# Hard X-ray Imaging Capabilities at NSLS-II

*MIT-NSLS-II Workshop*

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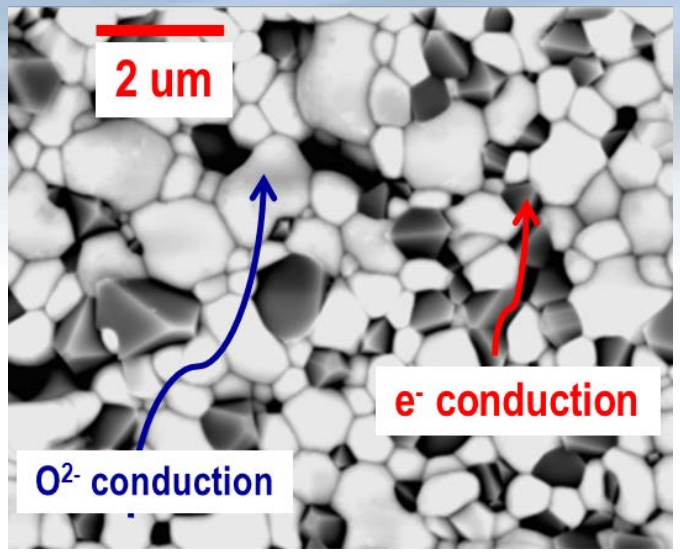
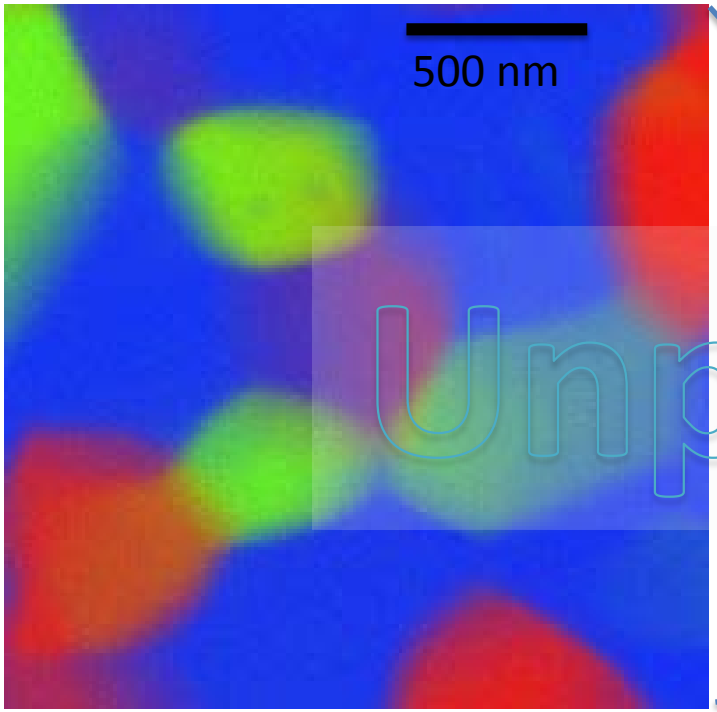
# Hard X-ray Imaging Beamlines at NSLS-II

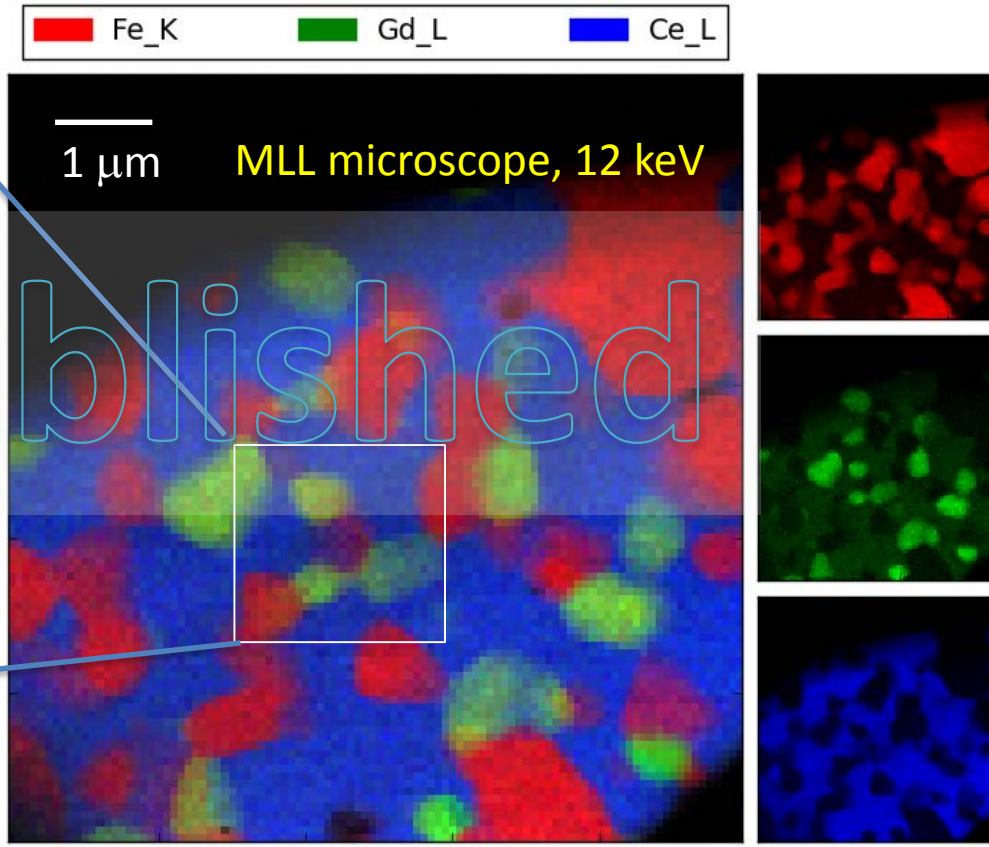
- Hard X-ray Nanoprobe (HXN) Beamline (in operation)
  - Nanoscale imaging at 10 nm.
  - Composition, morphology, crystalline domains/strain
  - Considerable sample preparation required.
- Submicron Resolution X-ray Spectroscopy (SRX) Beamline (in operation)
  - ~0.5 um resolution.
  - Composition, oxidation states
  - Little sample preparation required.
- Full-field X-ray Imaging (FXI) Beamline (first beam in Fall 2017)
  - ~30 nm resolution.
  - Fast 3D imaging, well-suited for in-situ
  - Moderate sample preparation required.
- X-ray Fluorescence Microprobe (XFM) Beamline (first beam in Fall 2017)
  - ~5 um resolution. Composition.
  - Little sample preparation required.
- Bragg CDI (BCDI) Beamline (under conceptual design)

# Phase boundary imaging of Ceramic membrane ($Ce_{0.8}Gd_{0.2}O_2$ + $CoFe_2O_4$)

- Solid oxide fuel cell (SOFC) & Li-air batteries
- Production of $O_2$
- Reduction of $CO_2$ emissions from coal
- Potential application for reprocessing spent nuclear fuel

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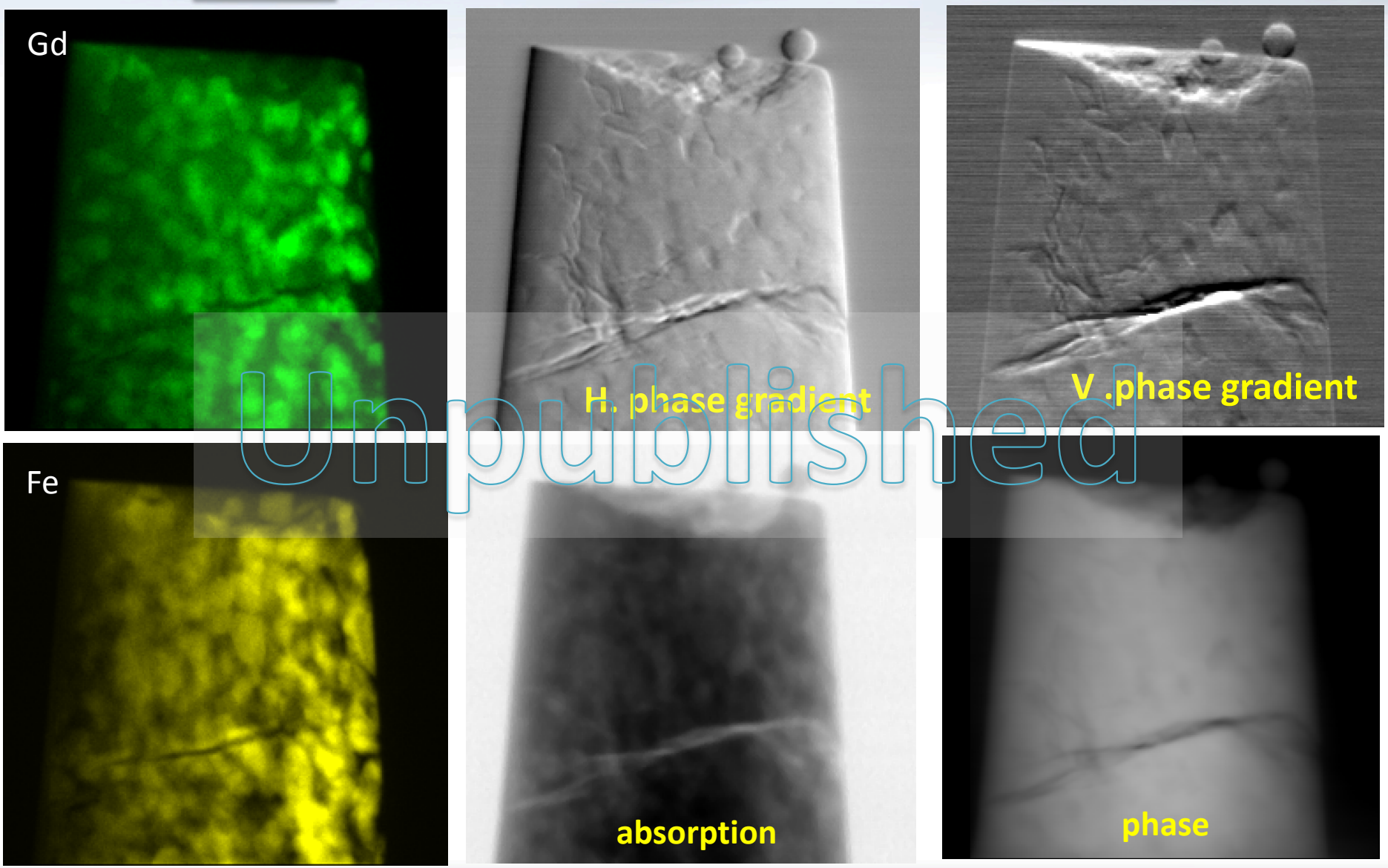
*Wilson Chiu, U. of Conn.*

# Multi-modal Imaging

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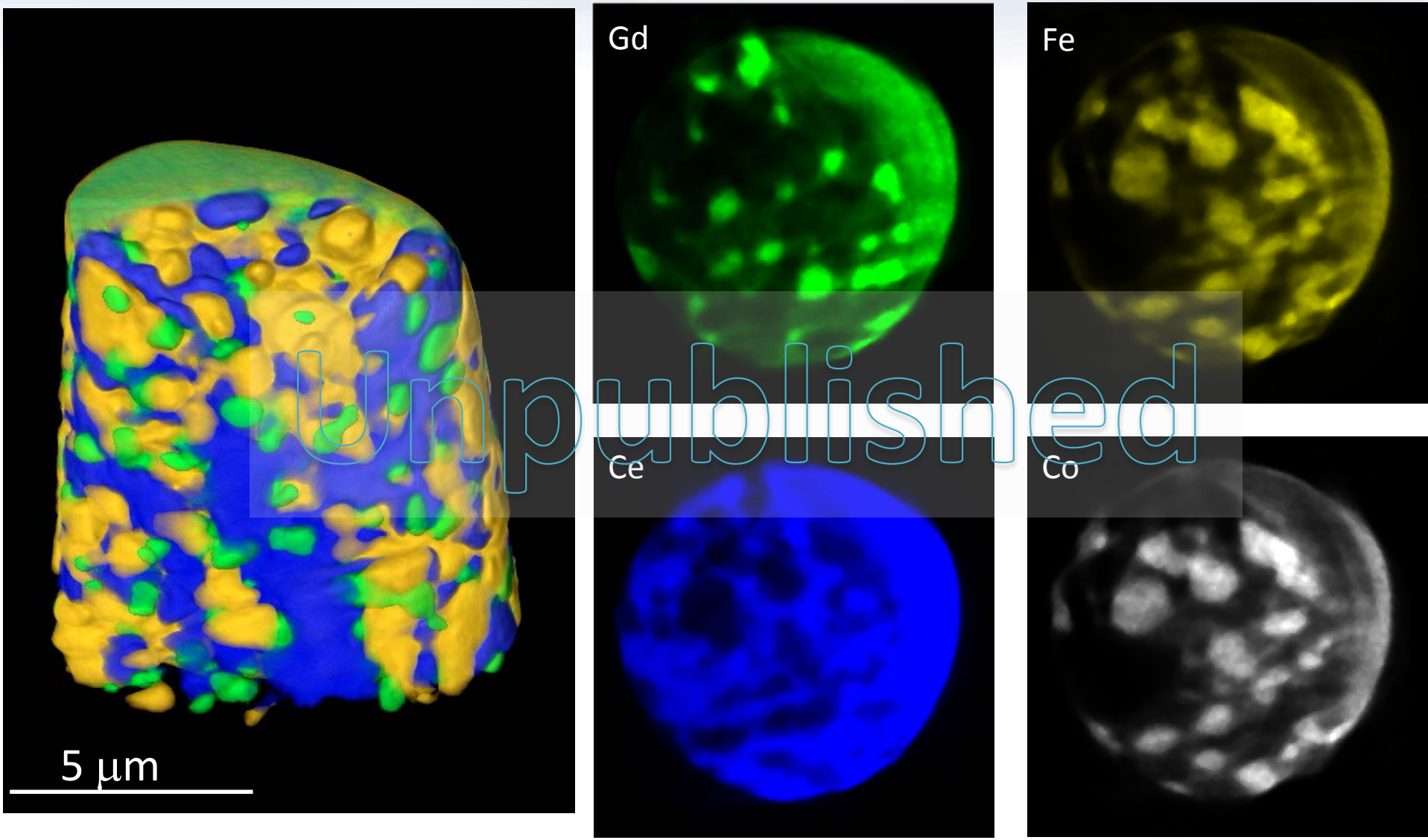
# 3D Grain Boundary Imaging of Ceramic membrane ($Ce_{0.8}Gd_{0.2}O_2$ + $CoFe_2O_4$)

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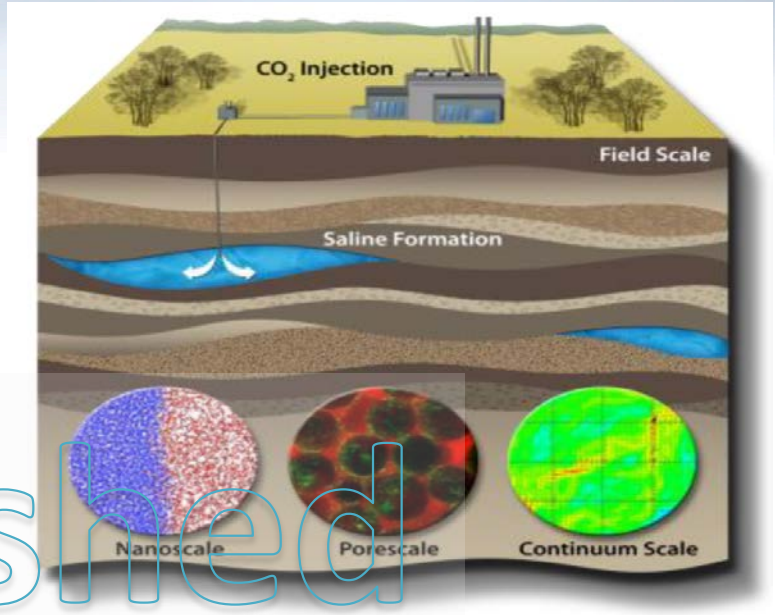
# Spectroscopy Imaging of Barite Nanoplates

Understand arsenate ($As^{[V]}O_4$) and arsenite ($As^{[III]}O_3$) uptake kinetics during barium sulfate precipitation in idealized high salinity wastewaters.




*From Wheeler, UT Austin*

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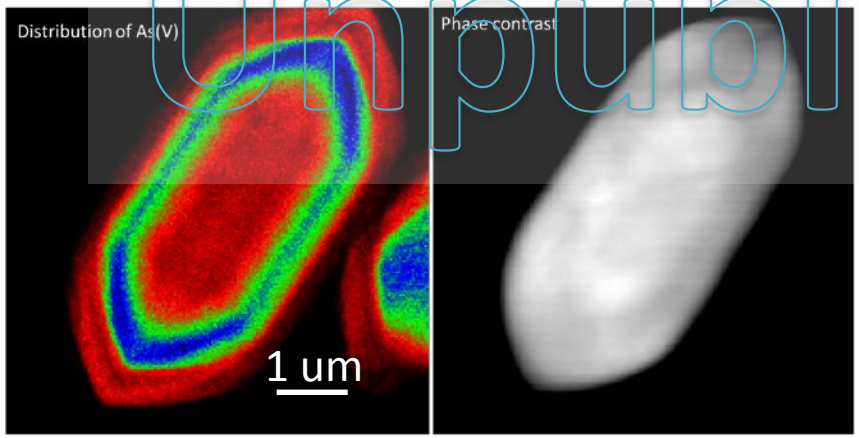
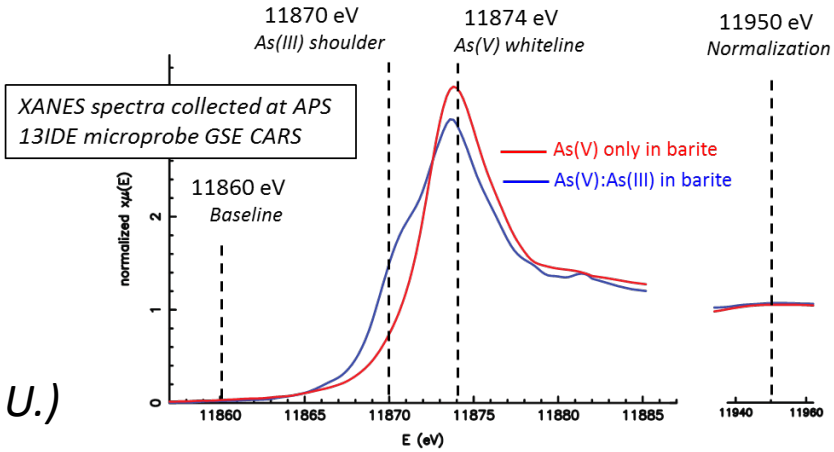
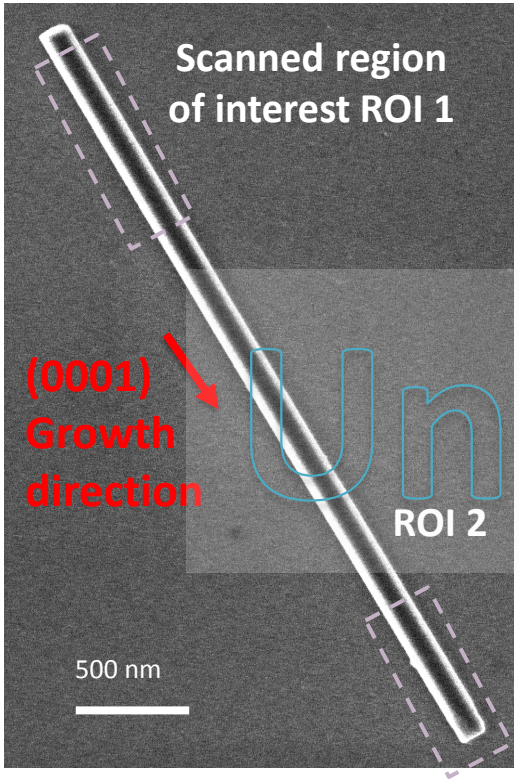



*Antonio Lanzirotti (U. Chicago), Jeffrey Fitts (Princeton U.) and Catherine Peters (Princeton U.)*

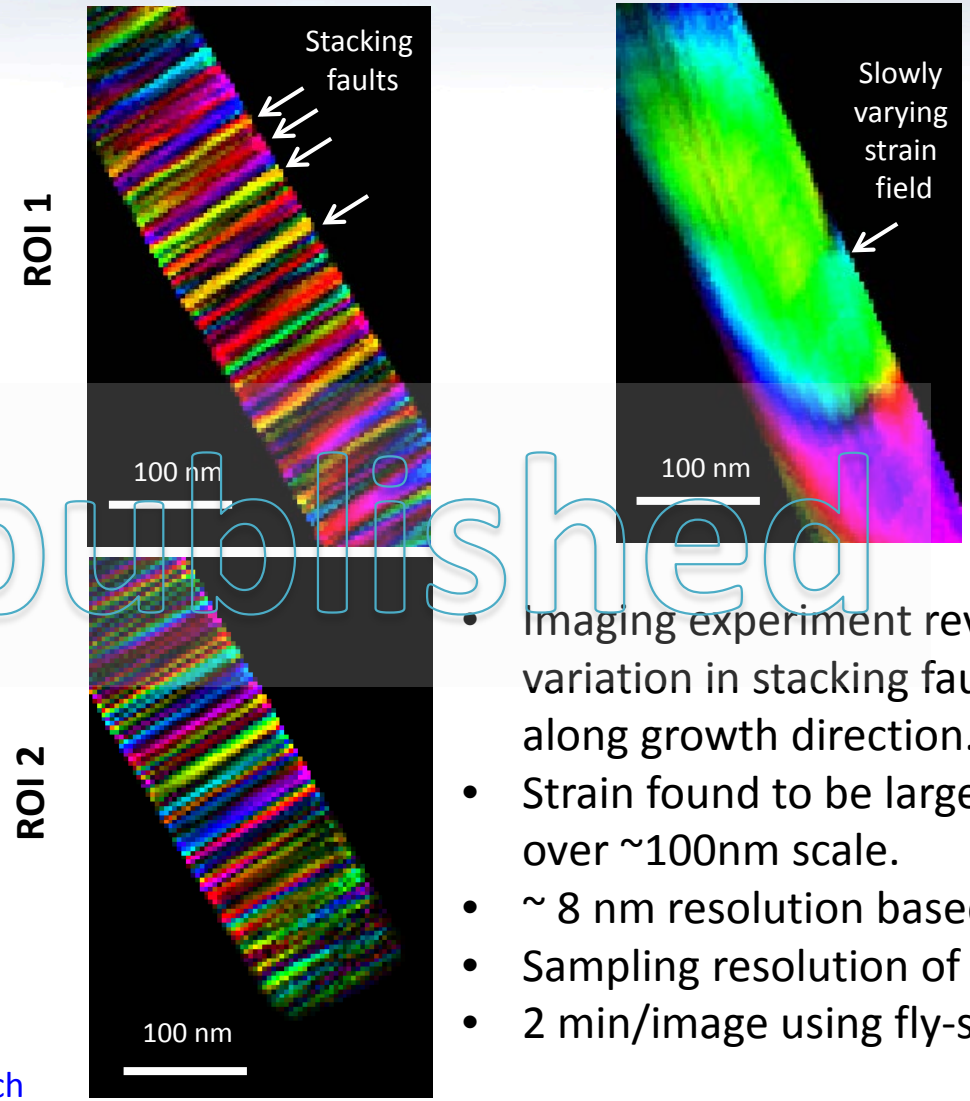
# Imaging Strain and Stacking Faults (SF)

$In_{1-x}Ga_xAs$ nanowires (x=0.14)
Catalyst-free, site-selective growth by MBE on Si(111)




**(20-20): sensitive to SF**

**(2-1-12) insensitive to SF**

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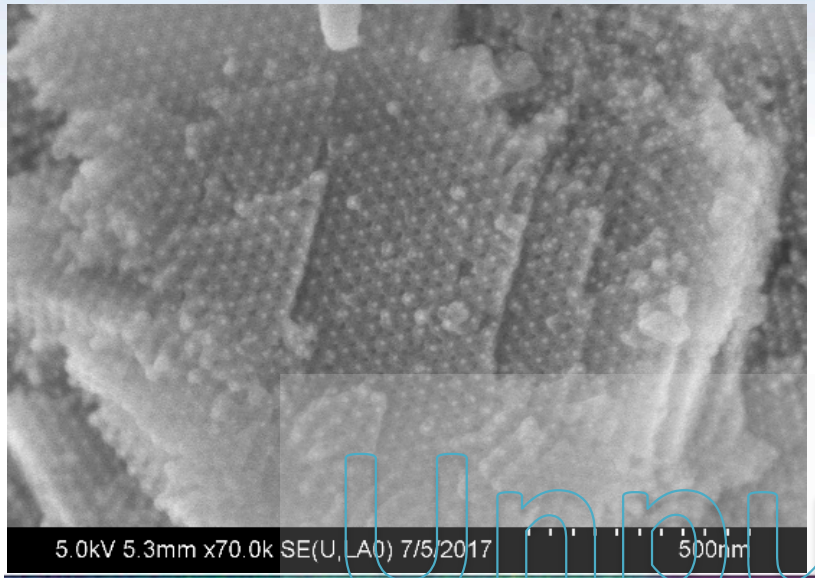
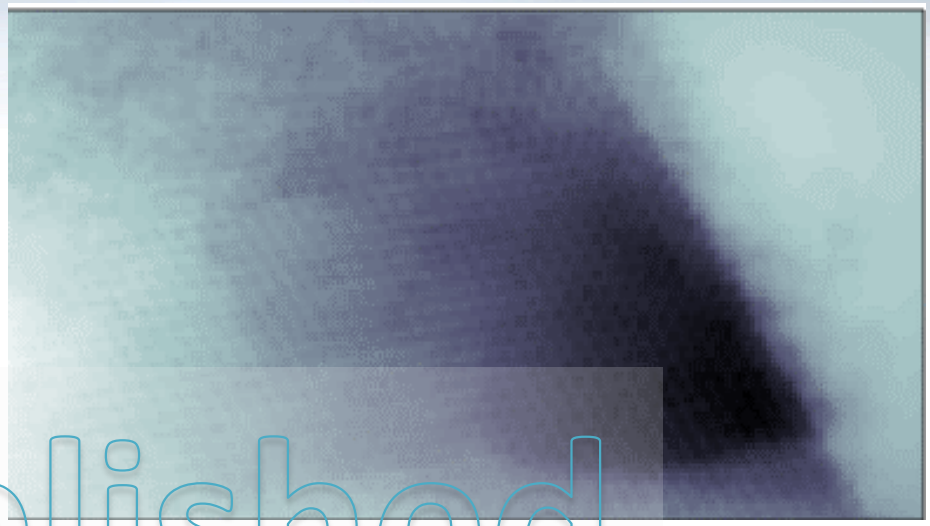
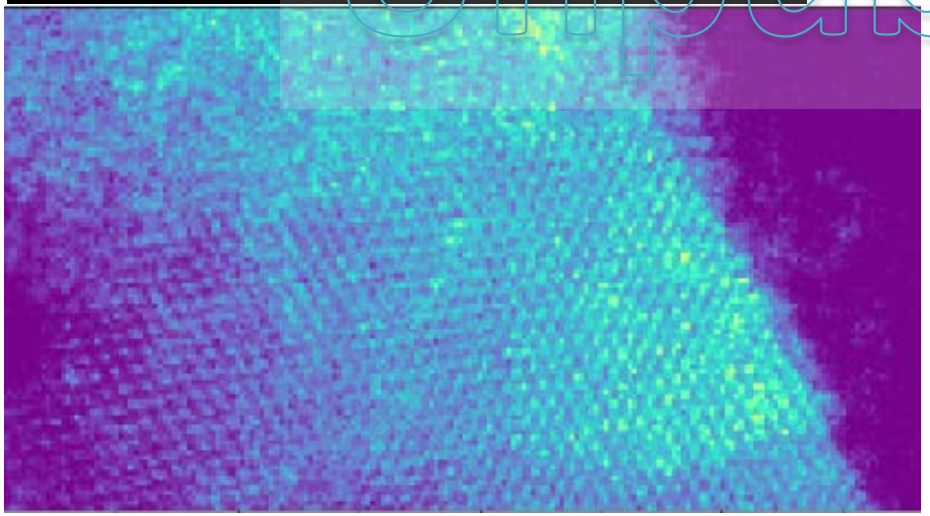
- Imaging experiment reveals variation in stacking fault density along growth direction.
- Strain found to be largely uniform over ~100nm scale.
- ~ 8 nm resolution based on signal.
- Sampling resolution of 3.6nm/pixel.
- 2 min/image using fly-scan.

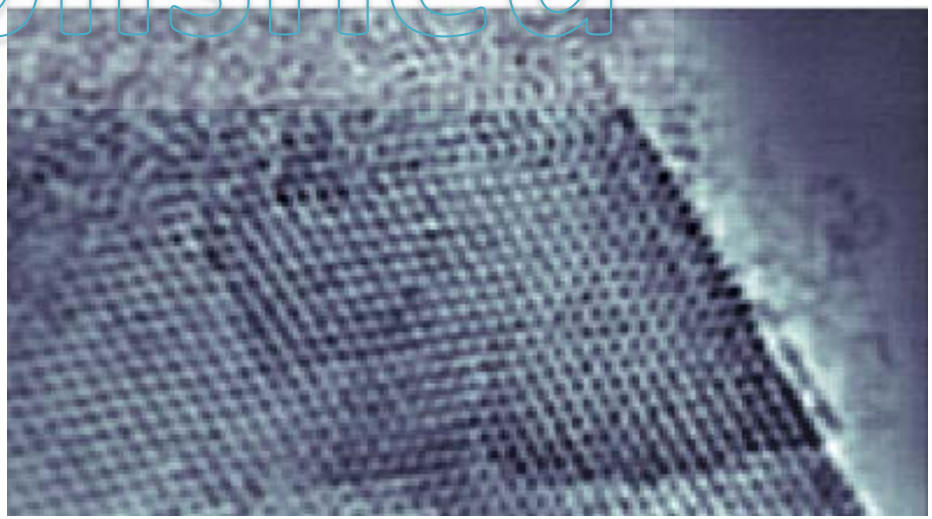
Hruszkewycz, Holt, Stephenson – ANL
Hill, Lauhon – Northwestern U.
Huang, Yan, Chu, Nazaretski – NSLS-II
Treu, Morkötter, Koblmueller – TU Munich

# Sub-10 nm Imaging







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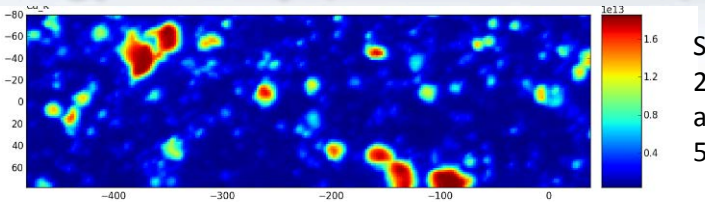
# *SRX: In Operando* Studies on Energy Storage Materials

## Li-S battery: Delivers significantly higher energy density (Gravimetric 6x)

**Goal:** Addressing the critical challenge in Li-S: **low conductivity of S cathode**

→ improved by using **hybrid cathode:** CuS-S

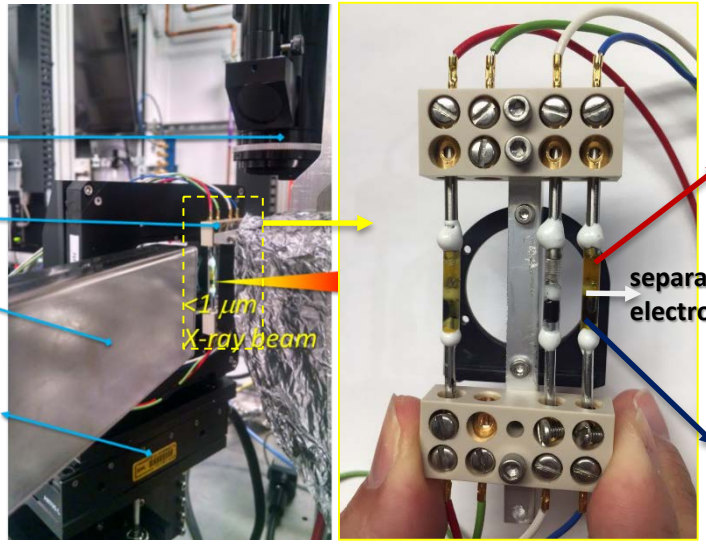
- Issues: dissolution of Cu & re-deposition



Step size: 2 μm
area: 520 x 80 μm²

***Cu mapping in Cu-S hybrid cathode***

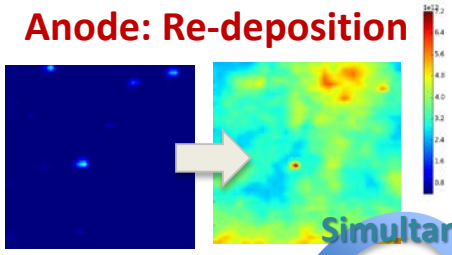
First time **direct visualization** *in operando*



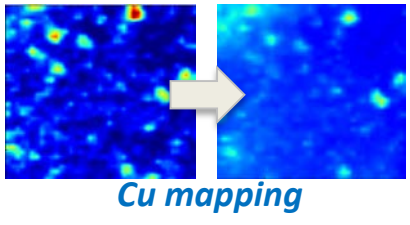

@Sub-micron Resolution X-ray Spectroscopy (SRX) Beamline

**A. Elemental Distribution Evolution** (XRF)

**Anode: Re-deposition**



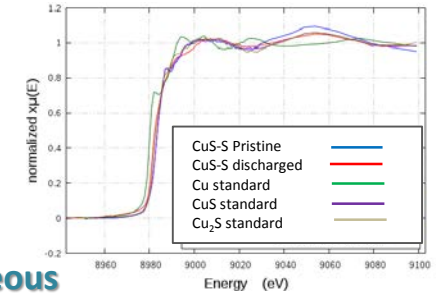
**Cathode: Dissolution**



***Cu mapping***

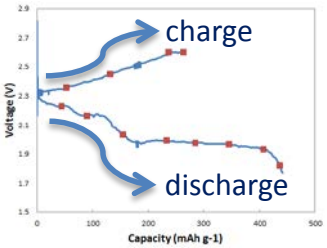
*Pristine vs Discharged*

**B. Chemical Evolution** (XANES)




Simultaneous Correlation

**C. Electrochemical Evolution**

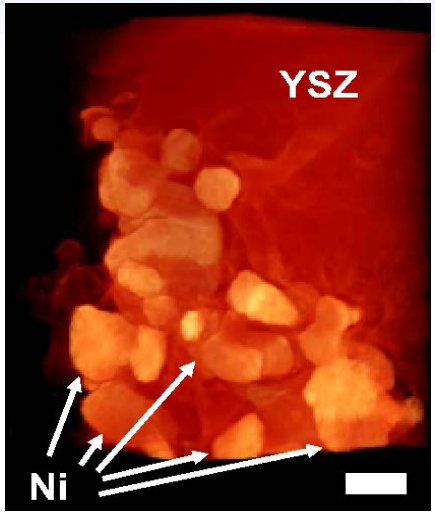



**Key Future Development**

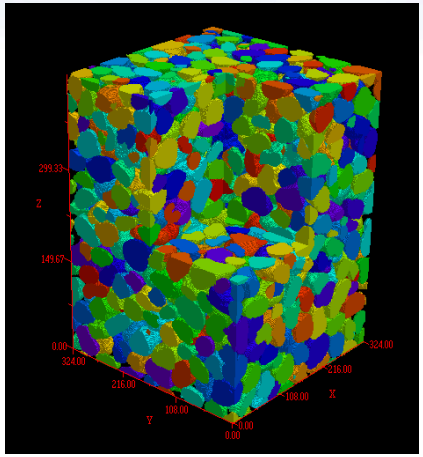
1) Investigating **alternative additives** at SRX *in operando* to resolve dissolution issue: $FeS_2$, $TiS_2$
2) Studying the key role of S by utilizing **future beamline capabilities with tender x-ray** spectroscopy
3) Applying **diffraction techniques at NSLS-II** to study structure and crystal-amorphous transition

*In collaboration with Hong Gan and Ke Sun (Sustainable Energy Department, BNL), Chonghang Zhao (Stony Brook U.)*

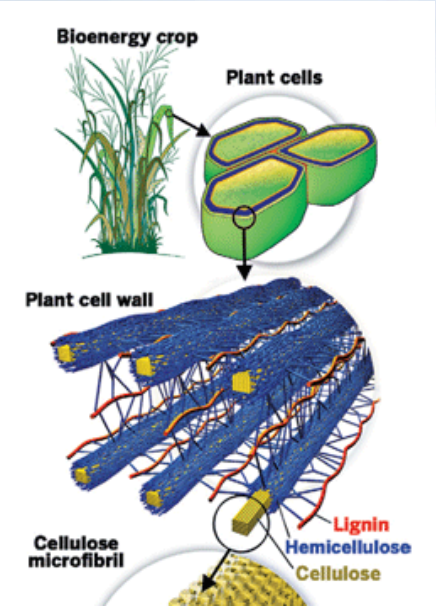
# FXI (full-field imaging) Science applications



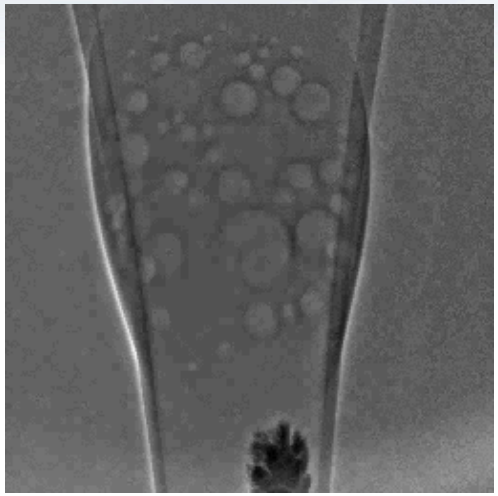

SOFC electrodes



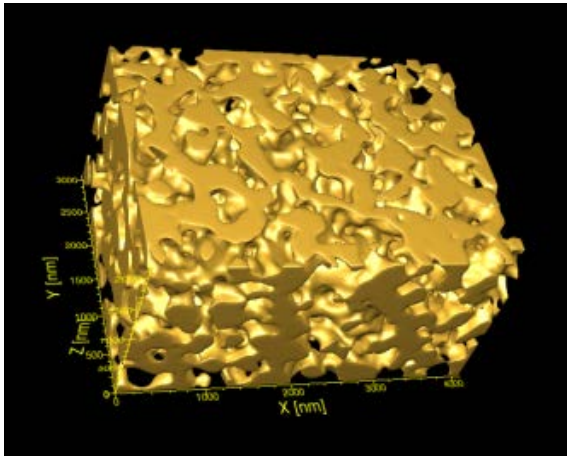
Geological porous media



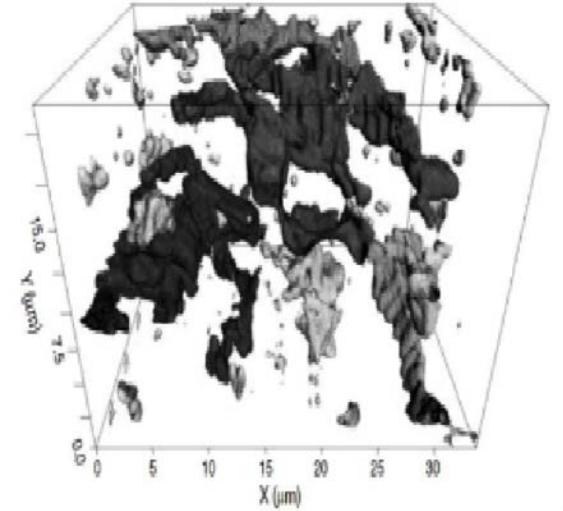

Structure of biomass



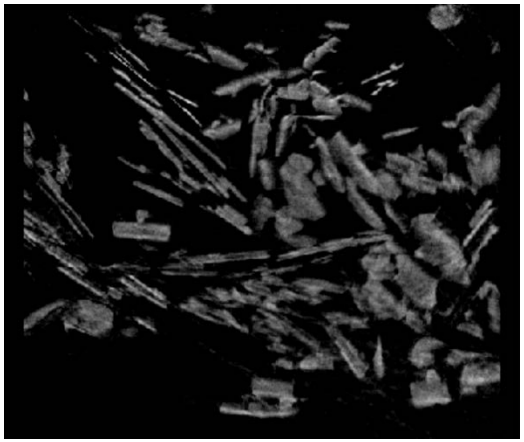
In-situ electrochemistry




Nanoporous alloys

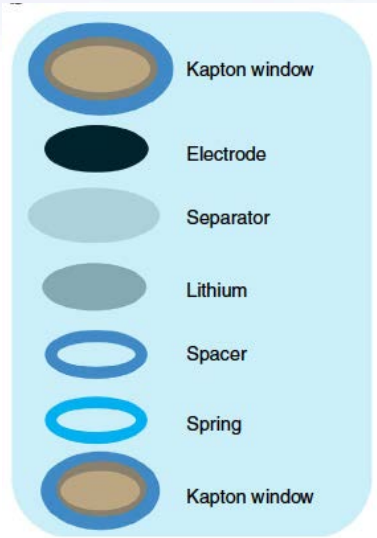


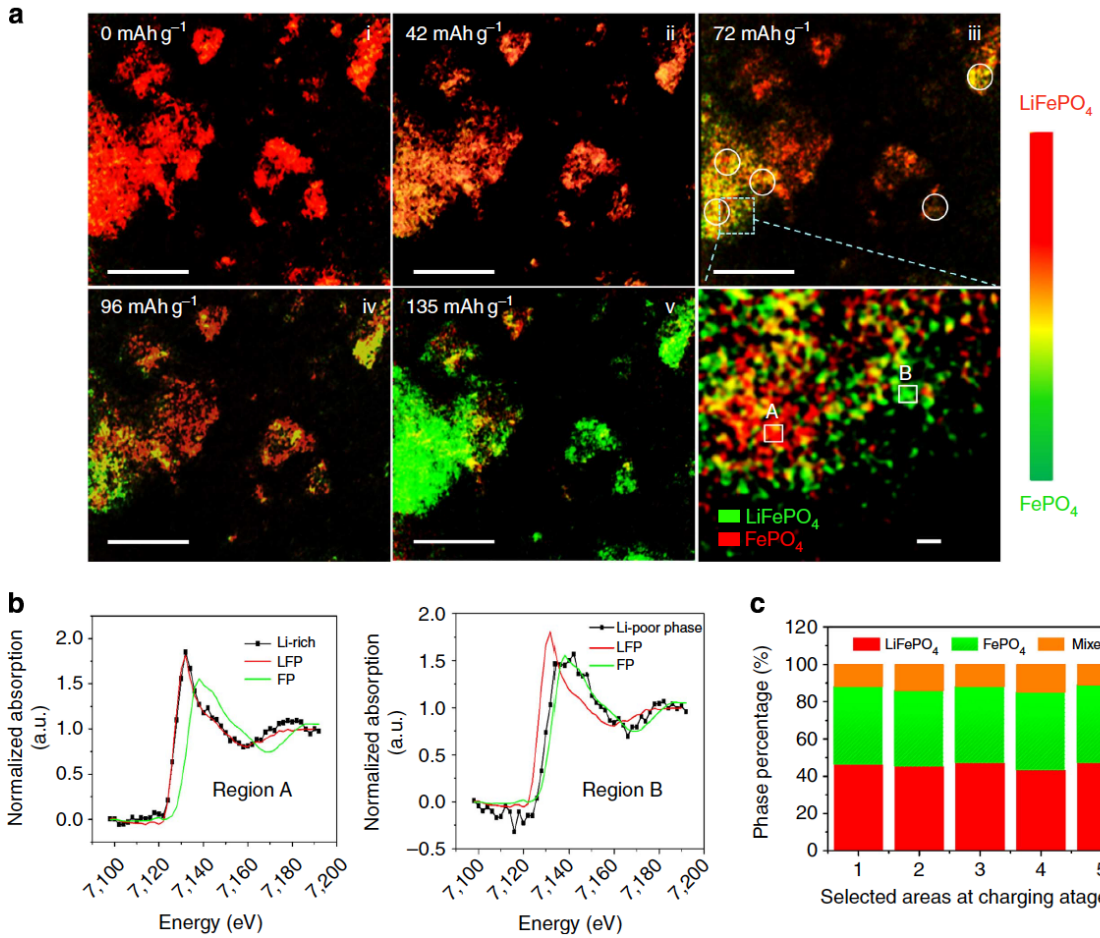

FeNiS under high T and P



Biomaterials for bone scaffold

# In operando 2D tracking of phase transformation in a battery



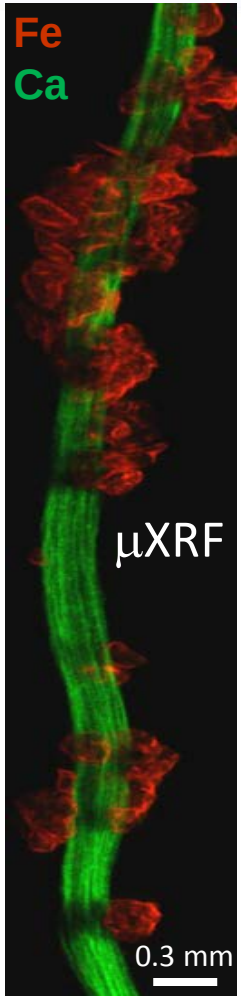

- Coin cell
- 2D chemical states
- 10 s per image

X8C

**Figure 2 | *In operando* 2D chemical mapping of multi-particle LFP system at a fast charging rate of 5C.** (**a**) Two-dimensional chemical mapping; (**b**) XANES at selected regions (square A and B) at charging stage iii; (**c**) histograms of statistical analysis of phase compositions at five selected regions (round zones) at charging stage iii. Scale bar: 10 μm for the five 2D maps, 200 nm for the enlarged map.

J. Wang et al, Nature Communications, Feb 2014

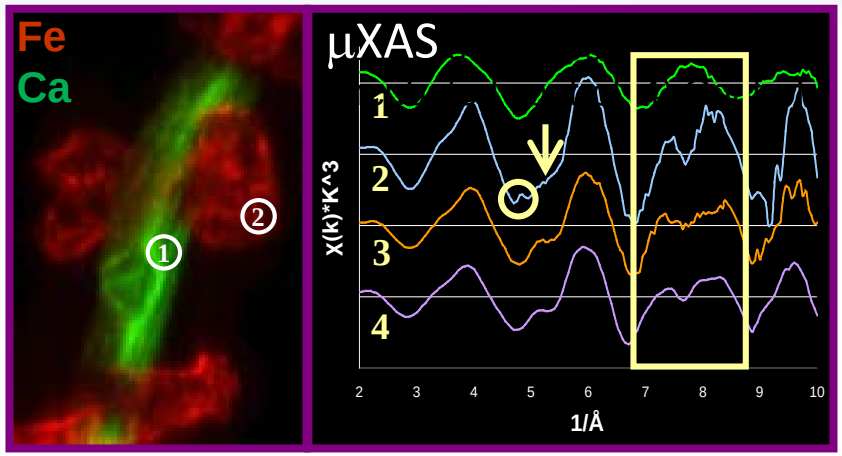
# XFM: Optimizing Yield of Biofuels Crops on Marginal Lands

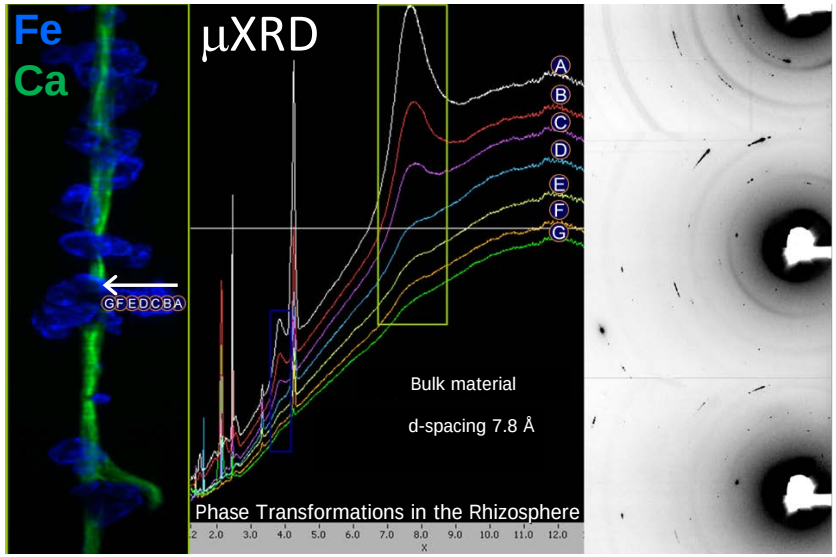



- Iron (Fe) is a trace element required for photosynthesis in all plants, but is often a limiting micronutrient due to the low solubility of Fe(III) in soils. Plant roots and associated micro-organisms have developed various strategies to acquire Fe from sparingly-soluble pools.

- **XFM beamline** is optimized for μXRF imaging coupled with μXAS and μXRD, which can be used to evaluate how plants (+/- microorganisms) modify the chemical form of Fe in their rhizosphere (soil-root environment) and absorb nutrients from soil.

Figures. **Left:** X-ray fluorescence image of Fe (red) and Ca (green) in rhizosphere showing Fe-coated soil particles attached to the Ca-rich root; **Upper right:** XRF image of root-soil interface showing the locations for Fe K-edge μXAS spectra 1&2 in the adjacent plot which altogether reveal root-induced transformation of solid Fe phase in model soil; **Lower right:** transect of μXRD patterns (a-g) from the root-soil interface indicating the phase transformation of the solid, crystalline form to a non-crystalline form occurs nearest the root surface.



# Thanks You For Your Attention