# Coupling Electrochemical Testing with Non-Destructive Evaluation for Safe Lithium Metal Batteries

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Advanced Materials and Processing Branch
NASA Langley Research Center

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# Challenges and Proposed Solutions

Challenges:

- ❖Lithium (Li) metal is one of the most promising anode material for high-energy lithium-ion batteries (LIBs)
- ❖However, LIBs are highly prone to fires and explosions due to organic liquid electrolytes

Proposed Solutions:

- ❖In-situ monitoring of hazards (dendrites, thermal runaway) using embedded sensors can detect incoming disasters beforehand
- ❖The aim is to prevent rather than cure disasters

# Previous Intern Work History

**Ashley Lam (Summer 2022)**

❖Establishing the testing procedures

❖Cycling methodologies and initial testing on the pouch cells

**Daniel Caiceido (Fall 2022)**

❖Cycling (charged and discharge) pouch cells

❖Scanning electron microscopy (SEM) on the end-of-life lithium anodes

**Aoife Zuercher (Spring 2023)**

❖Ex-situ non-destructive evaluation (NDE) and electrical impedance spectroscopy (EIS) on the cells every two cycles (charge and discharge)

❖SEM on the end-of-life lithium anodes.

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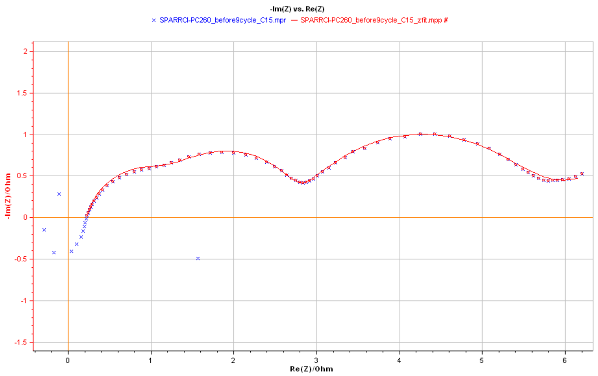
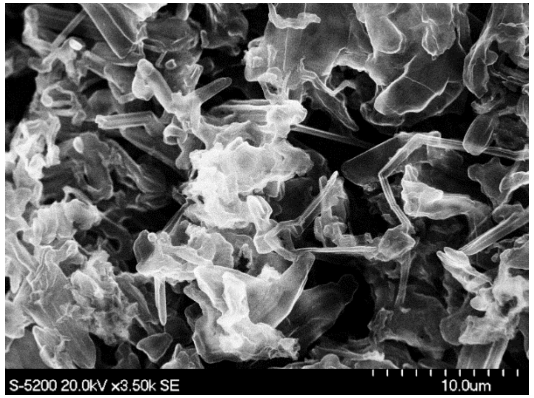
# Objectives

## Impedance Modelling



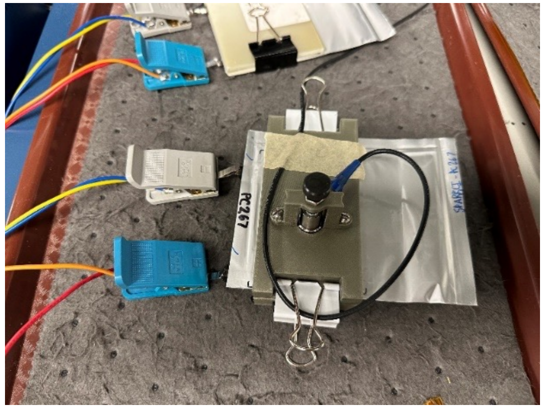

Finding changes in resistances of different battery components

## SEM at different C-rates




Observing dendrites for cells cycled at different C-rates

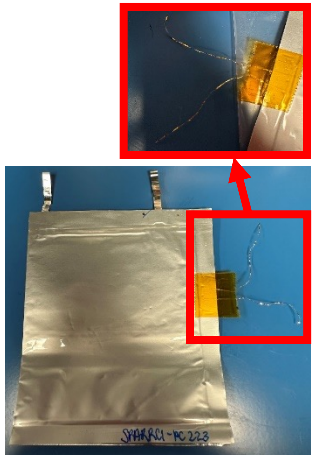
## In-situ NDE analysis



Live monitoring of battery health

## Random Walk

## Sensor cell analysis



Credit: Aoife Zuercher

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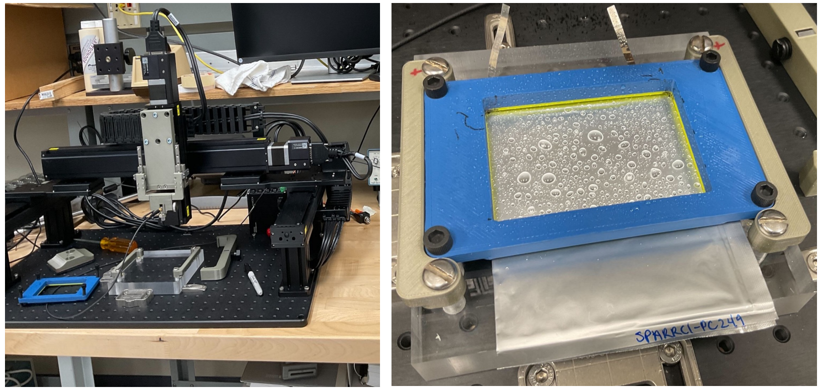
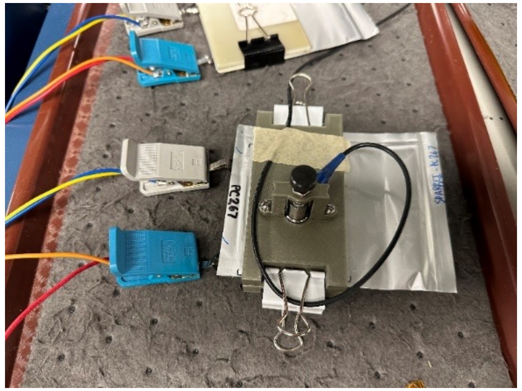
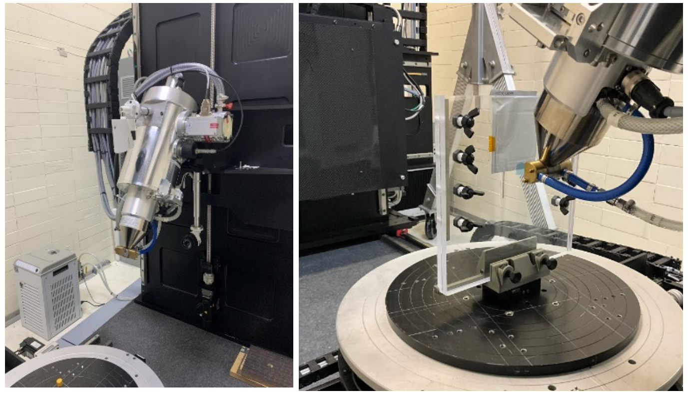
# Characterization Techniques



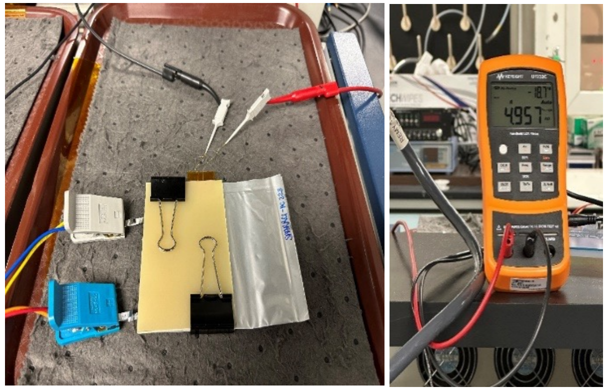
Ultrasound Testing (UT)



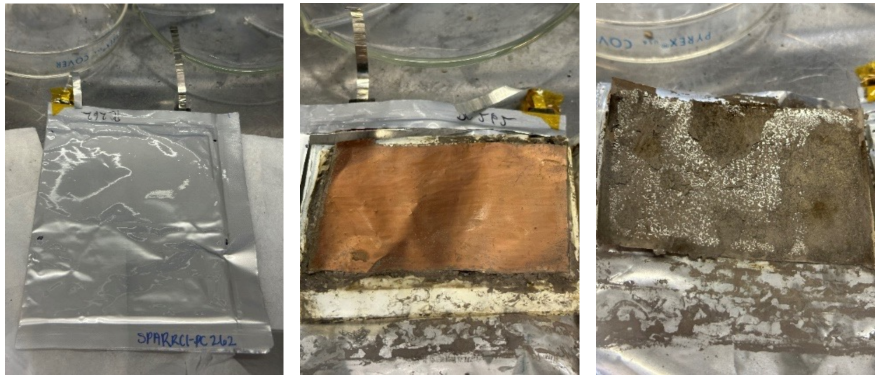
In-Situ Resonance



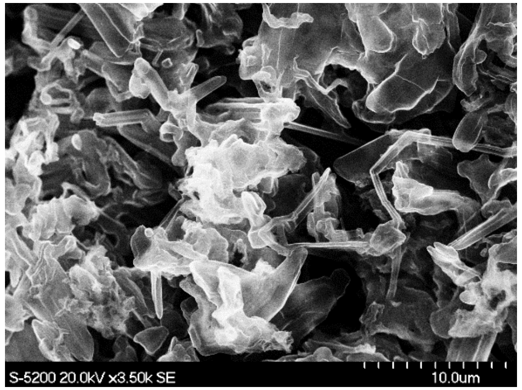
Digital Radiograph (DR)



In-Situ Sensing

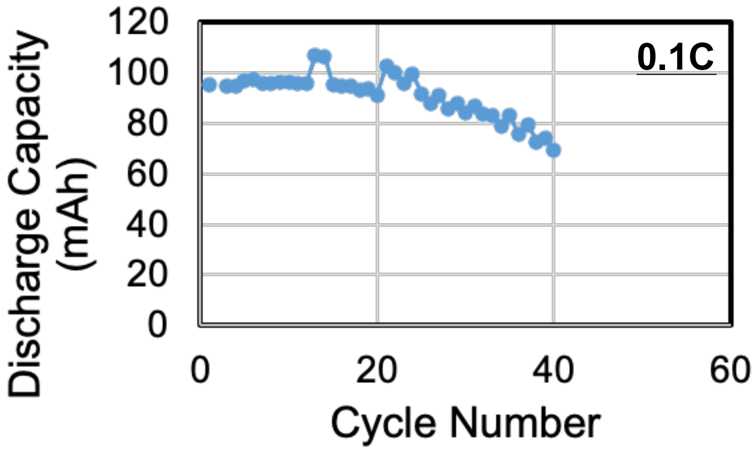


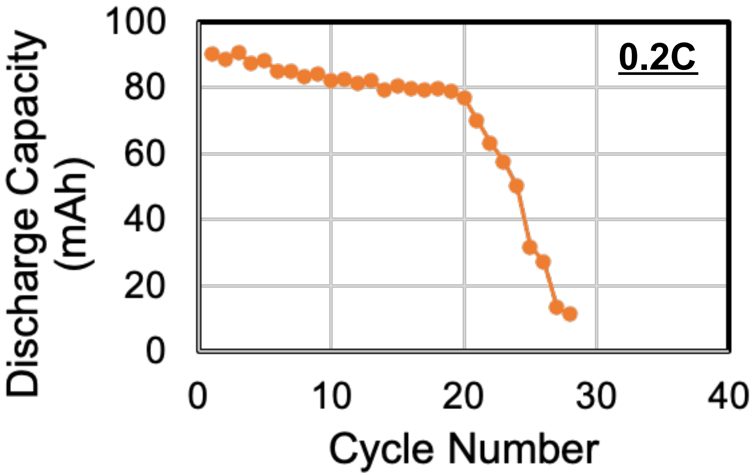
Destructive Physical Analysis (DPA)

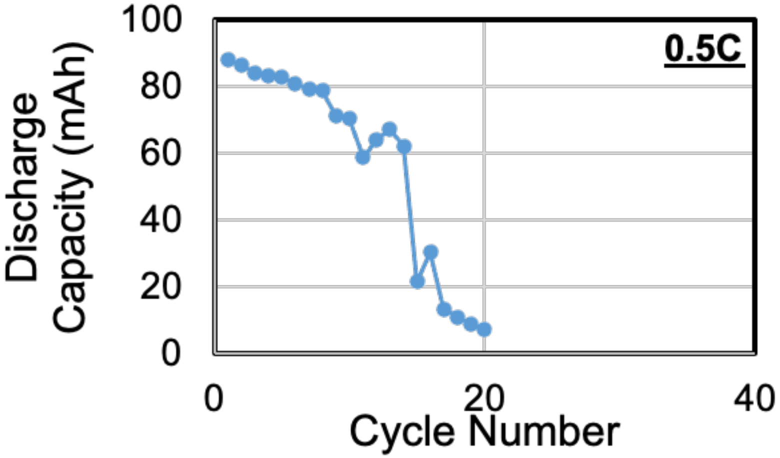



Electron Microscopy

# Task 1: Ultrasound Health Monitoring of Pouch Cells




❖Pouch cells were cycled at moderate C-rates.

❖Ultrasound scanning data were taken every two cycles.

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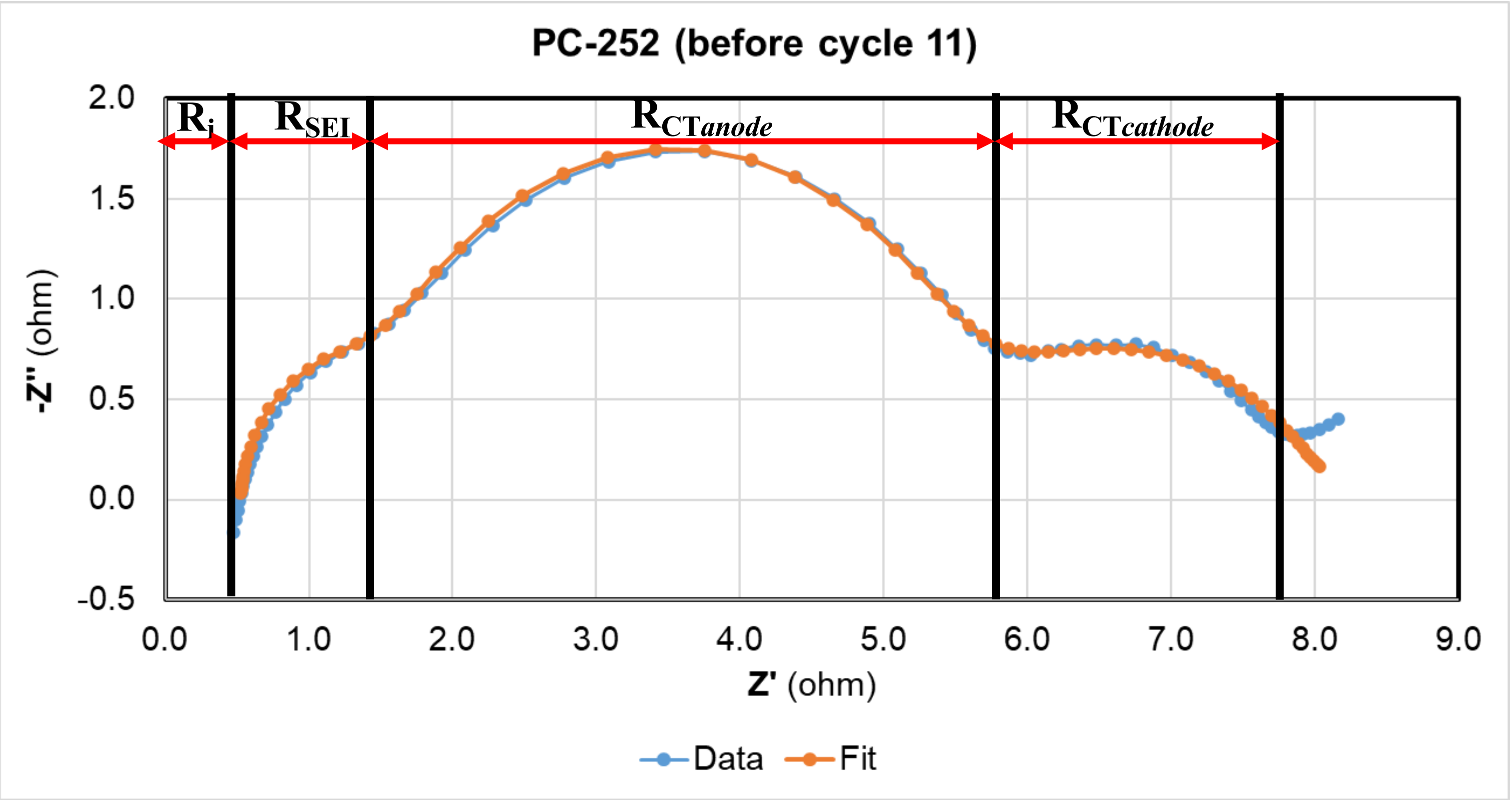
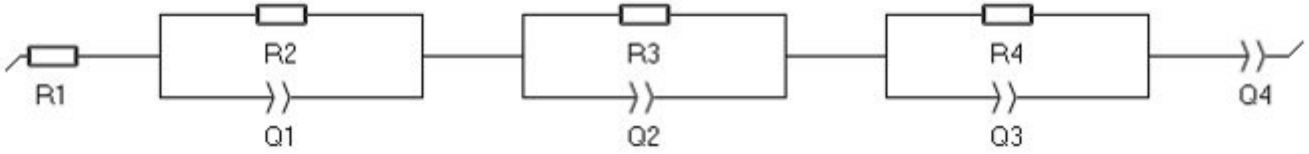
# EIS Modeling




$R_i$ – Internal Resistance
$R_{SEI}$ – Solid Electrolyte Interface Resistance
$R_{CT}$ – Charge Transfer Resistance

$R1 = R_i = 0.52$ Ohm
$R2 = R_{SEI} = 0.87$ Ohm
$R3 = R_{CT\mathit{Anode}} = 4.28$ Ohm
$R4 = R_{CT\mathit{Cathode}} = 1.74$ Ohm

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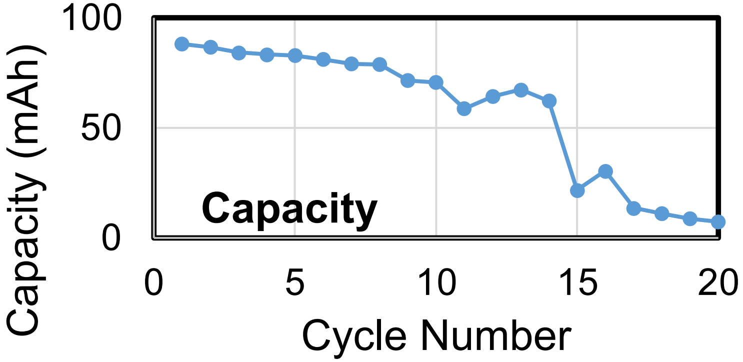
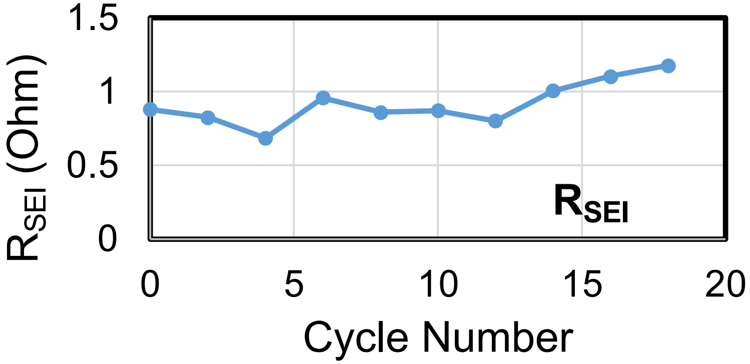
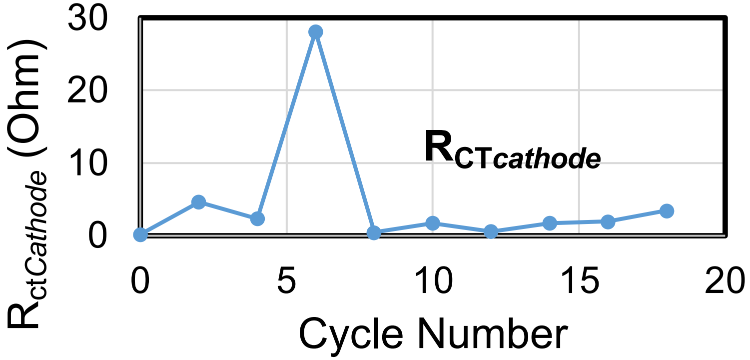
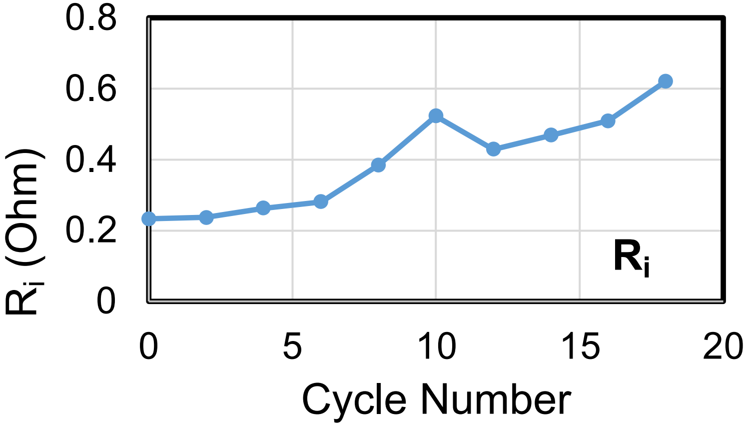
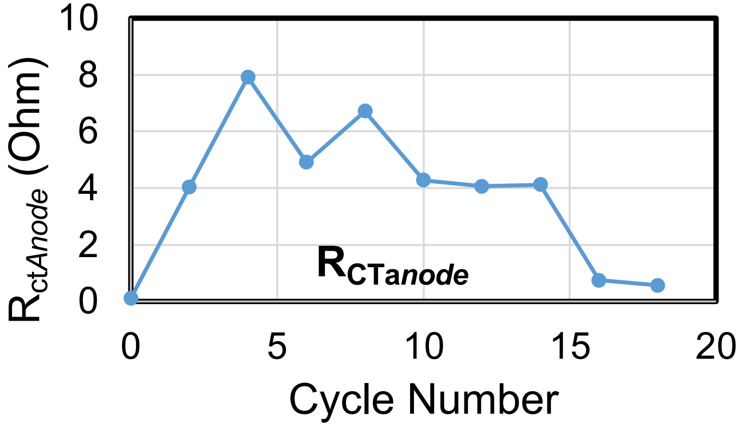
# EIS Results



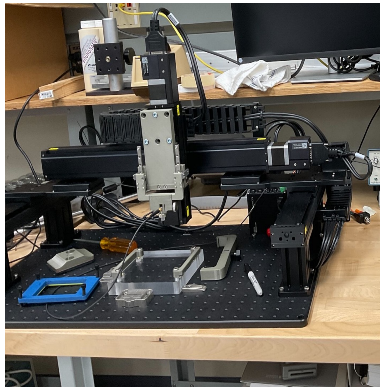



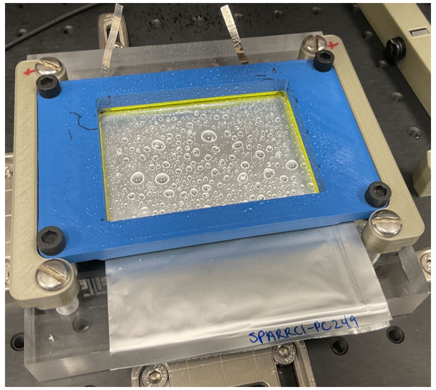



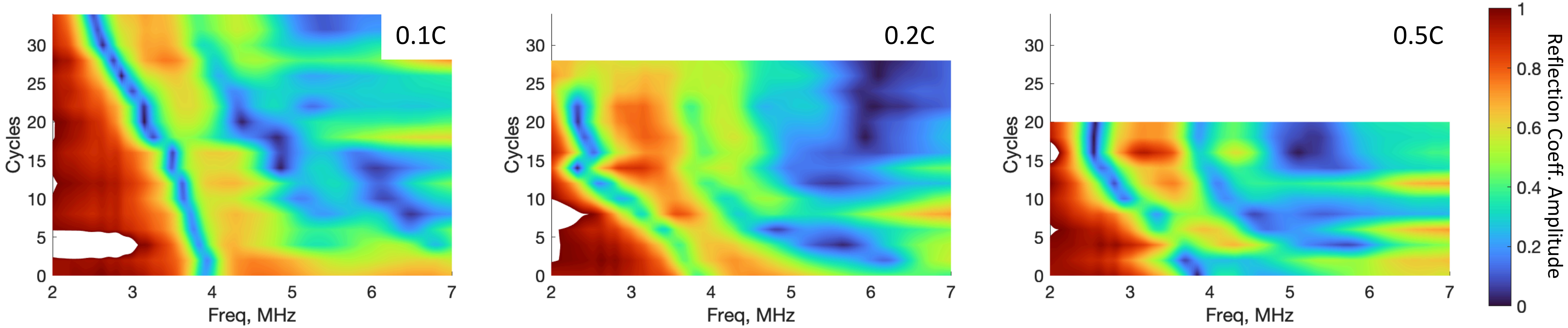

- Internal resistance increases linearly with number of cycles
- SEI resistance increases steadily with number of cycles and sharply at end of life
- $R_{CT}$ shows no distinct relationship with number of cycles at both anode and cathode
- This cell was cycled at <u>0.5C</u>

# Ultrasound Testing Data

- ❖ Significant shift in both first and second resonance observed
  - ❖ Stage 1: Fast change in second resonance, steady change in first
  - ❖ Stage 2: Steady change in first and second resonance
  - ❖ Stage 3: Little change in first or second resonance, dominant higher frequency resonance



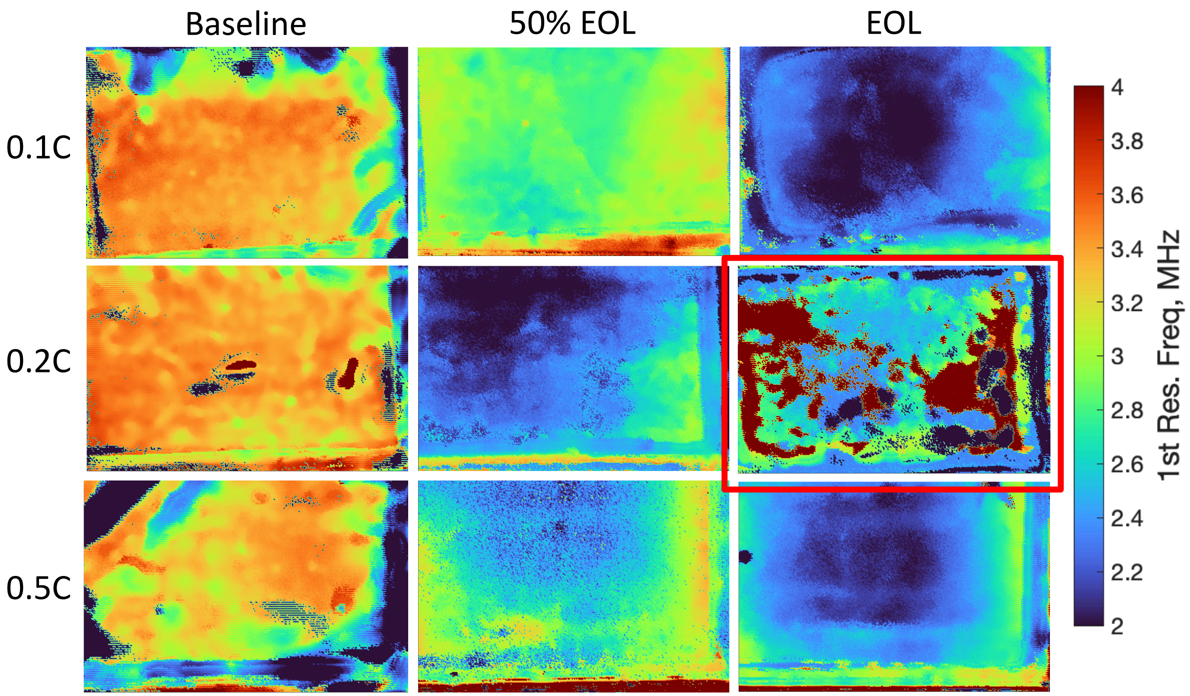




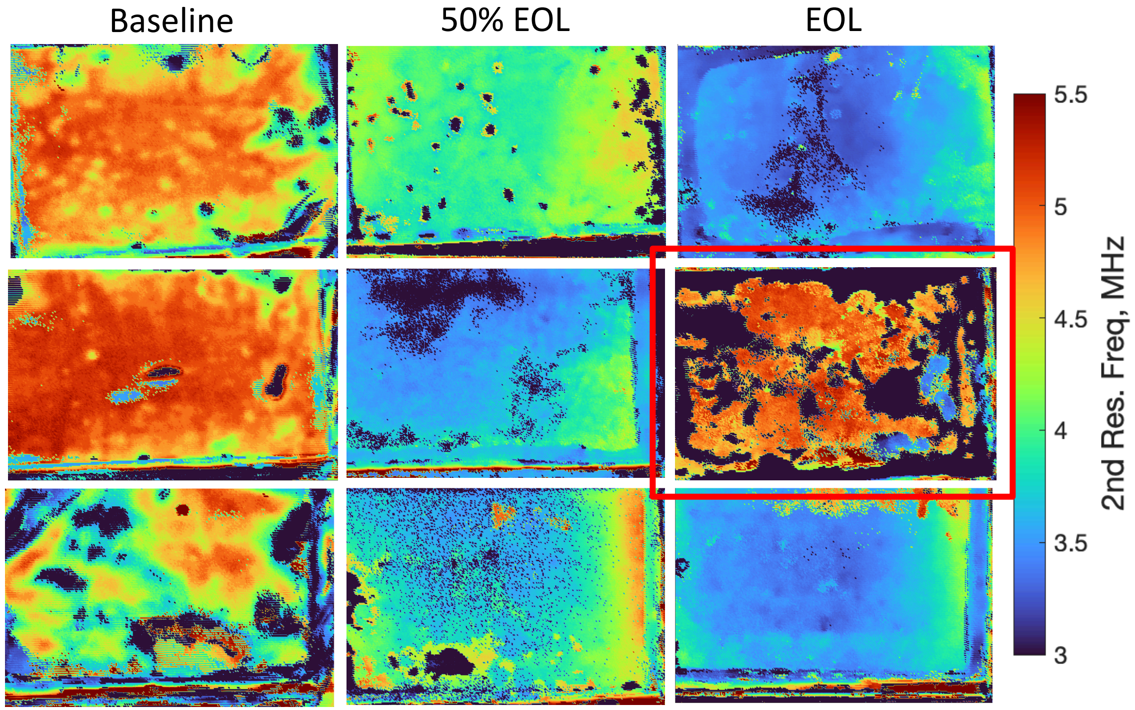

Analysis credit: Matt Webster

# Scanning Results from Benign Tests

- ❖Maps of 2nd resonance frequency appear to better show finer details of degradation.
- ❖This could be an indicator of battery health over successive cycles.




**1st Resonance Frequency**




**2nd Resonance Frequency**

Analysis credit: Matt Webster

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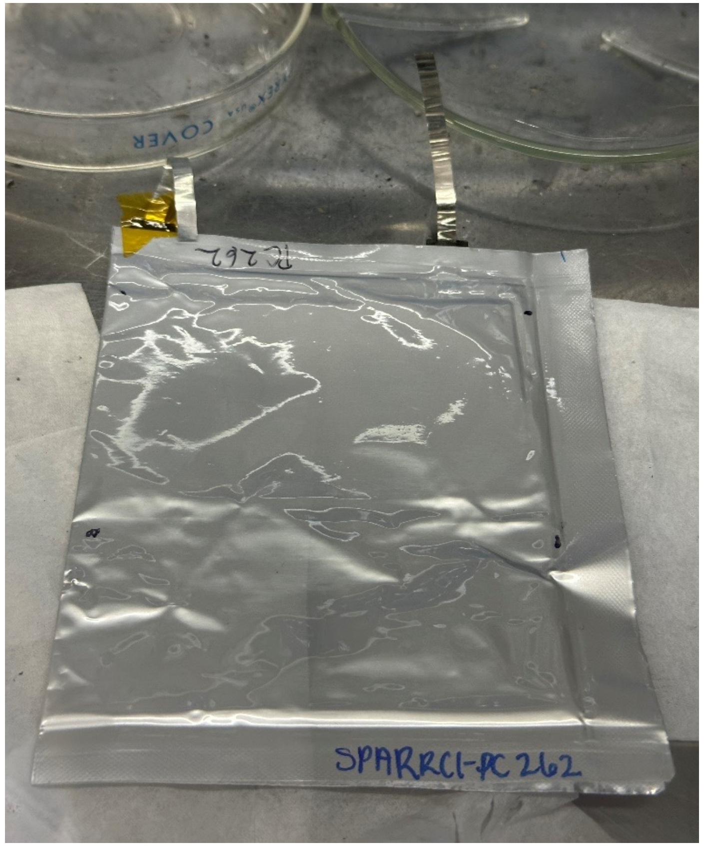
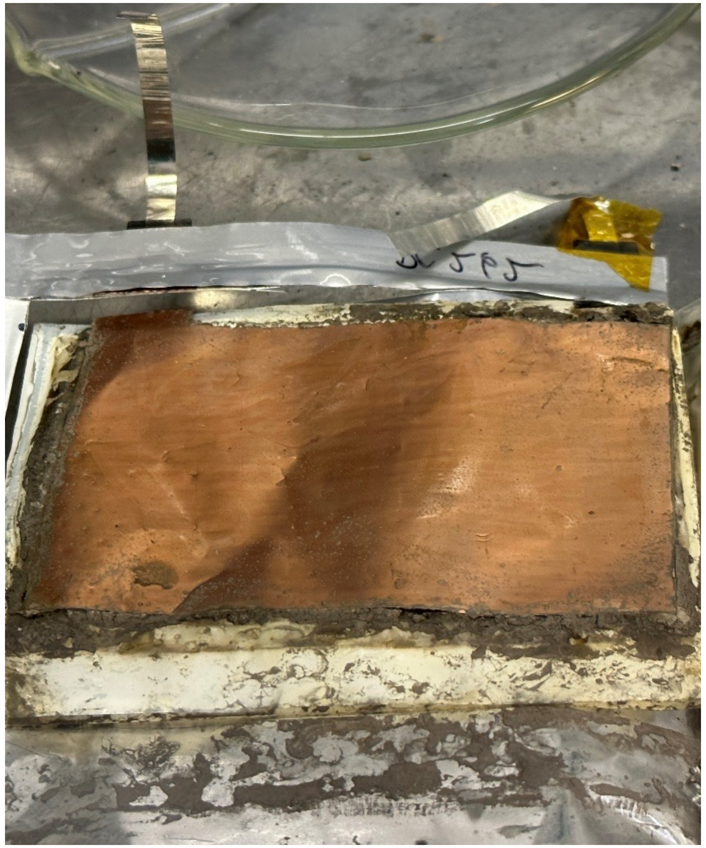
# Destructive Physical Analysis

Before Opening



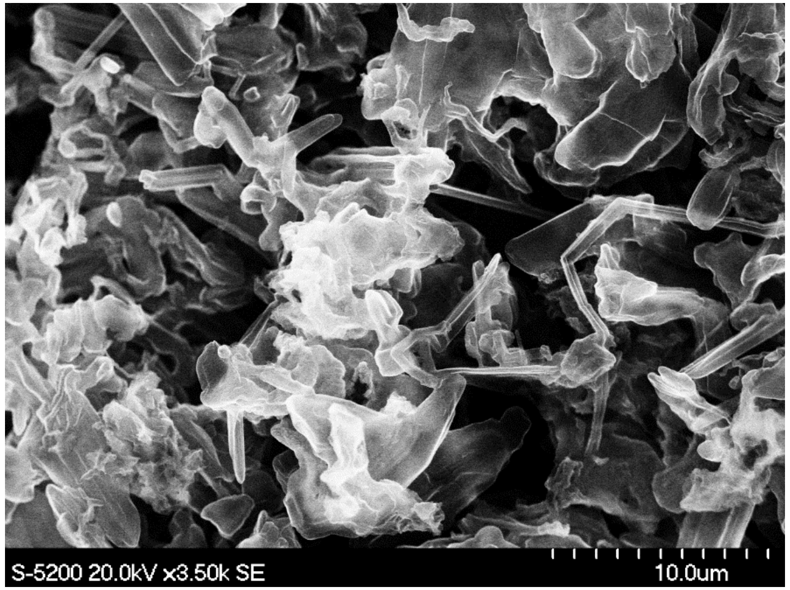

Copper



Lithium

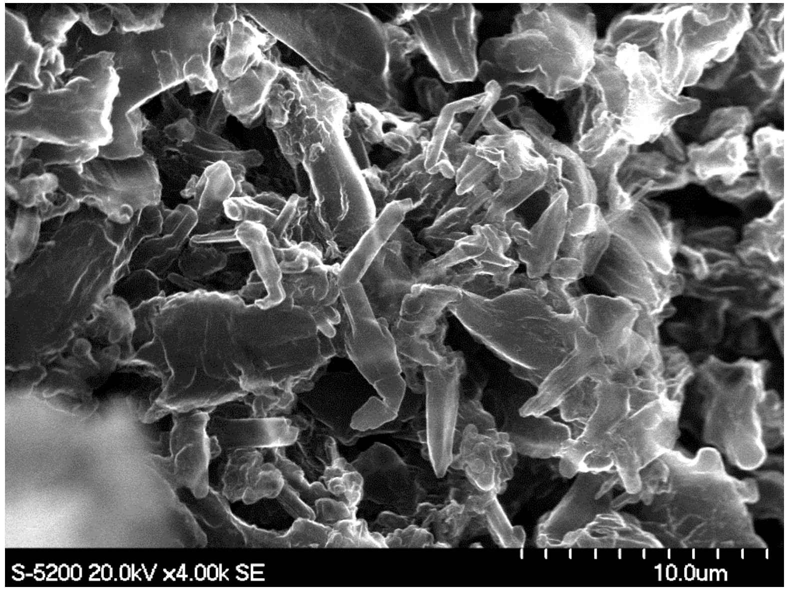
- This cell was cycled at 0.2C.

# SEM of Li Anodes Cycled at Different C-rates




**0.1C**


**0.2C**

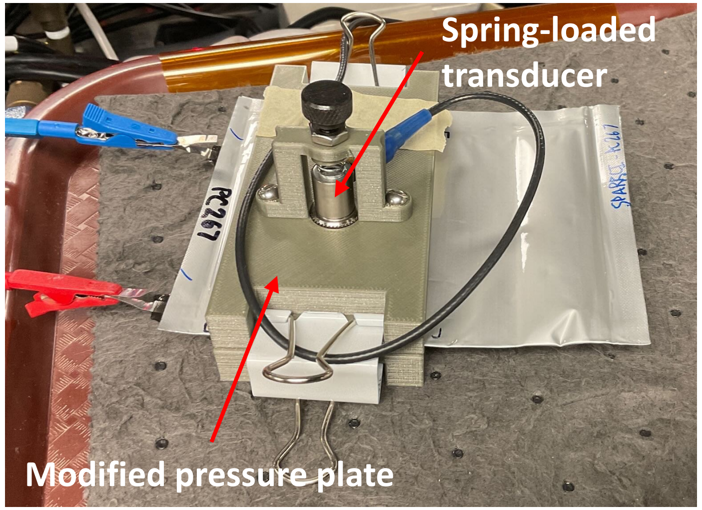


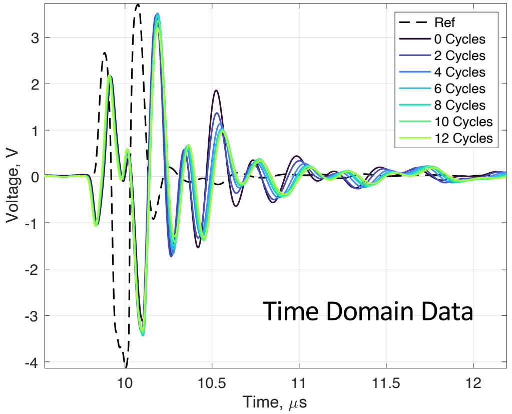

**0.5C**

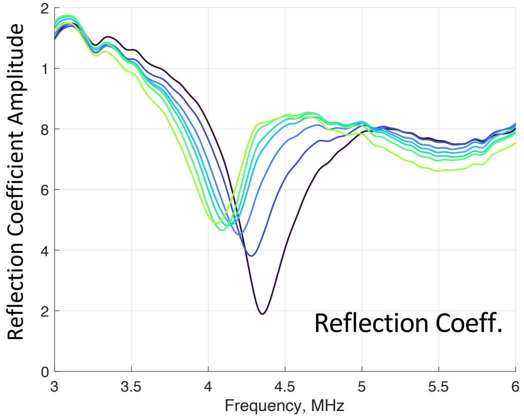
- Dendrites seem to grow bigger and thicker as C-rate increases. They also become more numerous
- C-rate of 0.1 means the theoretical current required to charge a battery in 10 hours, 0.2 means 5 hours, and 0.5 means 2 hours

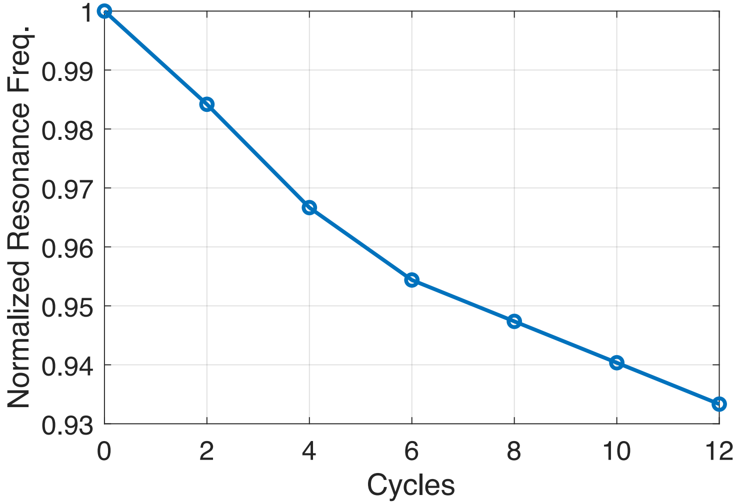
# Task 2: In Situ Resonance

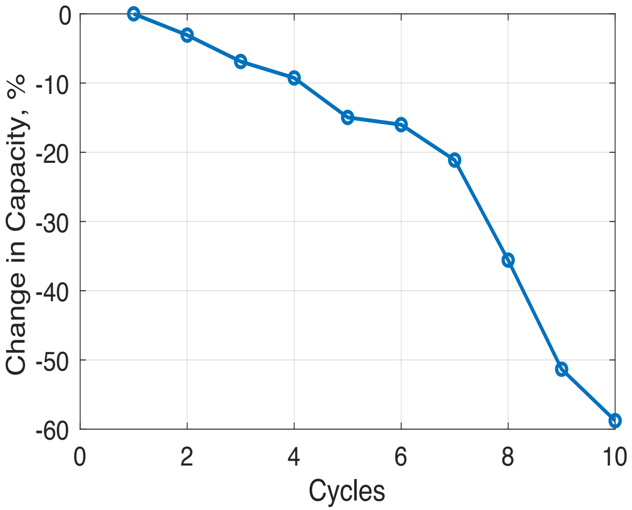
- In situ resonance testing completed on one cell
  - PC267 cycled at rate of 0.5C
  - Inspections made in two cycle increments
- Results show improved consistency in both time signatures




Analysis credit: Matt Webster

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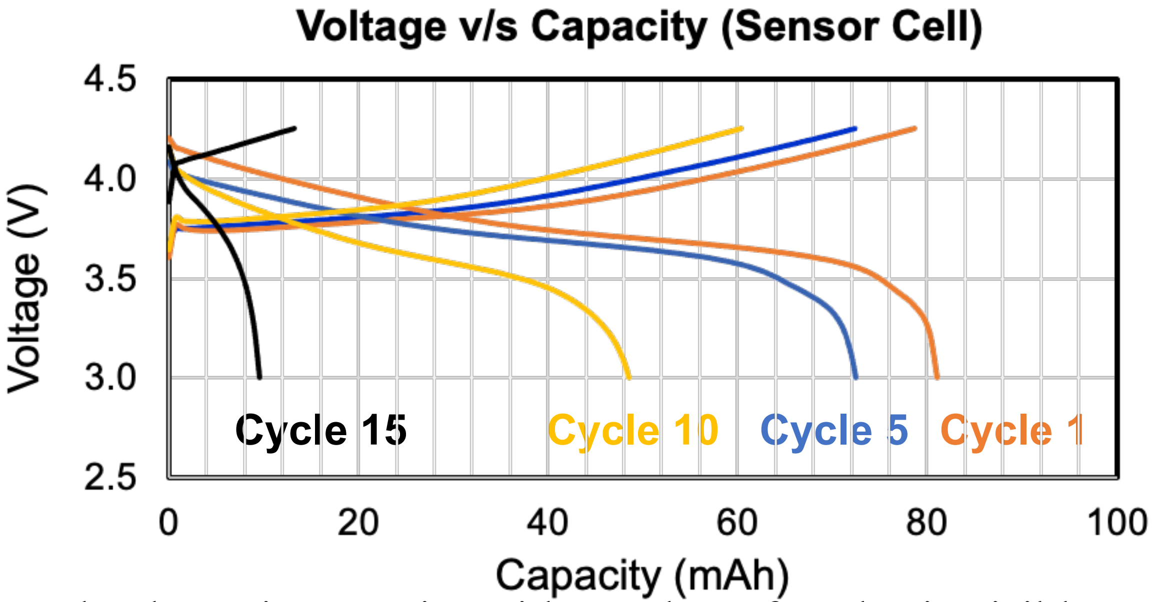
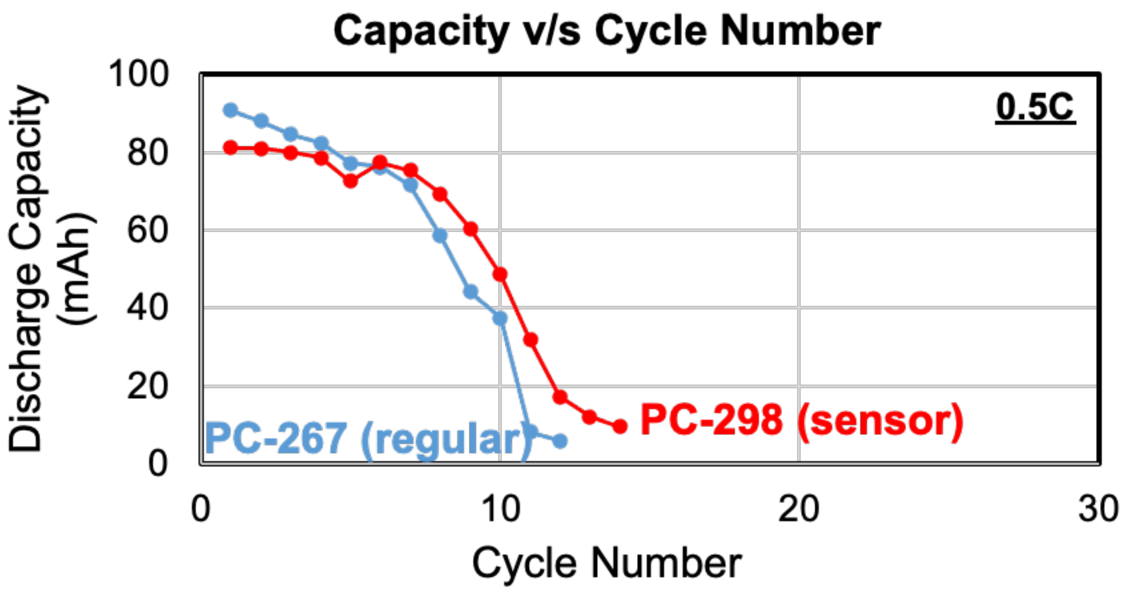
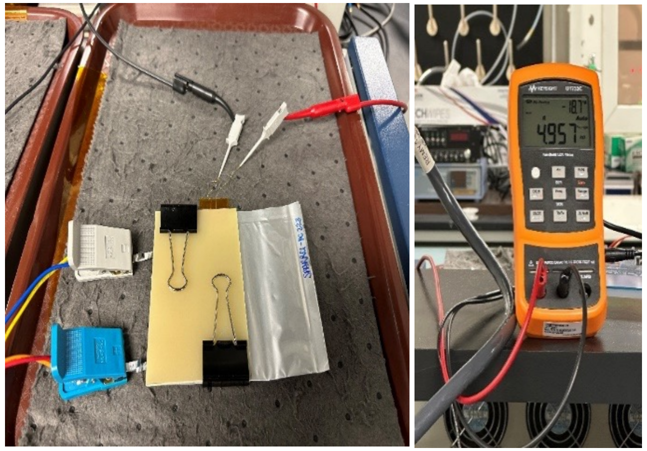
# Task 3: In Situ Sensing Tests




- The decay in capacity with number of cycles is visible
- The cell was cycled between 3 V and 4.25 V and reached end of life at 14 cycles
- Sensor is embedded in the separator of the battery
- The connected LCR meter measures the resistance across the sensor
- The sensor has negligible effect on battery performance



- **LCR**: Inductance, Capacitance, Resistance

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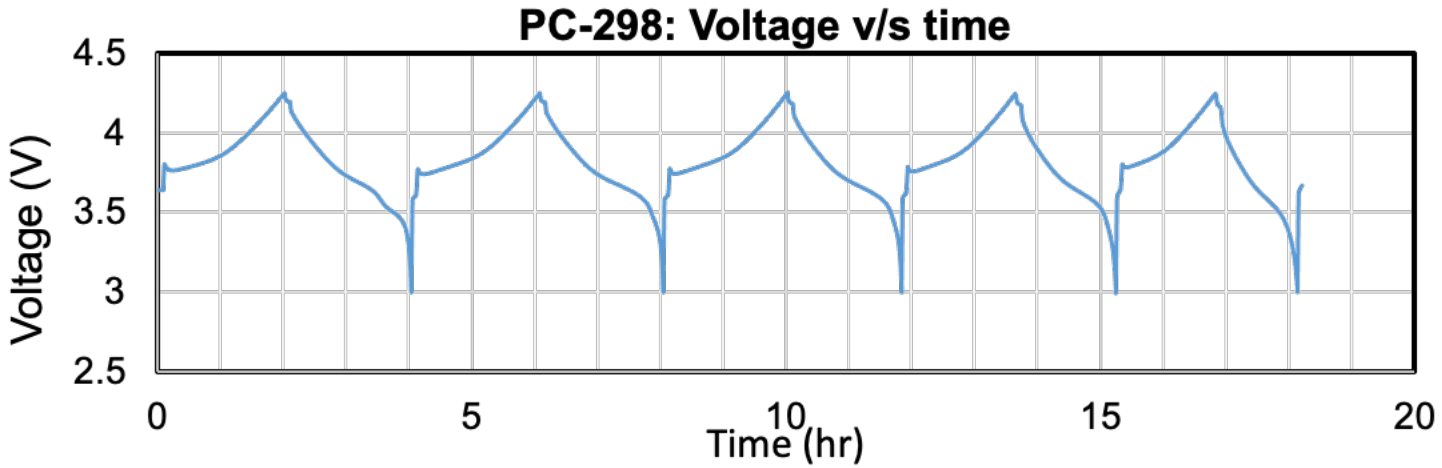
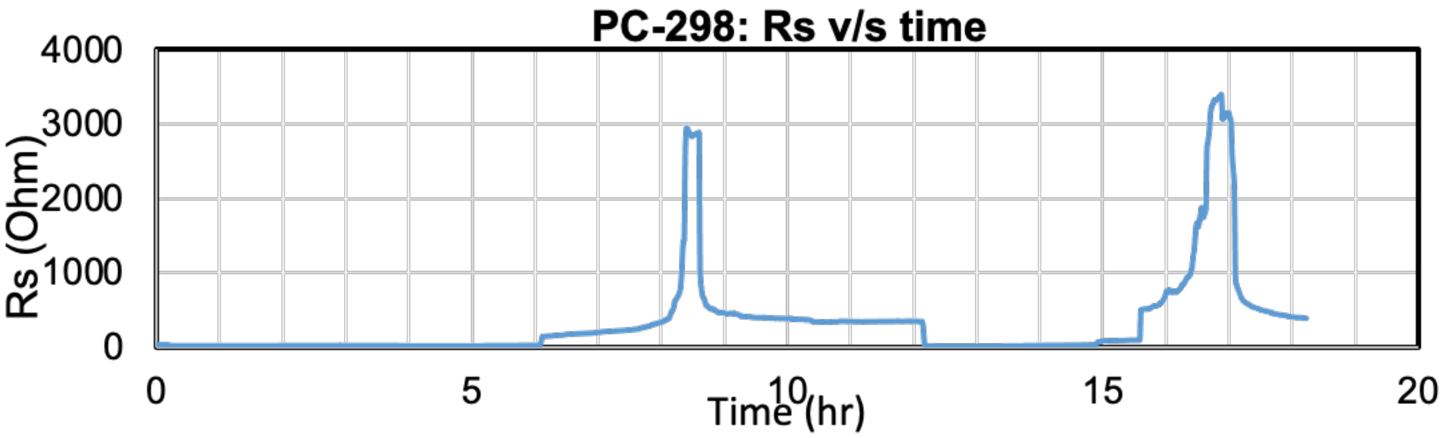
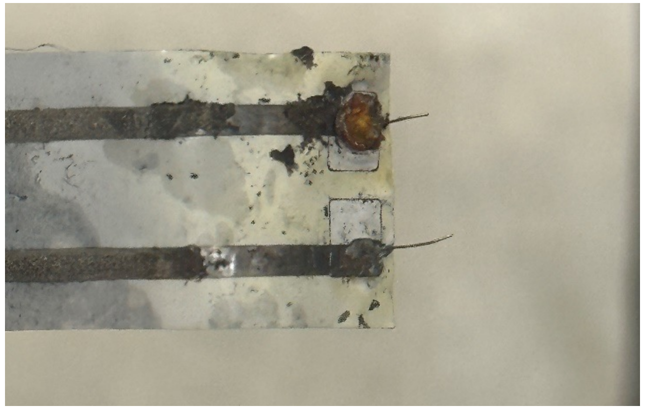
# In-Situ Sensing Results

Cycles 10-15 at C rate of 0.5C



- There were lithium metal deposits on the sensor after cycling. This could be the cause of its failure.

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# Summary

- ❖Performed cell cycling and electrochemical analyses (such as EIS) to correlate with ultrasound health monitoring
  - ❖DPA and SEM were conducted on cells at end-of-life
- ❖Performed in-situ resonance studies on pouch cells during cycling
- ❖Acquired in-situ sensing data from sensors embedded within cells during cycling

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# Next Steps/Outlook/Future Work

- ❖Finish the DPA and SEM on the remaining cells and observe dendrite growth in them
- ❖Model the sensor data and co-relate it to temperature changes into the cell. Program the in-situ NDE set-up to take readings continuously
- ❖This project is extremely crucial to sensor-based prognostics and battery safety as we can continuously monitor battery health and predict thermal runaway
- ❖This approach can provide a basis for identifying battery failure in advance and prevent catastrophic fires and explosions

# Acknowledgements


- Yi Lin, Daniel Perey
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- Brianne Demattia (SPARRCI PI at GRC)
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- Intern Co-Ordinator: Ashley Gonzalez.



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# Thank you! Any questions?