

Evaluating Degradation in Sodium-Ion Battery Cathodes Using Operando XRD

Marissa Wood¹, Tom Braun¹, Marcos Lucero², Fredrick Omenya², Nikhil Rampal¹, Liwen (Sabrina) Wan¹, David Reed², Xiaolin Li², Jonathan Lee¹



¹ Lawrence Livermore National Laboratory; ² Pacific Northwest National Laboratory



Introduction

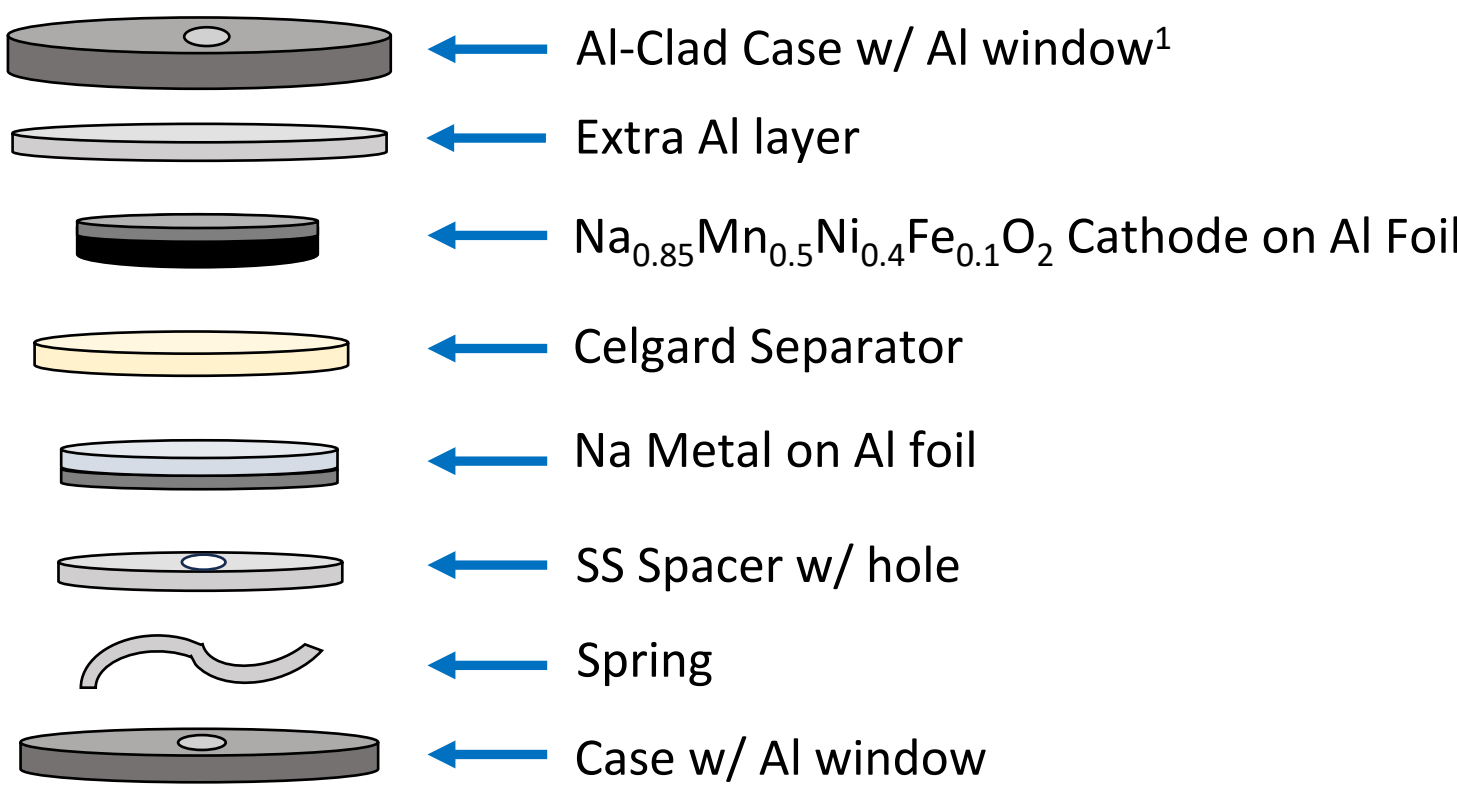
Project Profile

Topic: Sodium Batteries
Vertical: Materials and Systems Innovation
Strategic Pathway: Grow
Project Start and Finish: FY25-FY26

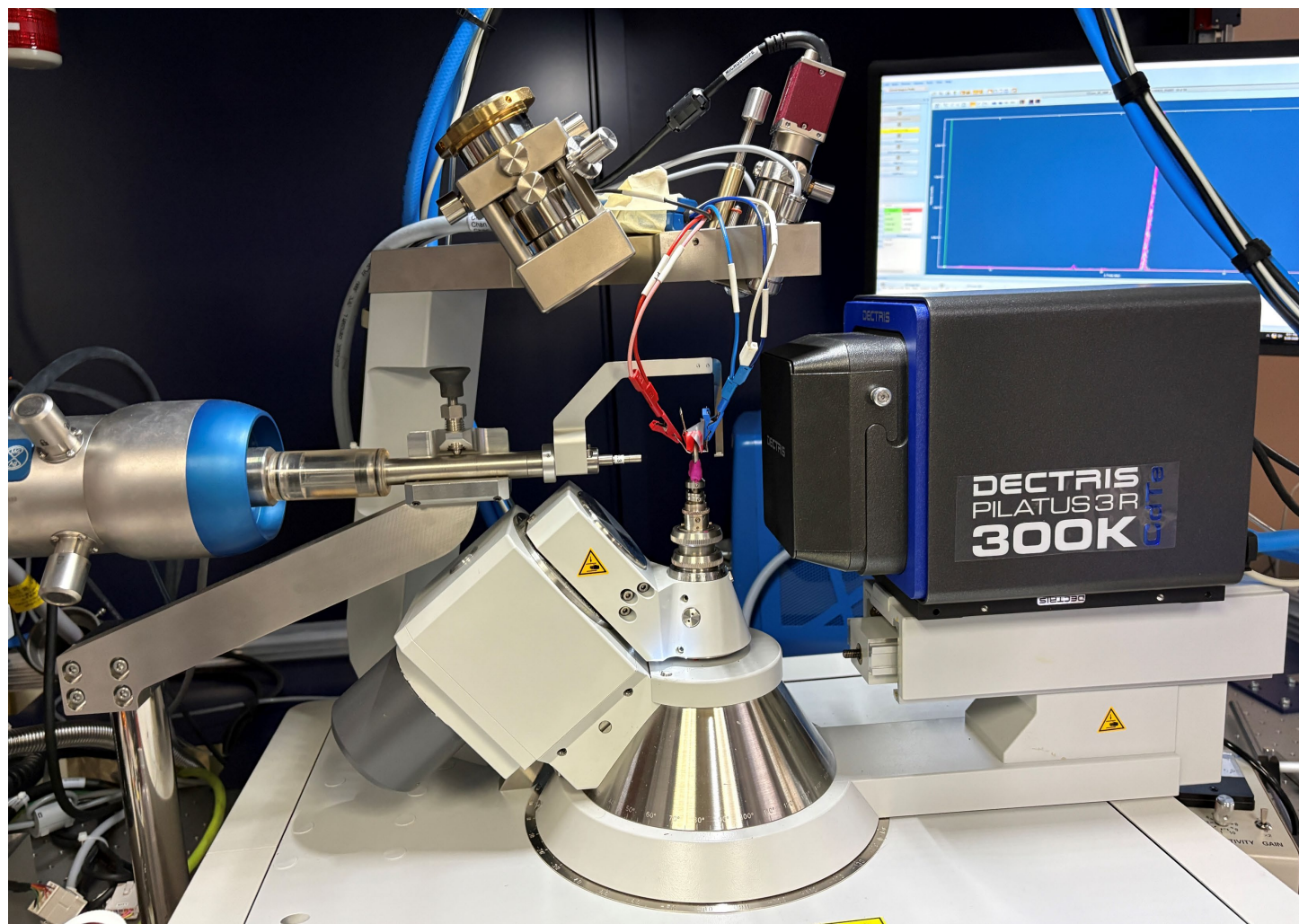
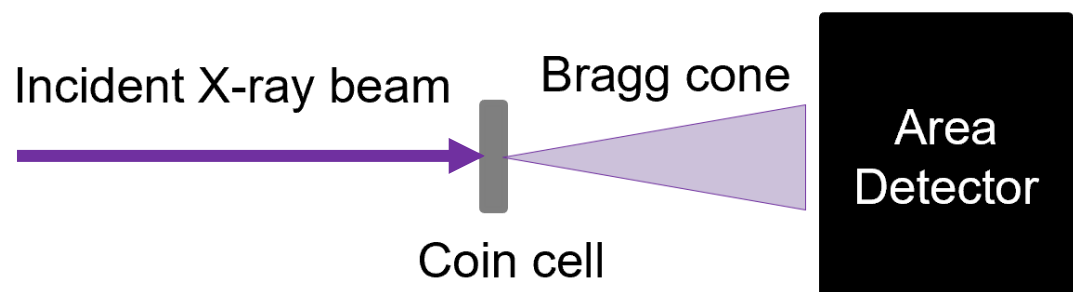
- Na-ion batteries offer a promising energy storage solution made with inexpensive, earth-abundant materials.
- Na-ion battery cathodes have historically suffered from structural instability and poor long-term cyclability.
- This work uses operando XRD to investigate degradation mechanisms in $\text{Na}_{0.85}\text{Mn}_{0.5}\text{Ni}_{0.4}\text{Fe}_{0.1}\text{O}_2$ cathodes during cycling by monitoring structural changes over time.

Experimental Setup

Coin Cell Configuration for Operando XRD

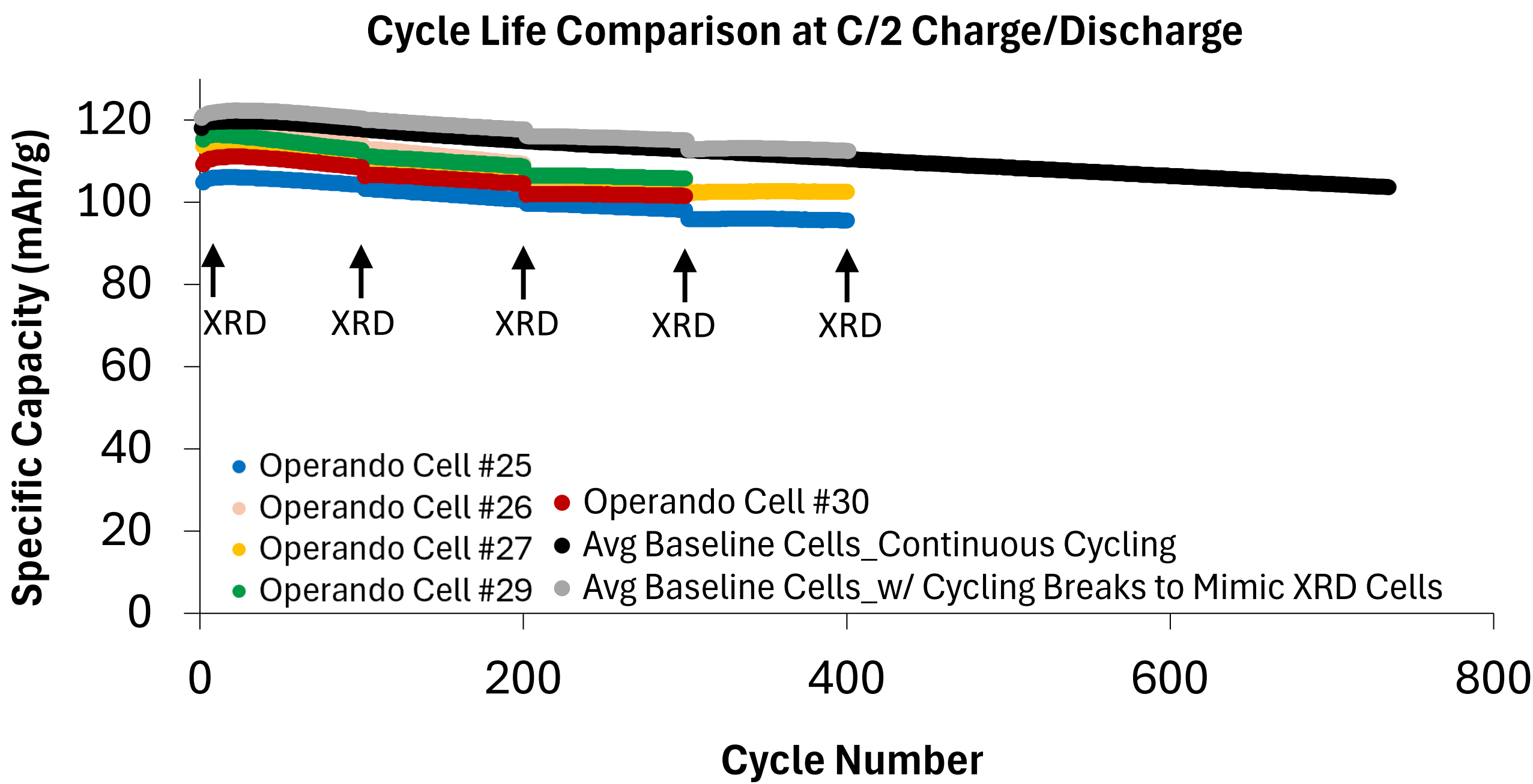


XRD Setup at LLNL



- Made 5 coin cells with XRD window & 4 baseline coin cells without window for comparison

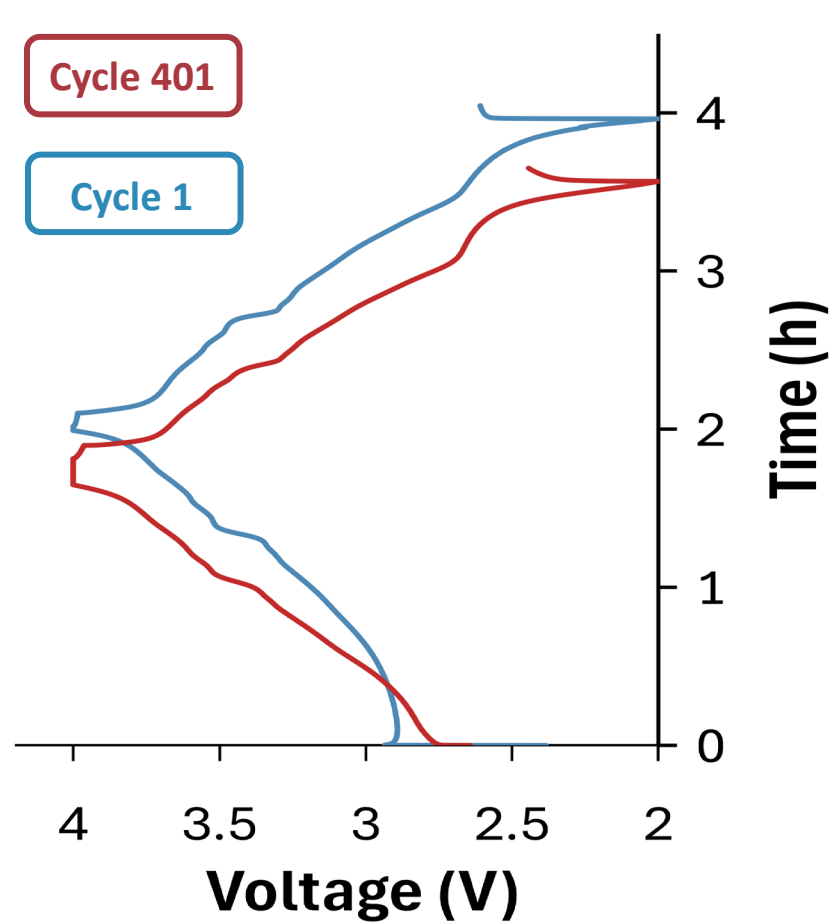
Electrochemical Cycling Results



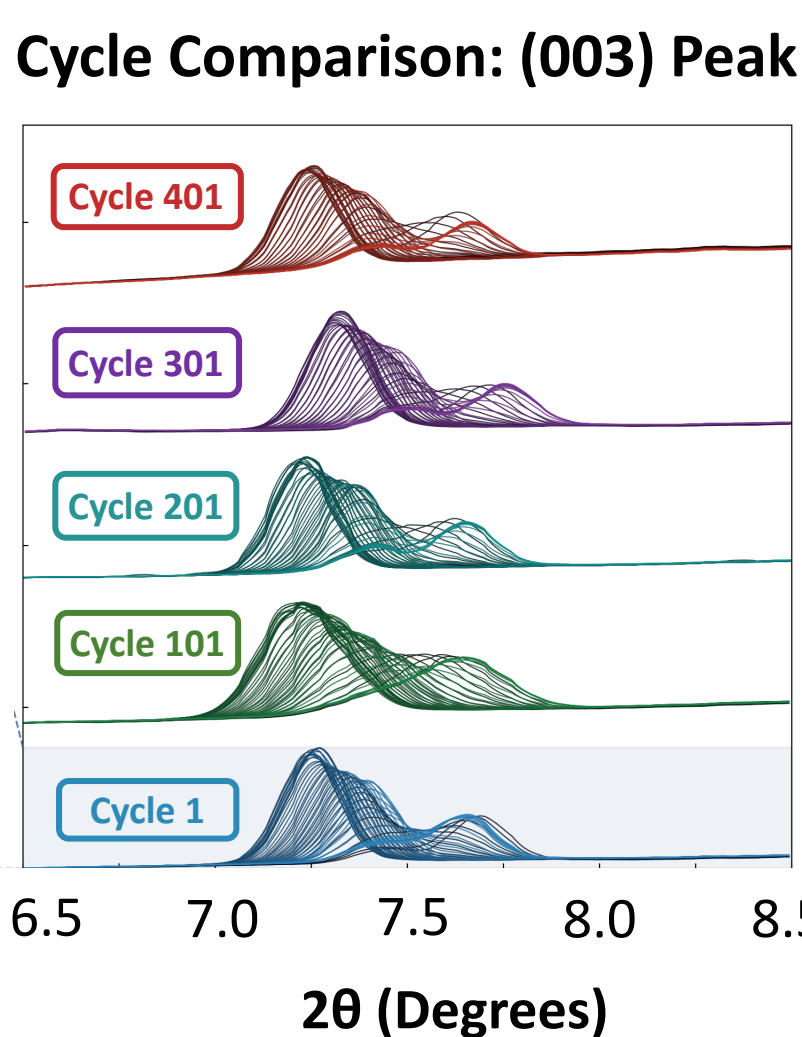
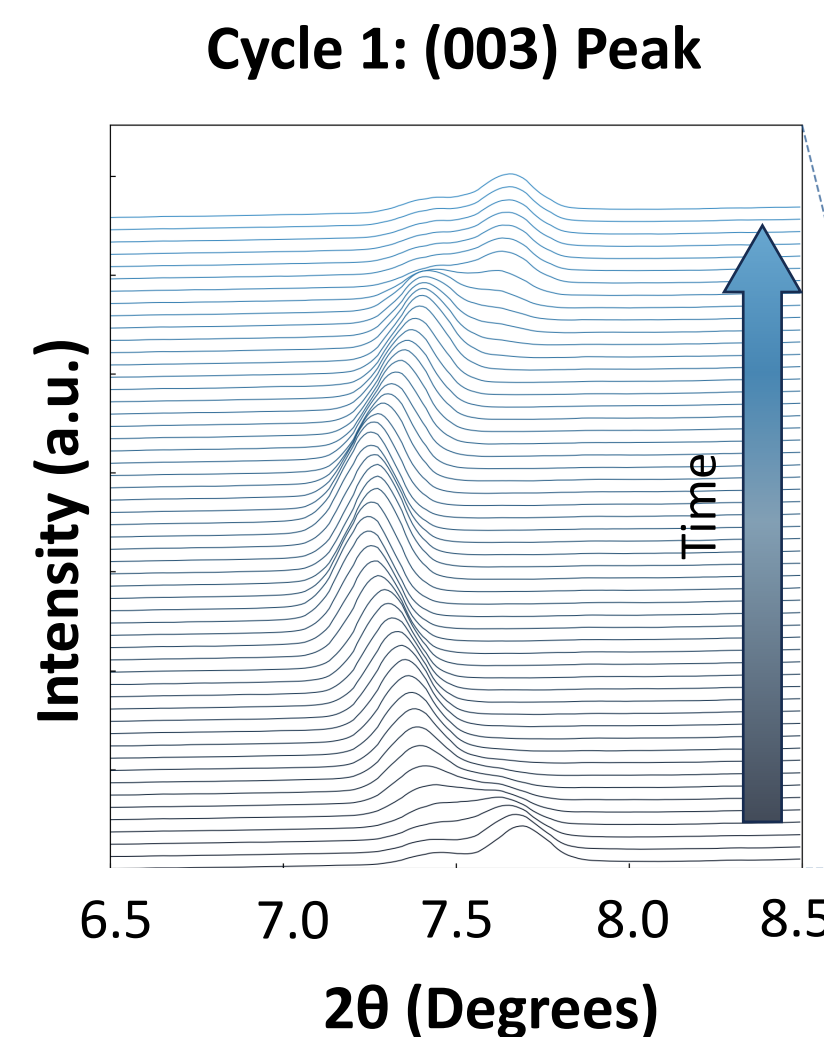
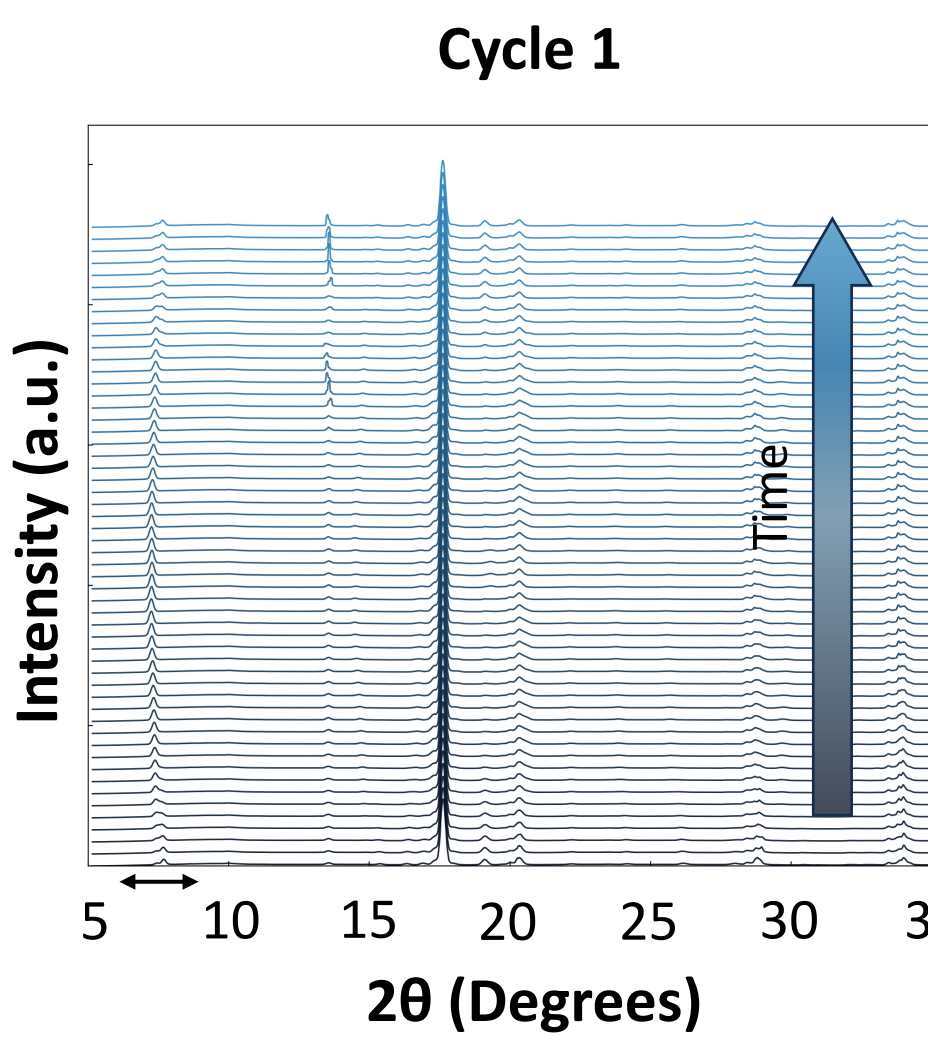
- Ran operando XRD for 1 cycle every 100 cycles.
- All cells demonstrated good capacity retention after hundreds of cycles.
- Cells made with XRD window showed similar degradation rate to those w/o XRD window—slightly lower capacity may be due to use of different spacer w/hole.

Operando XRD Results

Voltage Profiles



XRD Time Evolution Per Cycle



- Measured XRD every 5 minutes during charge and discharge at C/2.
- Saw expected, repeatable peak shifts with each cycle due to lattice expansion/contraction.
- No significant structural changes were observed after 400 cycles, but started to see indications of minor changes.

Conclusions & Future Directions

- Developed a successful approach to track degradation of $\text{Na}_{0.85}\text{Mn}_{0.5}\text{Ni}_{0.4}\text{Fe}_{0.1}\text{O}_2$ cathodes during battery cycling via operando XRD.
- XRD window in coin cells is stable over time.
- $\text{Na}_{0.85}\text{Mn}_{0.5}\text{Ni}_{0.4}\text{Fe}_{0.1}\text{O}_2$ cathodes showed stable cycling performance and no significant structural changes through 400 cycles, but showed indications of minor changes.
- More significant degradation is expected to occur in later cycles—will continue to measure XRD through 1000 cycles to gain insight into degradation mechanisms.

[1] L. Su. *et al. MethodsX* 2021, 8, 101562.