Operando X-ray characterization of Li-ion batteries

Johanna Nelson Weker
she/her/hers

https://sites.slac.stanford.edu/wekergroup/
jlnelson@slac.stanford.edu

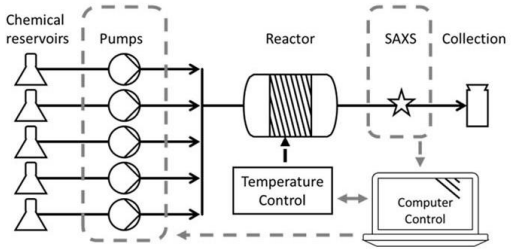
U.S. DEPARTMENT OF ENERGY
Office of Science

SLAC NATIONAL ACCELERATOR LABORATORY

In situ & operando hard X-rays characterization

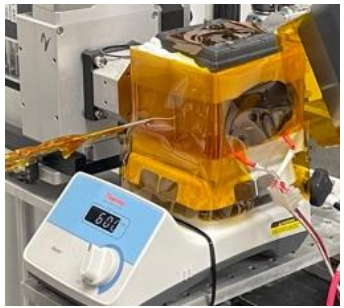
SLAC

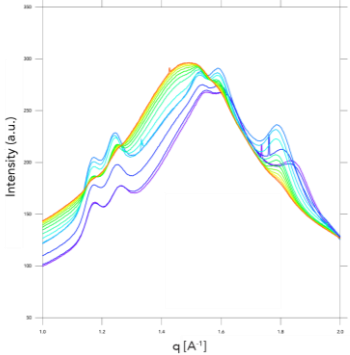
SAXS + synthesis to optimize atomically precises catalysts



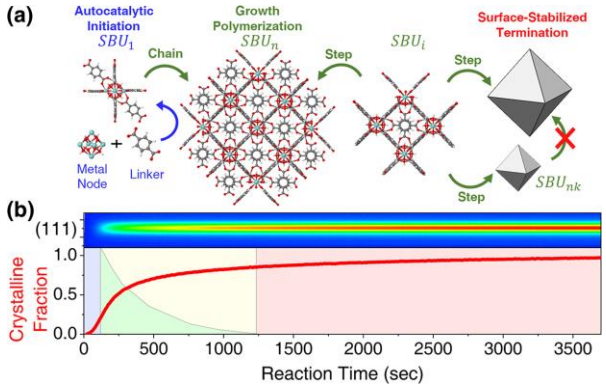
Fong et al. J. Chem. Phys. **154**, 224201 (2021)

SAXS + deconstruction of polymers (upcycling)



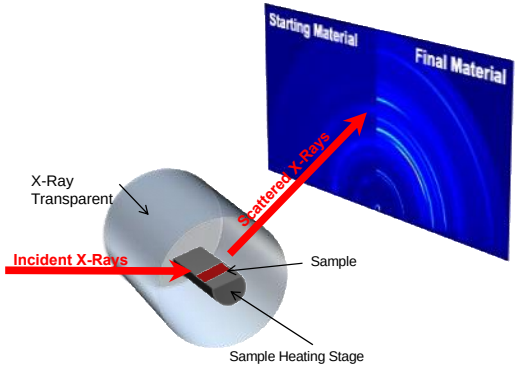


XRD + synthesis of metal–organic frameworks

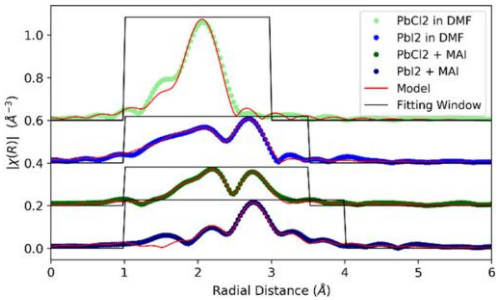


Dighe et al, JACS Au, 2, 2, 453–462 (2022)

XRD & EXAFS + annealing of perovskite PV

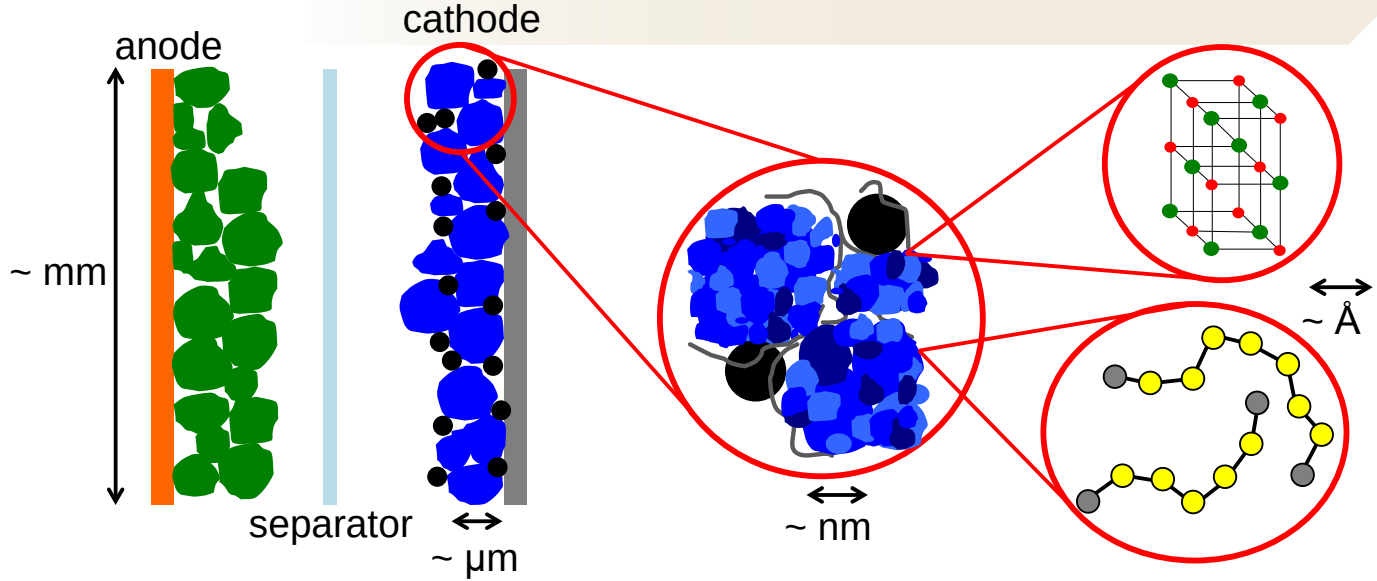


Stone et al., Nat. Commun. **9**, 3458 (2018).



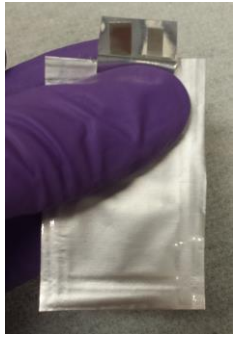
Thampy & Stone, Inorg. Chem. **49**, 18, 13364 (2020).

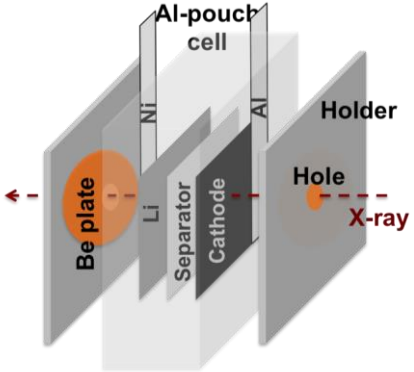
Operando X-ray characterization of Li-ion batteries

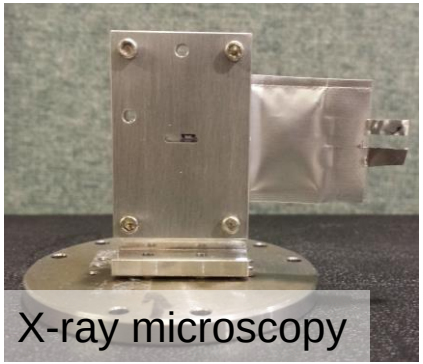


- Heterogeneous, hierarchical dynamic systems
- Need to characterize **dynamics** on all length scales
- **X-ray characterization** allows us to extract **chemical, structural, and morphological changes while cycle** energy storage systems

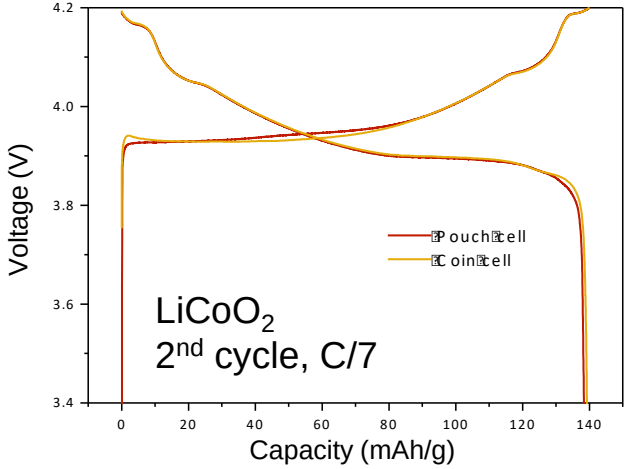
Optimized cells for long term cycling across techniques

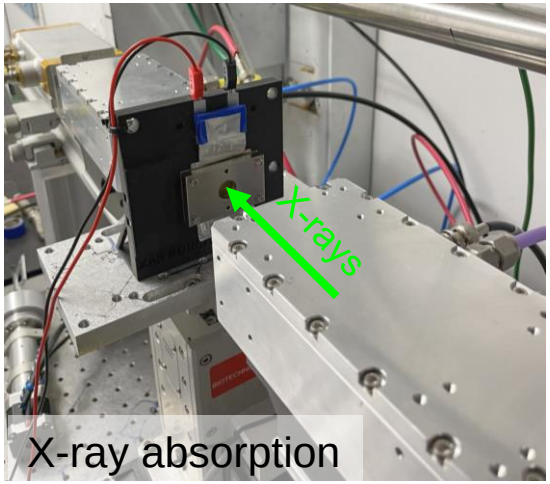




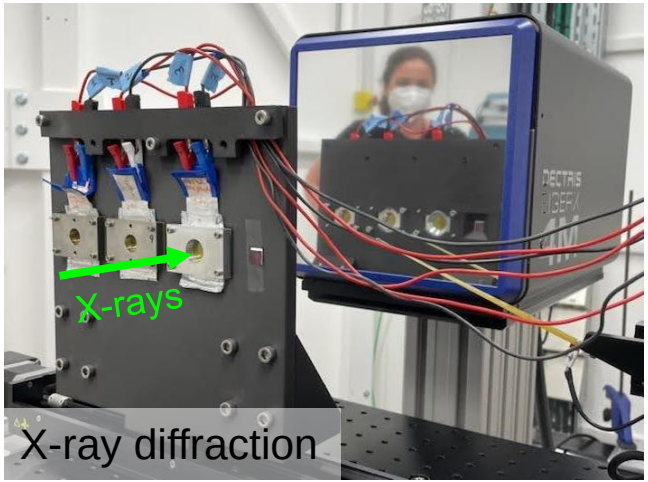

X-ray microscopy

Nominally identical cycling on and off the beamline





X-ray absorption



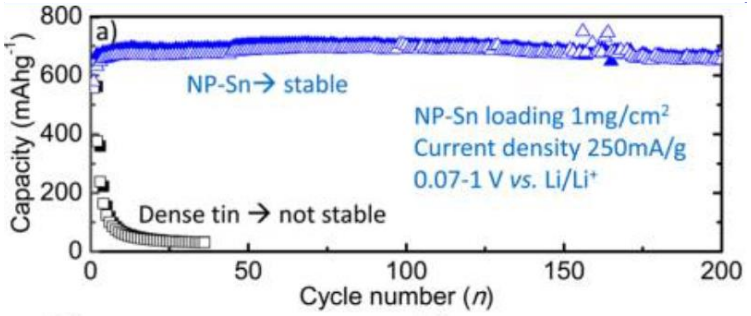
X-ray diffraction

Example 1: Stabilizing alloying anodes

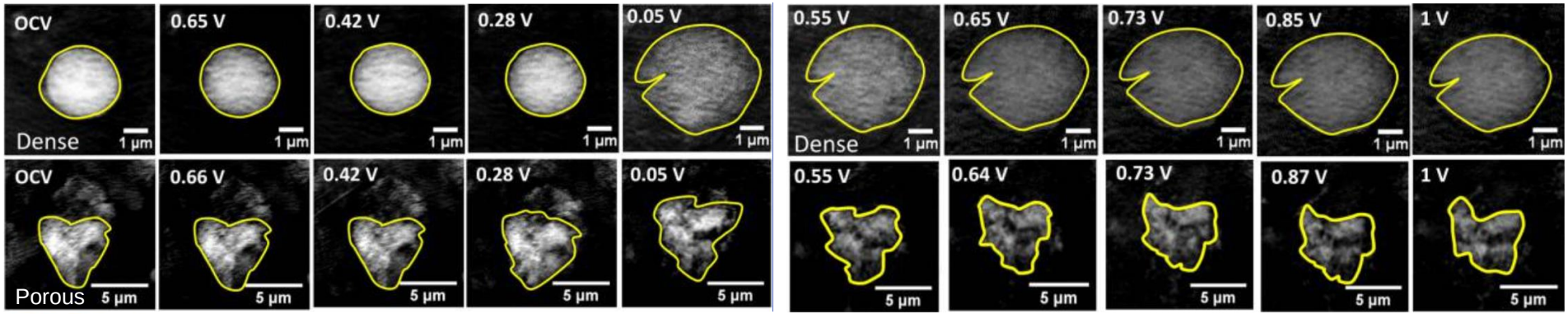
SLAC

Operando X-ray microscopy

- Our prior work showed nanoporous Sn reduced irreversible volume expansion
- But irreversible morphology changes in 1st cycle



lithiation → delithiation

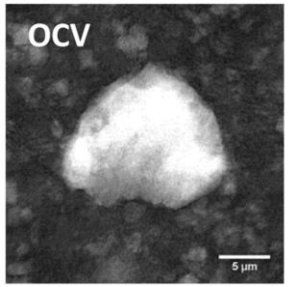


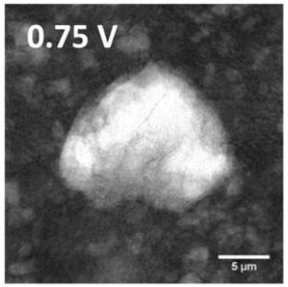
UCLA

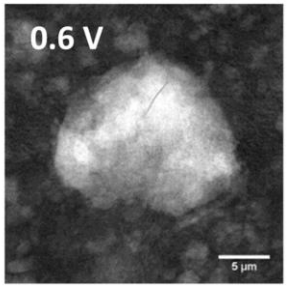
Cook, et al., *Nano Lett.* **2017**, *17*, 870-877.

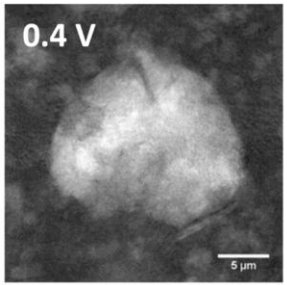
Porous SnSb alloy has stable particle morphology

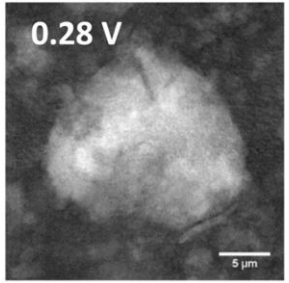
Lithiation → particle growth and crack →

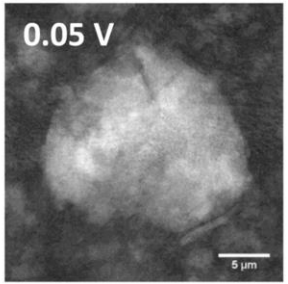




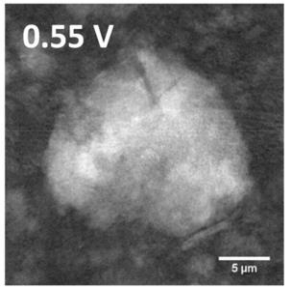


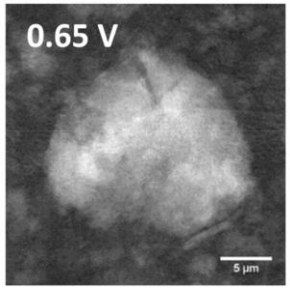


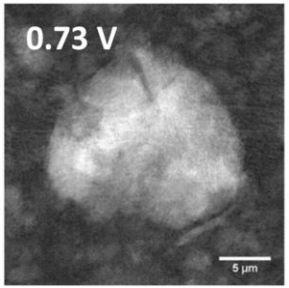


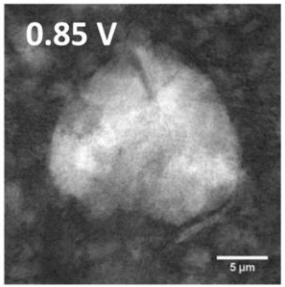


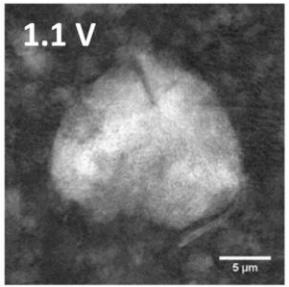
Delithiation → particle shrinks →

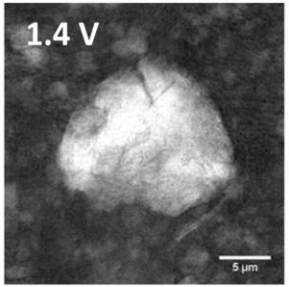








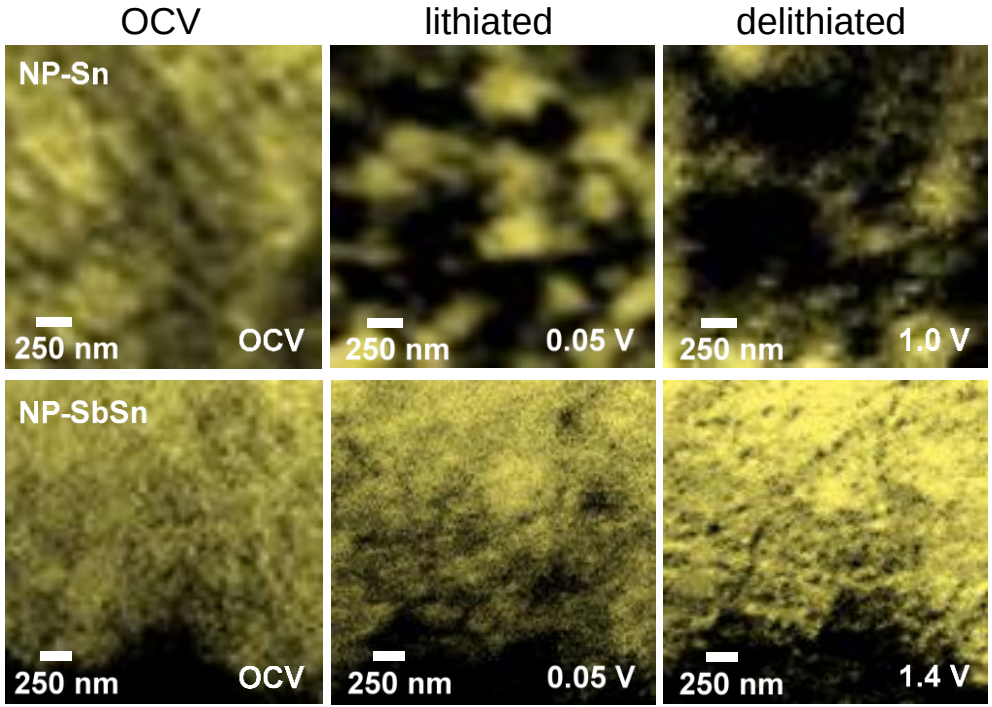




Lin, Dawson, King, Yan, Ashby, Mazzetti, Dunn, Nelson Weker, Tolbert, ACS Nano, 14, 11, 14820 (2020)

Work supported as part of the Synthetic Control Across Length-scales for Advancing Rechargeables (SCALAR), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science

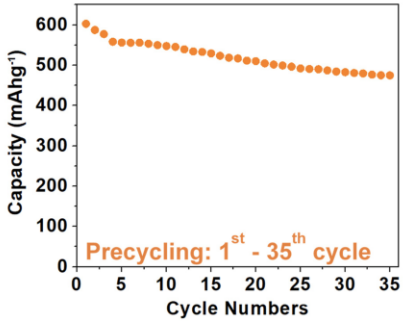
NP-SnSb alloy more stable pore structure



Lin, et al., ACS Nano, 14, 11, 14820 (2020)

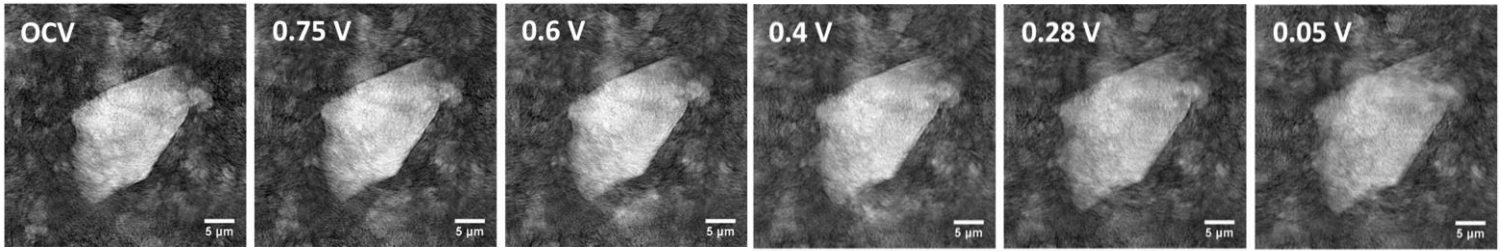
Work supported as part of the Synthetic Control Across Length-scales for Advancing Rechargeables (SCALAR), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science

NP-SnSb alloy structure still stable in 36th cycle

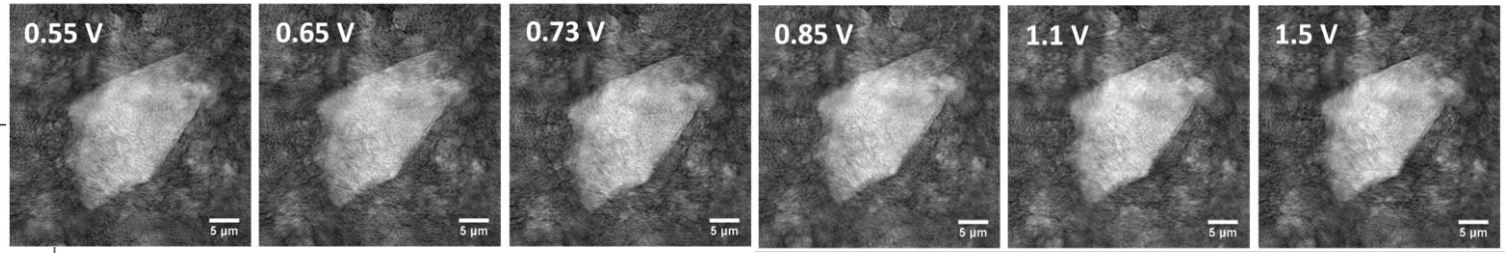


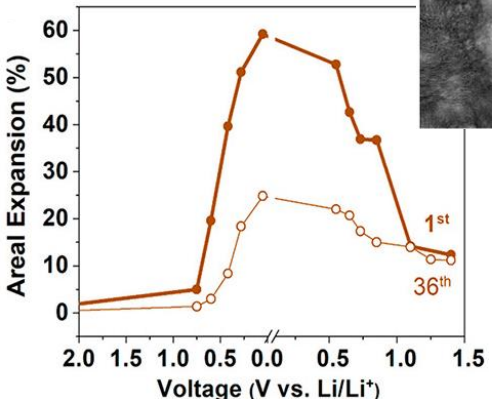
Lithiation

Lin, et al., ACS Nano, 14, 11, 14820 (2020)



Delithiation

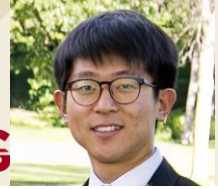




Pouch cells with external pressure enable long term cycling

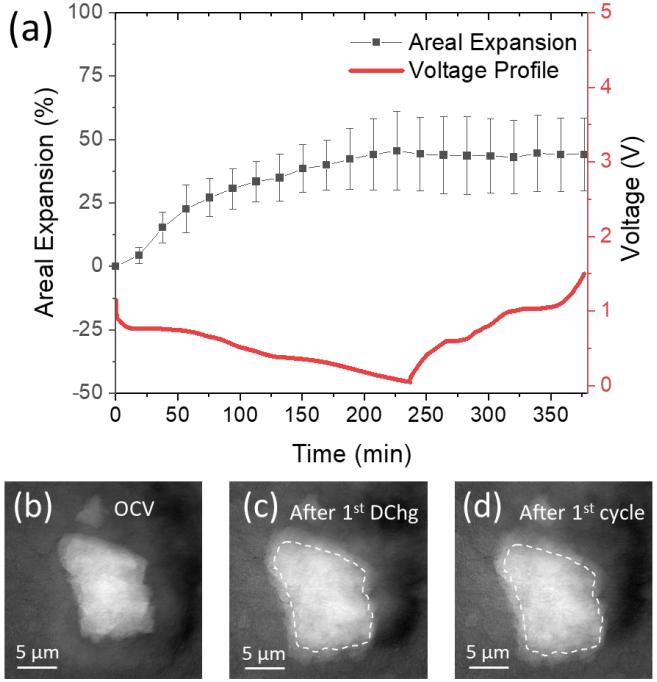
Work supported as part of the Synthetic Control Across Length-scales for Advancing Rechargeables (SCALAR), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science

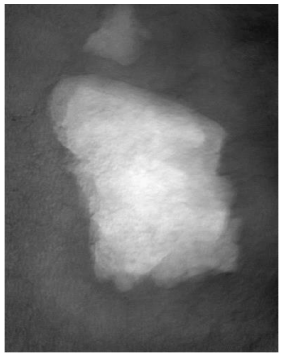
Interfacial engineering

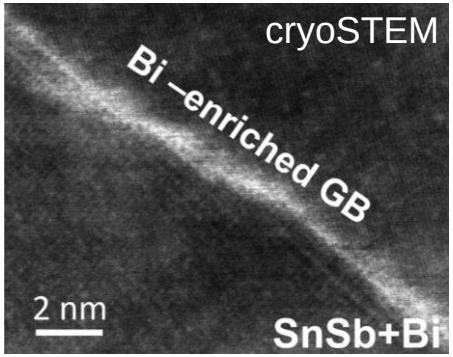


Qizhang Yan
(UCSD)

Stabilize SnSb alloy structure with Bi-enriched, liquid-like interfacial phase

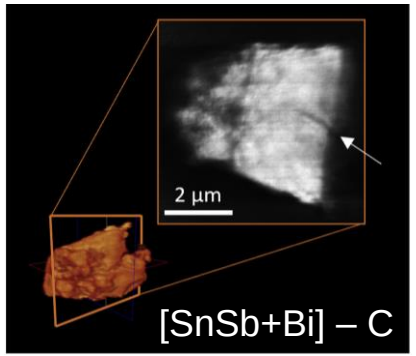


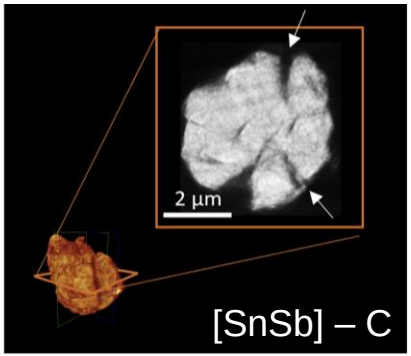




SCALAR
UC San Diego
UCLA

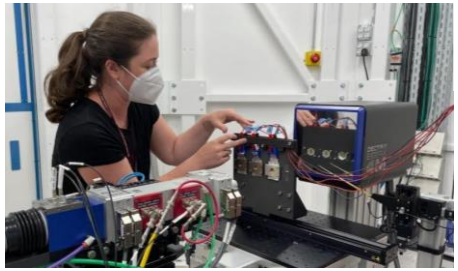
ex situ tomography after 20 cycles



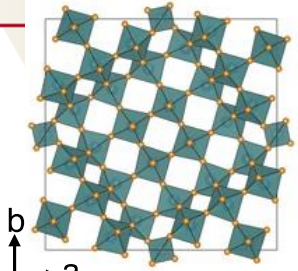


Q. Yan, S.-T. Ko, A. Dawson, D. Agyeman-Budu, G. Whang, Y. Zhao, M. Qin, B.S. Dunn, J. Nelson Weker, S.H. Tolbert, J. Luo, Cell Reports Physical Science, 3, 100694 (2022)

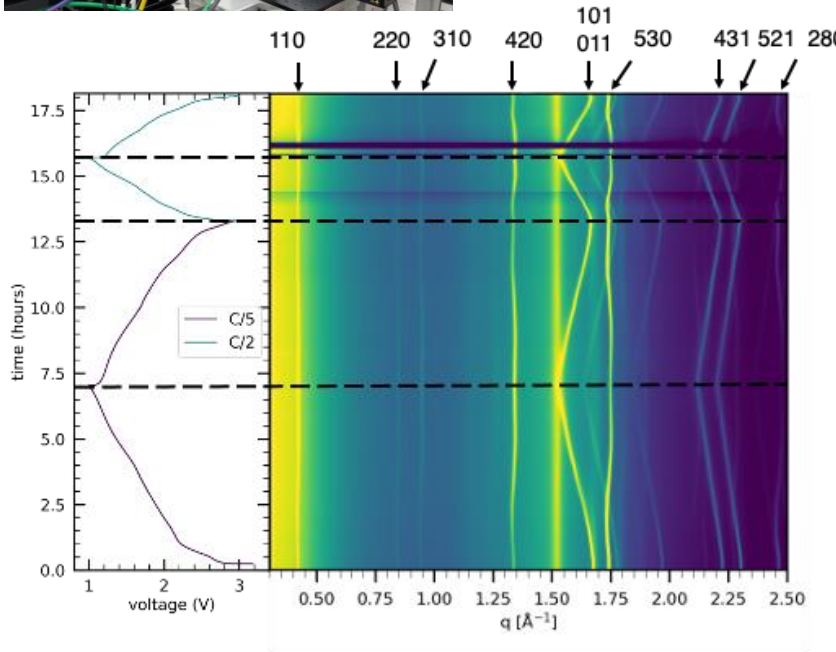
Example 2: Probing fast cycling anode materials



Dr. Molleigh Preefer
on new BL 17



➢ Wadsley–Roth/Bronze Phases†:
Class of materials that cycle ***very fast*** (up to 1 min/charge)



- *Operando* X-ray diffraction (XRD) to track structural changes
- Lattice expansion and contraction primarily along the c-axis
- Little change in the a-b plane, likely due to the edge-sharing octahedra
- XRD: collected up to 60C (1 min/charge)

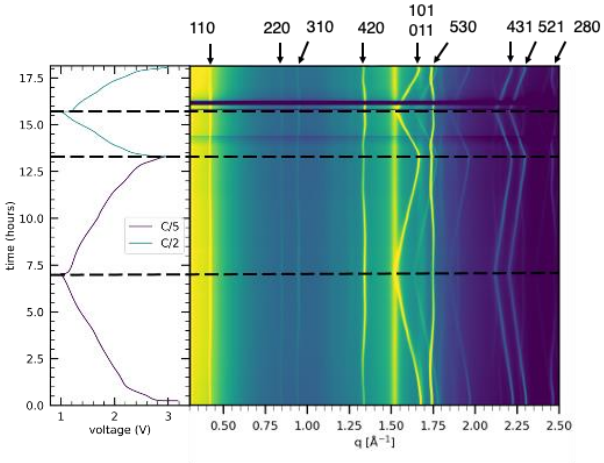
†*J. Am. Chem. Soc.* 2016, 138, 28, 8888–8899

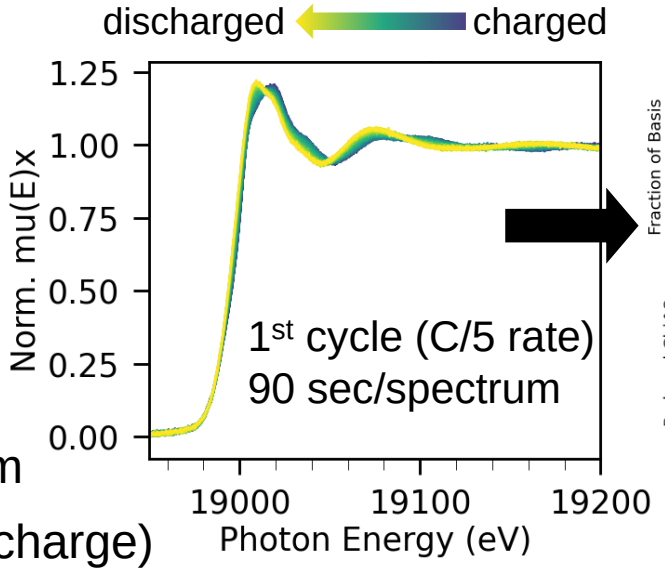
Example 2: Probing fast cycling anode materials

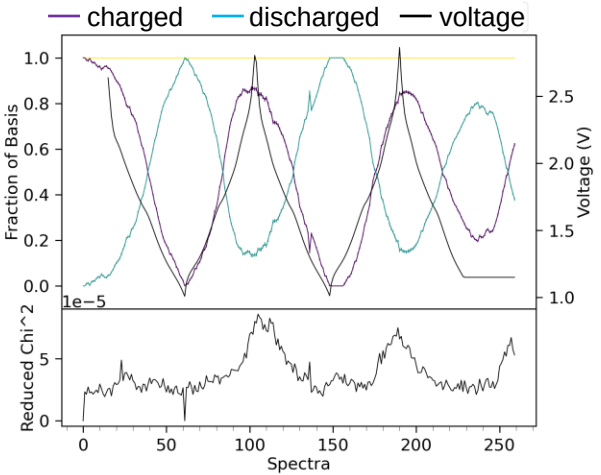

Dr. Molleigh Preefer

- Leveraging complementary techniques
- X-ray absorption spectroscopy (XAS) to track chemical changes


Emily Dunn
SULI student







- Quick-XAS: 10 sec/spectrum
- Collected up to 20C (3 min/charge)

Summary of *operando* goals at SSRL

- Robust *operando* cell design for long term cycling
- One cell design for multiple techniques (scattering, spectroscopy, microscopy)
- Fast data collection
  - faster cycles (1 min charging/discharging)
  - characterize multiple cells simultaneously