Advancing Research with Combined Synchrotron Techniques

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03/20/2024

Outline

Introduction

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

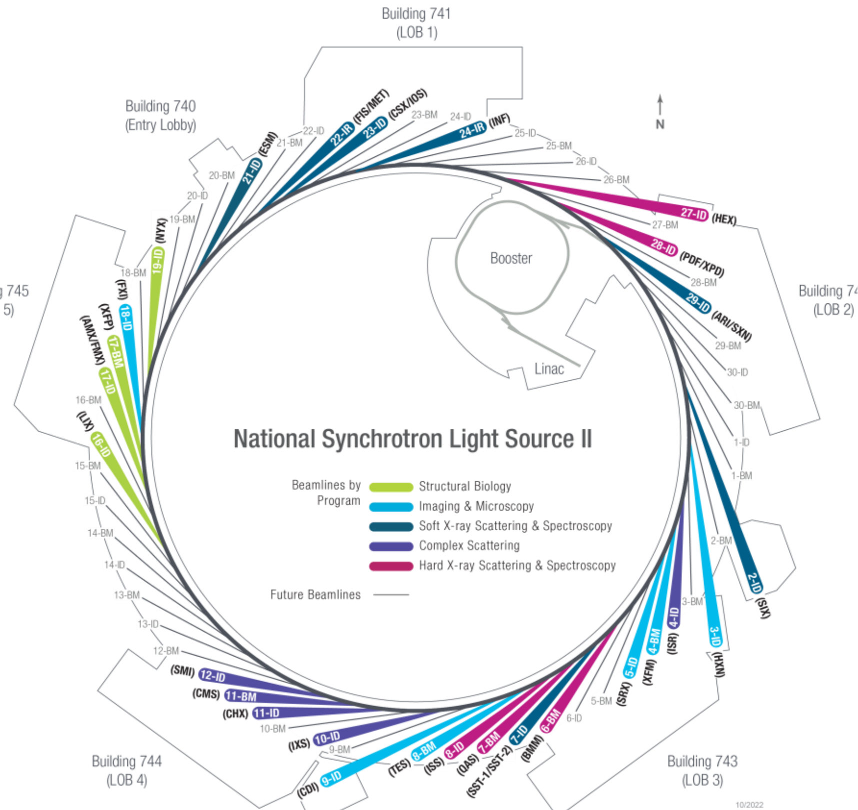
Combining Techniques

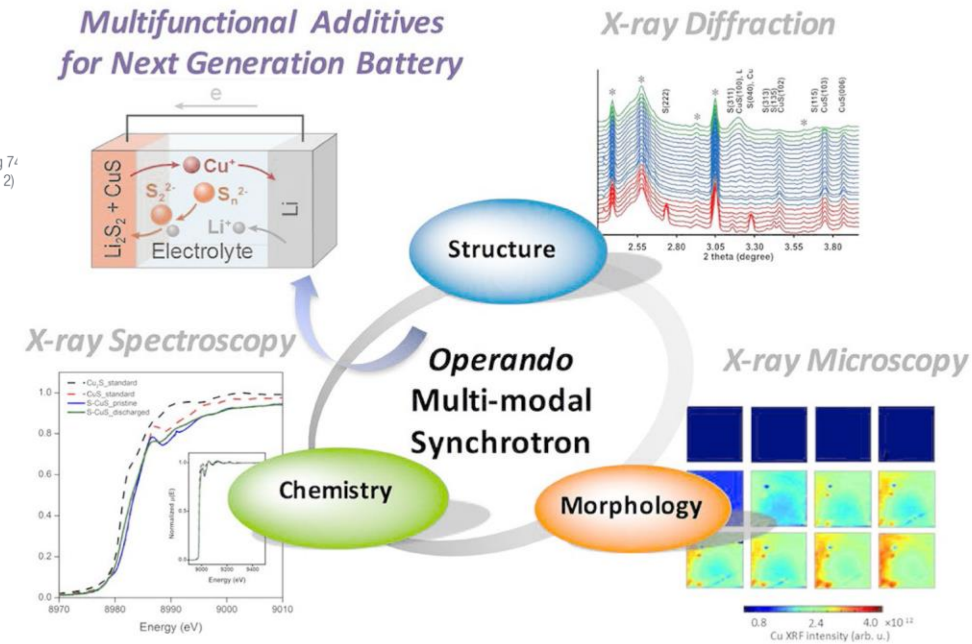
- XAS + XRD: Comprehensive structural analysis
- XAS + DRIFTS: Catalyst insights

GI

- Total electron yield

Introduction





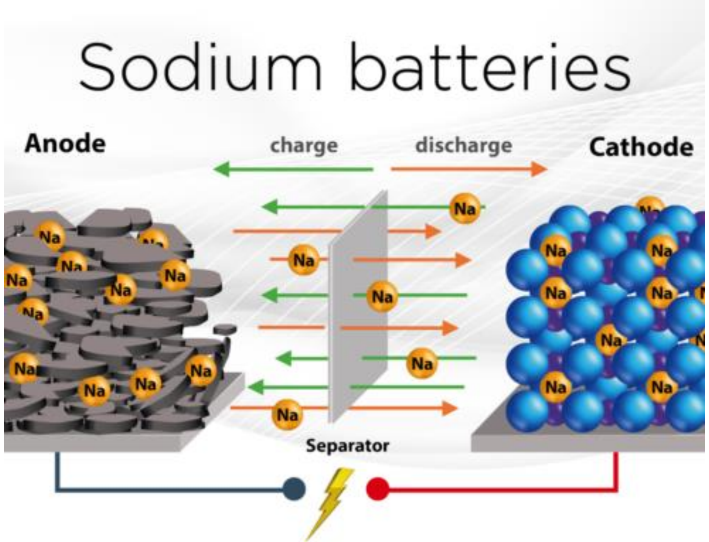
- Comprehensive Analysis
- Real-Time Monitoring
- Maximize data yield from valuable beamtime
- Reveal unexpected properties or phenomena
- Bridge gaps between research fields.

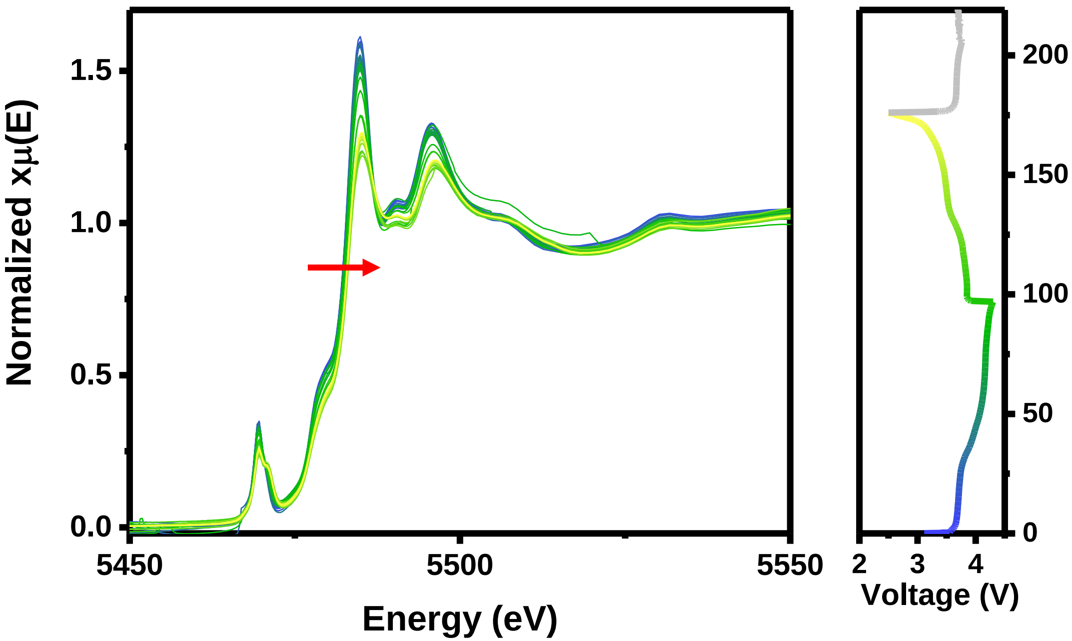
Brookhaven National Laboratory

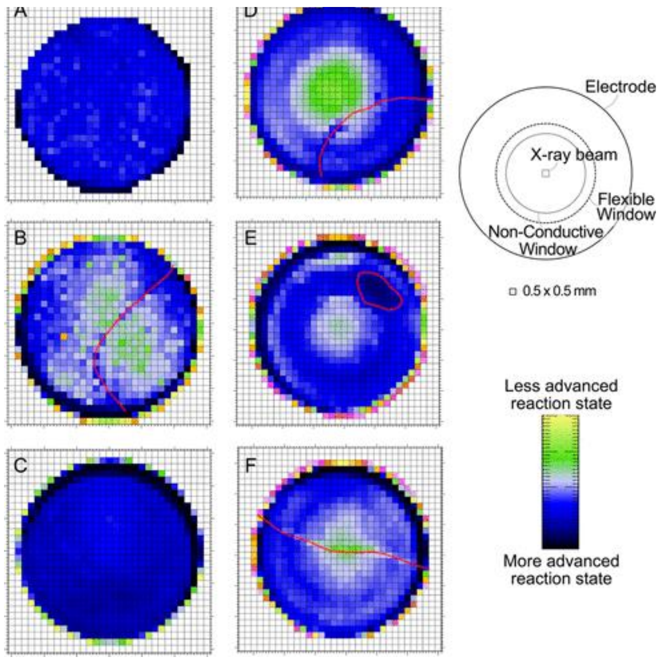
Sci. Rep. 7, 12976 (2017)

Introduction

Na-ion battery, slow reaction??







No Cross-Validation!

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

Sci. Rep. 7, 12976 (2017)

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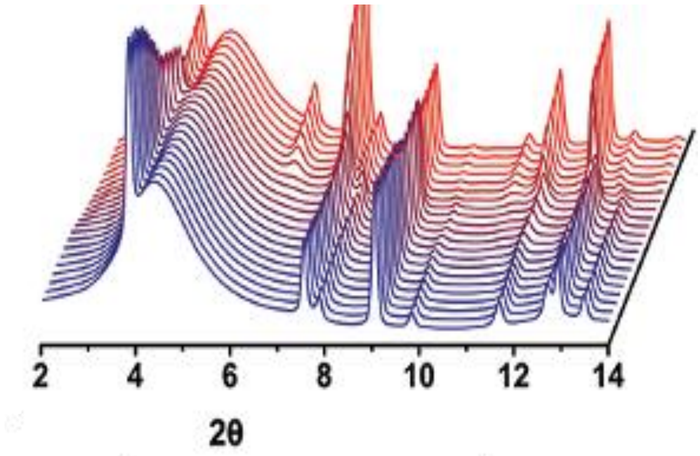
Combined XAS & XRD

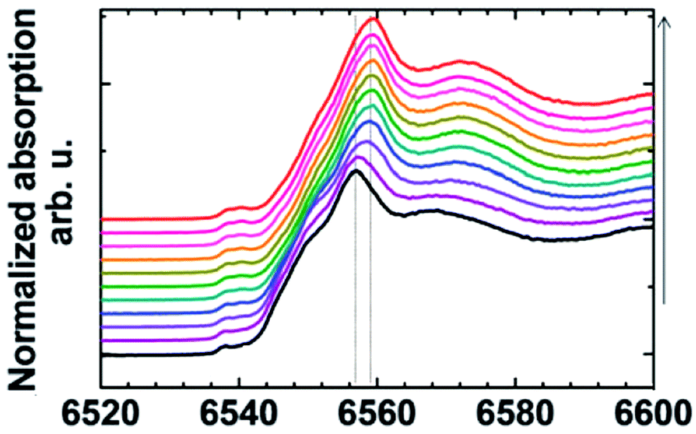
X-ray diffraction (long-range order):

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



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

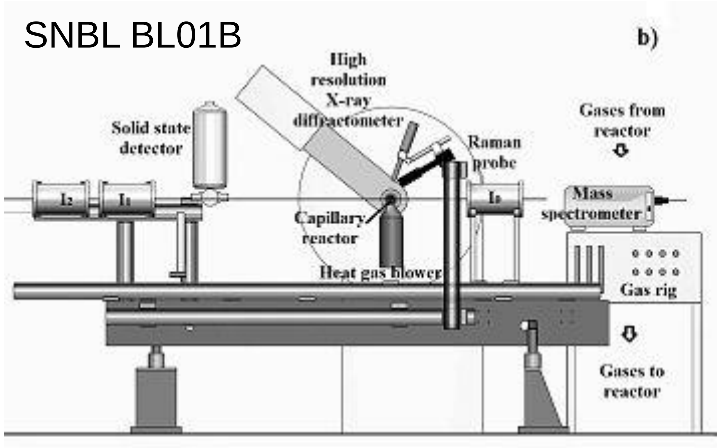
- Electronic structure and oxidation state of specific elements
- Local coordination geometry and bond lengths.



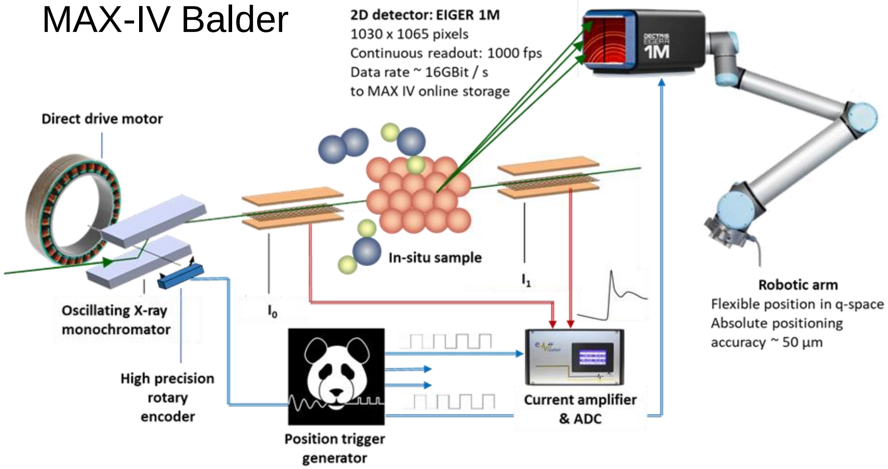
Combined XAS & XRD

NSLS II 7-BM

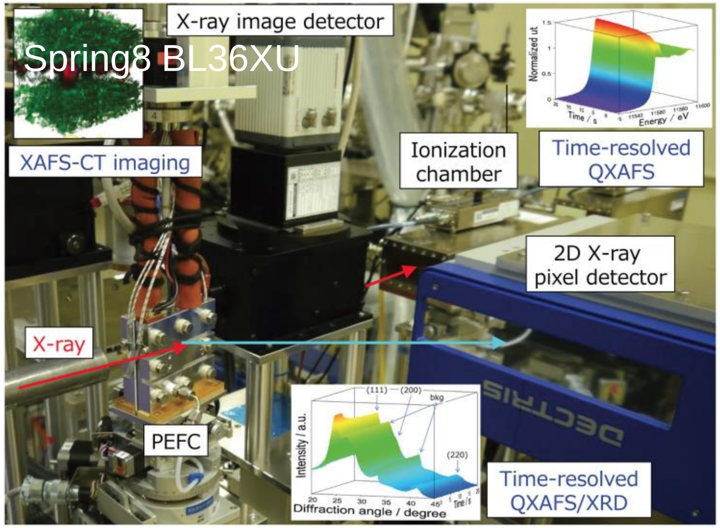
SNBL BL01B



MAX-IV Balder



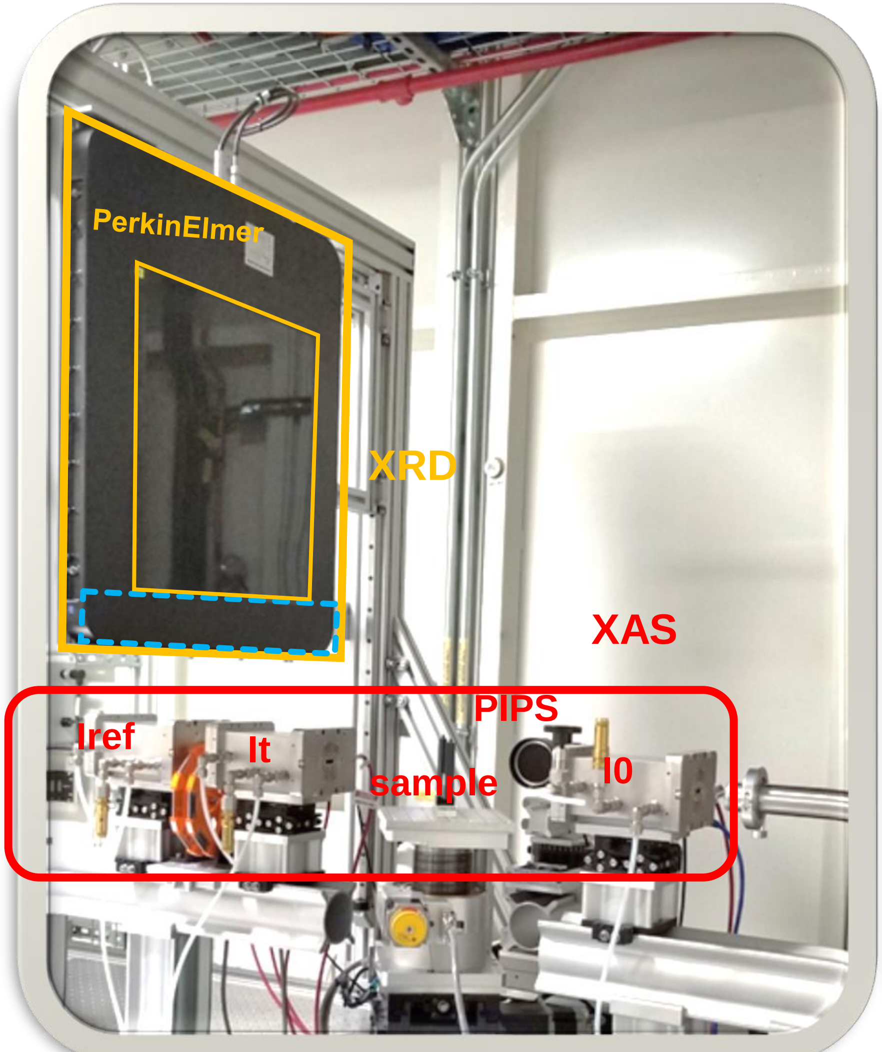
Spring8 BL36XU

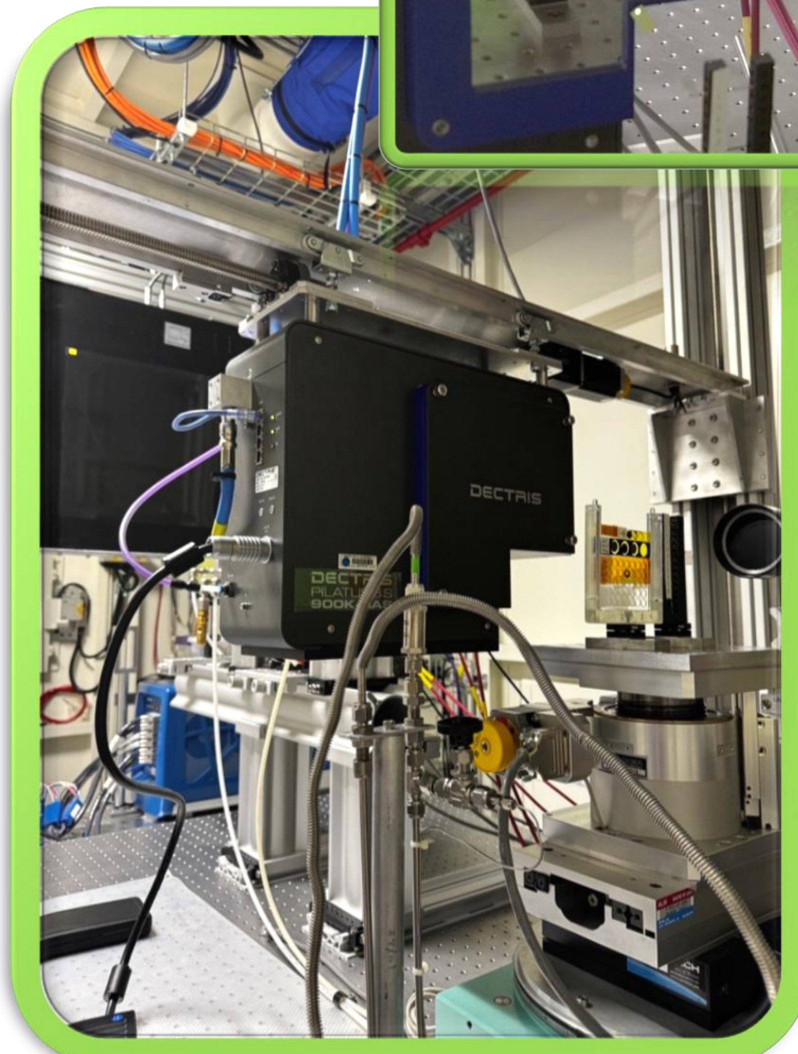


J. Appl. Cryst. (2014). 47, 449–457.
J. Phys. Chem. C 2017, 121, 18202−18213.

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

Combined XAS & XRD

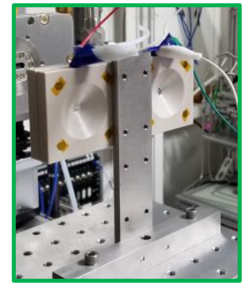




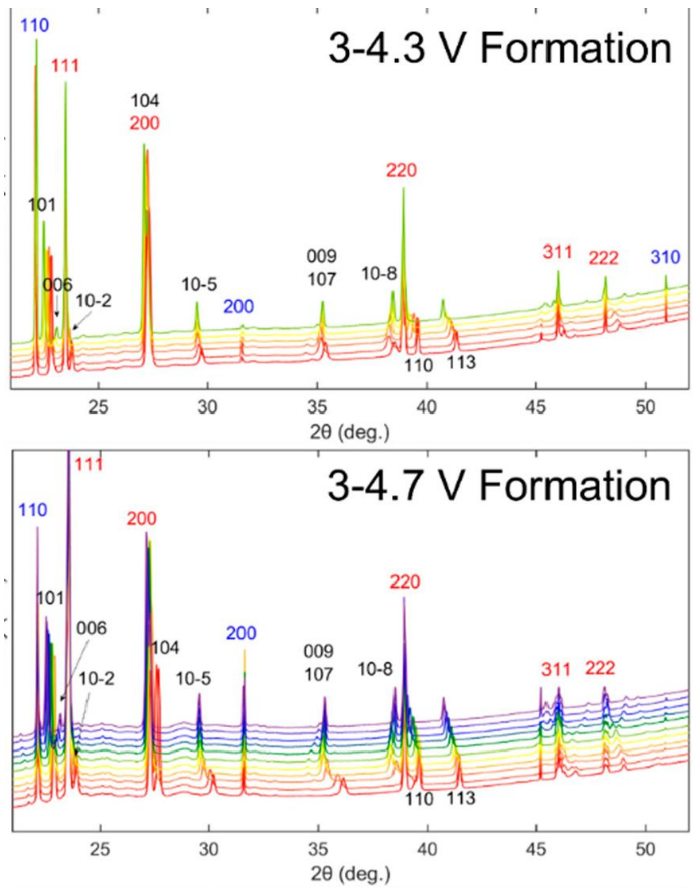
Combined XAS & XRD

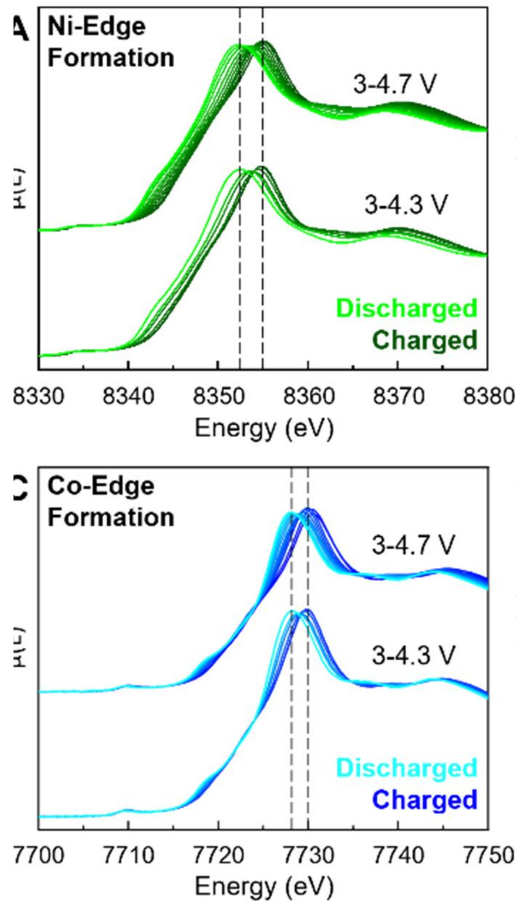
- Case study 1a: Battery

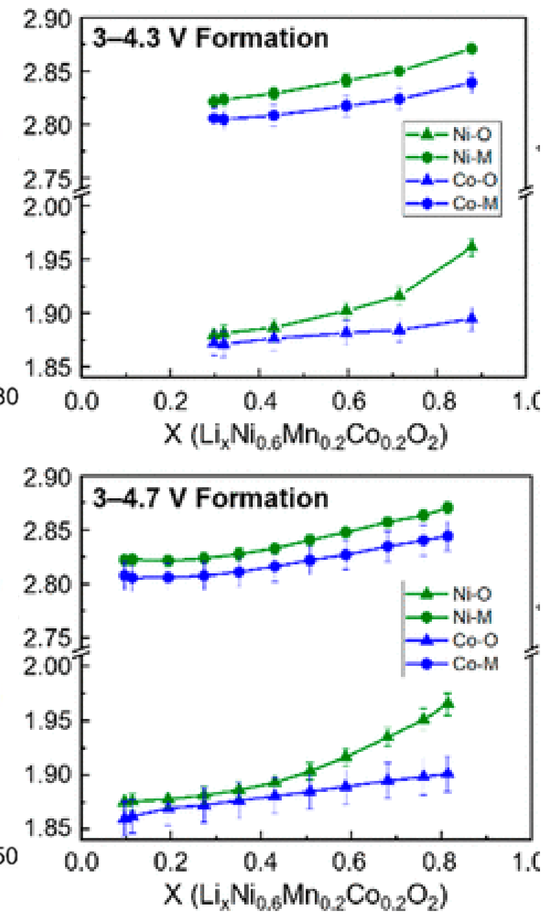
C/5 rate
XAS: Mn, Co, Ni K-egde
XRD: λ = 0.9547 Å



David Bock, BNL





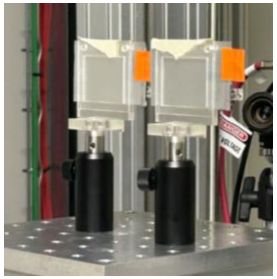


ACS Appl. Mater. Interfaces 2021, 13, 43, 50920−50935.

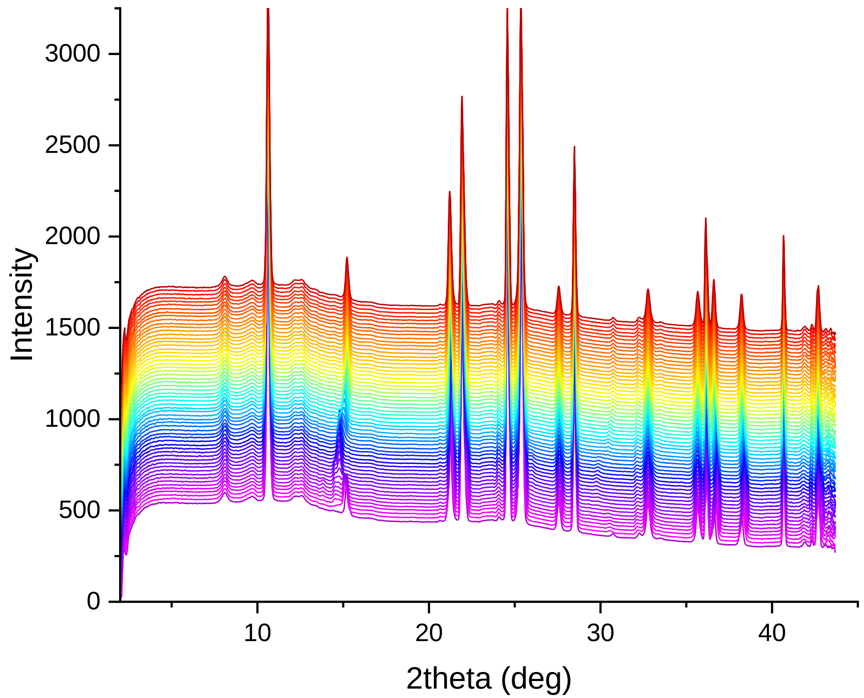
Combined XAS & XRD

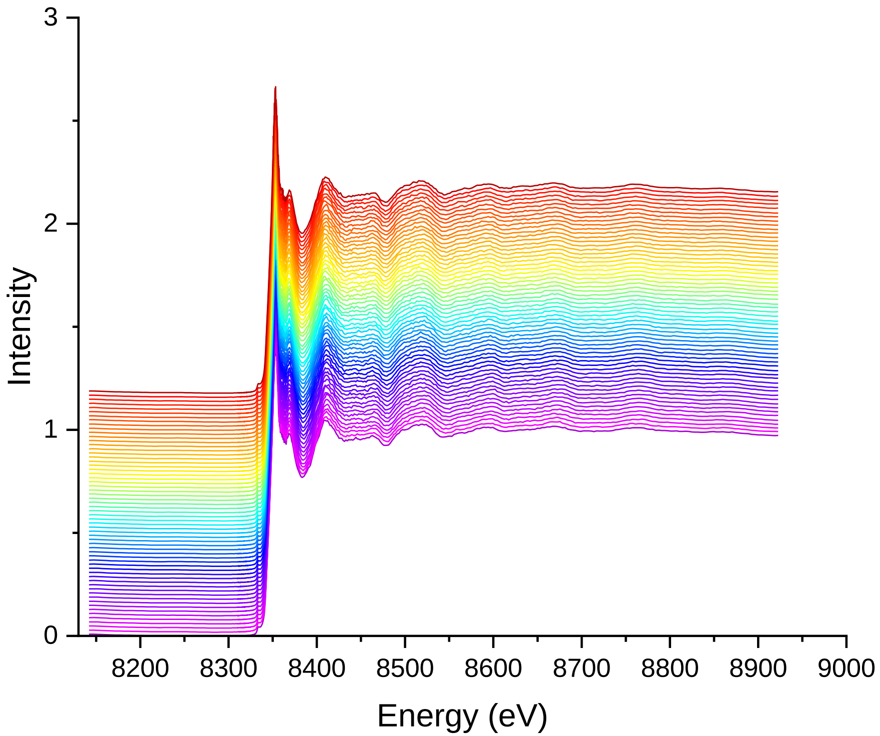
- Case study 1b: Fast- charging Battery

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



Tianyi Li, ANL





XCEL
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

Unpublished

Combined XAS & XRD

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XAS: Ni K-egde
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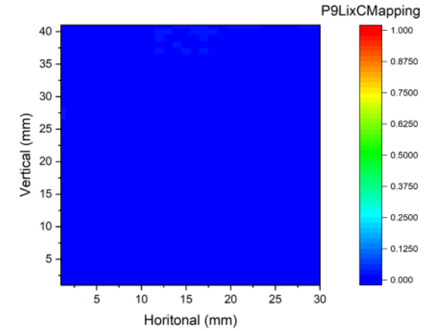

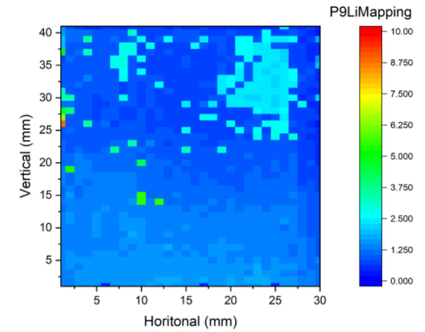

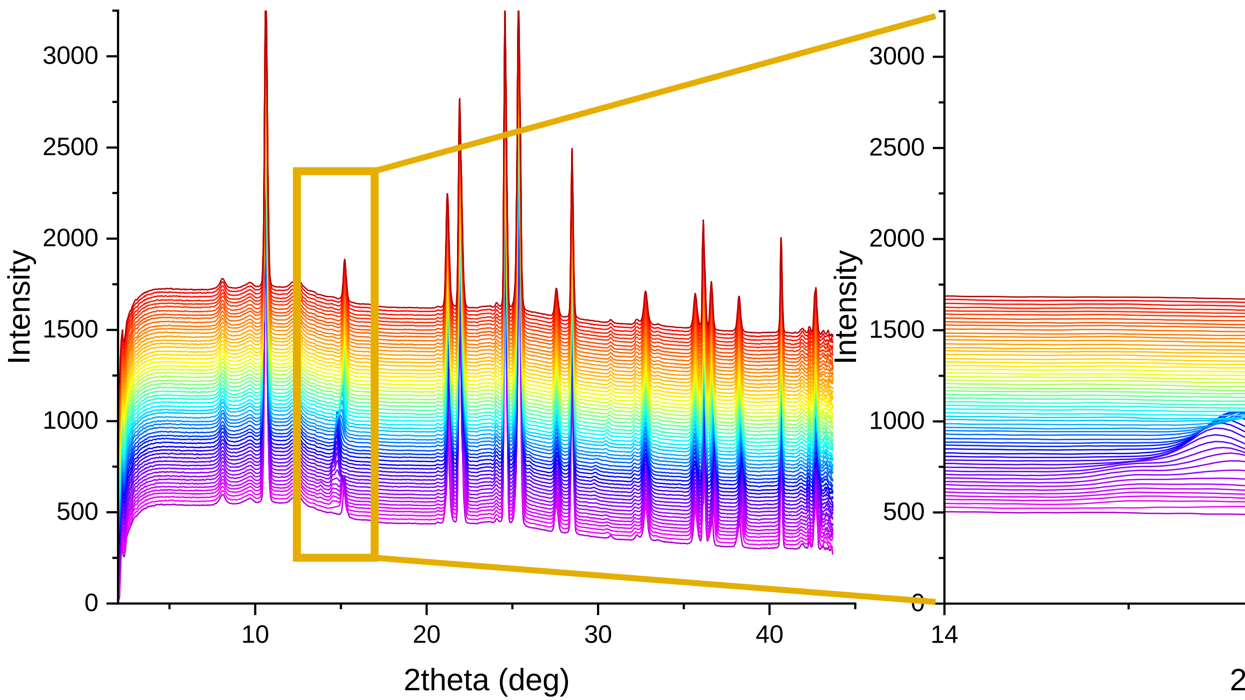

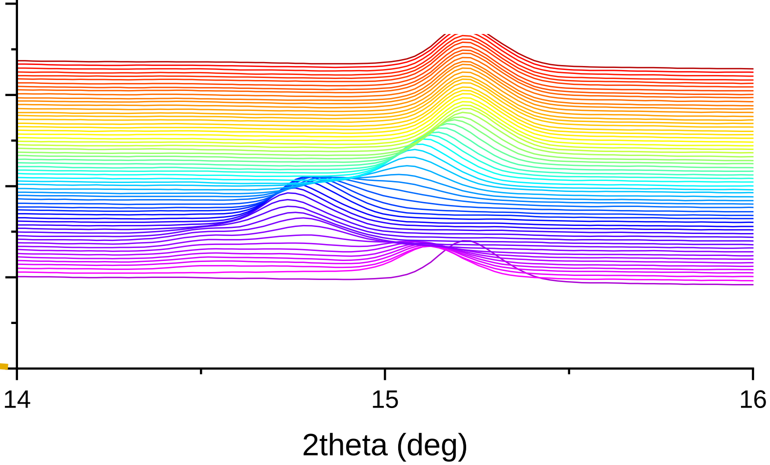

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Combined XAS & XRD

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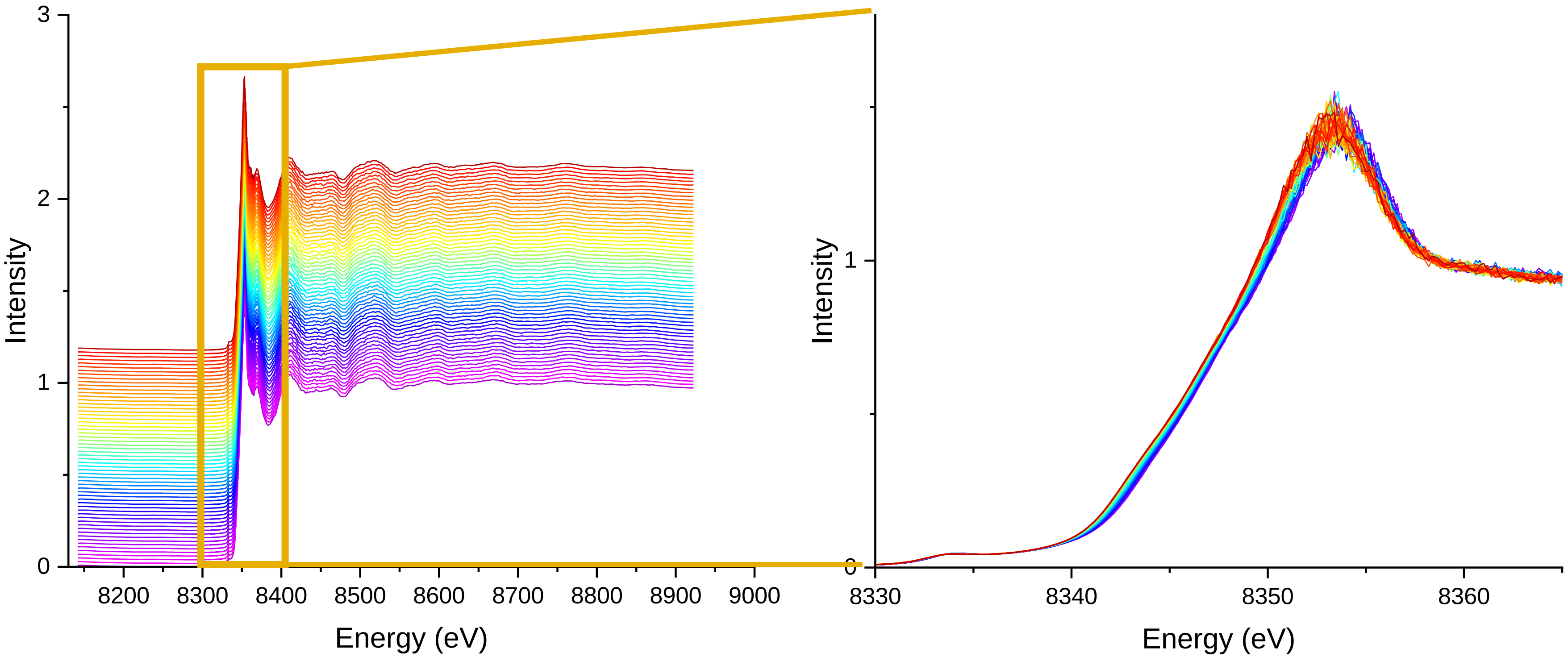
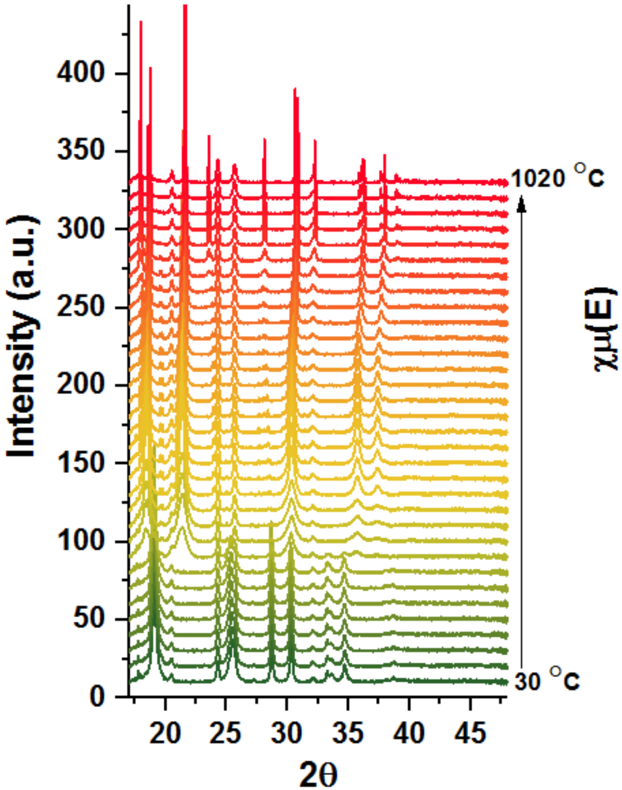


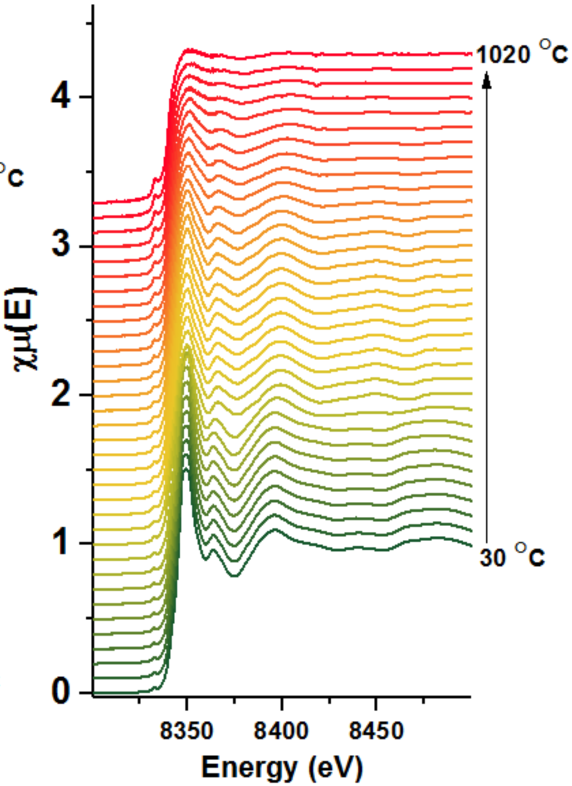
XCEL
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

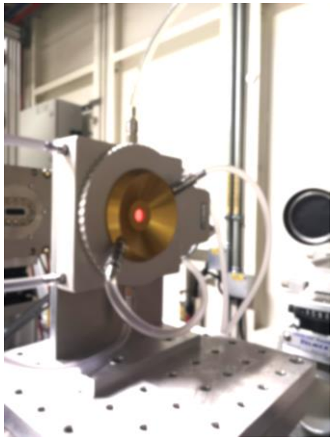
Unpublished

Combined XAS & XRD

- Case study 2: Linkam stage heating



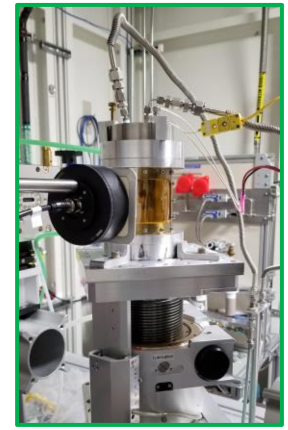


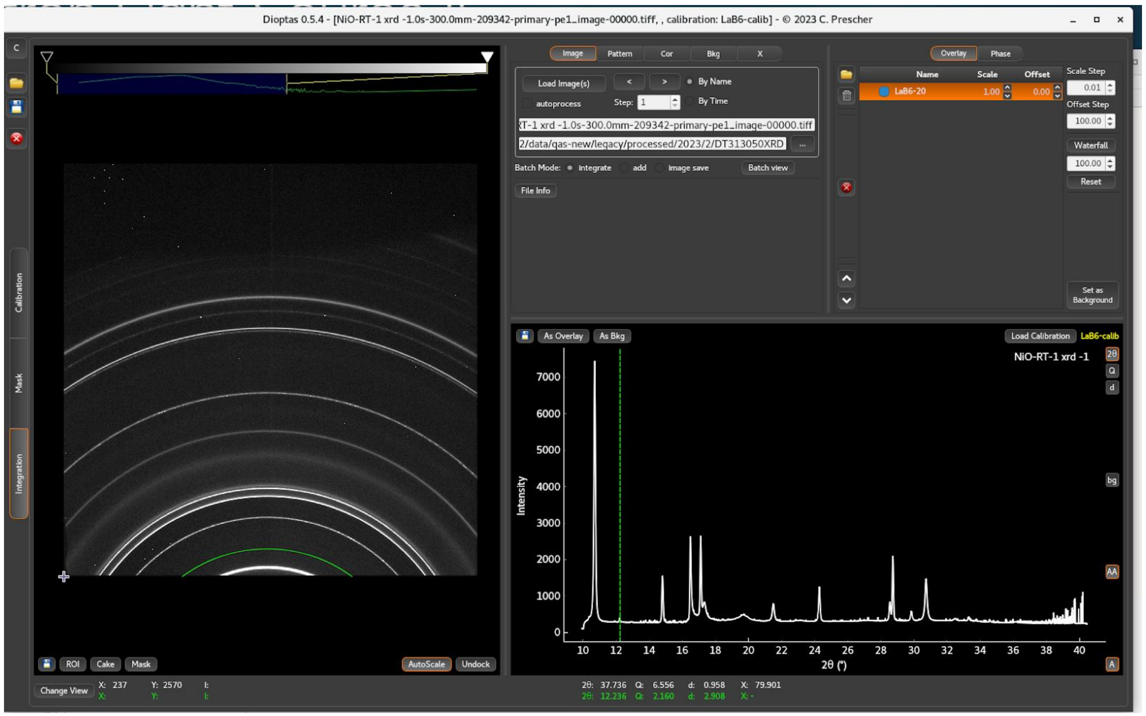


Unpublished data by S. Liu

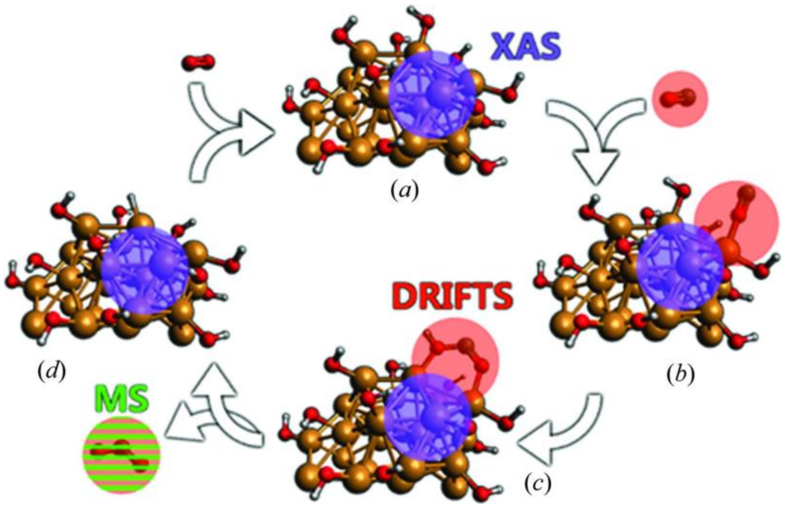
Combined XAS & XRD

- Also works with Nashner-Adler cell





Combined XAS & DRIFTS



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

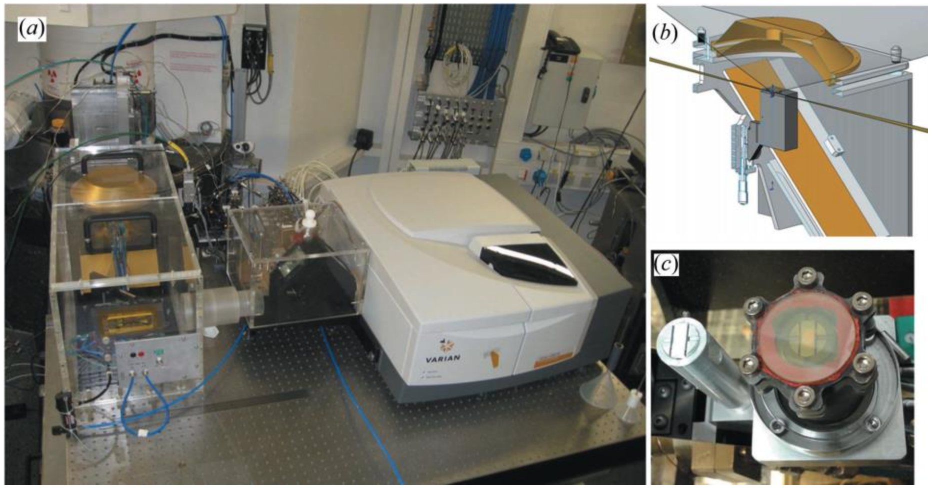
DRIFTS is sensitive to molecular vibrations and offers insight into surface species, functional groups, and adsorbed molecules on catalyst surfaces.

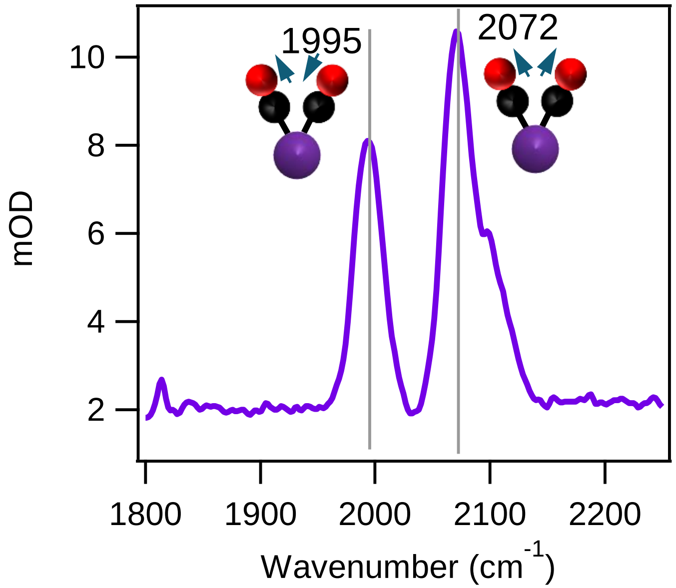
Combined XAS/DRIFTS: simultaneously monitor changes in the metal center (via XAS) and the organic species or reactants/products (via DRIFTS) during a reaction.

J.SynchrotronRad.(2019).26,801–810.

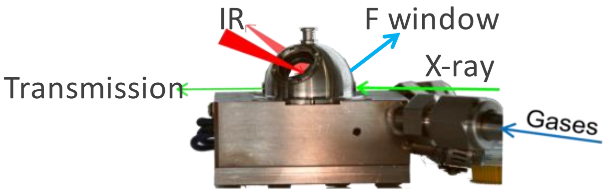
Combined XAS & DRIFTS

ESRF ID24

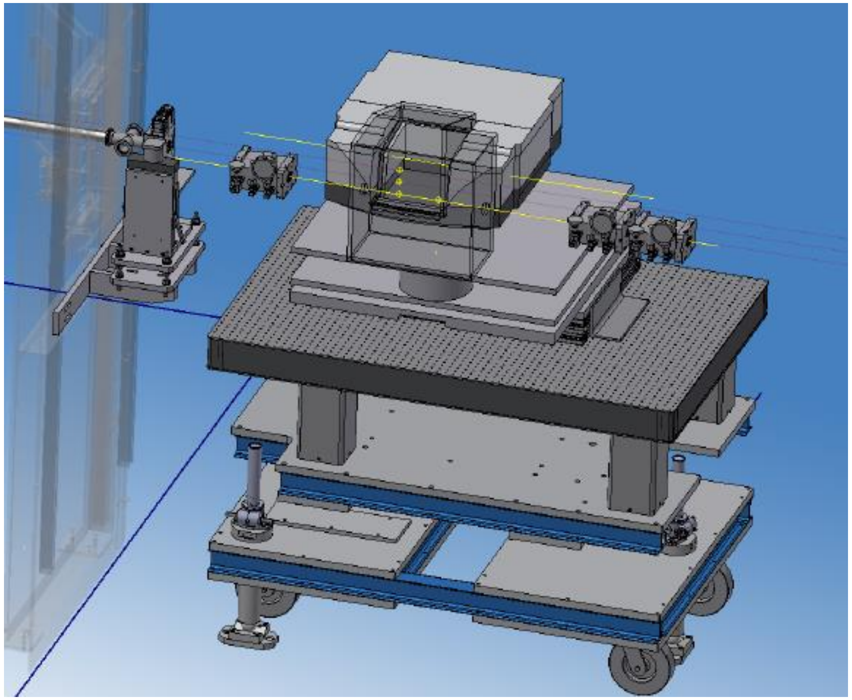




APS 9-BM

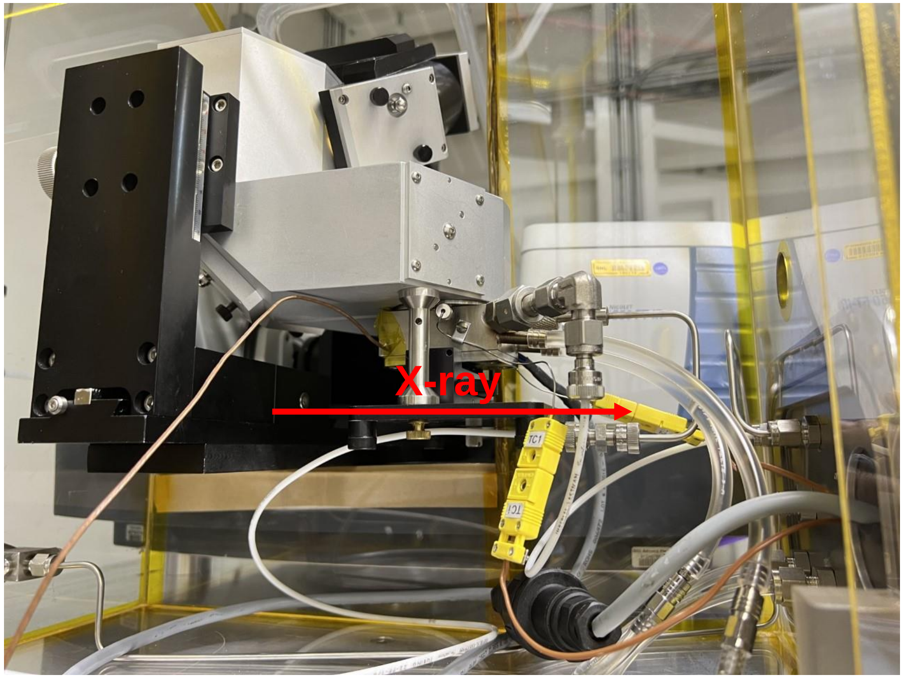


QAS hutch C endstation -- DRIFTS



- Combined XAS and DRIFTS measurements with gas flow
- Thermo-Nicolet iS-50 IR spectrometer and Harrick cell

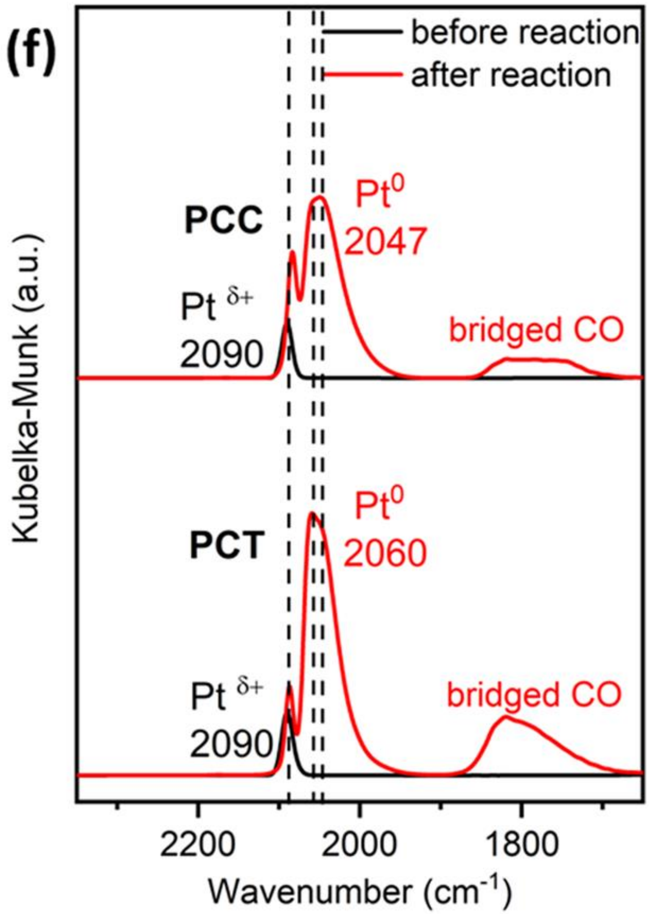
QAS hutch C endstation -- DRIFTS

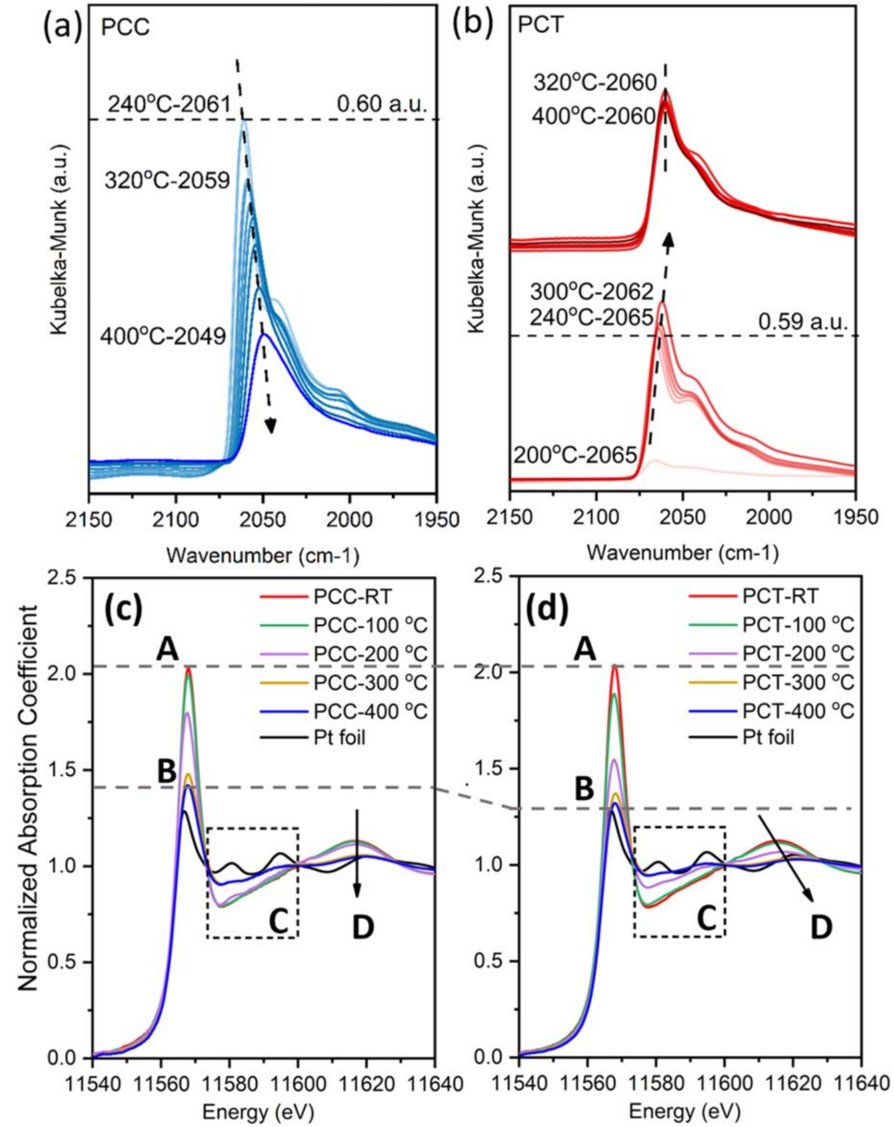


- Plexiglass box to avoid influence from the air for DRIFTS measurement.
- The water line and gas line are hooked up to Harrick cell.

Combined XAS & DRIFTS

- Science commissioning result

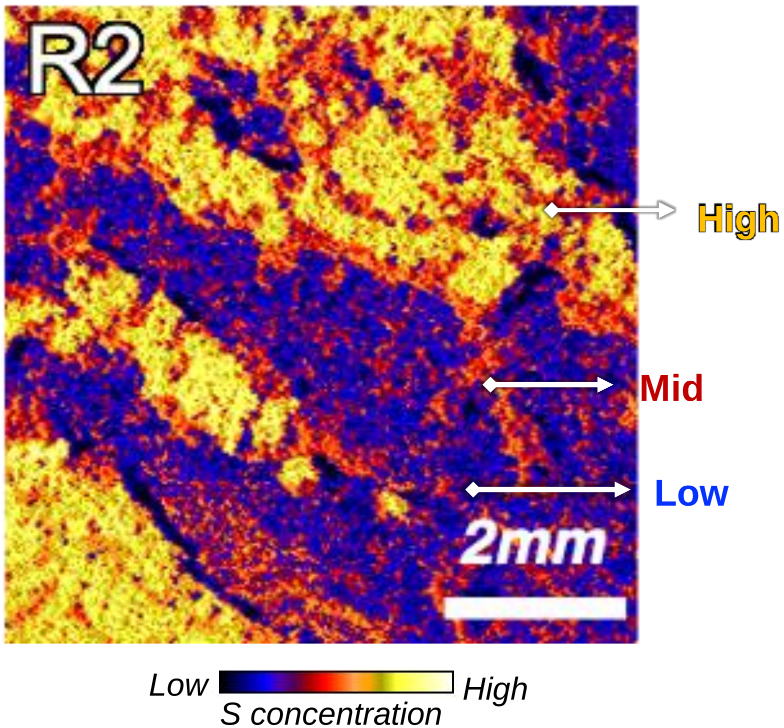


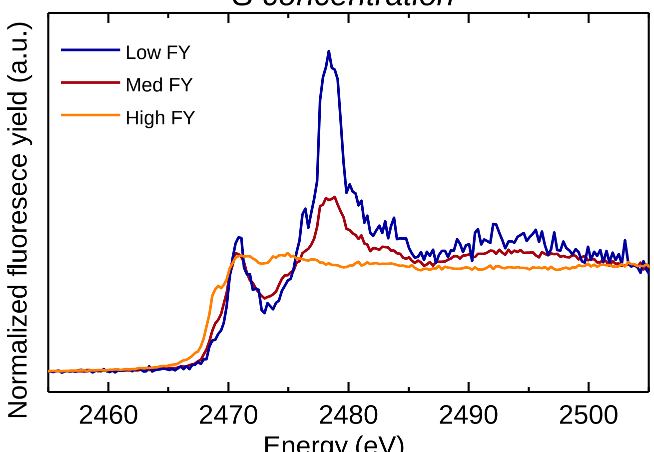


Commun Chem 6, 264 (2023)

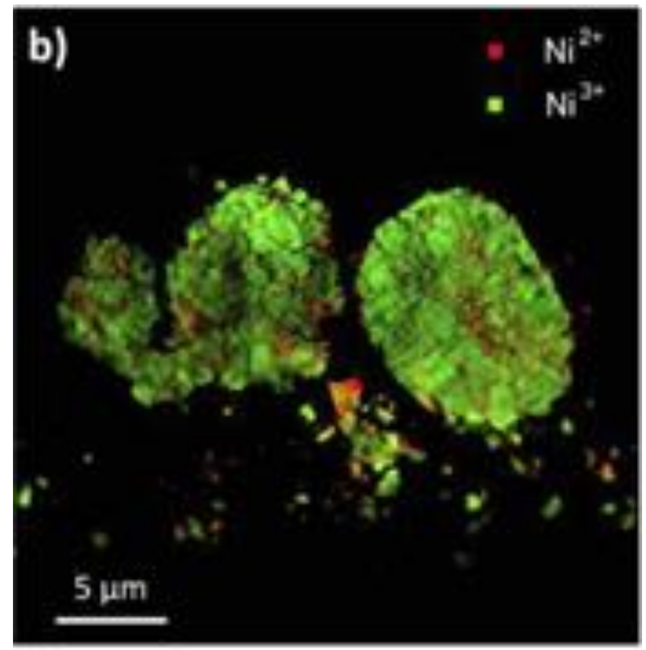
Other combined techniques with XAS

XRF mapping





XANES tomography

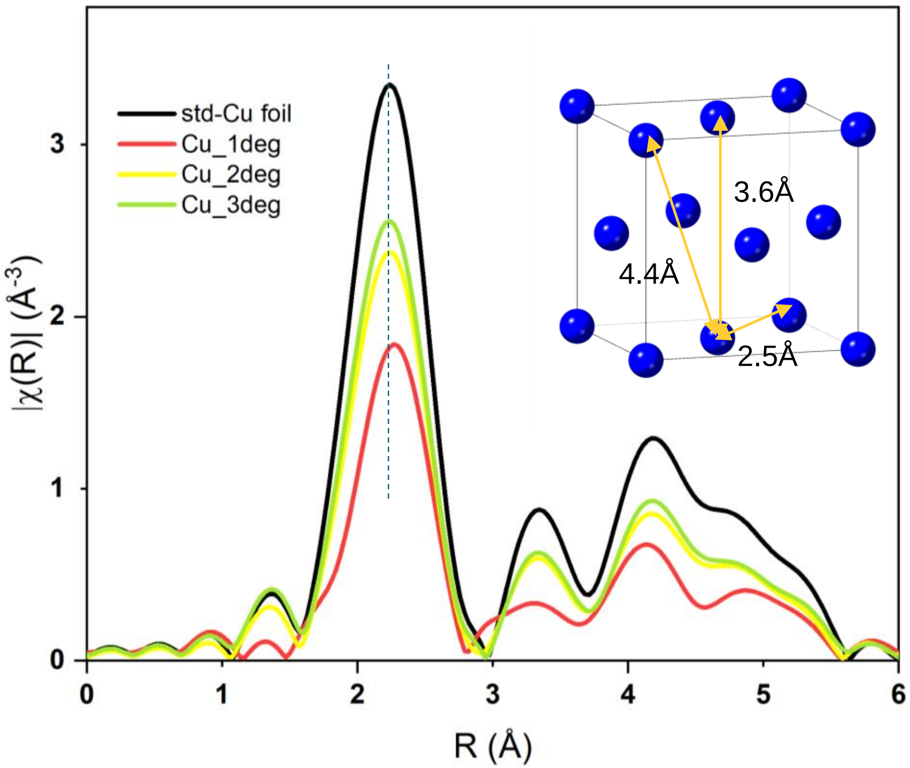


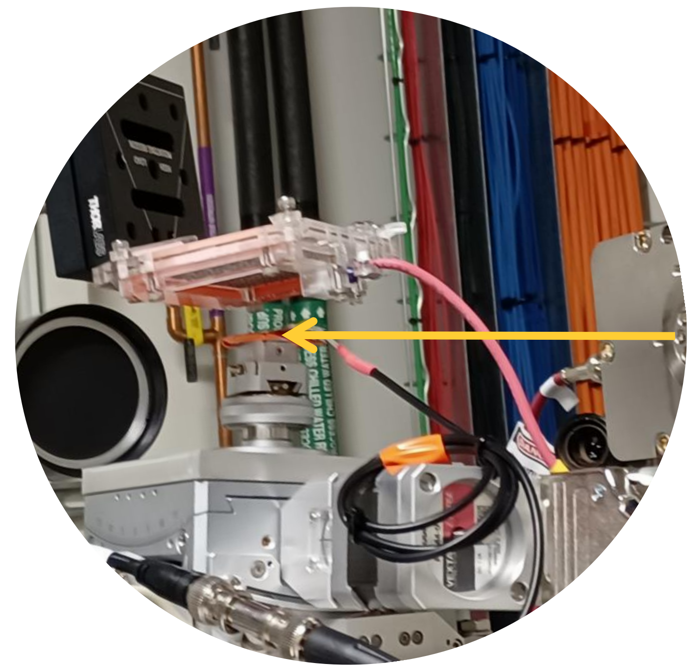
Energy Environ. Sci. 13, 3620-3632 (2020)

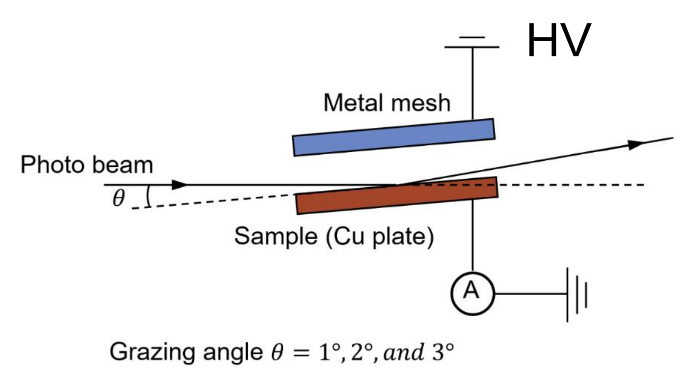
GI-TEY

In collaboration with TES LBS Dr. Yonghua Du

Surface sensitive measurement with TEY



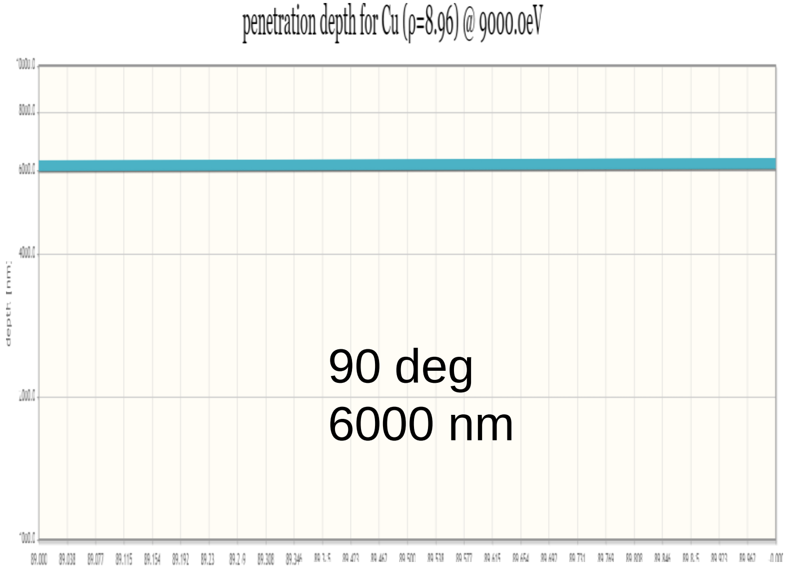


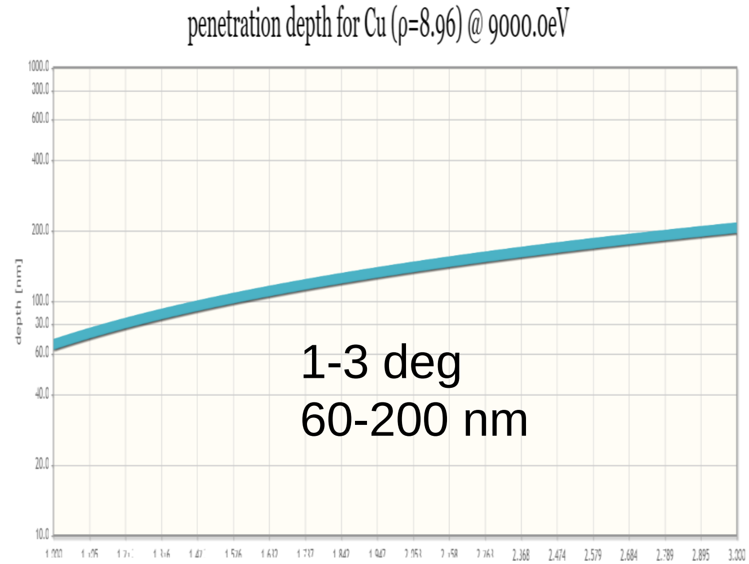


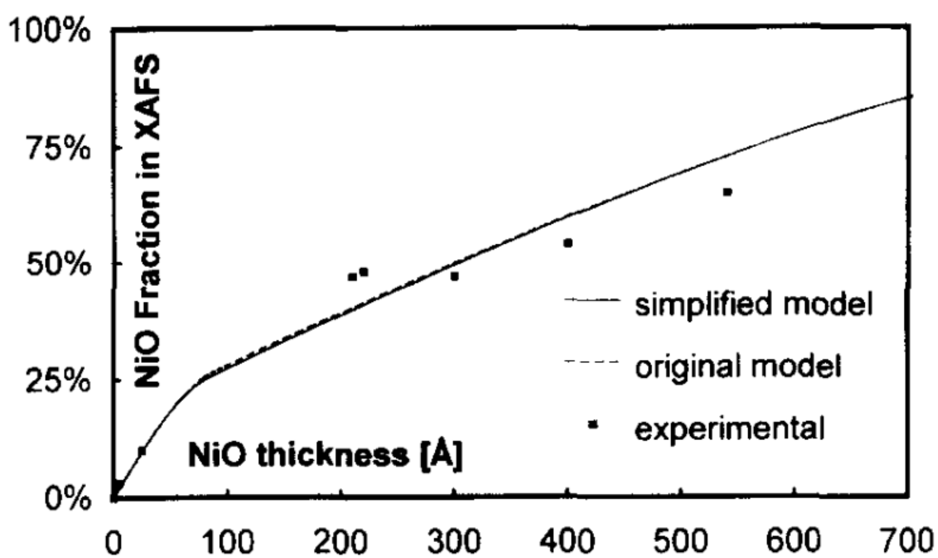
With incident angle of 1 deg

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

GI-TEY







Surface Science 324 (1995) L371-L377