Advancing Research with Combined Synchrotron Techniques

Lu Ma
QAS Lead Scientist
NSLS II, Brookhaven National Laboratory

03/12/2025

Outline

Introduction

- Overview of synchrotron X-ray characterization techniques
- Importance of combined techniques

Combining Techniques

- XAS + XRD: Comprehensive structural analysis
- XAS + DRIFTS: Catalyst insights

Pseudo grazing incidence measurement

- Total electron yield for surface analysis

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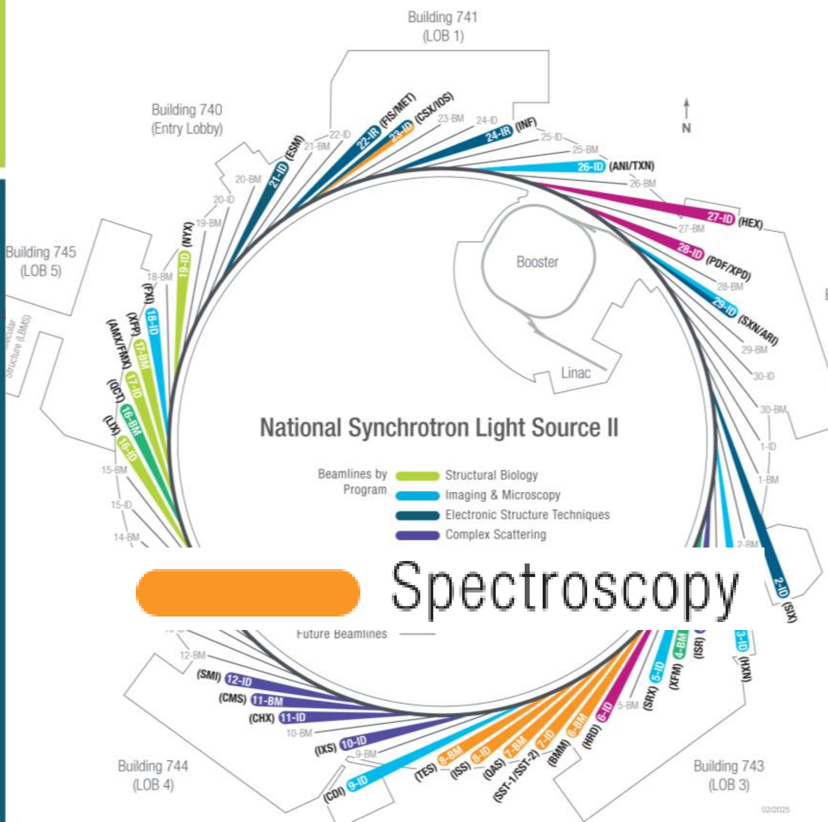
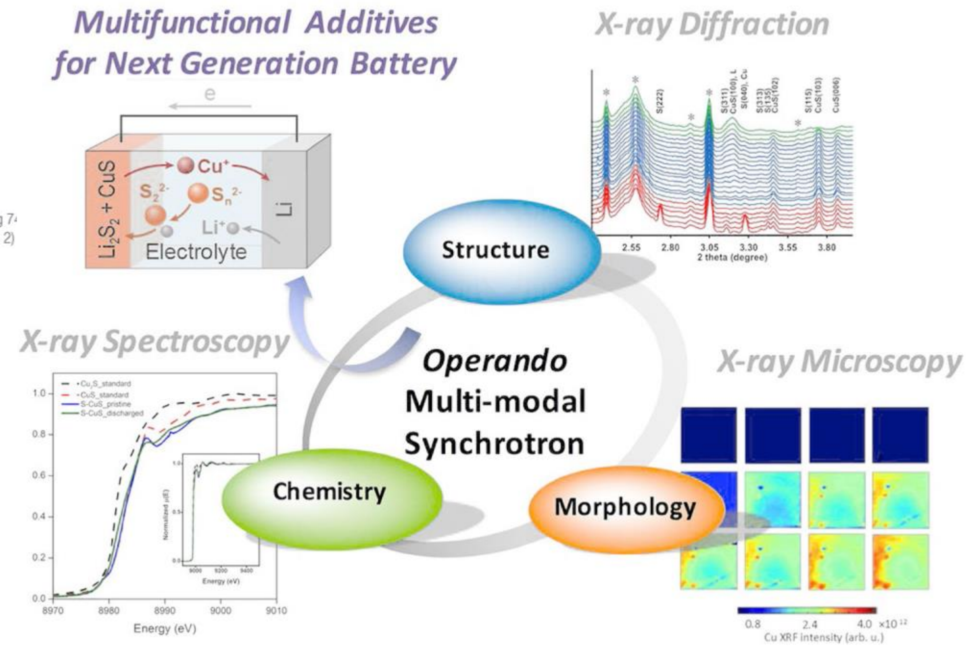
Introduction





- Comprehensive Analysis
- Real-Time Monitoring
- Maximize data yield from valuable beamtime
- Reveal unexpected properties or phenomena
- Bridge gaps between research fields.

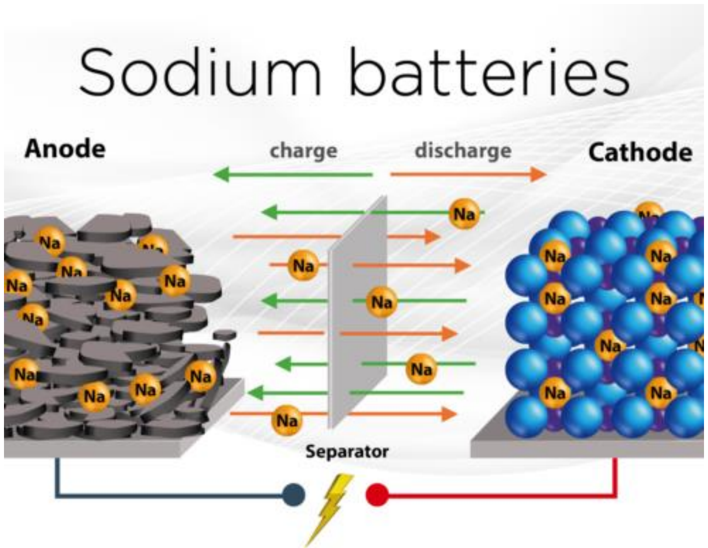
Brookhaven National Laboratory

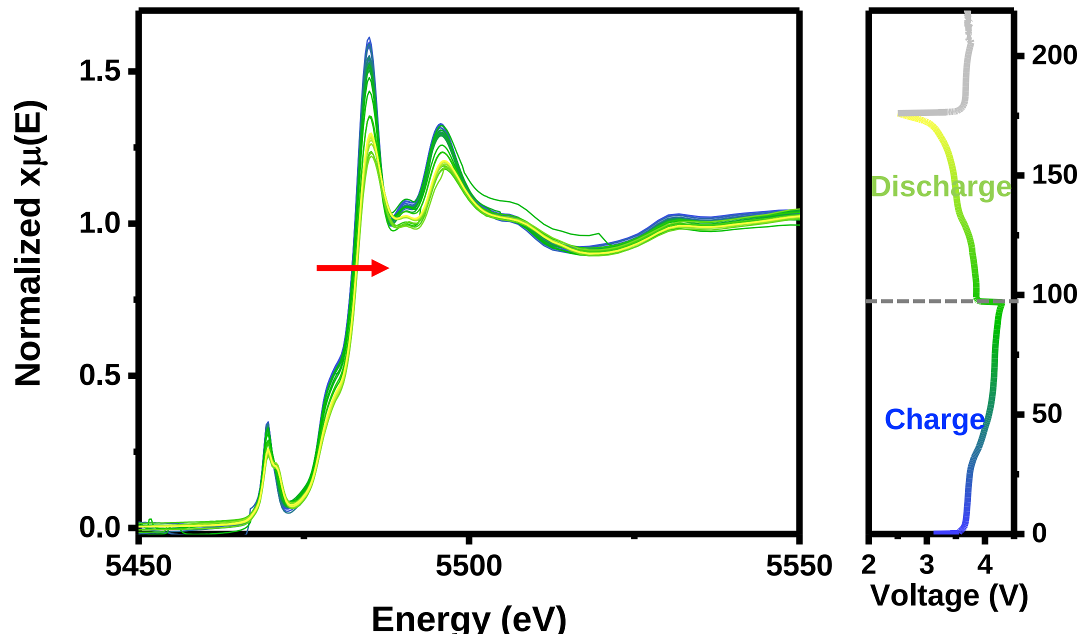
Sci. Rep. 7, 12976 (2017)

Introduction

Na-ion battery

Was that a slow reaction? Why did that happened?





During Charge:
Na^+ extraction, oxidation at cathode

During Discharge:
Na^+ insertion, reduction at cathode

Lack of Cross-Validation when using a single characterization technique!

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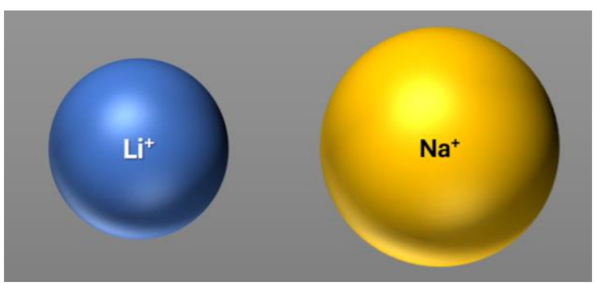
Introduction

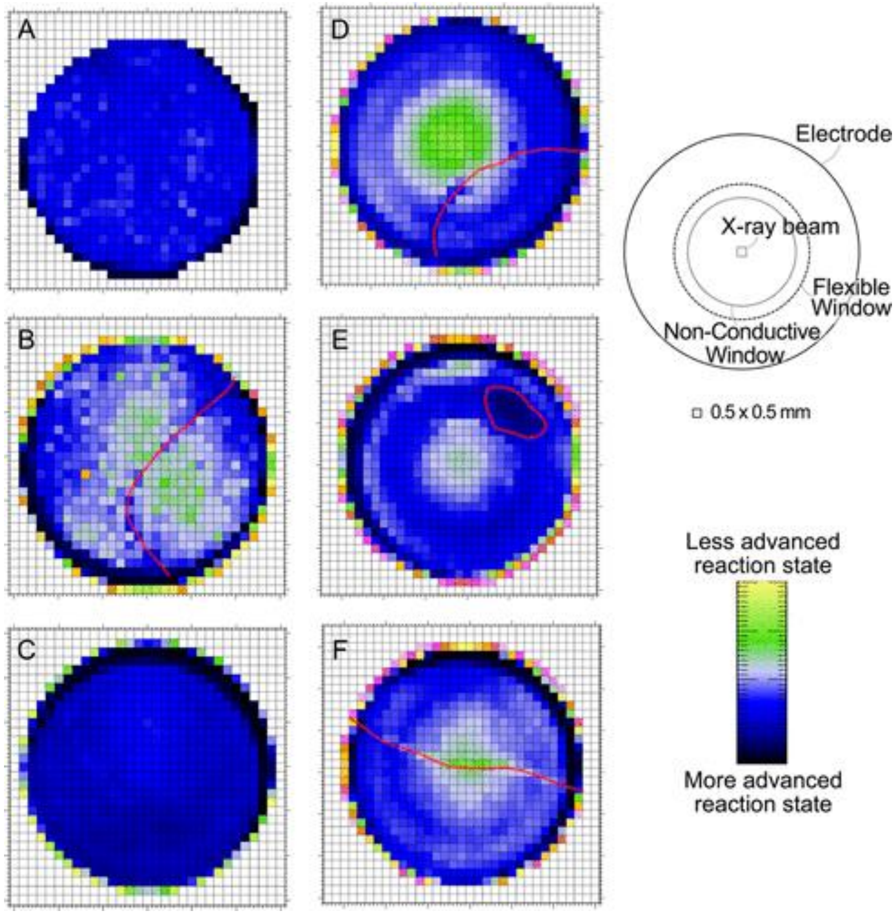
Coin cell in real life

Coin cell at the beamline

Modified with a hole in the middle to allow the X-ray penetration



76 pm 102 pm

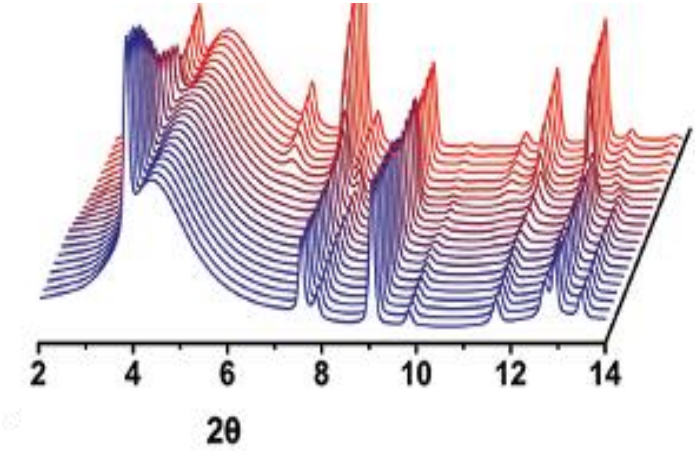


J. Phys. Chem. Lett. 2015, 6, 11, 2081–2085

Combined XAS & XRD

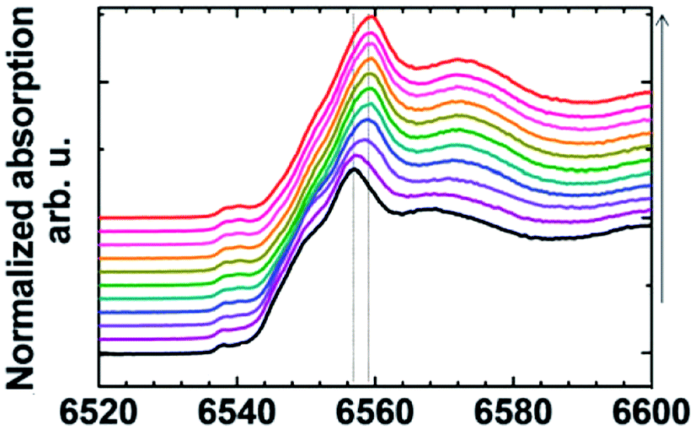
X-ray diffraction (long-range order):

- Phase identification of crystalline materials
- Crystal structure
- Particle size, strain, and other microstructural properties



X-ray absorption spectroscopy (short-range order):

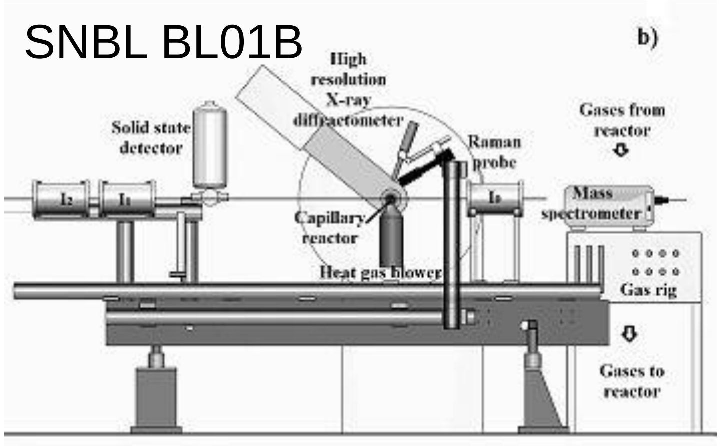
- Electronic structure and oxidation state of specific elements
- Local coordination geometry and bond lengths



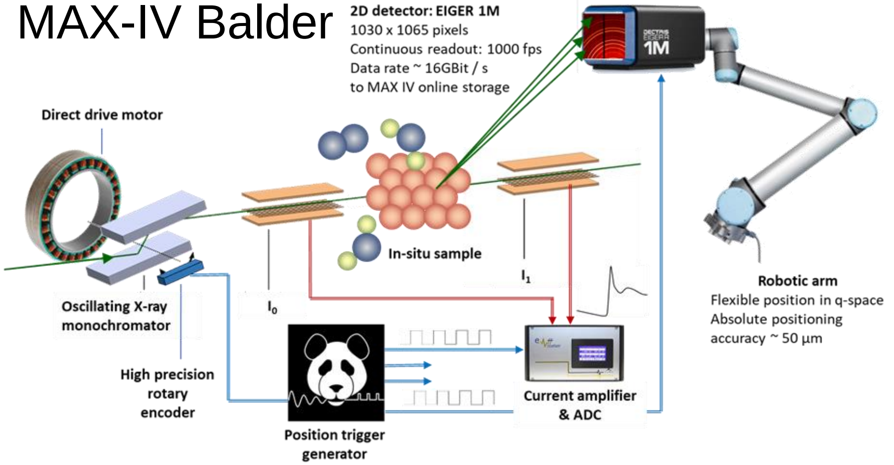
Combined XAS & XRD

NSLS-II
7-BM

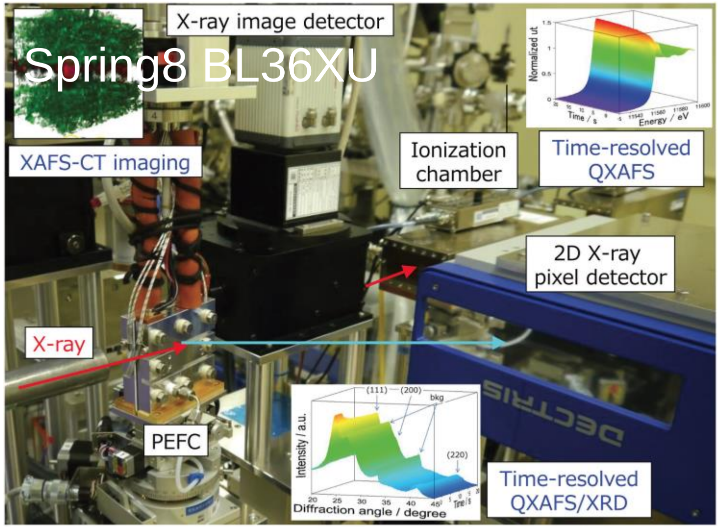
SNBL BL01B



MAX-IV Balder



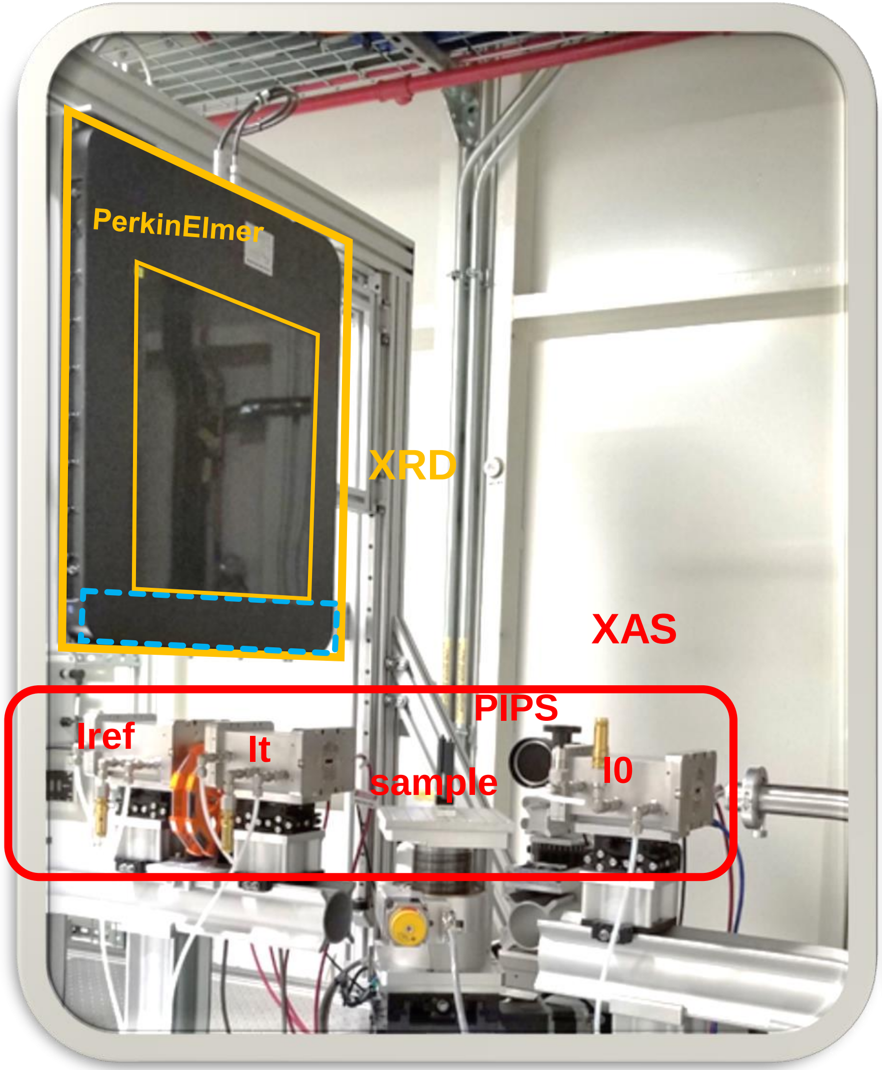
Spring8 BL36XU

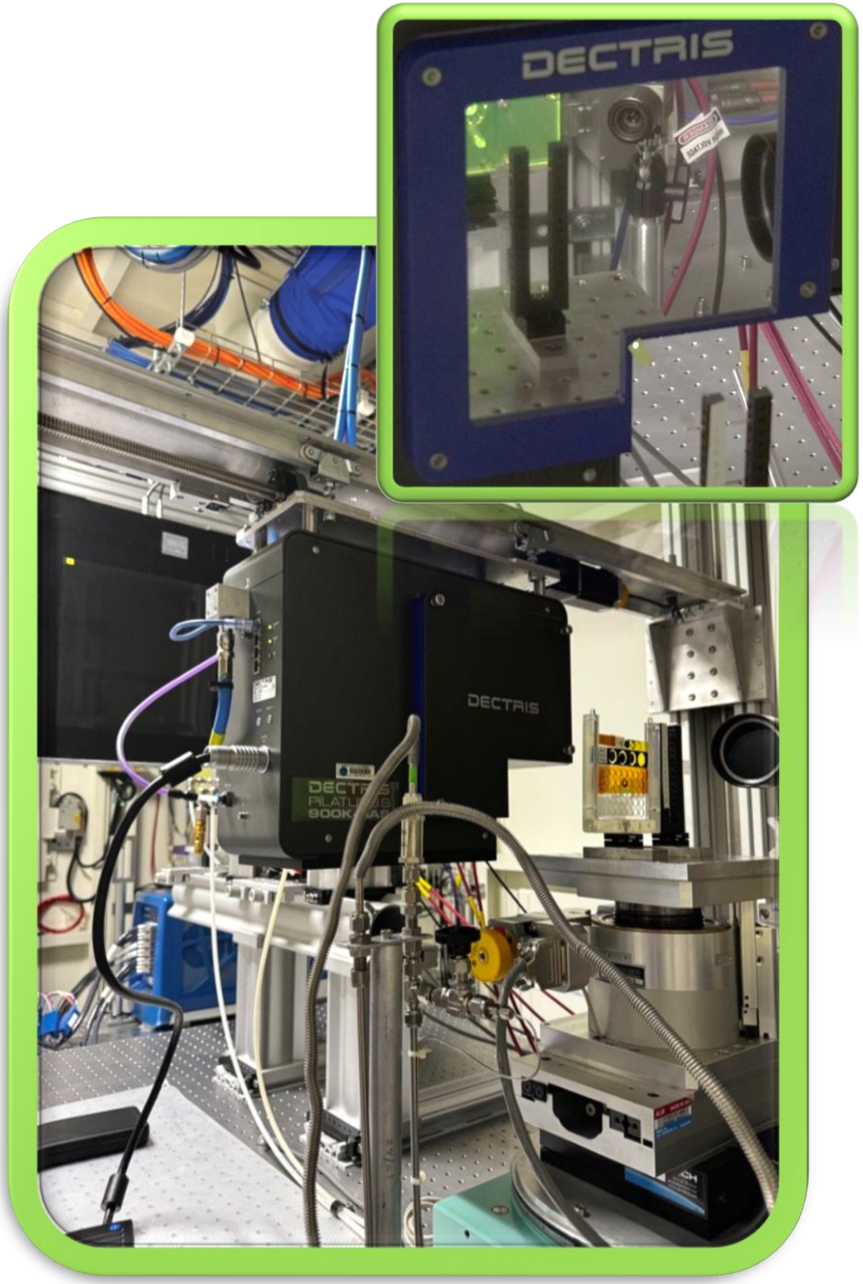


J. Appl. Cryst. (2014). 47, 449–457.
J. Phys. Chem. C 2017, 121, 18202−18213.

Chem. Rec. 2019, 19, 1444–1456.
ACS Sustainable Chem. Eng. 2017, 5, 3631−3636.

Combined XAS & XRD

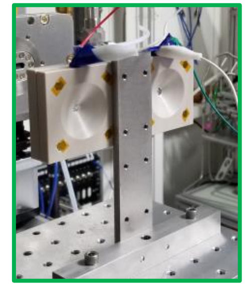




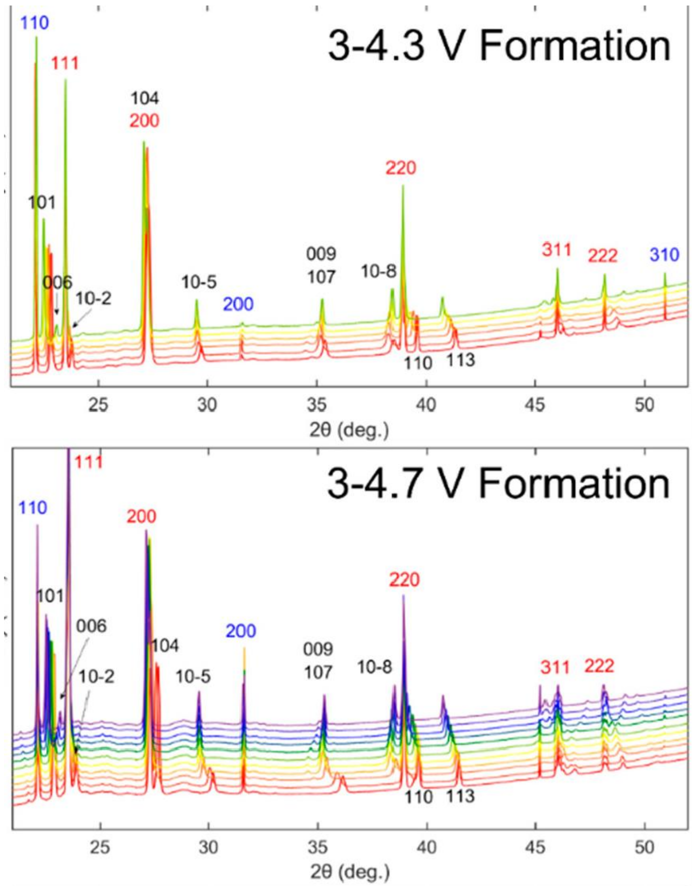
Combined XAS & XRD

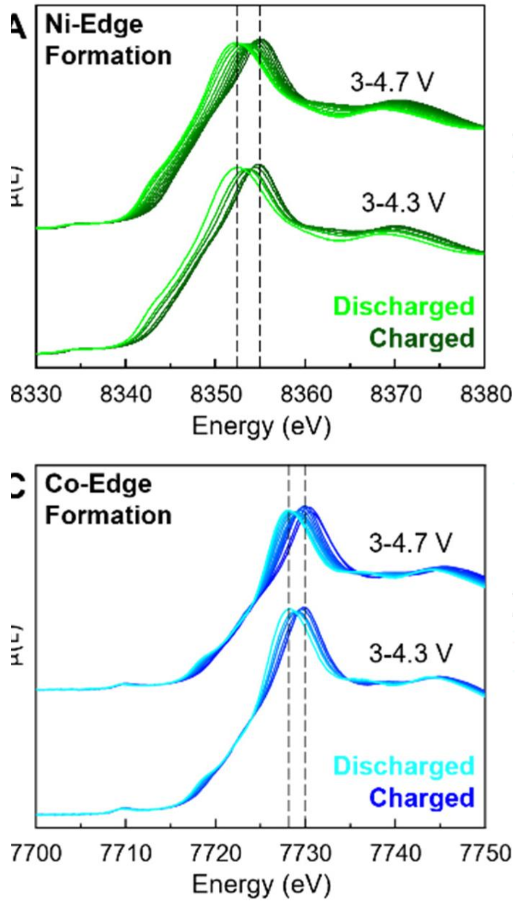
- Case study 1a: Battery

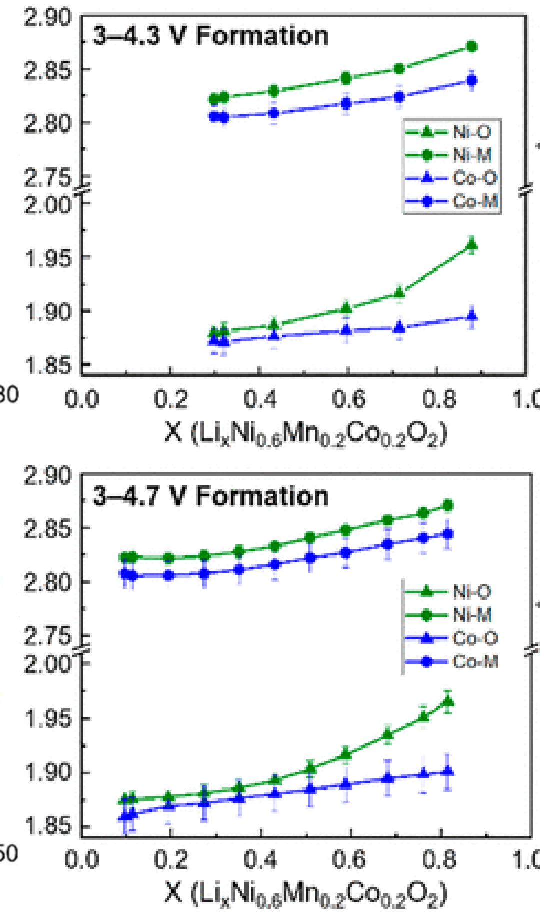
C/5 rate
XAS: Mn, Co, Ni K-egde
XRD: λ = 0.9547 Å



David Bock, BNL



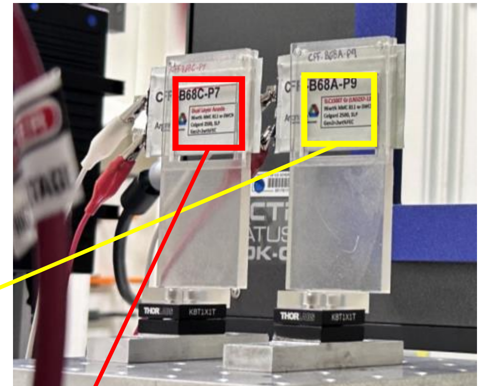




ACS Appl. Mater. Interfaces 2021, 13, 43, 50920−50935.

Combined XAS & XRD

- Case study 1b: Fast- charging Battery

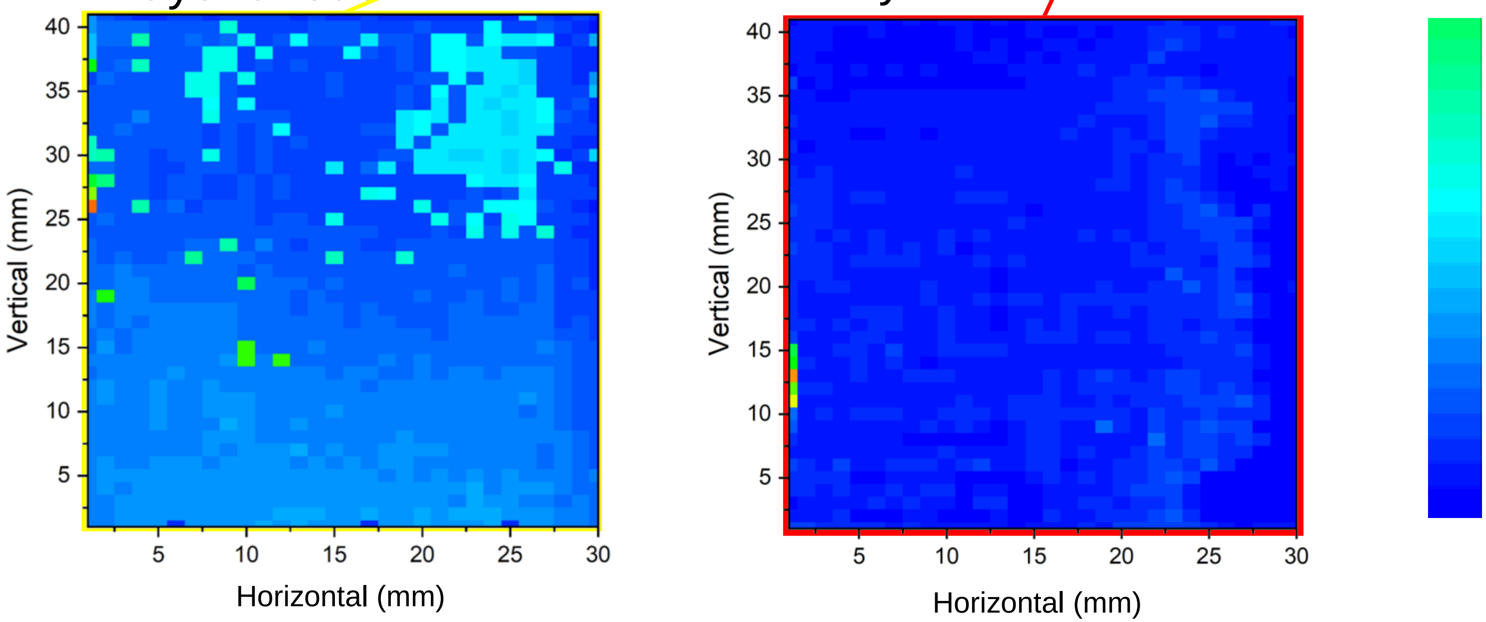


Tianyi Li, ANL

Lithium intensities after 900 fast charging cycles

1 layer anode

2 layers of anode



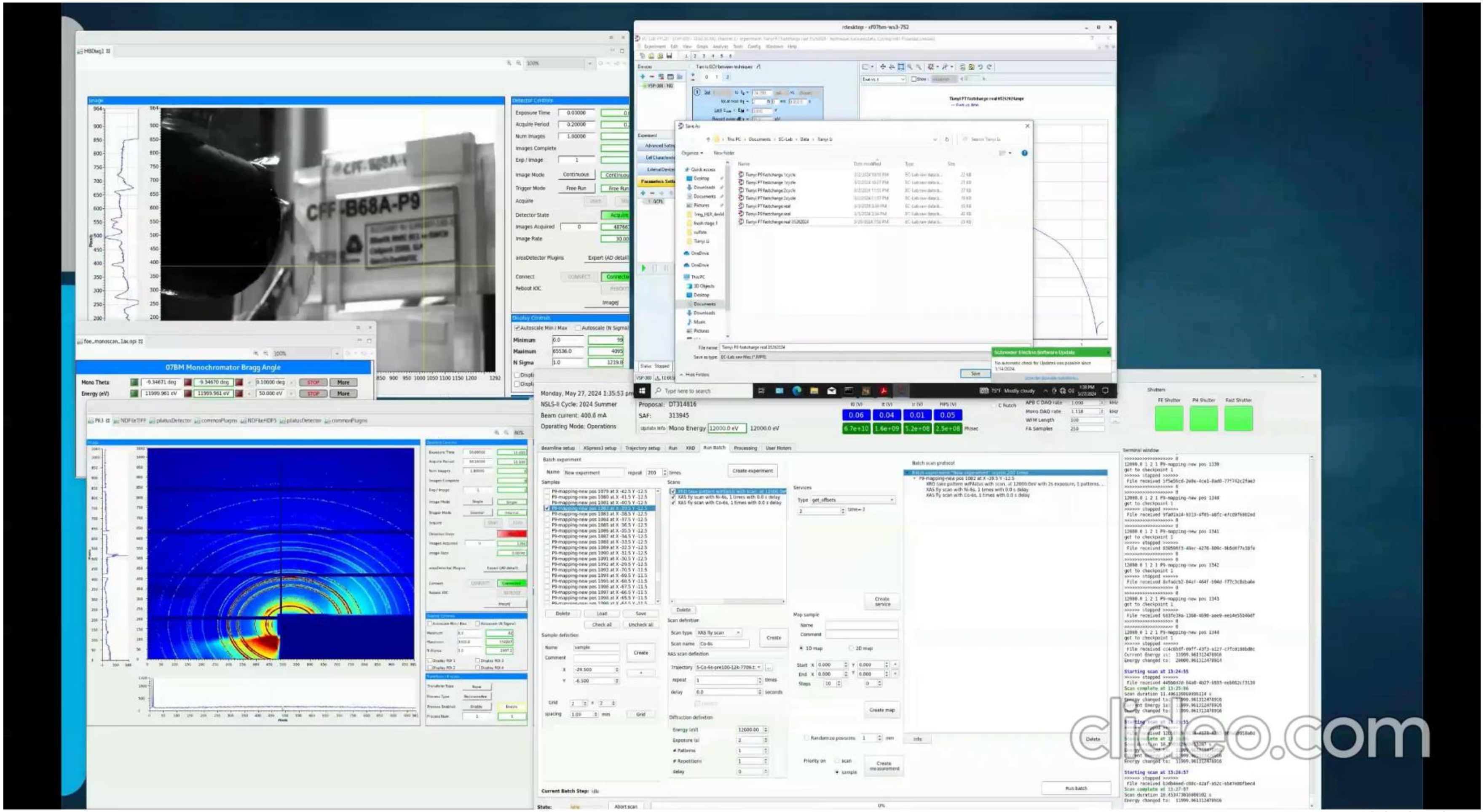
XCEL
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

Unpublished

Combined XAS & XRD

- Case study 1b: Fast- charging Battery
- Real time monitoring, 64X speed

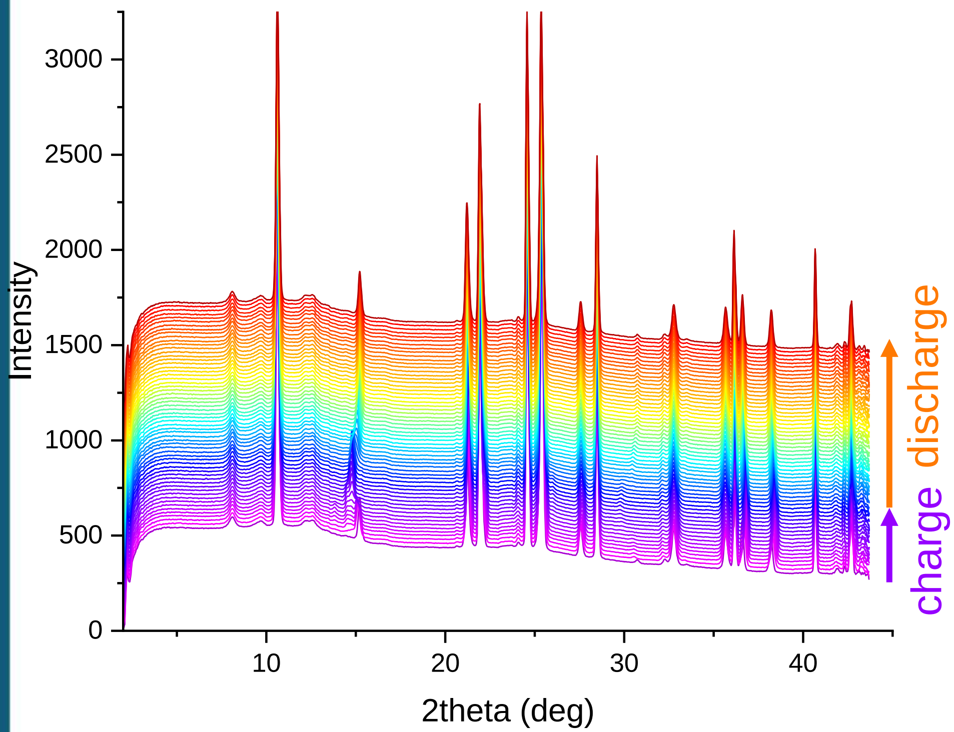
6C rate, 2 spots
XAS: Ni K-egde
XRD: λ = 0.8856 Å

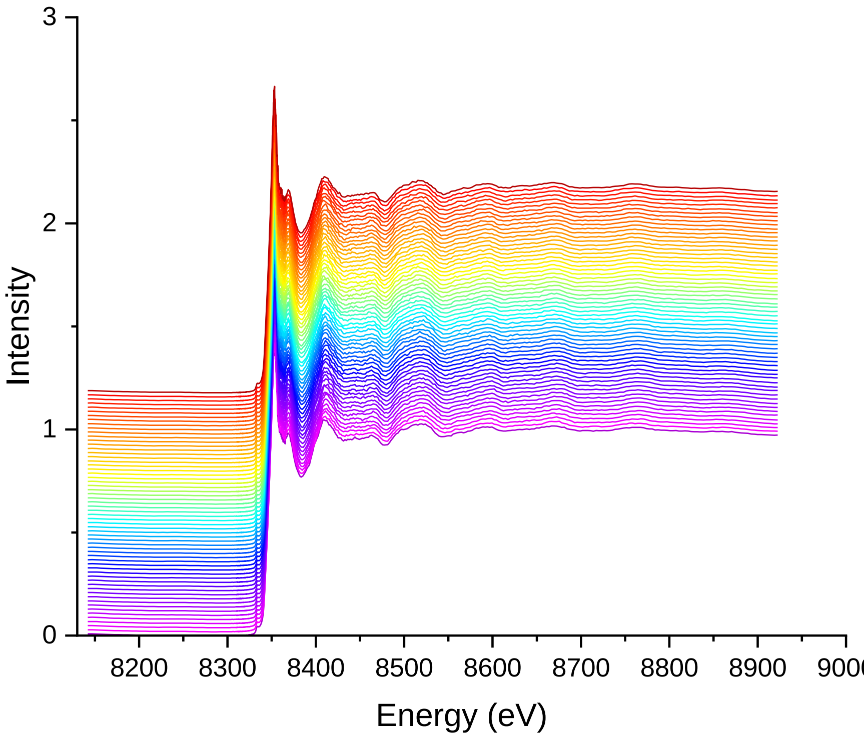


Unpublished

Combined XAS & XRD

- Case study 1b: Fast- charging Battery





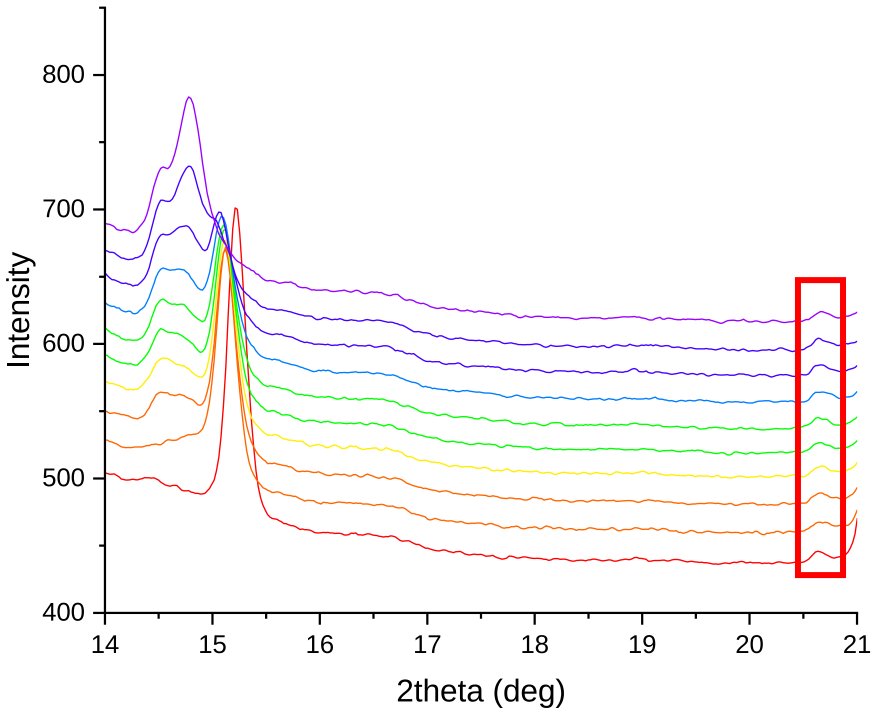
Unpublished

eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

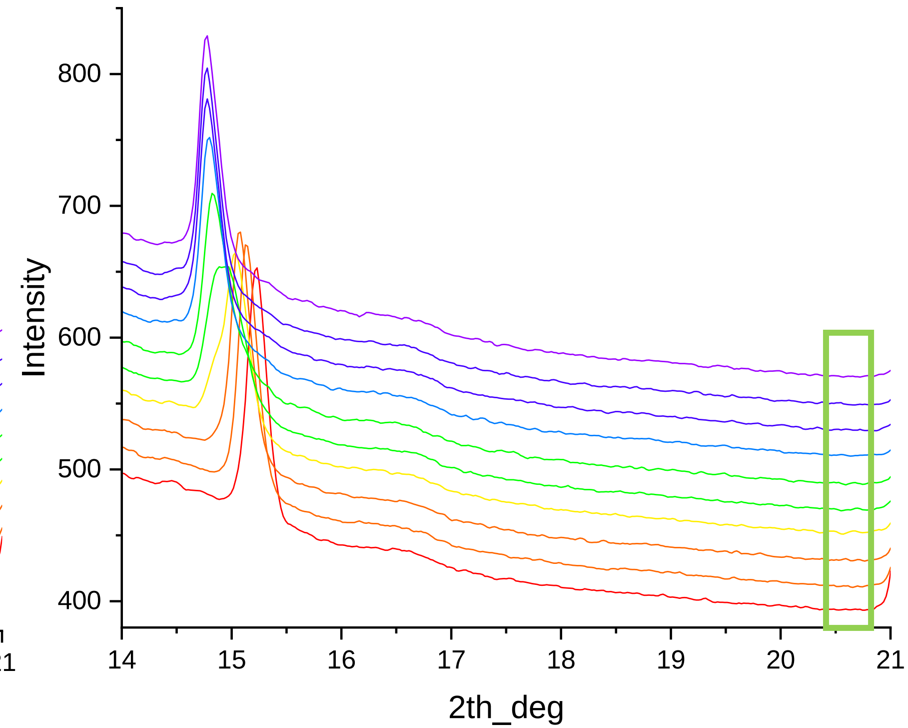
Combined XAS & XRD

- Case study 1b: Fast- charging Battery

Bad spot of 1 layer
Presence of dead lithium



Good spot of 1 layer
No dead lithium

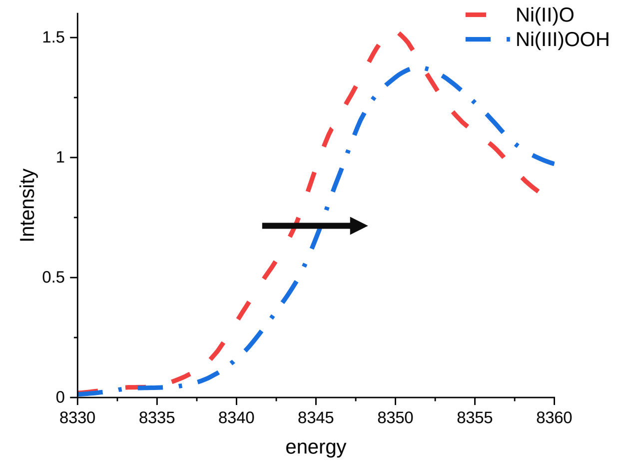


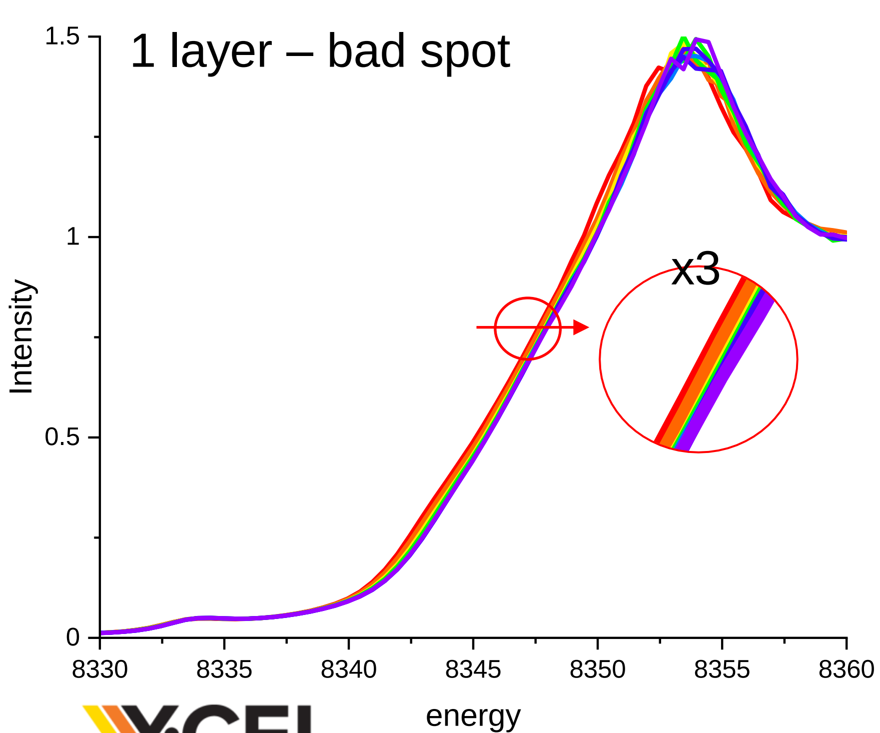
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

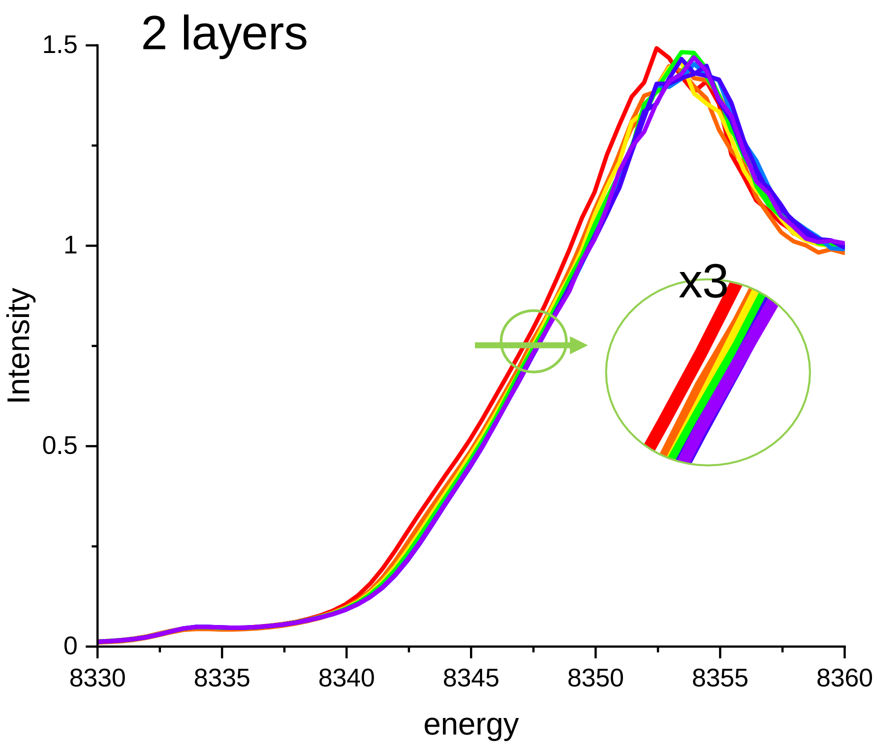
Unpublished

Combined XAS & XRD

- Case study 1b: Fast- charging Battery





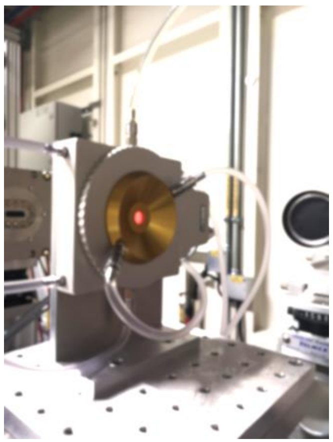


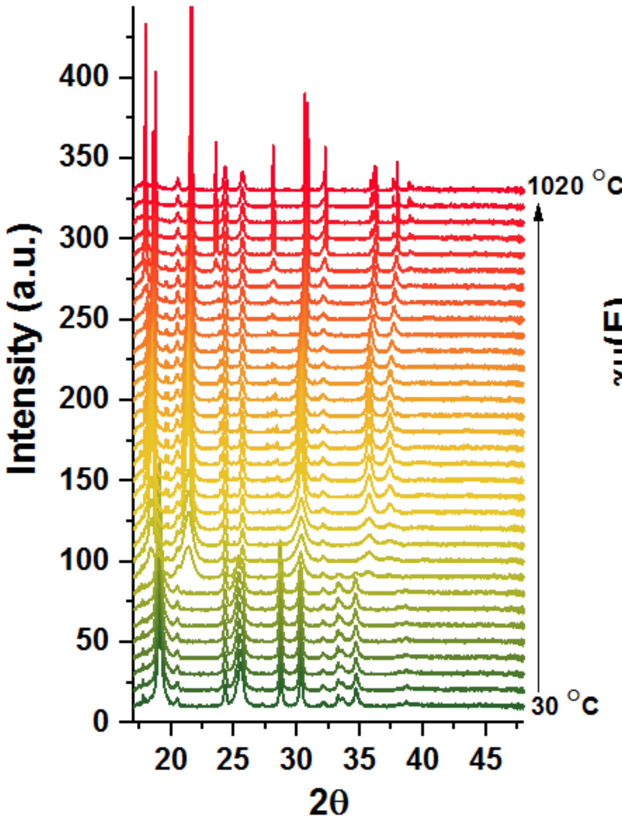
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

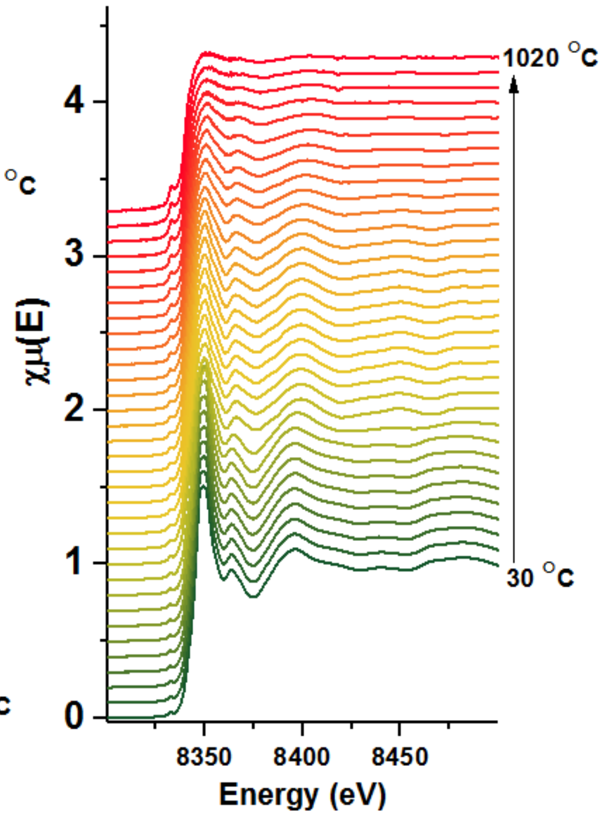
Unpublished

Combined XAS & XRD

- Case study 2: Linkam stage heating







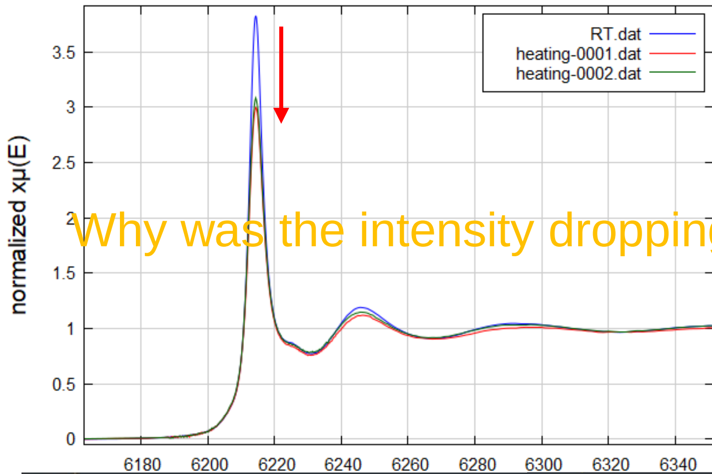
Sizhan Liu, BNL

At 330^{o}C (after temperature correction),
A layer structure begun to form, and Ni was reduced.

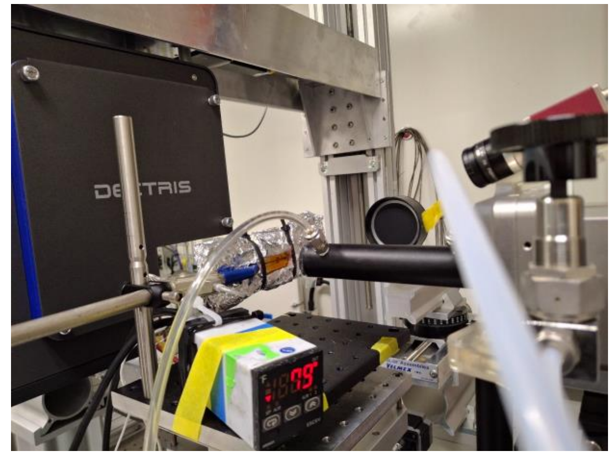
Under review

Combined XAS & XRD

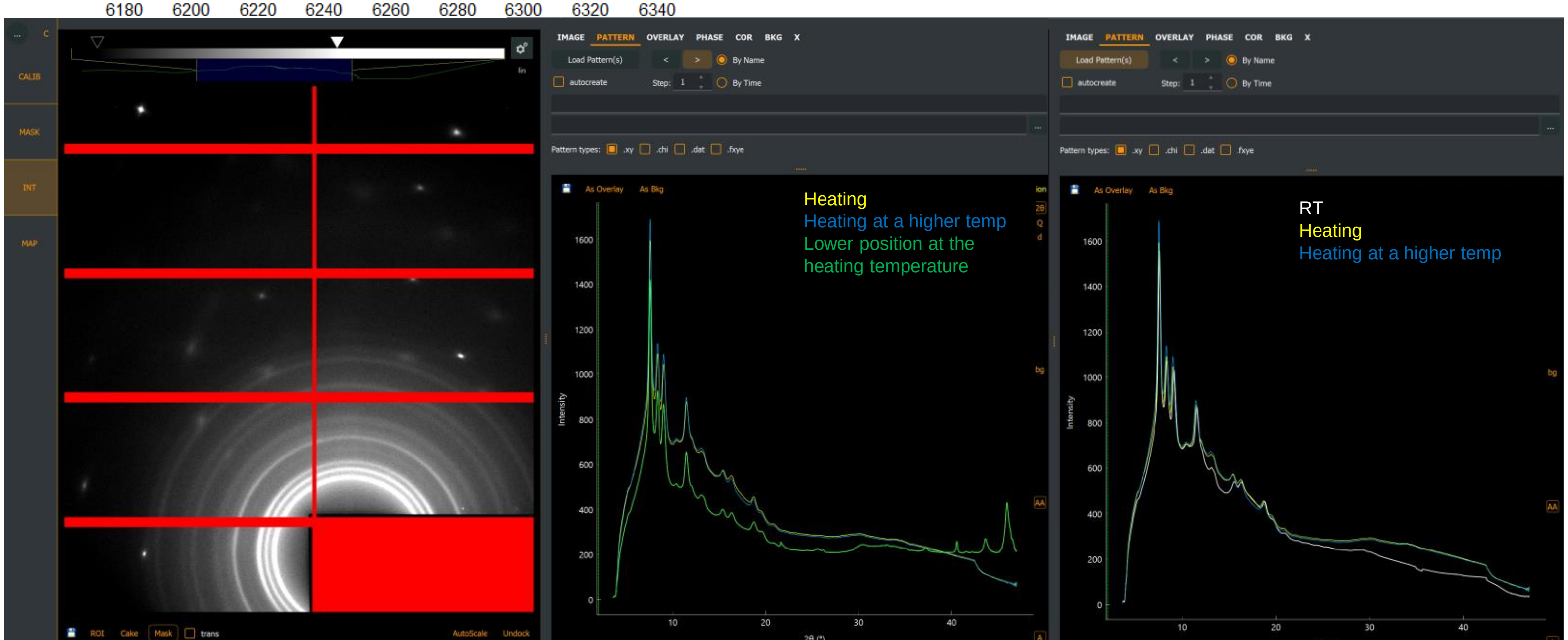
- User's hydrothermal setup



Why was the intensity dropping?

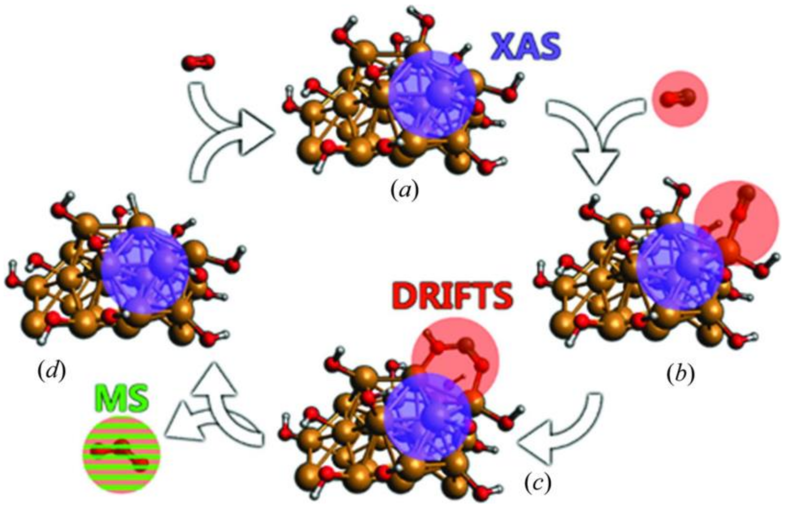


Xin Zhang, PNNL



Unpublished

Combined XAS & DRIFTS



XAS provides information on the oxidation state, electronic structure, and local coordination environment around specific elements.

DRIFTS is sensitive to molecular vibrations and offers insight into surface species, functional groups, and adsorbed molecules on catalyst surfaces.

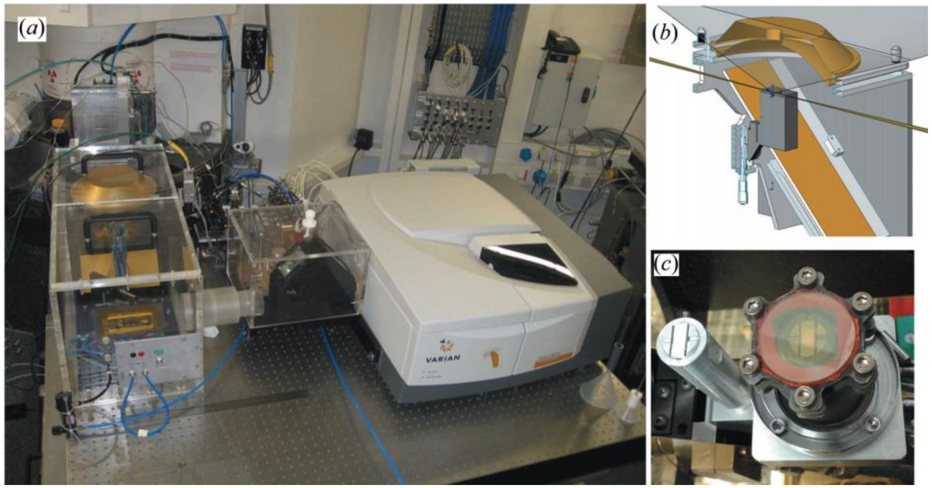
Combined XAS/DRIFTS: simultaneously monitor:

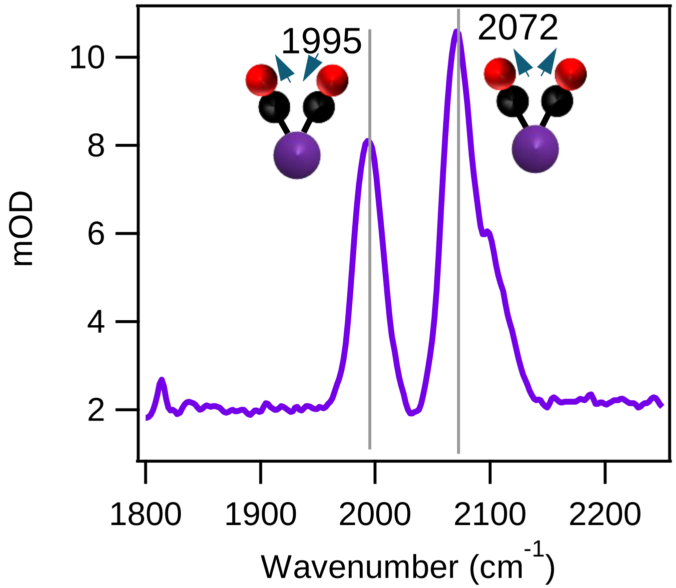
- Changes in the metal center (via XAS)
- Organic species or reactants/products (via DRIFTS) during a reaction.

J.SynchrotronRad.(2019).26,801–810.

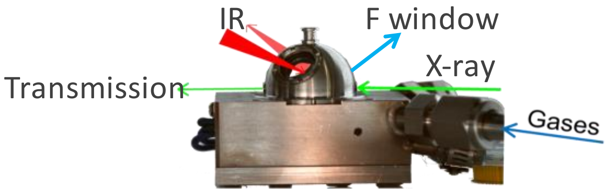
Combined XAS & DRIFTS

ESRF ID24

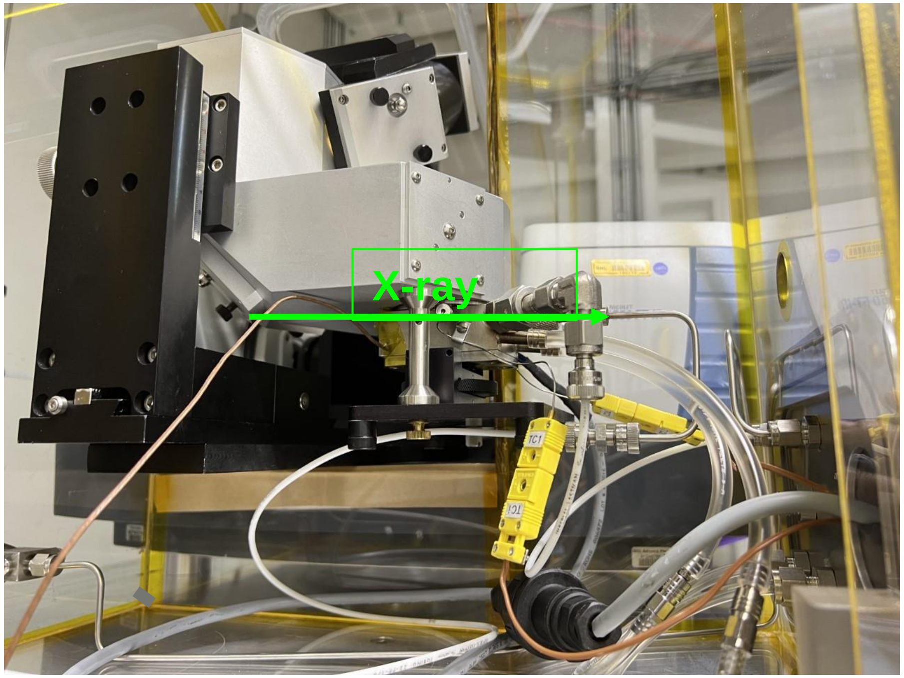




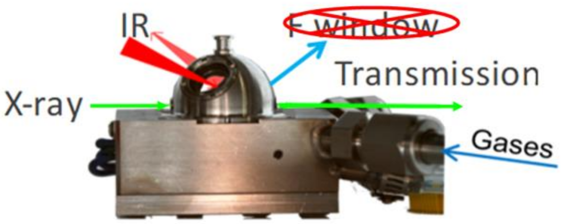
APS 9-BM



QAS hutch C endstation -- DRIFTS



- Thermo-Nicolet iS-50 IR spectrometer and Harrick cell
- Plexiglass box to avoid influence from the air for DRIFTS measurement
- Transmission XAS only, fixed sample depth of 3mm
- Suitable for high-energy measurements but not for diluted samples or low-energy measurements below 12 keV.

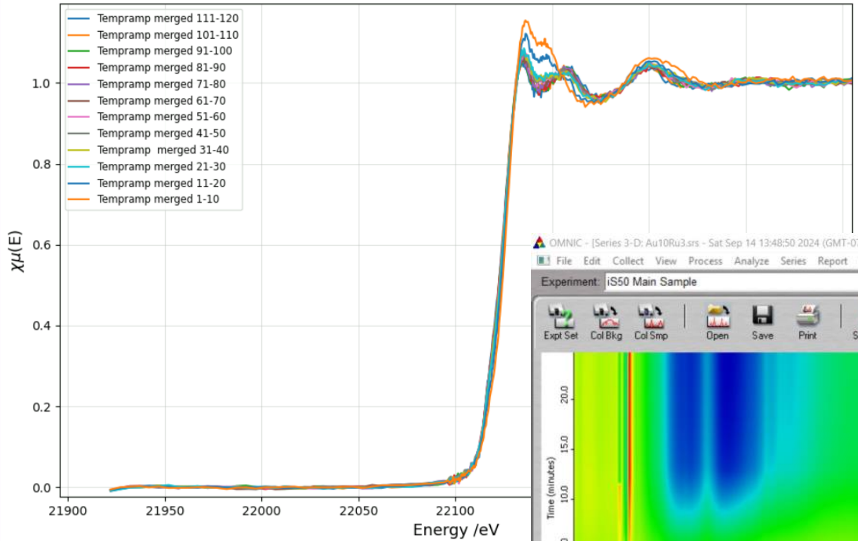


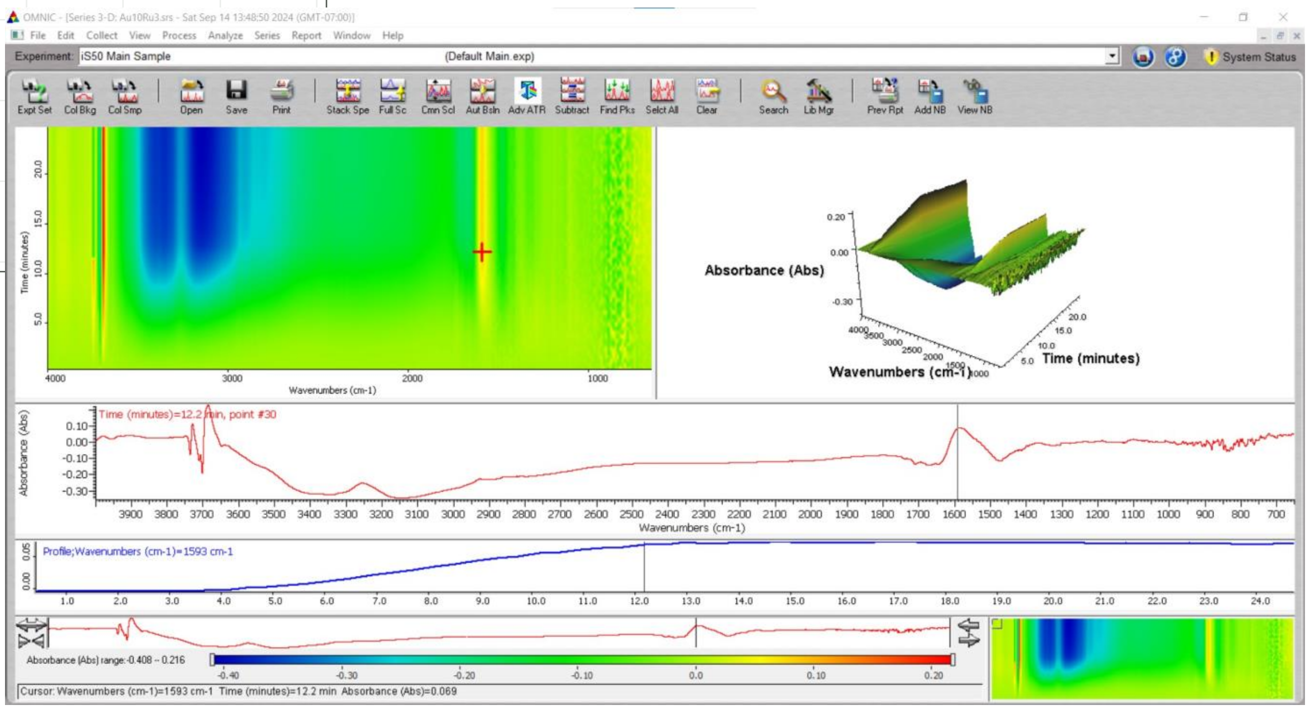
Combined XAS & DRIFTS

- Science commissioning result

~5wt.% Ru

Lin Yuan,
Standford University



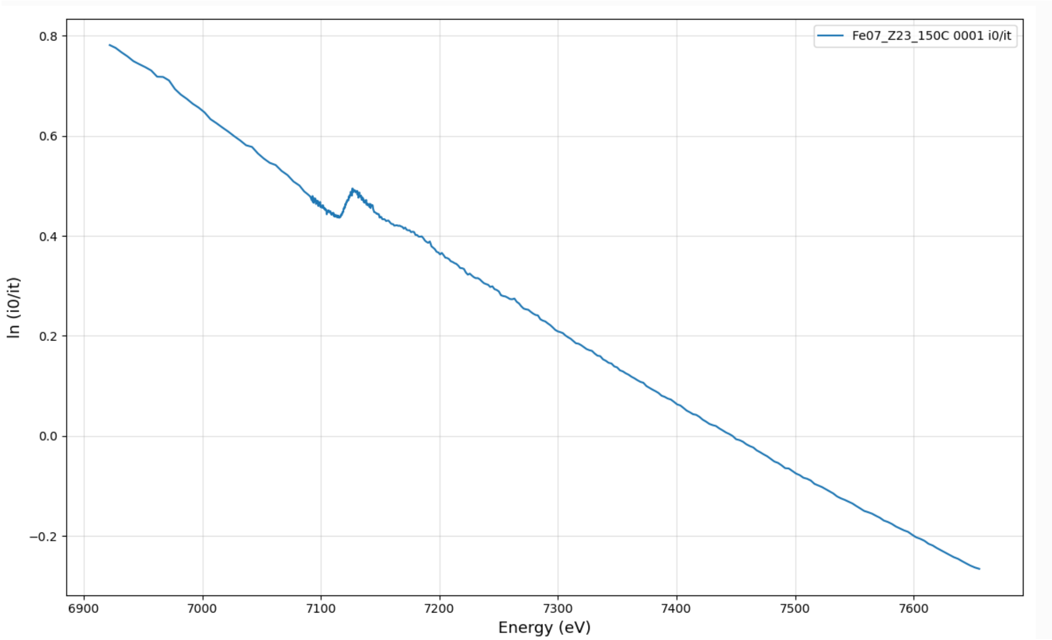


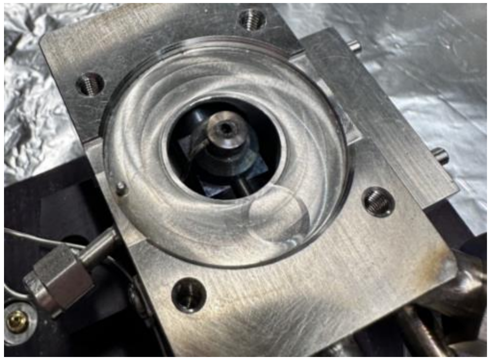
Combined XAS & DRIFTS

- Science commissioning result

0.7wt%Fe samples
Reduced the depth to 1mm with a graphite insert

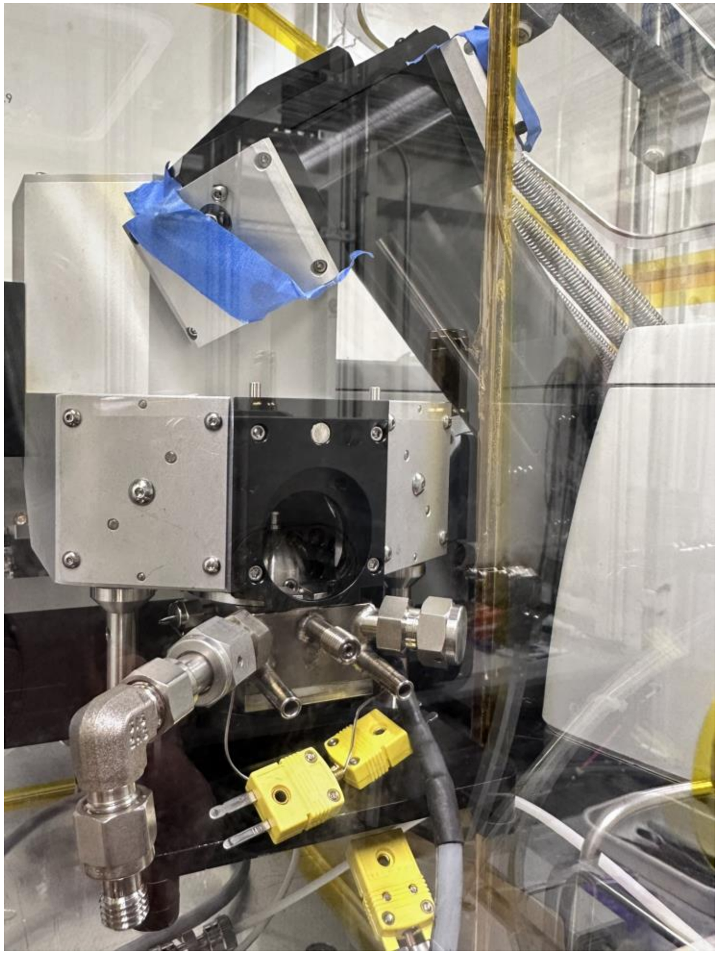
Dominik Wierzbicki,
TES, NSLS-II





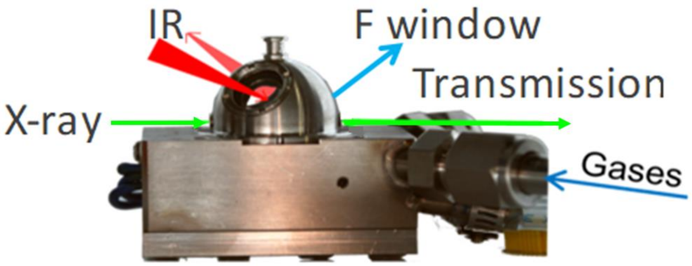
Combined XAS & DRIFTS

- Fluorescence capability under development



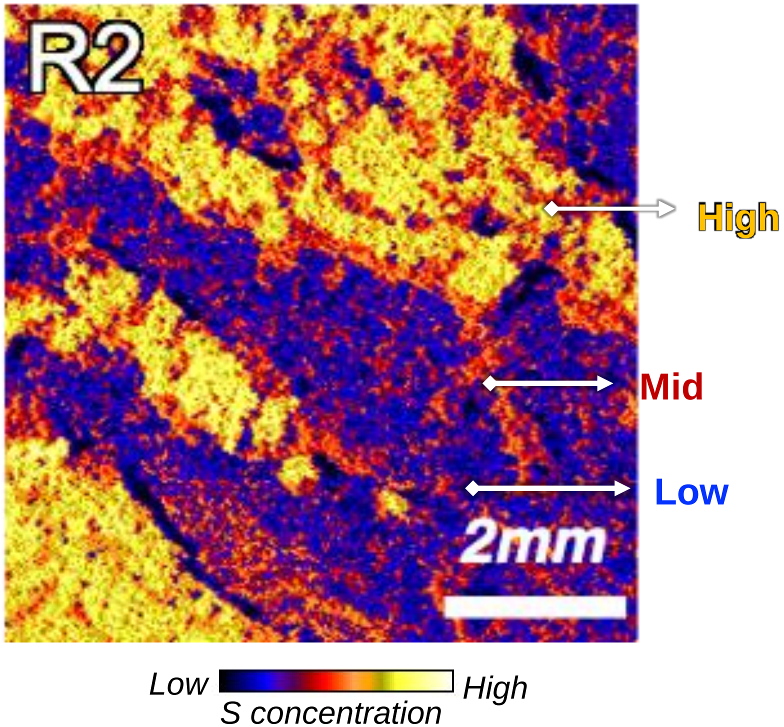
Front: X-ray in

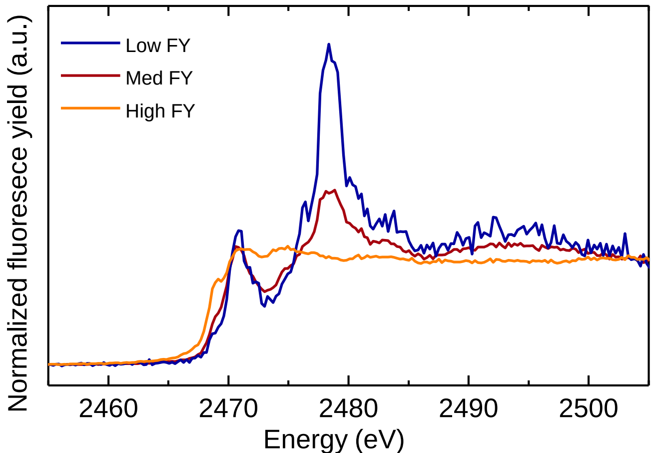
Back: Fluorescence



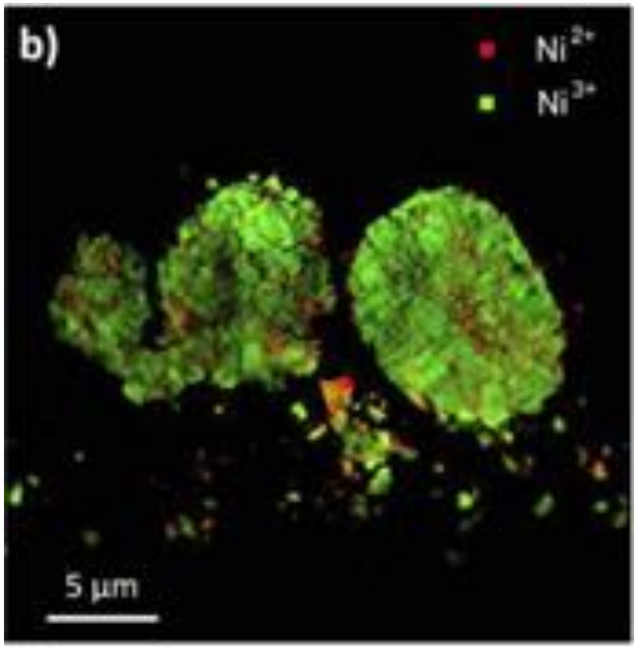
Other combined techniques with XAS

XRF mapping





XANES tomography

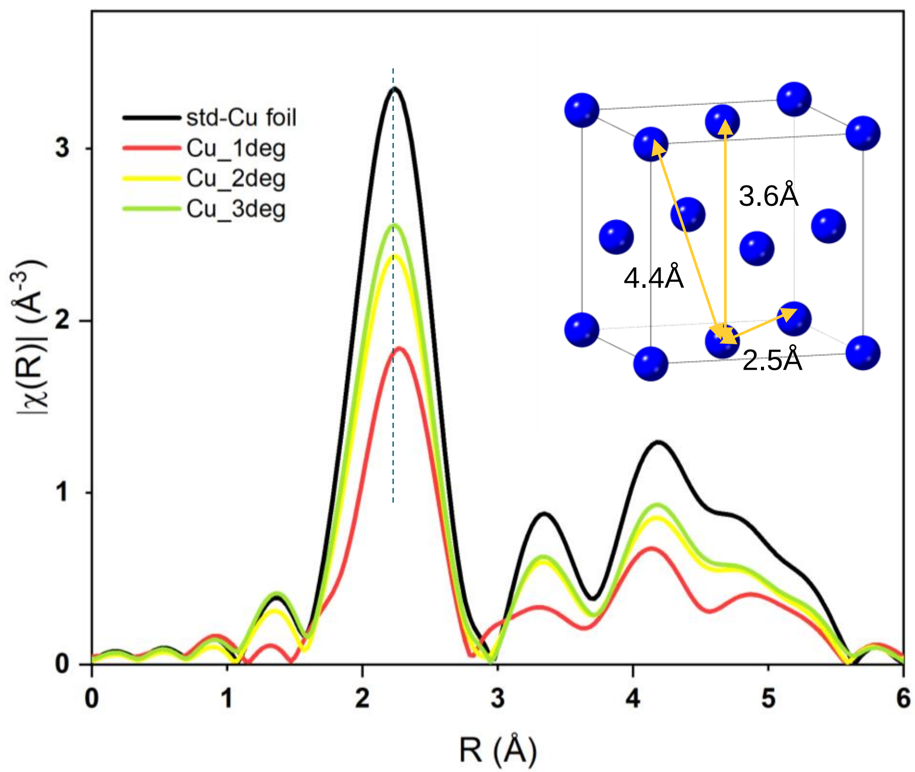


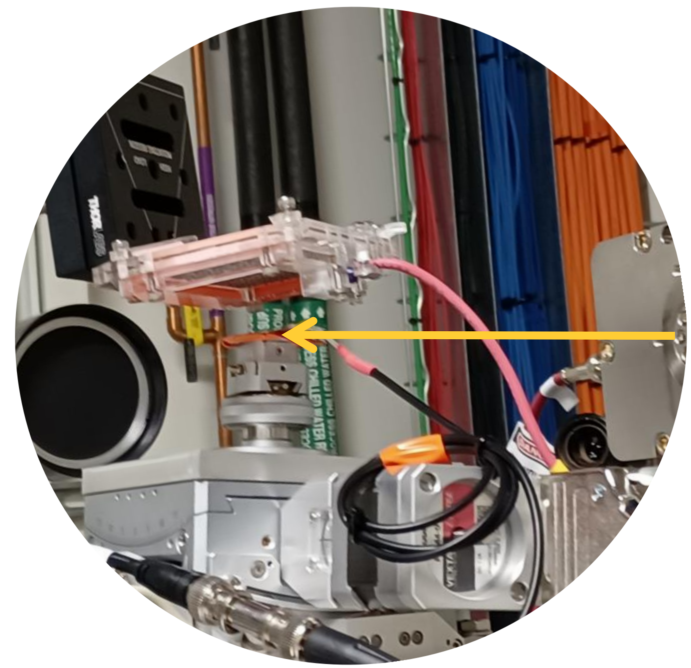
Energy Environ. Sci. 13, 3620-3632 (2020)

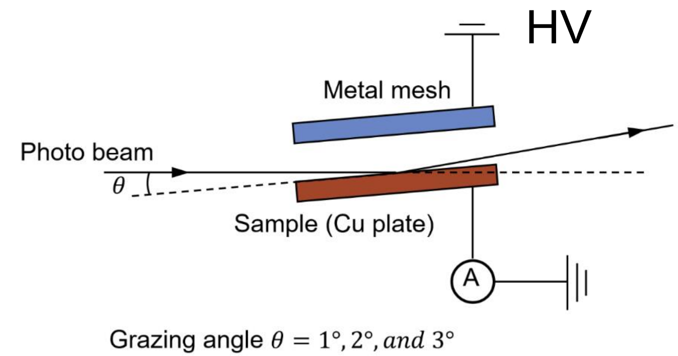
Pseudo grazing incidence -TEY

In collaboration with TES LBS Dr. Yonghua Du

Surface sensitive measurement with TEY



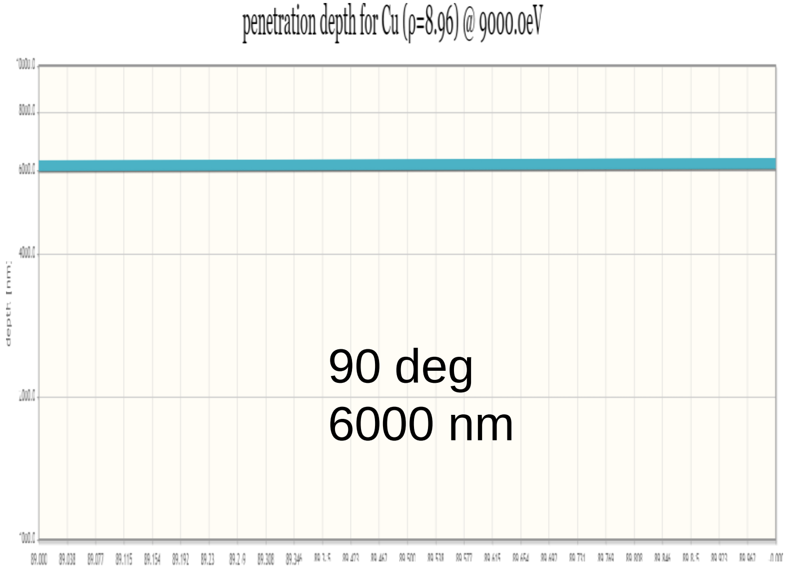


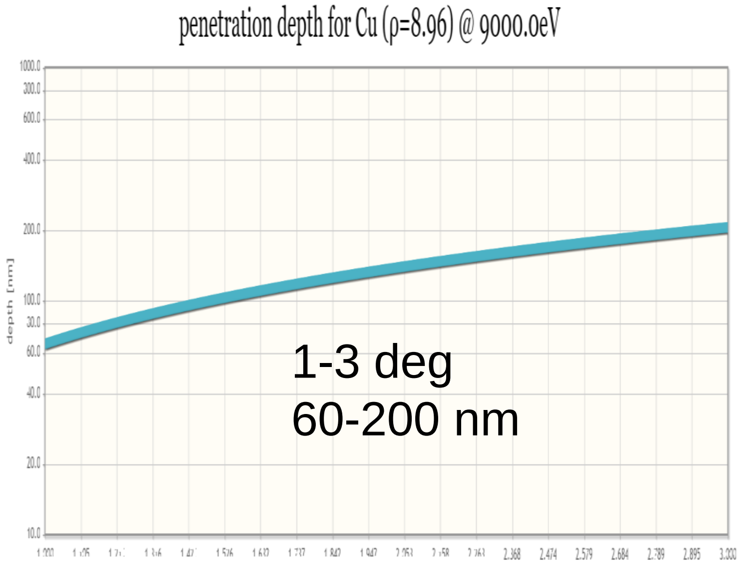


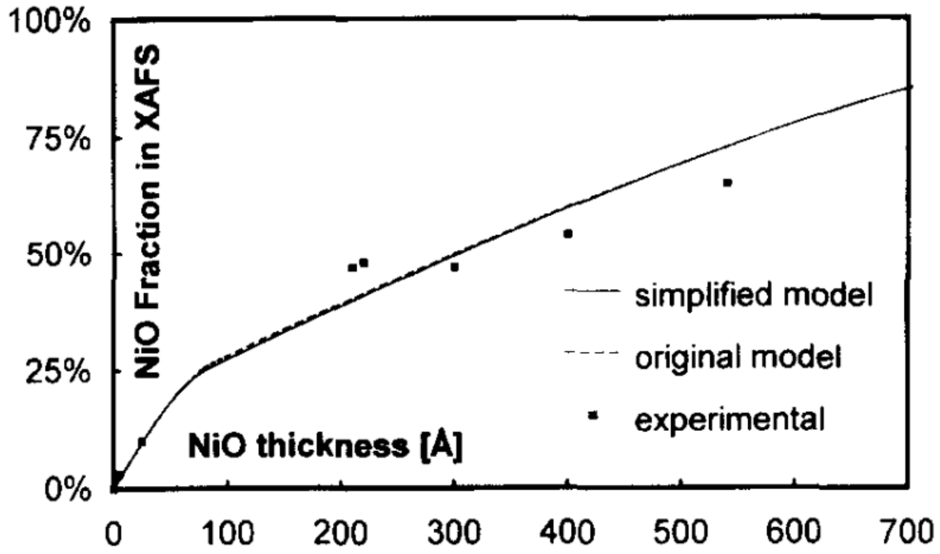
With incident angle of 1 deg

- Surface nearest Cu-Cu1 bond distance increase
- Coordination number of the first two Cu-Cu bonds decrease

Pseudo grazing incidence -TEY



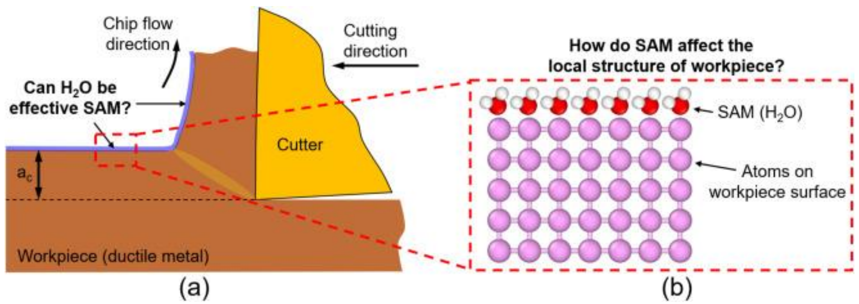


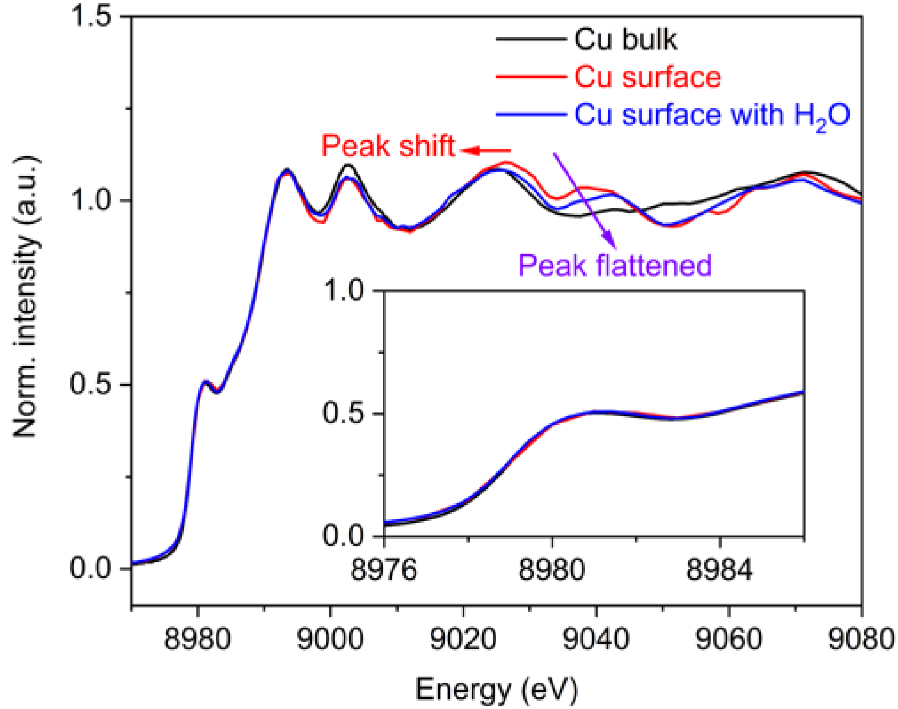


Surface Science 324 (1995) L371-L377

Pseudo grazing incidence -TEY

How water quickly effected the Cu surface and make the cutting much easier?





Journal of Cleaner Production 481 (2024) 143994

Take-home message

- **Combined synchrotron techniques in a single experiment at one beamline** provide complementary insights, offering a more comprehensive understanding of materials and their behaviors.

- However, integrating multiple techniques often requires **compromises**, as sample conditions need to accommodate both methods.

- **Careful experimental design** is crucial to ensure that conditions are feasible for both techniques.

- Despite the challenges, this approach significantly enhances **real-time, multi-dimensional characterization**.

Unpublished

Thank you!