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COLLEGE OF ENGINEERING
THE UNIVERSITY OF ALABAMA IN HUNTSVILLE

Structural Changes Within Anode Materials of Na^+ Batteries

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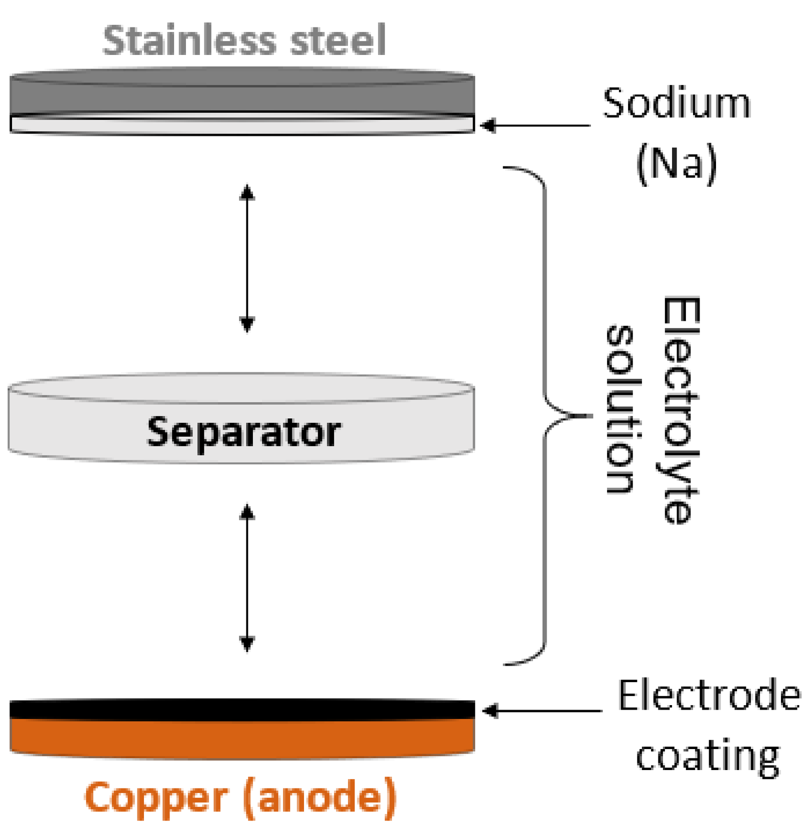
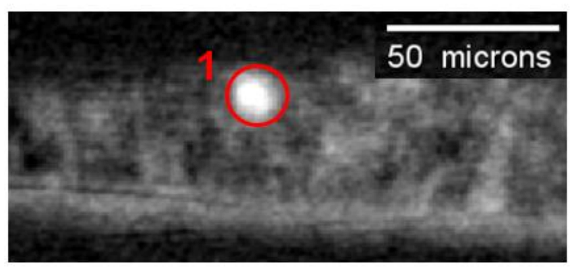
Overview: Sodium Ion Battery



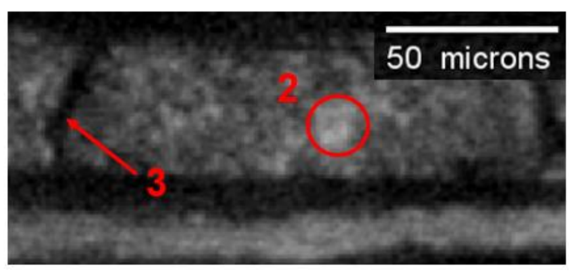
- Sodium (Na) is more earth abundant than lithium (Li), making Na-ion batteries a promising alternative to Li-ion batteries for some applications.
- Composite electrodes tested against Na and Li metal counter electrodes
  - Tin (Sn) active material
  - Carbon black conductive additive
  - Polyvinylidene fluoride (PVDF) binder
- The electrolyte solution (1 M $NaClO_4$ (70%) + FEC (30%) in PC) aids in transfer Na^+ to the electrode
- GF/F glass microfiber separator.

Cycling Effects on the Electrode's Porous Structure

Pristine X-Ray Cross section



Delithiated X-Ray Cross section



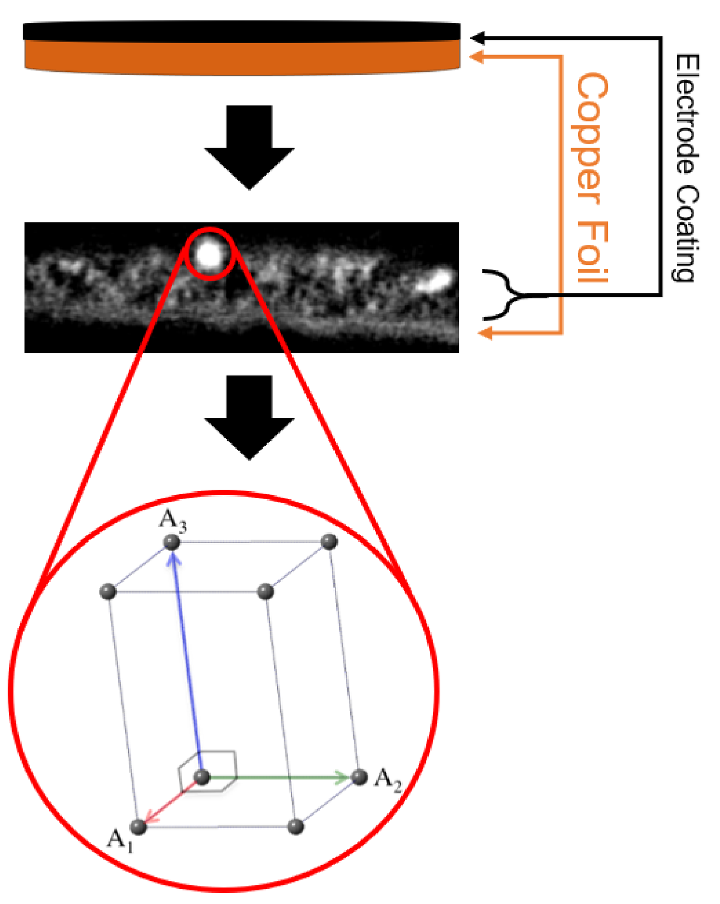
- Both X-ray cross sections have the same composition, 80% Sn, 10% CB, 10% PVDF
- Tin particle 2 is more fragmented than 1 due to lithiation and delithiation
- The cycled electrode contains cracks within its porous macrostructure (labeled by arrow 3)

Electrode Structural Hierarchy

General Cross section
(≈ 35 – 55 microns)

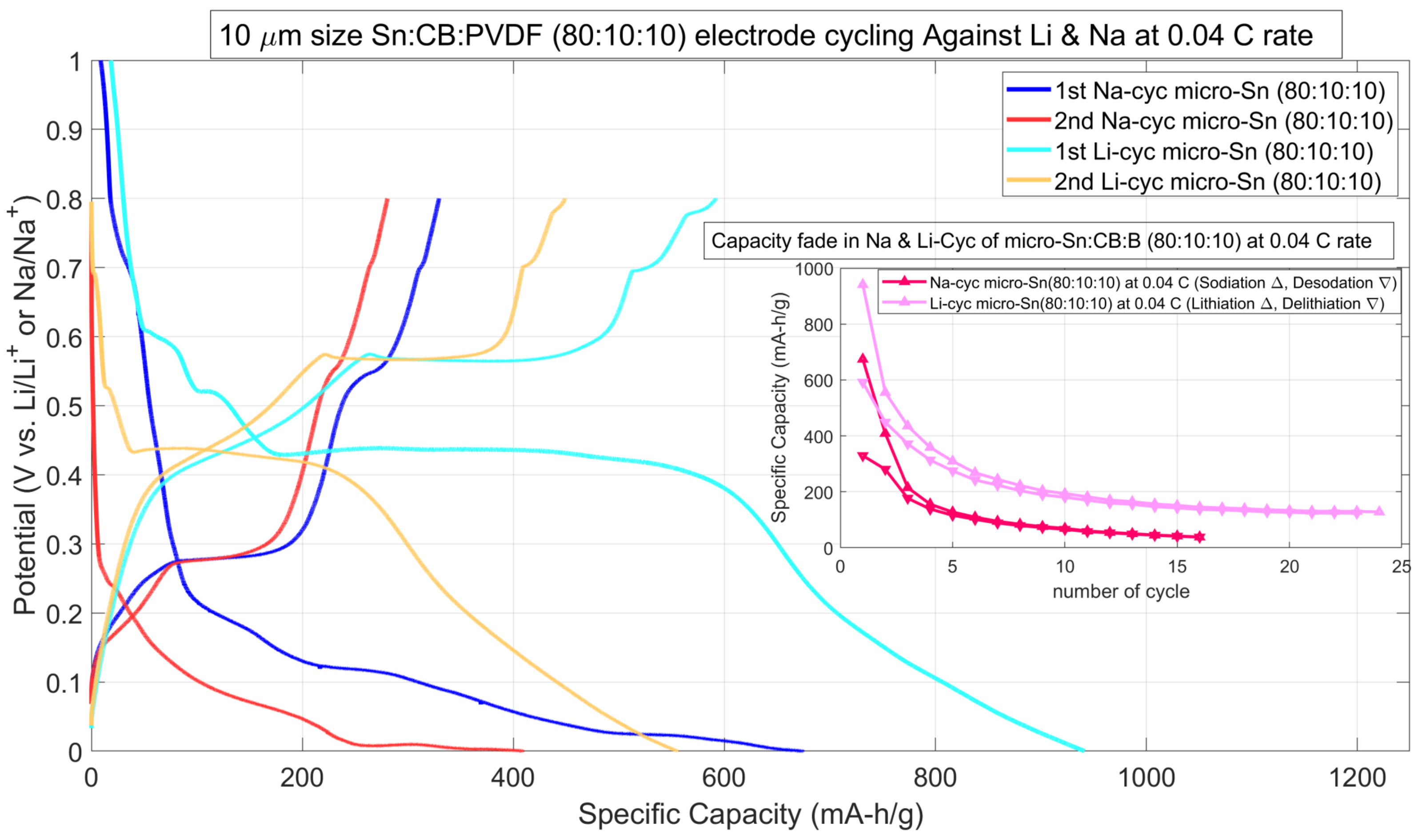
X-Ray Cross section
(≈ 35 – 55 microns)

Tin Tetragonal Unit Cell
(≈ 6 angstroms
0.0006 microns)



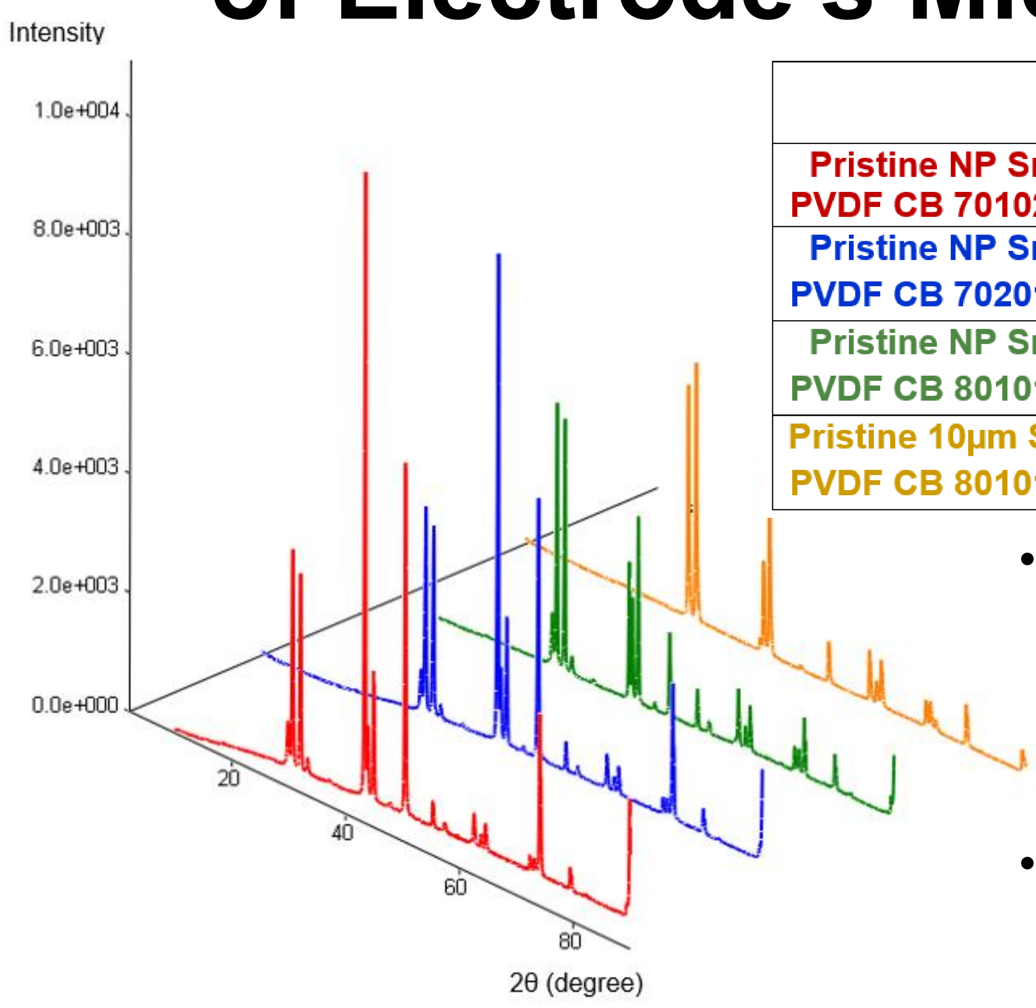
- With an applied current Na^+ integrates into the Sn crystal structure.
- Different phases of Na-Sn form at the distinct voltage plateaus.
- Na^+ ion insertion expands the tin particle ~420% at its fully sodiated phase, $Na_{15}Sn_5$.
- Galvanostatic Cycling with Potential Limitation (GCPL) cycling was performed on a BioLogic VMP potentiostat/galvanostat.
- X-ray tomographic cross sections were obtained at the Argonne National Laboratory Advanced Photon Source (Beamline 2-BM-A).
- X-ray diffraction measurements were performed on a Rigaku MiniFlex 600 X-ray diffractometer.

GCPL Cycling Curves



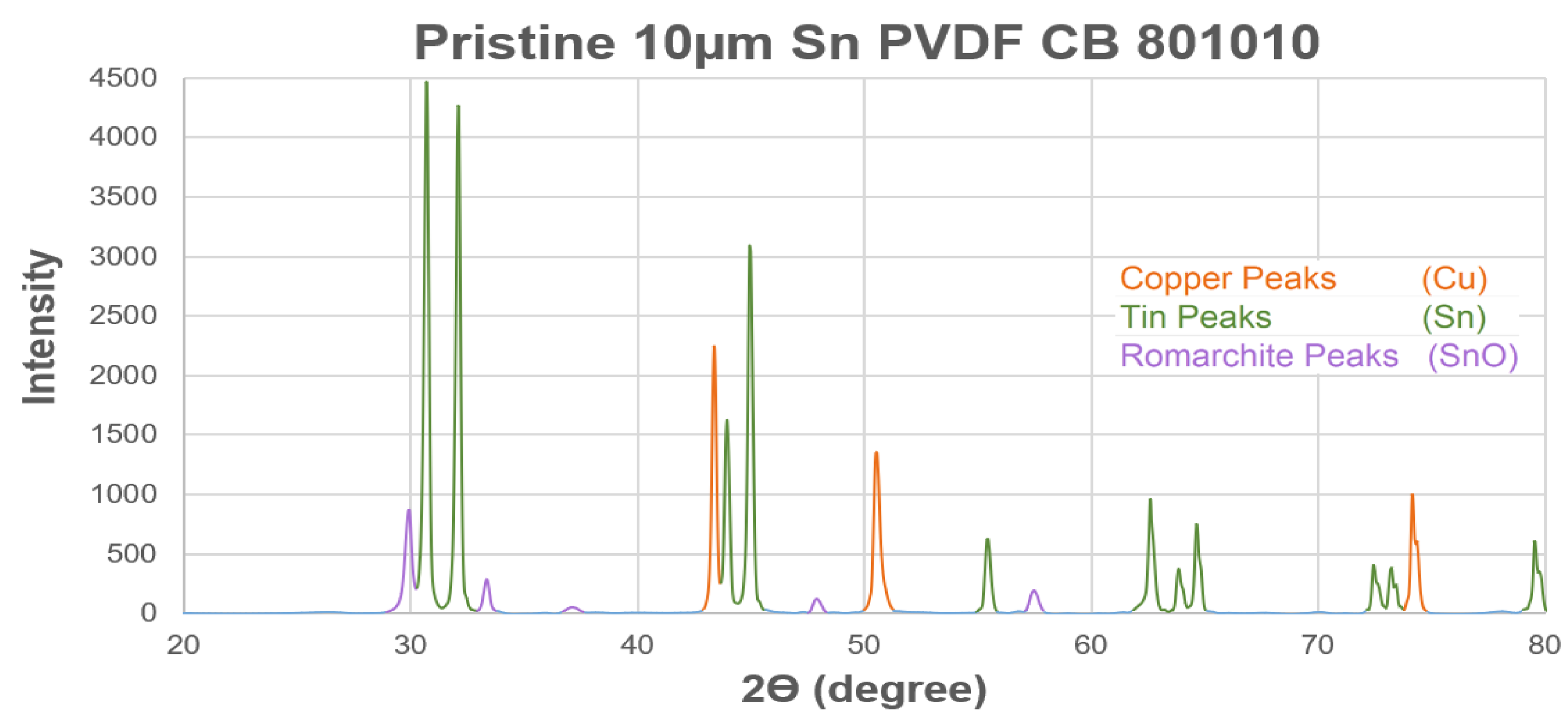
- Voltage plateaus correspond to different Na-Sn and Li-Sn phases. Some Na-Sn / Li-Sn phases formed in first cycle may not appear in later cycles due to irreversible mechanism of solid electrolyte interphase (SEI) formation.

X-Ray Diffraction Measurements of Electrode's Microstructure



	Copper (Cu)	Tin (Sn)	Romarchite (SnO)
Pristine NP Sn PVDF CB 701020	Cubic	Tetragonal	Tetragonal
Pristine NP Sn PVDF CB 702010	Cubic	Tetragonal	Tetragonal
Pristine NP Sn PVDF CB 801010	Cubic	Tetragonal	Tetragonal
Pristine 10μm Sn PVDF CB 801010	Cubic	Tetragonal	Tetragonal

- The intensity of the diffracted X-ray beam at a given angle depends upon the material's crystal structure.
- XRD data shows Sn active material, Cu foil substrate, and SnO impurities.



Conclusions/ Future Research

The above X-ray diffraction and tomographic data serves as a basis for pristine PVDF electrodes at various compositions. In the future we will apply these methods to explore the microstructural effects of cycling at various degrees of sodiation.

Acknowledgements

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References

1. Mehl, M J, et al. "Library of Crystallographic Prototypes." *Aflow*, Library of Crystallographic Prototypes: Part 1, Comp. Mat. Sci. 136, 2017, aflowlib.org/CrystalDatabase/tetragonal_lattice.html.