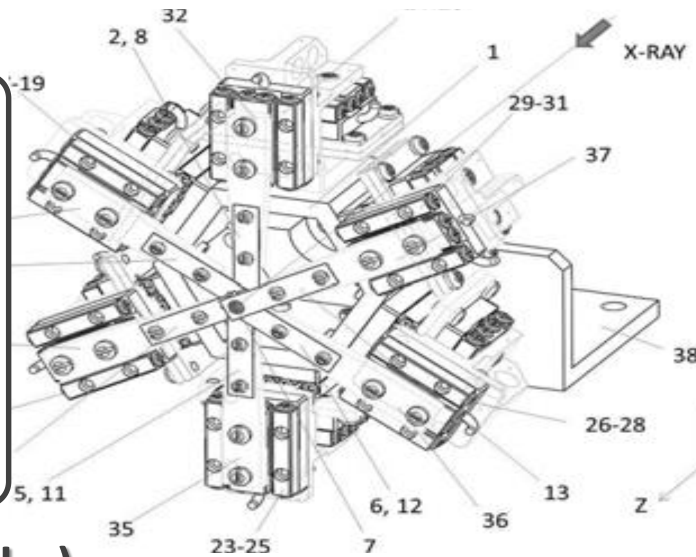
II. Instrument upgrade

4. Increasing efficiency with optics

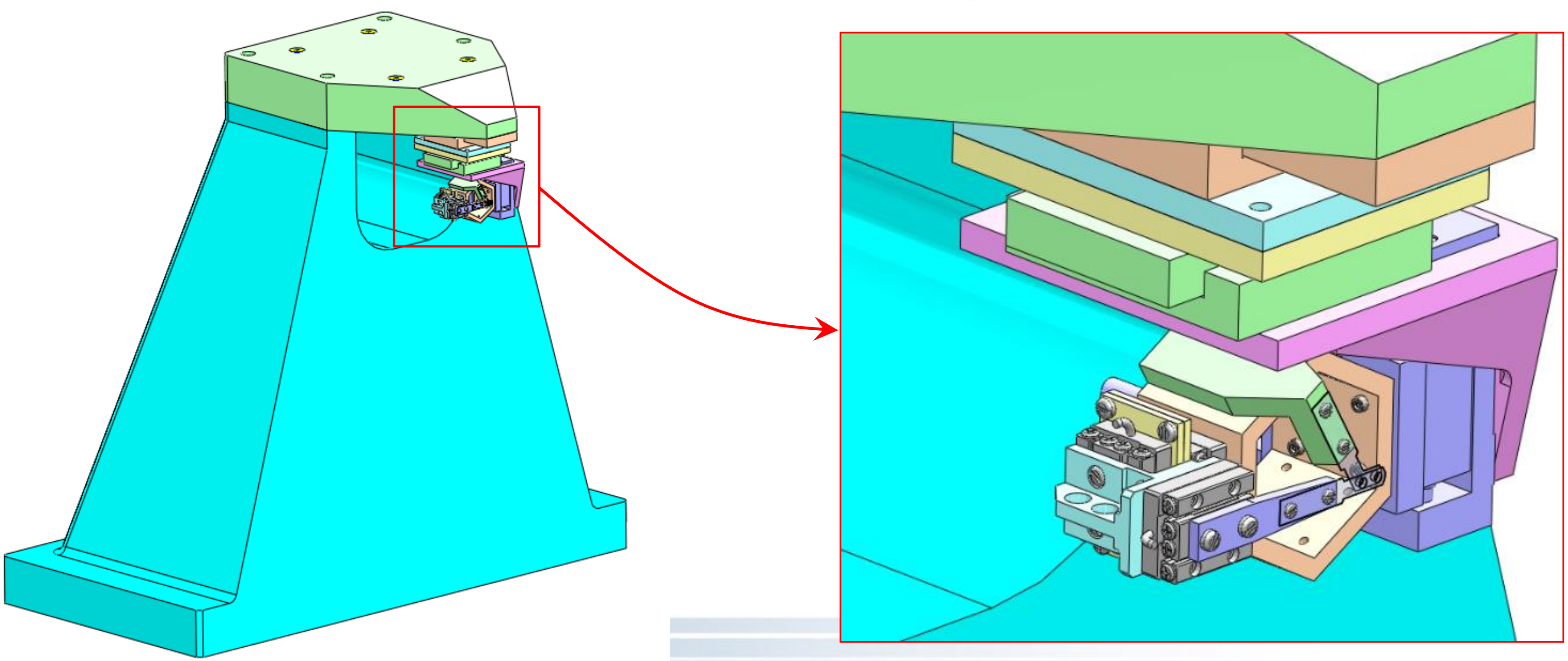
Transforming XIA filters system into a Transfocator:

R. Reininger simulations: expected flux increase from 5 to 10 times





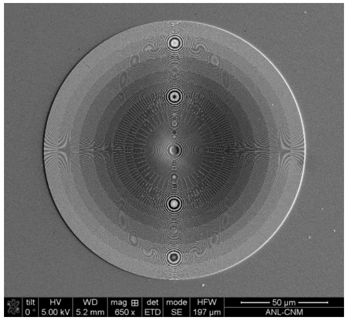
Zone plate stacking (D. Shu)



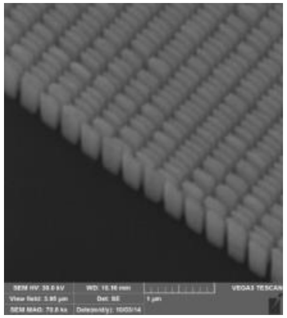
II. Instrument upgrade

4. Increasing efficiency with optics

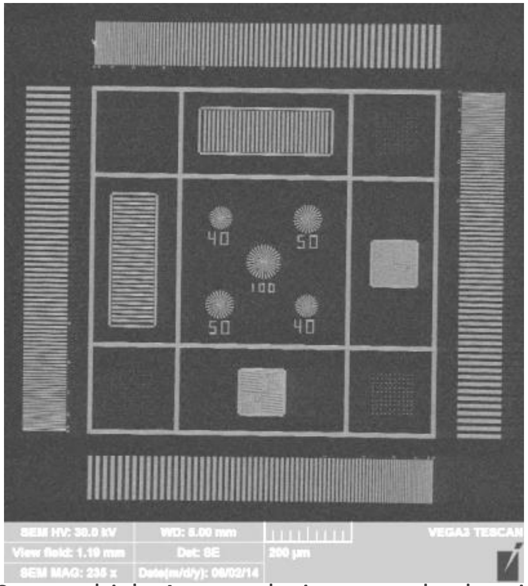
- Development of optics with higher efficiency and spatial resolution (M. Wojcik):
  - 16 nm Δr_n Fresnel **Z**one **P**late (ZP)
  - Stacking zone plates (D. Shu design)
  - beam shaping condenser for high quality phase contrast



20 nm Δr_n ZP fabricated using the zone doubling technique. Zones thickness: 550 nm, compo= Ir

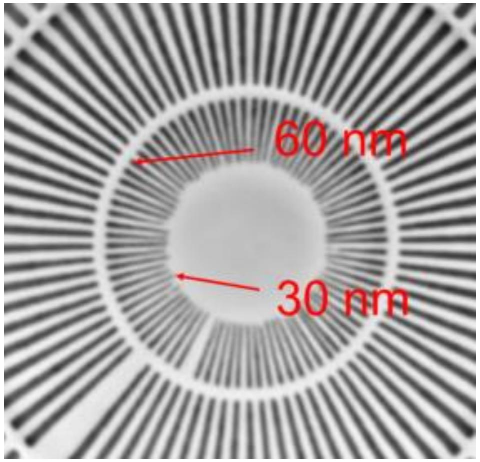


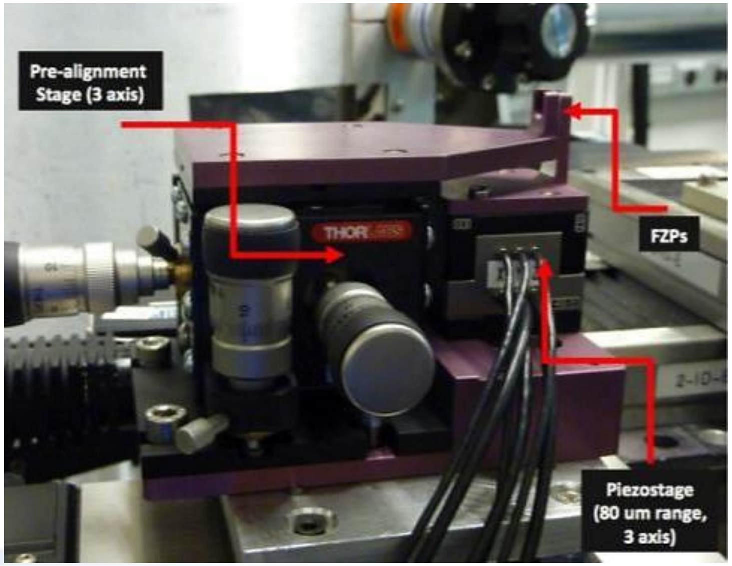
60 nm Δr_n ZP fabricated using a diamond mold & Au zones. Thickness: 1400 nm, ~18% efficiency @ 8 keV.



400 nm thick Au resolution standard, with 40, 50, & 100 nm Siemens stars in the center.

30 nm Siemens star imaged with 20 nm zone plate





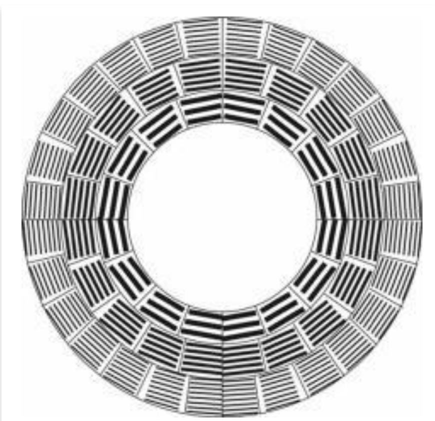
II. Instrument upgrade

4. Increasing efficiency with optics: beam shaping condenser (BSC)

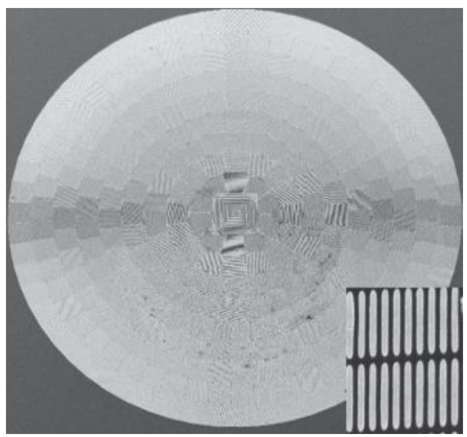
BSC = zone plate like grating

<u>BSC has numerous assets:</u>

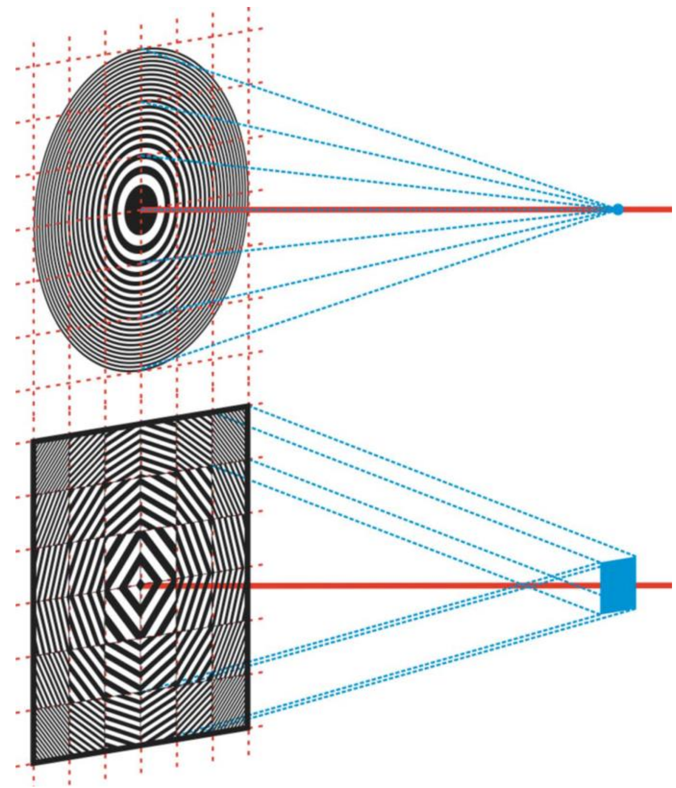
→ allow to control the illumination size
→ provide a flat illumination
→ small and stiff
→ easy to align (pitch and yaw)
→ very large beam acceptance (e.g., 2 x 2 mm^2)
→ "perfect" conic illumination for phase contrast



Beam shaping condenser design (Vogt et al., 2006)



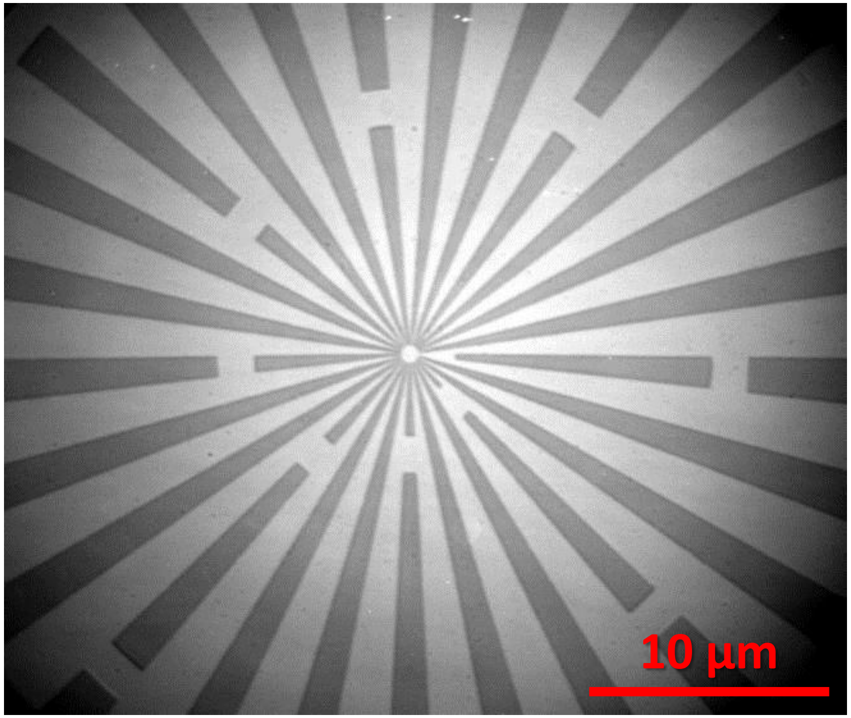
SEM image from PSI



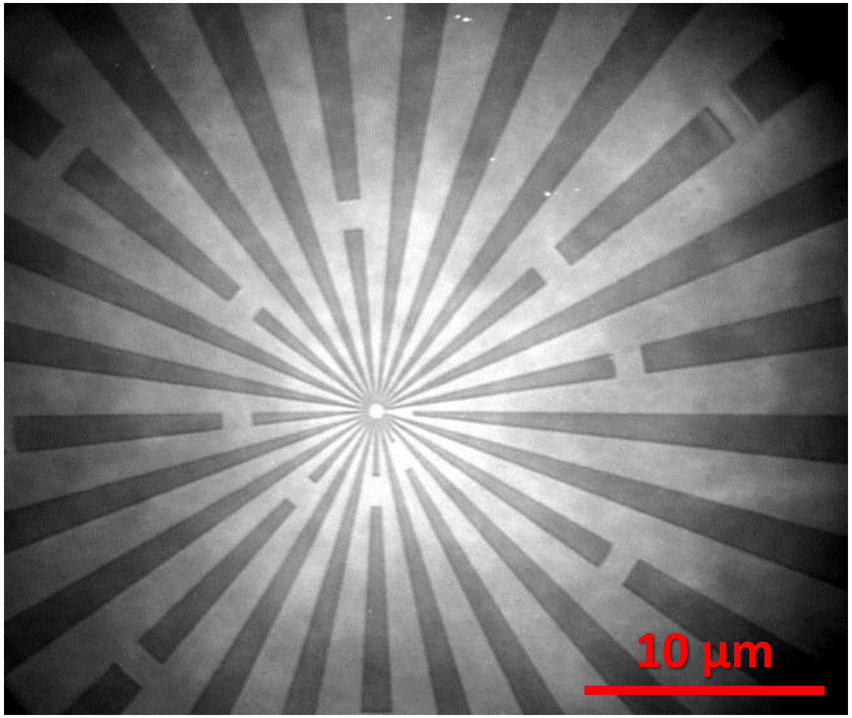
Used at SLS, Petra III and now APS

II. Instrument upgrade

4. Increasing efficiency with optics: beam shaping condenser (BSC)



50 nm Siemens star
60 nm Δr_n ZP, 10X objective lens
5 s exposure time
APS prototype BSC (60 nm Δr_n)



40 nm Siemens star
60 nm Δr_n ZP, 10X objective lens
5 s exposure time
Mono-capillary

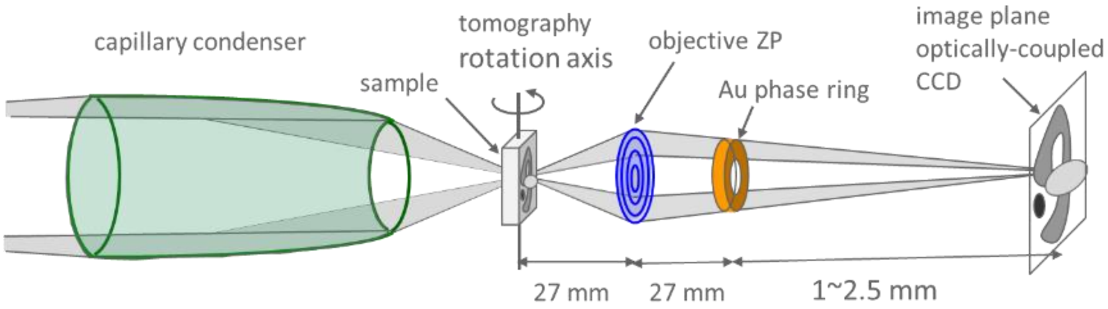
Efficiency gain with the BSC prototype @ 8 keV: 1.57

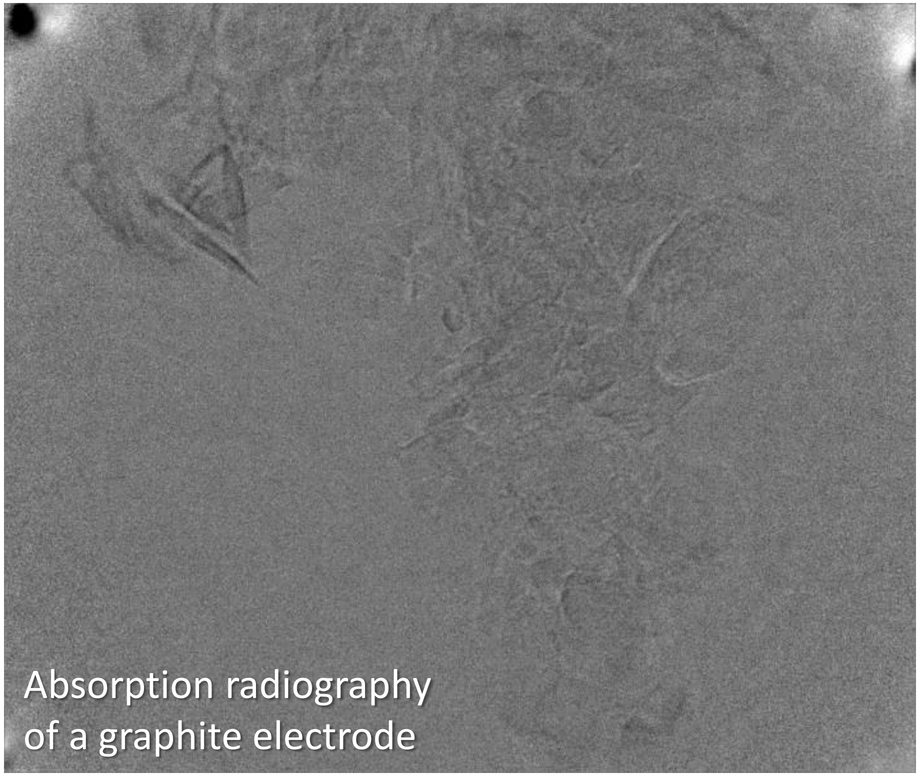
II. Instrument upgrade

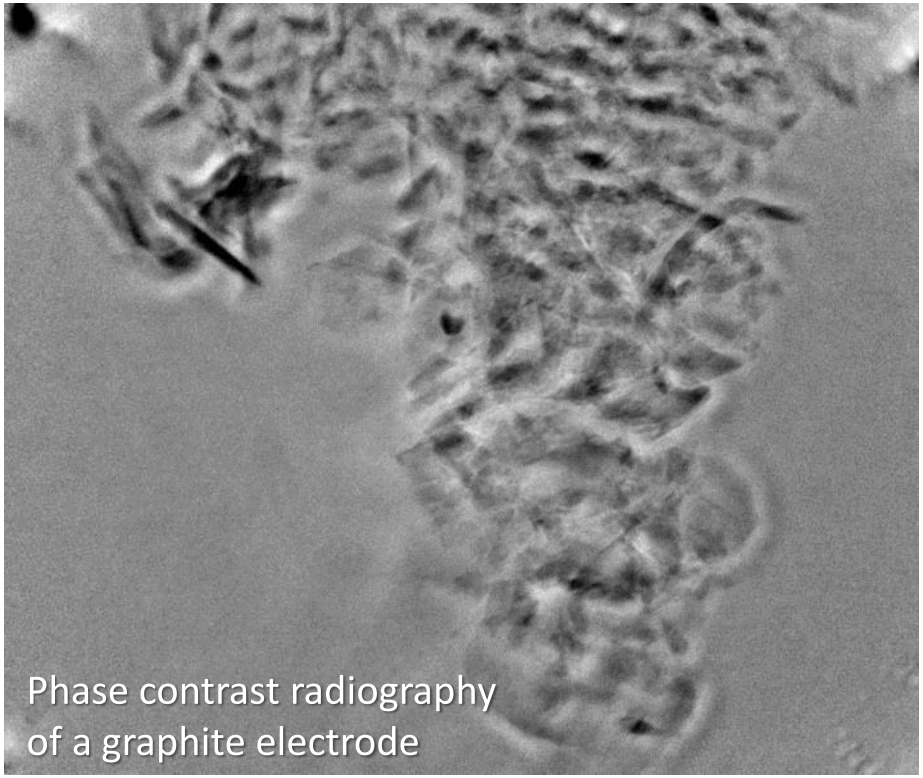
4. Increasing efficiency with optics: beam shaping condenser (BSC)

Improving Zernike phase contrast:

Phase contrast translate light variations in phase into corresponding changes in amplitude, which can be visualized as differences in image contrast

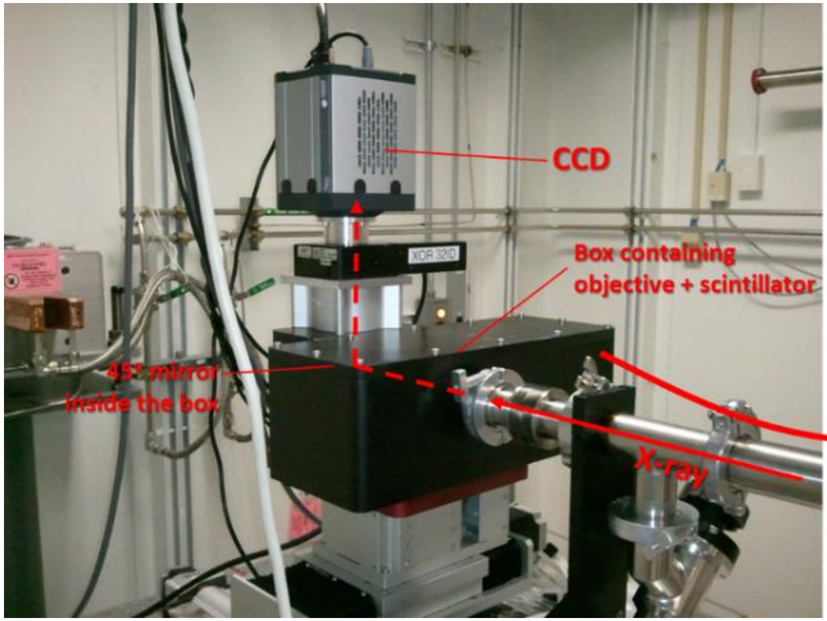


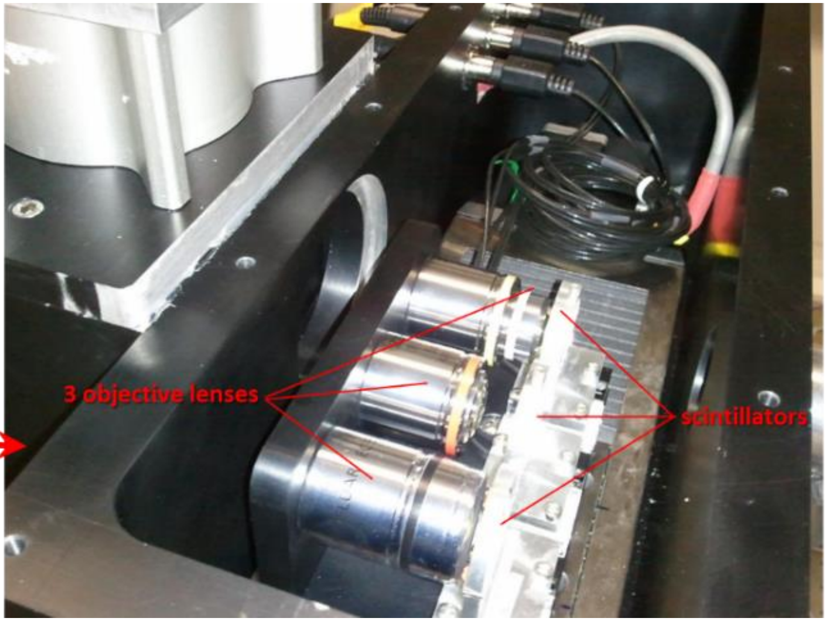

Absorption radiography of a graphite electrode


Phase contrast radiography of a graphite electrode

II. Instrument upgrade

5. Increasing detection efficiency





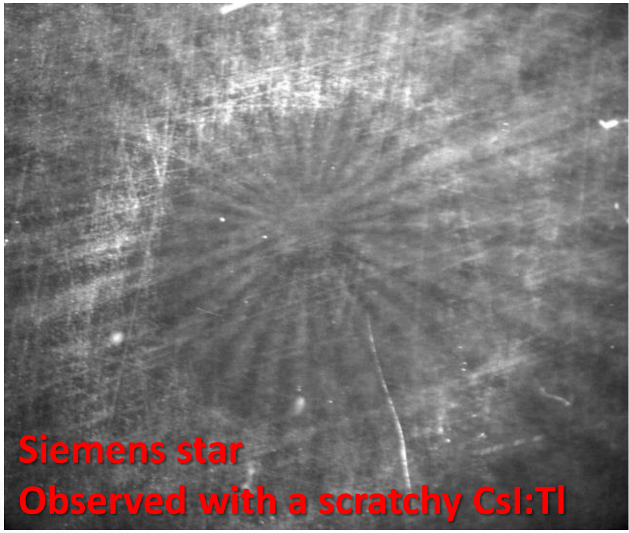
Still using LuAG, 20 years old technology:

- Alternative: CsI:Tl?

→ light yield 4-5 times higher, but very difficult to polish without scratches

→ reliable provider: ZEISS doesn't sell them anymore

Energy (keV)	8	8
Scintillator	LuAG	CsI:Tl
Scint. Thickness (µm)	20	20
Scintillator density	6.7	4.51
Scint. Absorption (%)	75	94
Andor Neo QE (%)	60	60
Objective NA	0.25	0.25
Obj. light collection (%)	0.4	0.4
Obj. light transmittance (%)	90	90
Scintillator photon yield (ph / MeV)	12500	56000
Photon yield @ working En	100	448
photon on CCD / incident X-ray	0.162	0.907



II. Instrument upgrade

6. Increasing efficiency with low-dose reconstruction methods

Traditional approach:

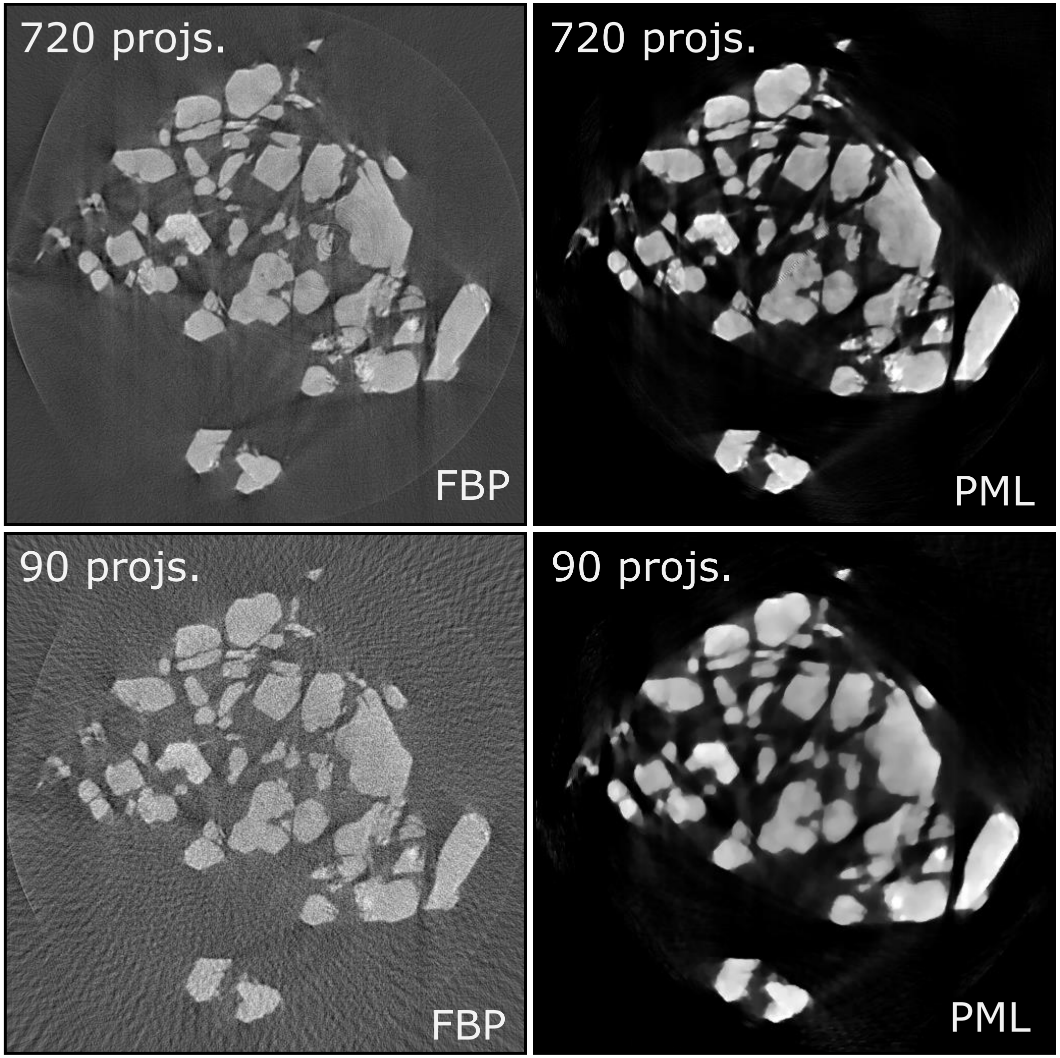
- Analytical, direct (FBP, Gridrec)
- Ideal geometry/sampling
- Simplified physics and noise
- Fast

Modern approach:

- Model-based, statistical, iterative
- Based on reasonable models for physics and statistics
- Usually slower

→ Enable low dose X-ray imaging

- *Both approaches are implemented in Tomopy*
- *Modern approach require supercomputer*
- *Recently, Astra toolbox (for tomographic computations using GPU and CPU) has been integrated to Tomopy*

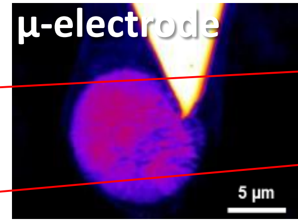


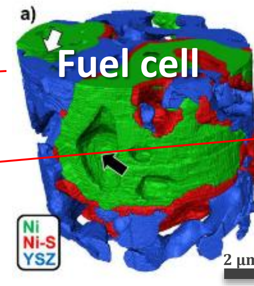
Algorithms implementation & reconstructions by D. Gursoy
Link APS – ANL supercomputer (Tekin Bicer)

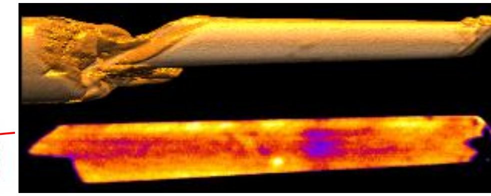
III. Overview of on-going scientific programs

Energy Science

- Fuel cell
- Battery
- UMo nuclear fuel

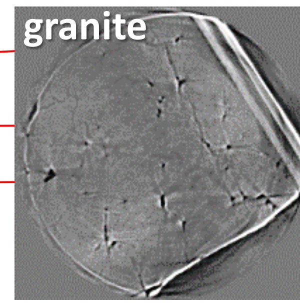


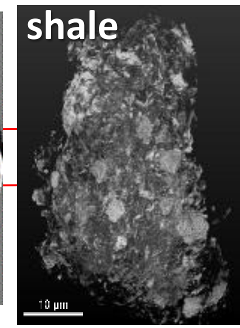


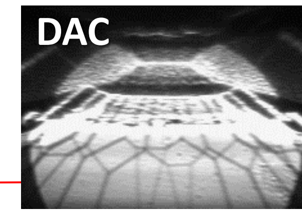


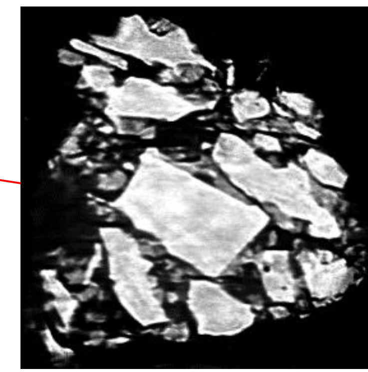
Earth and Environmental Science

- Melt formation
- Rock fracking
- High pressure experiments with DAC
- CO_2 storage
- Pollution / remediation



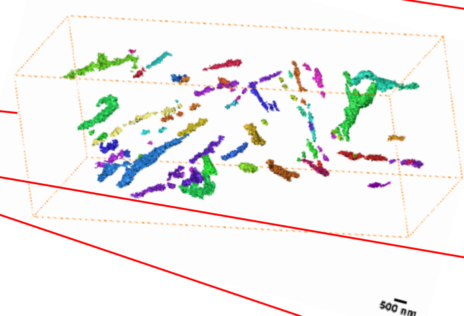


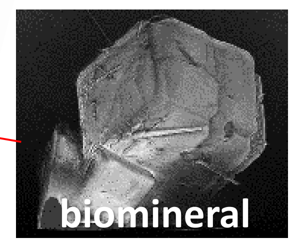




Material Science

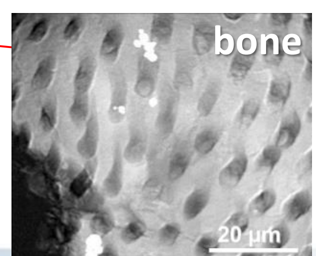
- Metallurgy
- Photonics
- Electronic industry
- Supraconductors

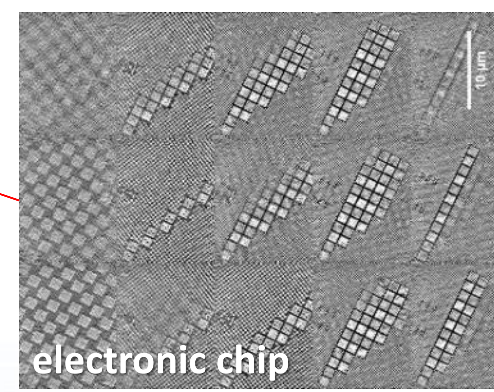




Biology

- Biomaterials
- Wood preservation
- Biology (brain, lungs)





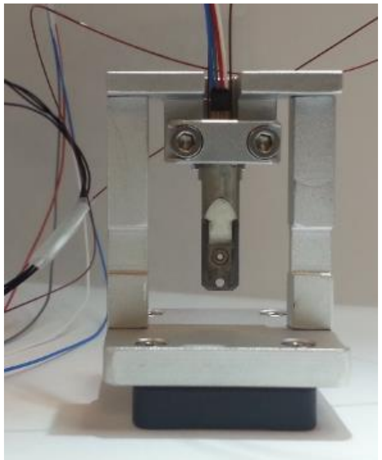
→ **total of 42 groups**

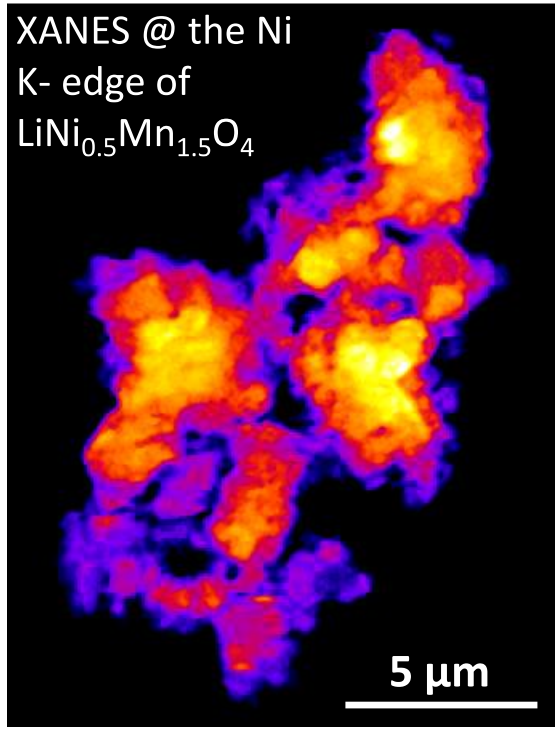
III. Overview of on-going scientific programs

Energy Science

Hummingbird in-situ cell testing

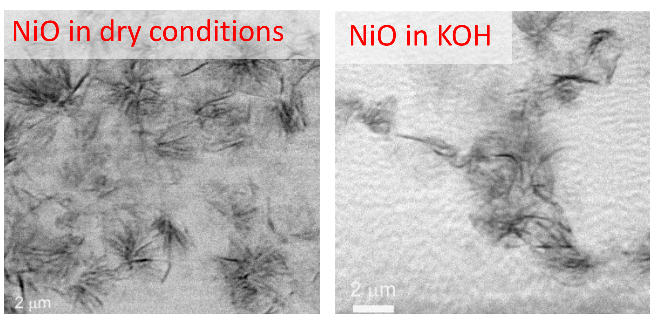
(M. Ge, Y. Chu, HXN, NSLS-II)

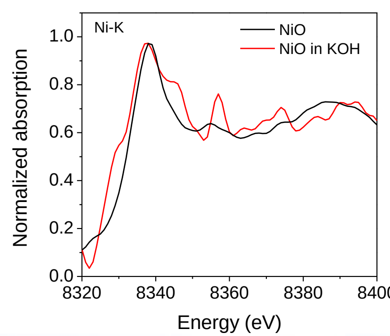




Cross-correlative experiments across X-ray & electron microscope platforms (TEM)

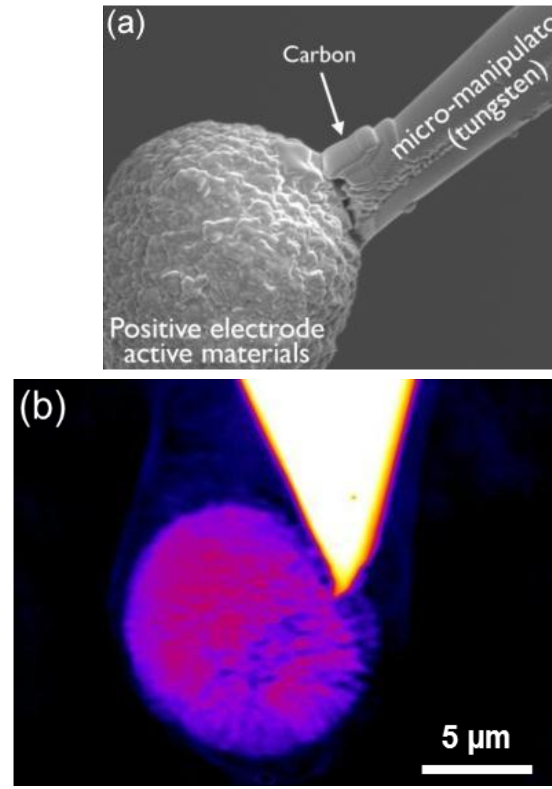
Study of lithiation process of NiO nanosheet





characterization of battery cathode: $Li\text{-}Ni_xCo_yMn_zO_2$ (D. Miller, A. Demortière, MSD)

→ aims to understand microstructure evolution and deformation process during the electrochemical cycling of Li-ion electrode



(a) FIB/SEM micro-scale set-up for in situ electrochemical cycling. (b) TXM radiograph of the µ-electrode

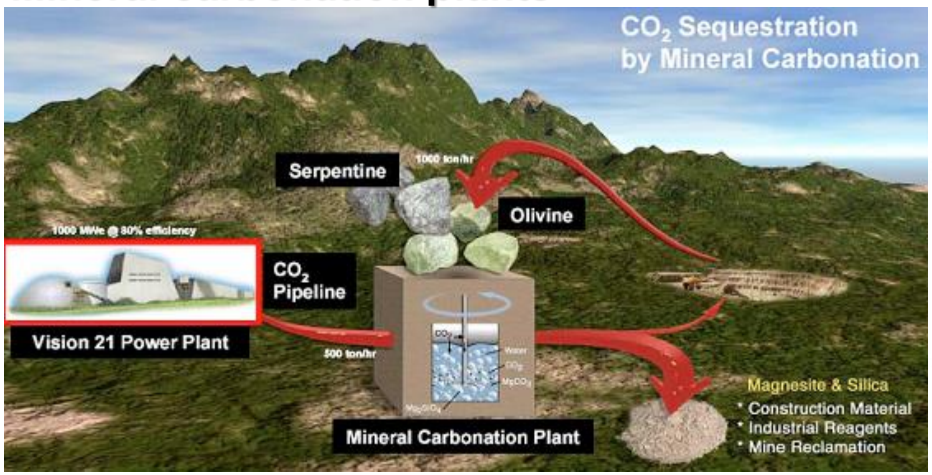
III. Overview of on-going scientific programs

Geoscience: Efficient CO_2 sequestration for halting the global warming

(Wenlu Zhu et al., University of Maryland*)*

- ***Mineral carbonation*** of ultramafic rocks is proposed as a ***safe** and **irreversible** CO_2 **sequestration*** method.

Mineral carbonation plants



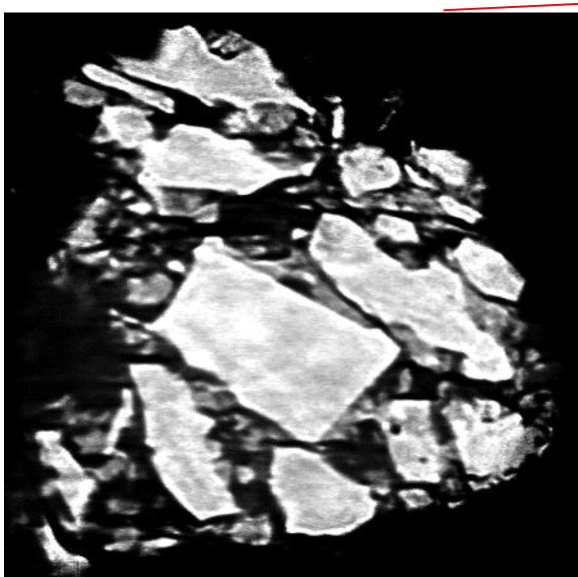
$$\underset{\text{(olivine)}}{Mg_2SiO_4} + \underset{\text{(gas or fluid)}}{2\,CO_2} = \underset{\text{(magnesite)}}{2\,MgCO_3} + \underset{\text{(quartz)}}{SiO_2}$$

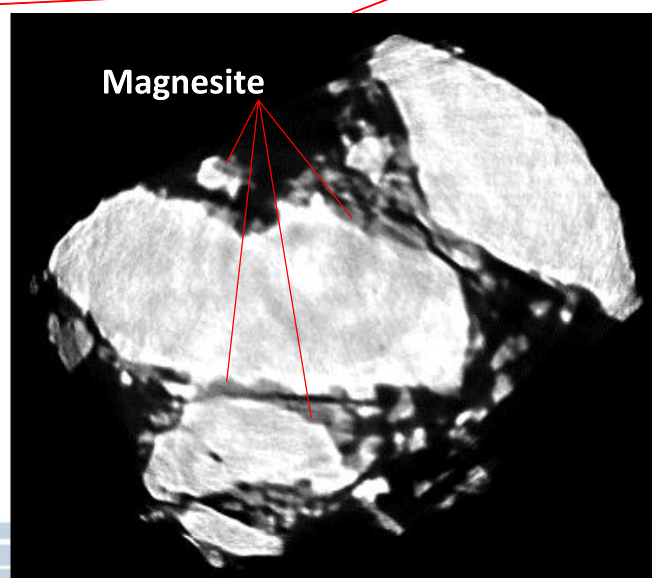
A viable process for CO_2 mineralization must achieve sufficient carbonation volumes

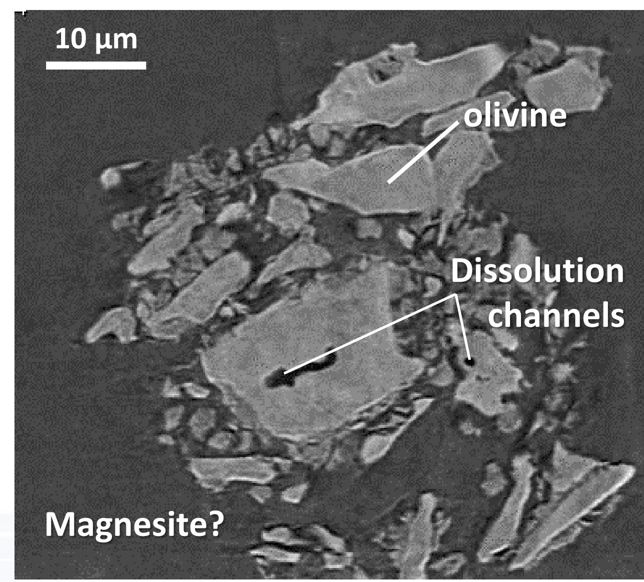
→ high rates, low energy penalties, minimum consumables & *self-sustaining process*

*Reconstruction with **PML***

*Access to MIRA **supercomputer** (T. Bicer)*





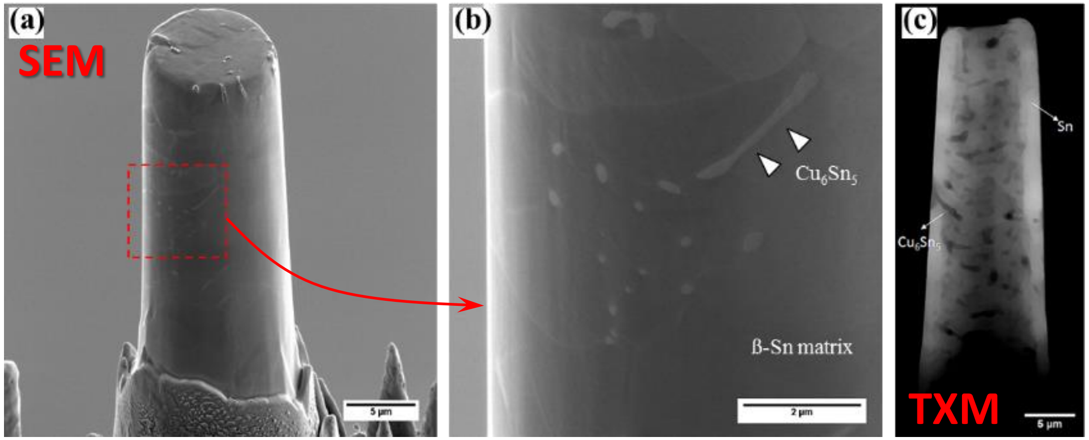


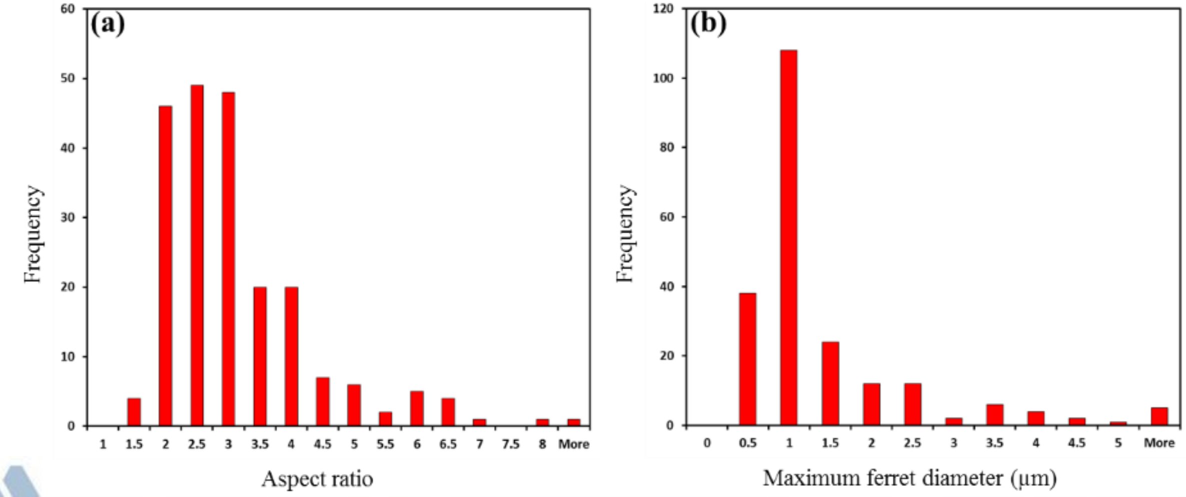
III. Overview of on-going scientific programs

Material Science: metallurgy

Nanoscale Three-Dimensional Non-Destructive Characterization of microstructures in Sn-rich Solder alloy using TXM *(S. Kaira, N. Chawla et al., submitted)*

- Study of Cu-Sn solder microstructures for microelectronic applications



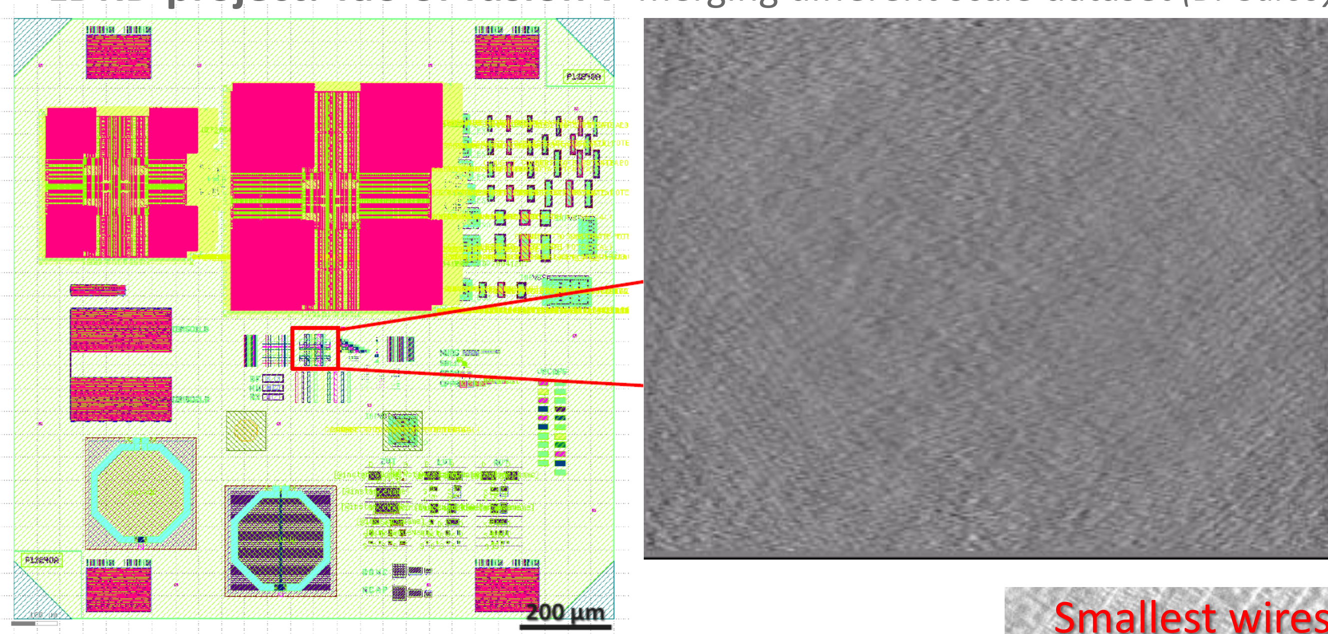


complex morphology of Cu_6Sn_5 phase IMC in the ß-Sn matrix

III. Overview of on-going scientific programs

Microelectrionic industry

- **LDRD project: Tao of fusion**→ merging different scale dataset *(D. Gursöy & E. Lavely, Y. San from BAE)*

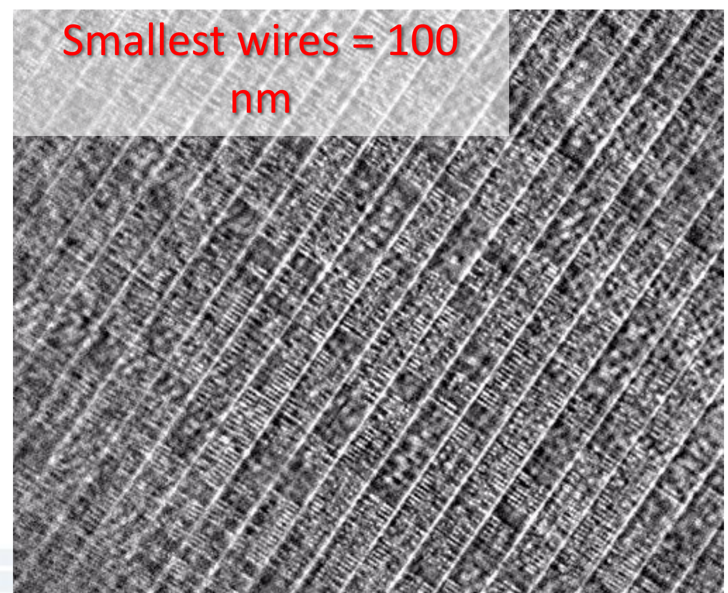


- **1 year PuP**

→ nano-tomography of full microchips

(*J. Rudati, M. Sutherland from*

D*efense* ***M****icro****E****lectronics* ***A****ctivity*)



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