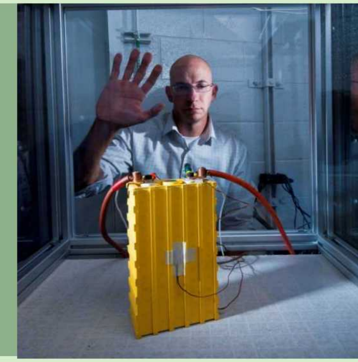
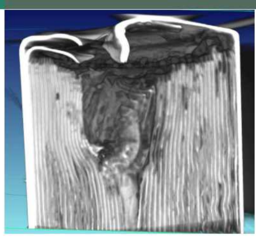
SAND2019-12426C

Investigations of the structural and electrochemical properties of overheated Li-ion batteries





PRESENTED BY

Loraine Torres-Castro, Joshua Lamb, Eric Deichmann, and Mohan Karulkar

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Outline

- **Introduction**
  - ✓Battery Abuse Testing Laboratory (BATLab) Capabilities
  - ✓Motivation
  - ✓Objective
- Methodology
- Results & Discussion
  - ✓NMC
  - ✓LFP
- Summary
- Acknowledgements

Capabilities and Infrastructure: Battery Abuse Testing Laboratory (BATLab)

Comprehensive abuse testing platforms for safety and reliability of cells, batteries and systems from mWh to kWh.

Mechanical abuse

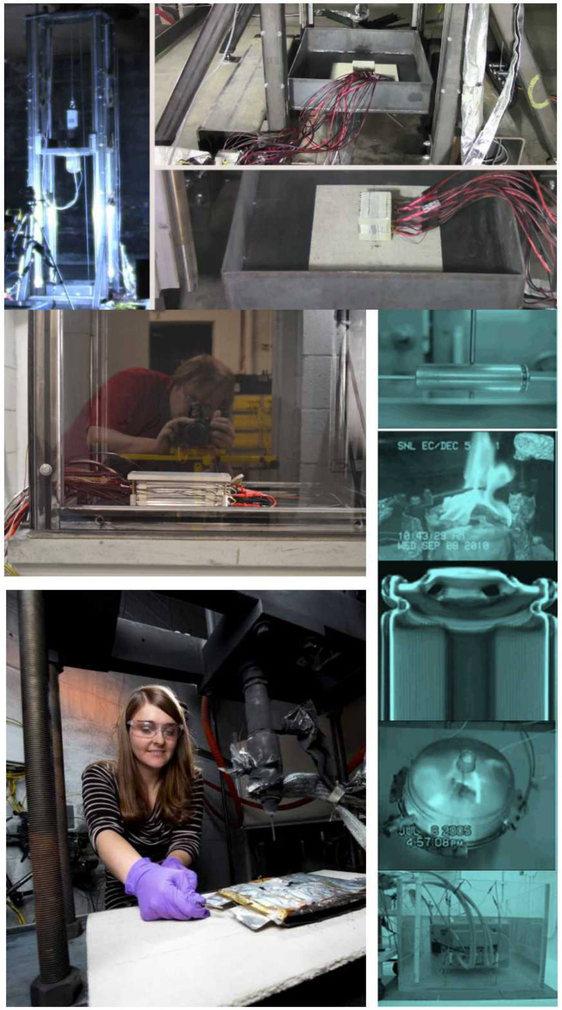
- Penetration (max. force 25 klbs, max speed 10 mm/s)
- Crush (max. force 100 klbs, max speed 2 mm/s)
- Impact (max. height 8”8’, max. drop weight 700 lbs)
- Immersion

Thermal abuse

- **Over temperature (250 ˚C, 5 ˚C/min)**
- Flammability measurements (250 ˚C, 5 ˚C/min)
- Calorimetry (405 ˚C, 5 ˚C/min)

Electrical abuse

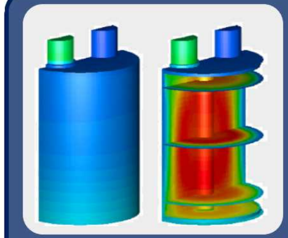
- Overcharge (max. current 300 A)
- Overdischarge (max. current 300 A)
- Short Circuit (max. current 2500 A)



Science and Diagnostics of Battery Failure

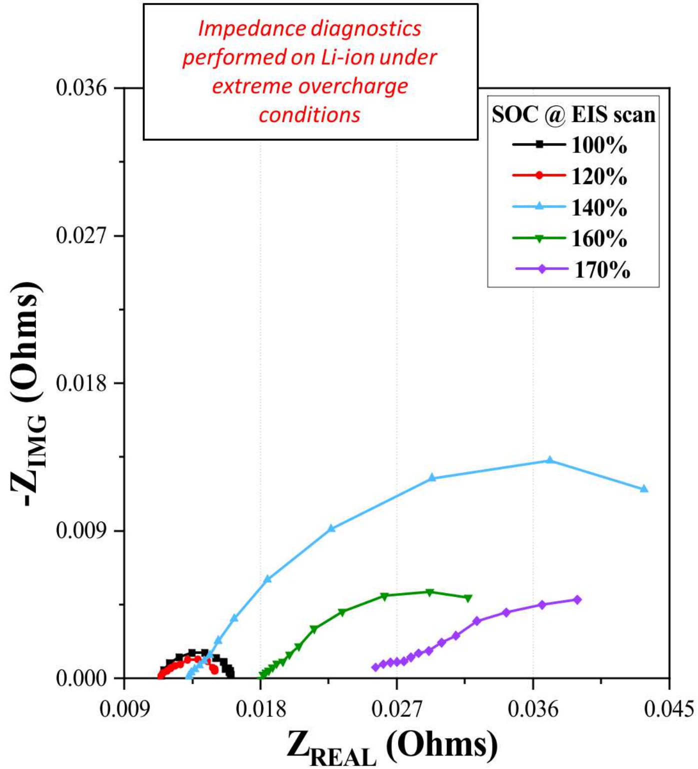
Materials R&D
- Non-flammable electrolytes
- Electrolyte salts
- Coated active materials
- Thermally stable materials
- Battery failure post mortem materials analysis

Testing
- Diagnostics during battery failure (pictured right)
- Gas analysis
- Battery calorimetry
- Electrical, thermal, mechanical abuse testing
- Failure propagation testing on batteries/systems
- Large scale thermal and fire testing (TTC)



Simulations and Modeling
- Multi-scale models for understanding thermal runaway
- Validating failure propagation models
- Fire Simulations to predict the size, scope, and consequences of battery fires

Procedure Development and Stakeholder Interface
- USABC Abuse Testing Manual (SAND 2005 3123)
- OE Energy Storage Safety Roadmap
- R&D programs to inform best practices, and policies.
- Hosted International Battery Safety Workshops and Energy Storage Safety Workshop



- Sandia is uniquely positioned to study the entire life cycle of a technology.
- Diagnostic tests can be performed under extreme failure conditions to understand the *how* and *why* of battery failure.

How do you know if a potentially abused battery is unsafe or unstable?

Voltage and temperature are often lagging indicators of a battery failure.

By the time a measurable trend is detected, it may be too late to arrest a catastrophic thermal runaway.

Batteries may also be unstable due to previous exposure to abusive conditions, but show little sign of problems during initial monitoring.

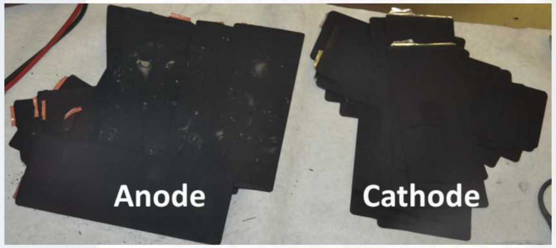
Objective Statements

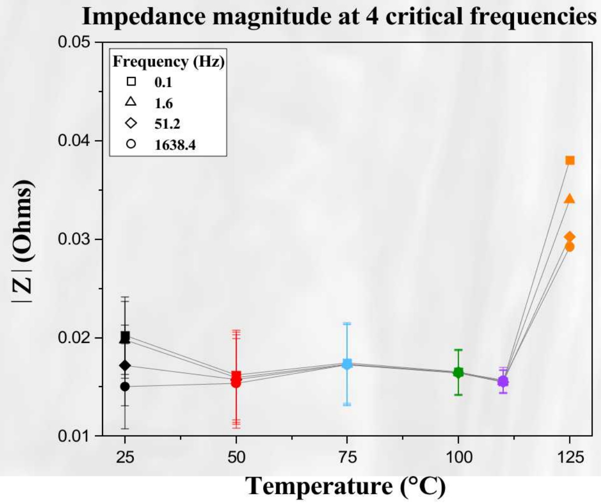
Objective 1

Provide insight into the development of an in-operando diagnostic technique to detect faulty cells within a pack.

Objective 2

Understand battery failure mechanisms during strenuous conditions to lead the design of more resilient and reliable energy storage systems that are inherently safe.





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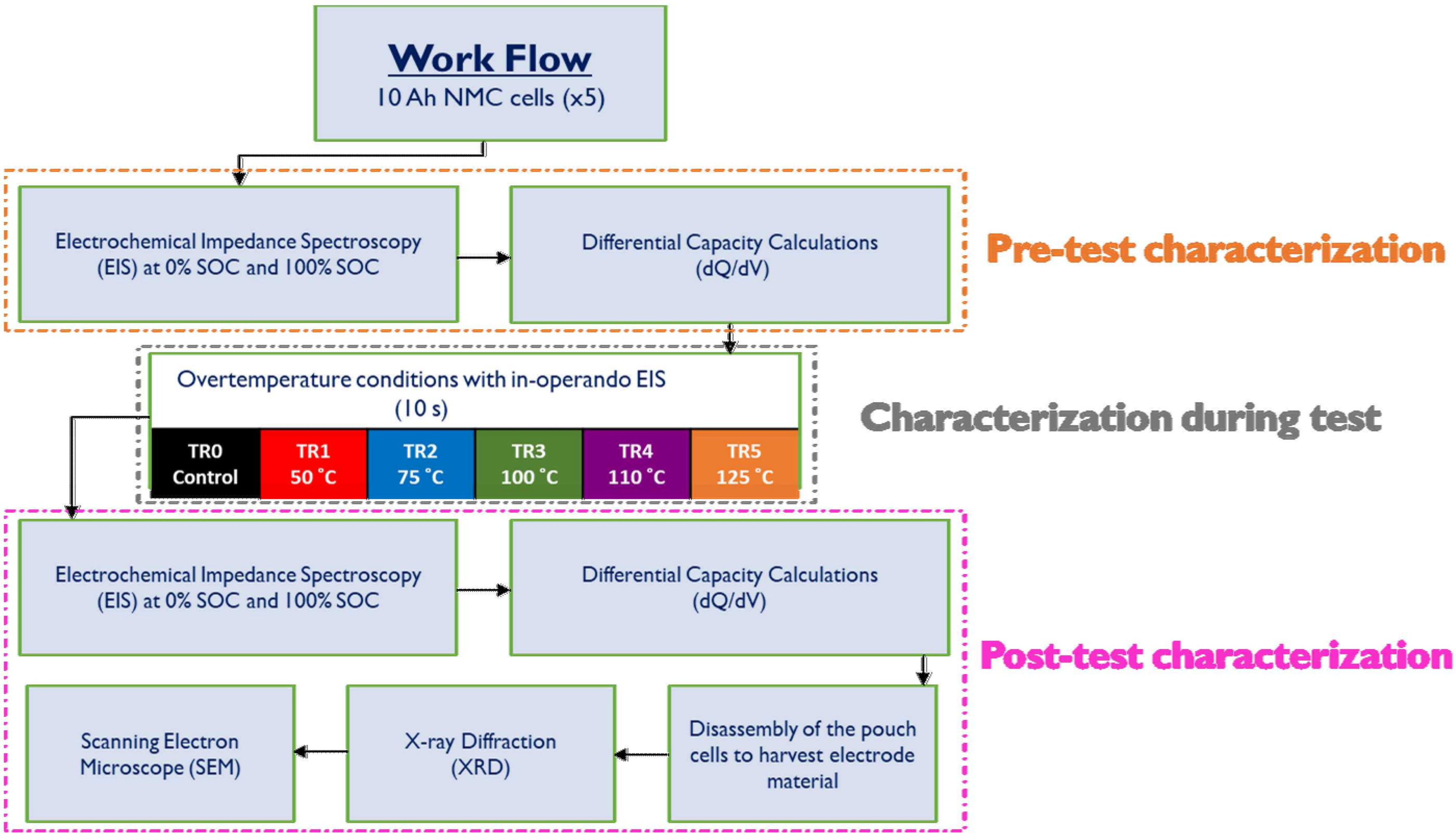
Methodology and Approach



TR – Thermal Ramp

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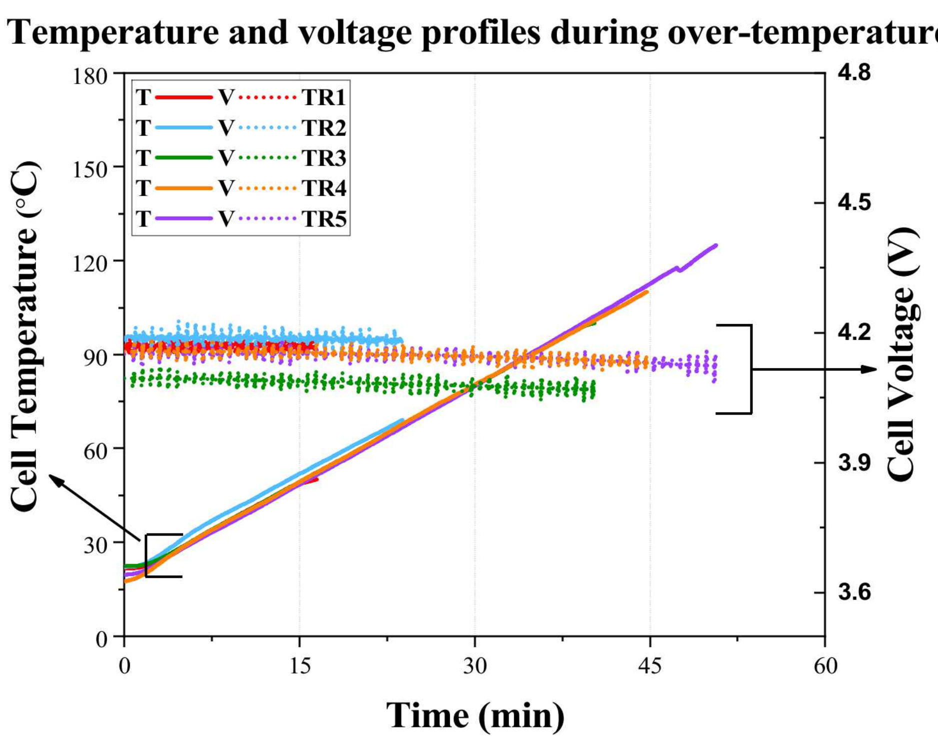
Over-temperature Effects to Cell Temperature and Voltage

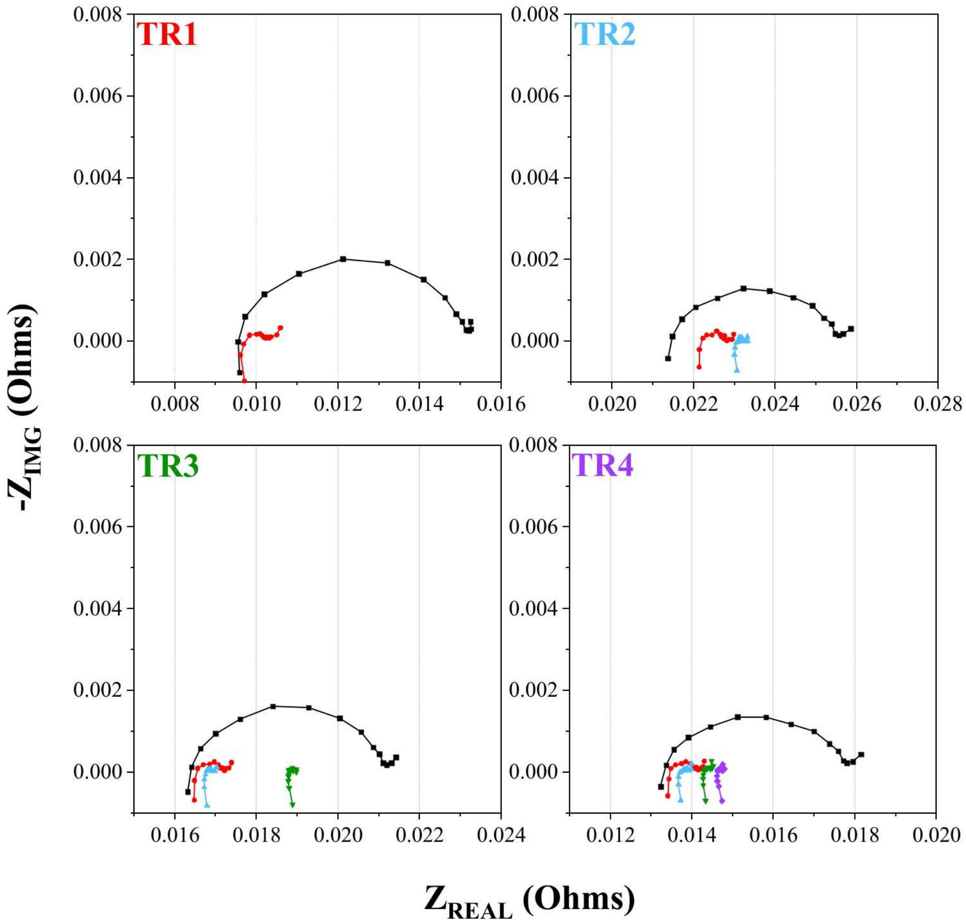


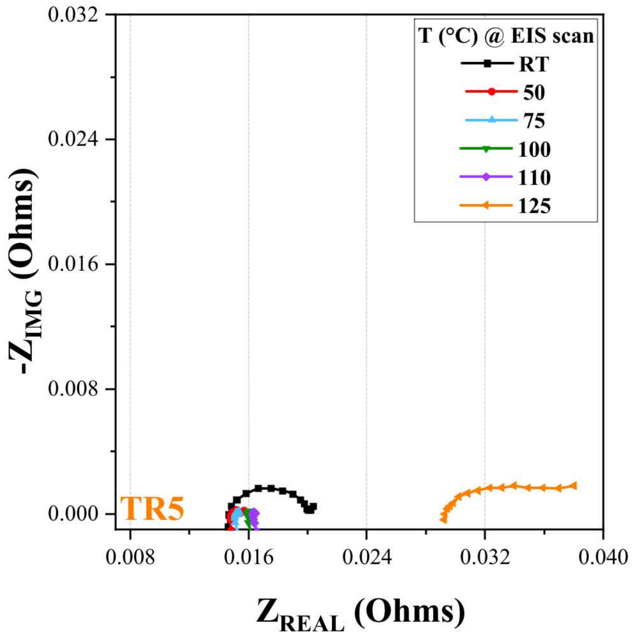
Five individual cells, TR1, TR2, TR3, TR4 and TR5 were heated to 50 ˚C, 75 ˚C, 100 ˚C, 110 ˚C and 125 ˚C, respectively.

Test parameters	
Heating rate	2 ˚C/min

	Initial T (°C)	Max. T (°C)	ΔT (°C)
TR1 - 50 ˚C	21.6	50	28.4
TR2 - 75 ˚C	22.1	75	52.9
TR3 - 100 ˚C	22.4	100	77.6
TR4 - 110 ˚C	17.6	110	92.4
TR5 - 125 ˚C	19.5	125	105.5

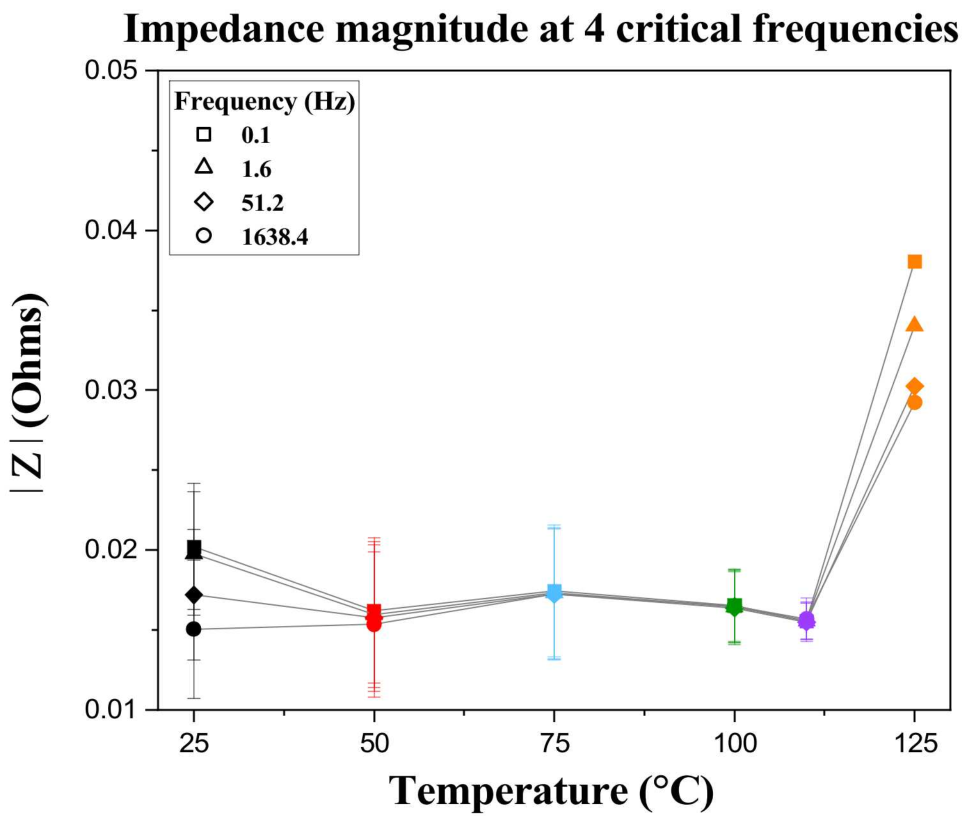
In-operando EIS





- Minor changes observed up to 100 ˚C, while significant changes began to develop in the EIS curves above that point.
- The technique is most effective at detecting temperatures that approach dangerous levels, and would likely not provide a signal at more moderate temperatures.

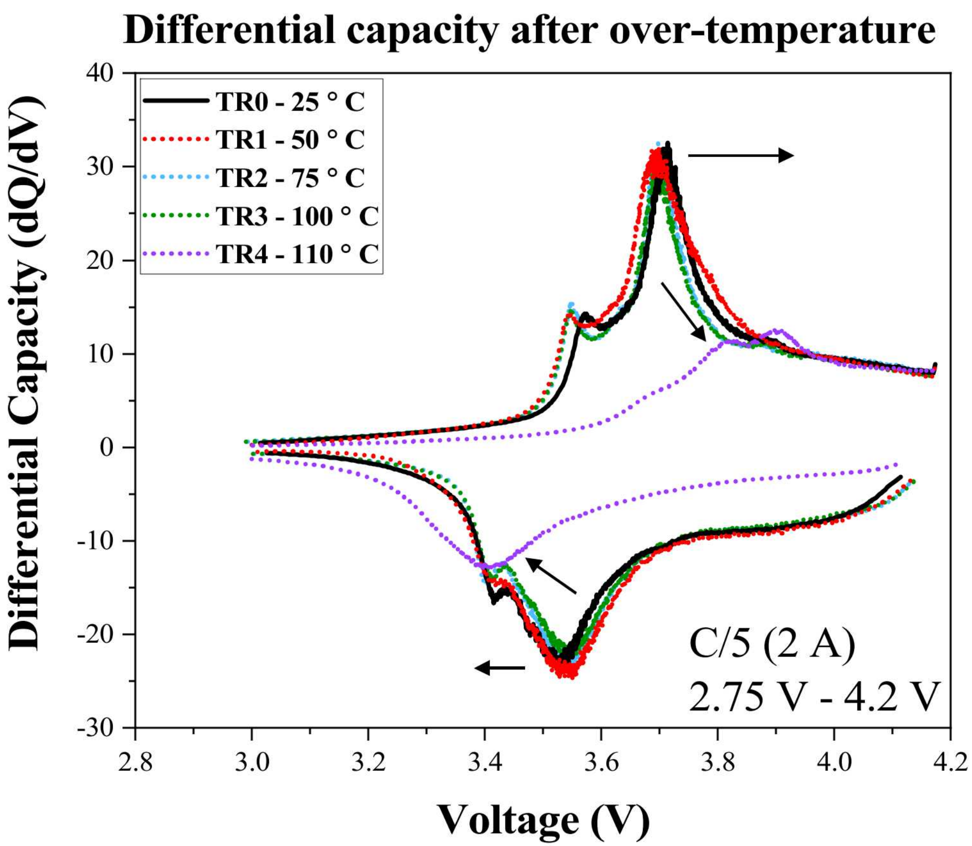
TR5-125°C : Magnitude of the complex impedance

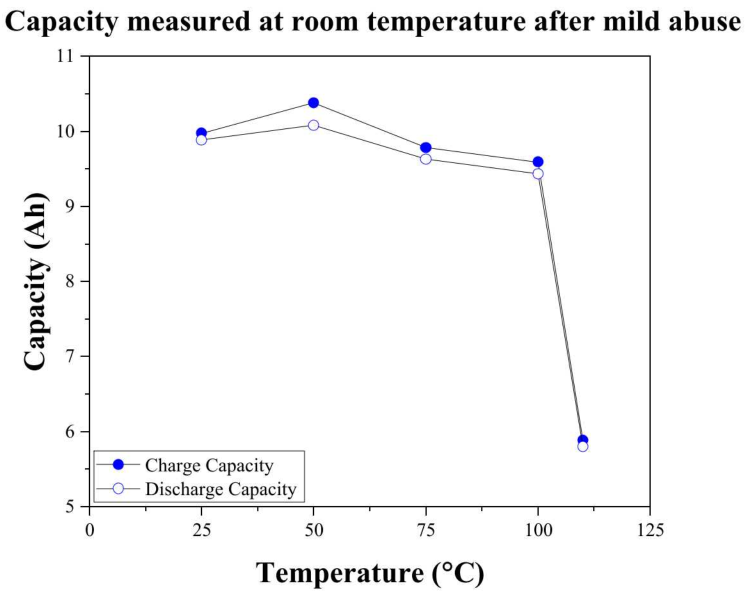


$$|Z| = \sqrt{Z_{REAL}^2 + Z_{IMG}^2}$$

- To better understand the changes after 100°C, we analyzed the magnitude as a function of T.
- The data showed significant increments in resistance after ~110°C for all frequencies.

Differential Capacity (dQ/dV)





- Minor changes observed until above 100 ˚C, where we begin to see signs of significant damage to the battery.
- TR5 (125 ˚C) was significantly damaged by the over-temperature procedure to the point of not being operational.

Disassembly Images

The electrodes were harvested at 0% SOC after the overtemperature procedure was completed (fully lithiated cathode).

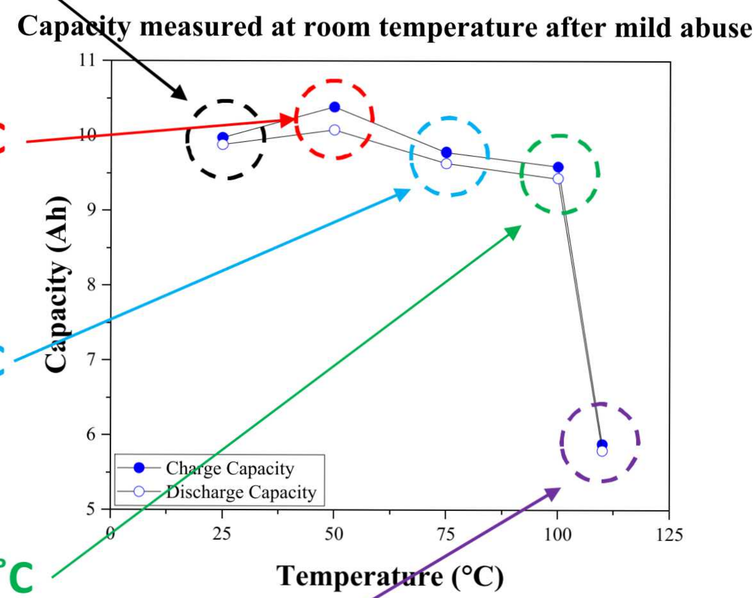
Control Cell

Heated to 50 ˚C

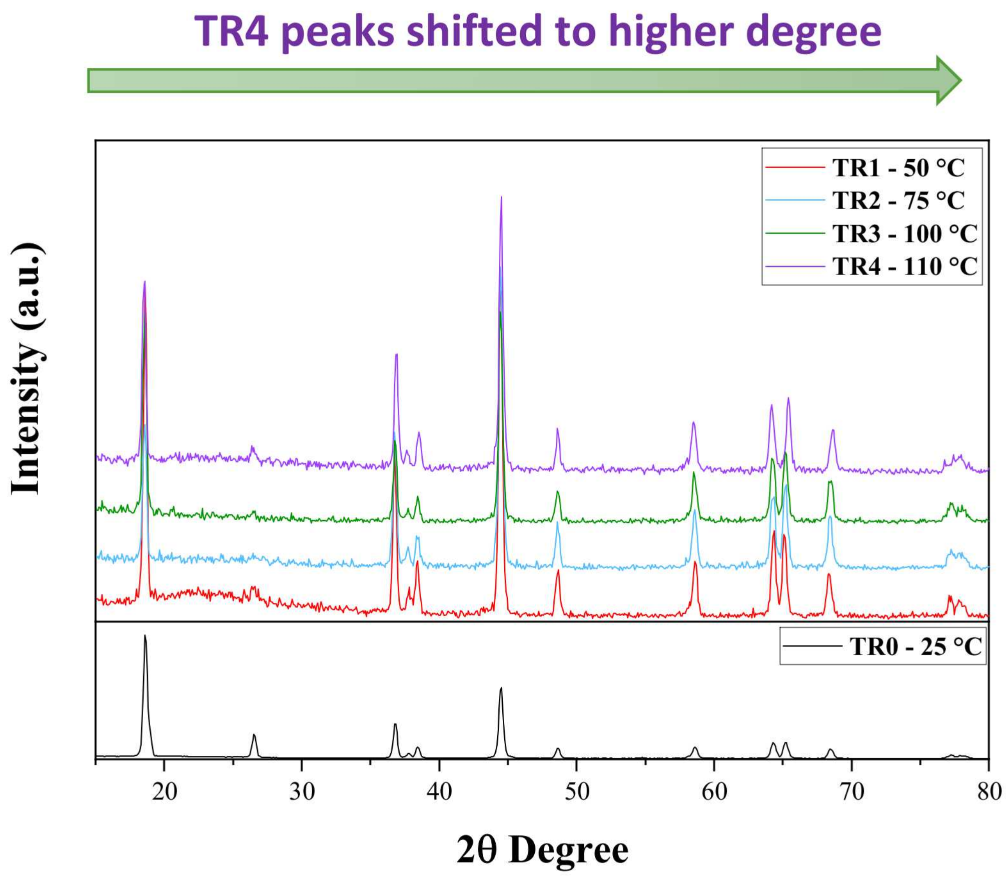
Heated to 75 ˚C

Heated to 100 ˚C

Heated to 110 ˚C

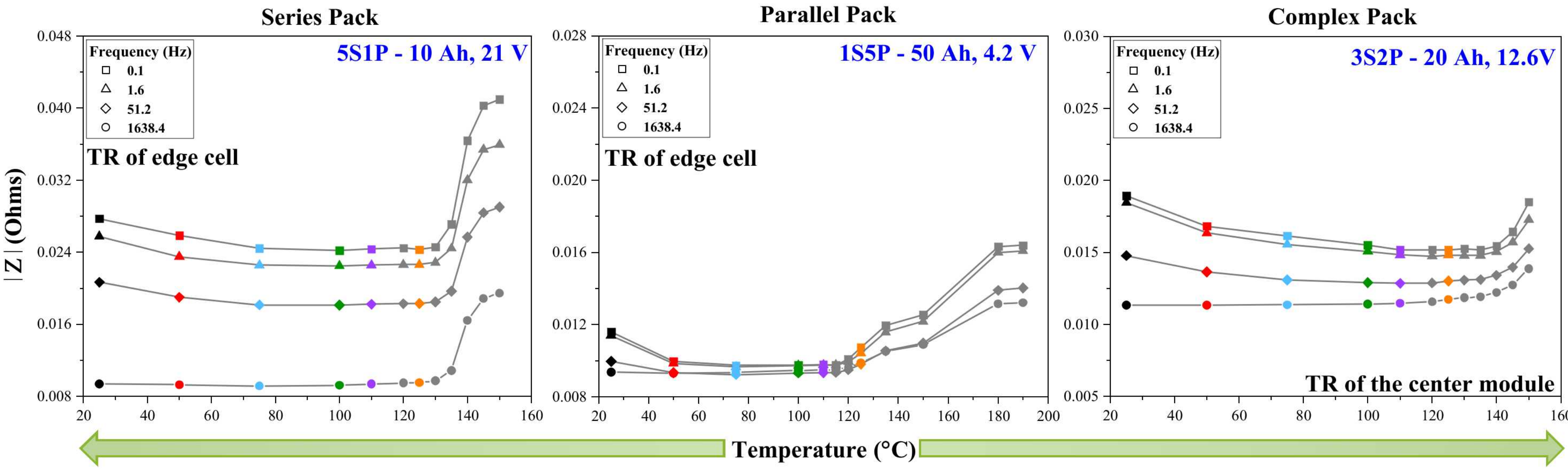


X-ray Diffraction (XRD): Cathode



- XRD diffractograms of the positive electrode revealed changes for T >100 ˚C.
- TR4 diffraction peaks were slightly shifted to higher degree values, indicating a general shrinkage of the lattice.

Identified marker applicable to battery packs in series, parallel or complex configurations?



- *Series Pack:* Signals of cell damage detected at high temperatures (130 ˚C and above).
- *Parallel Pack:* Increases in the scalar impedance were identified at temperatures above 120 ˚C. However, the parallel packs did further limit the detection capabilities.
- *Complex Pack:* At this level of complexity, we begin to see the signal becoming washed out as the resistances detected within the entire pack begin to counteract any behaviors observed due to cell failure

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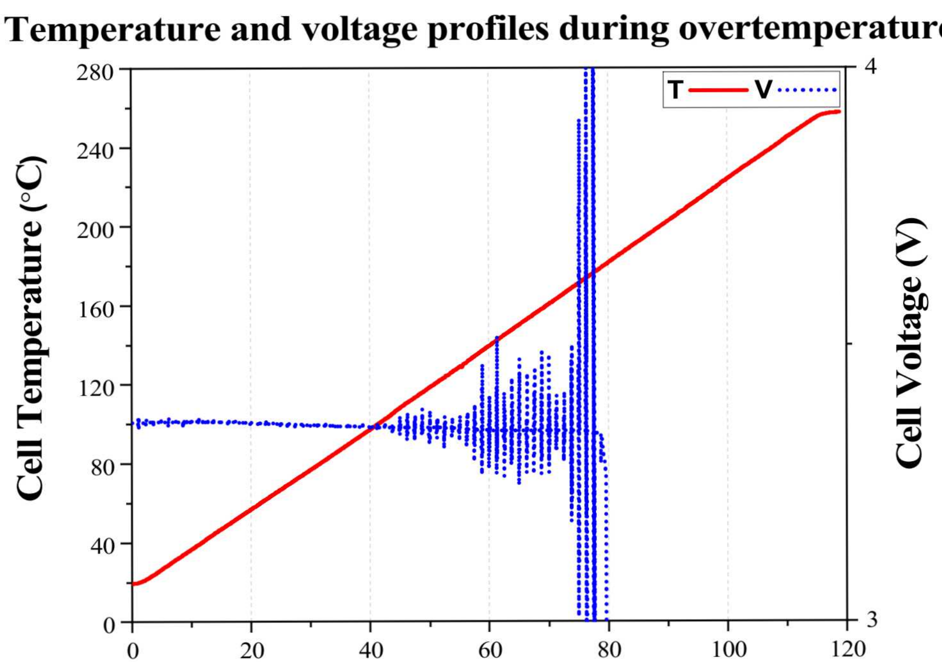
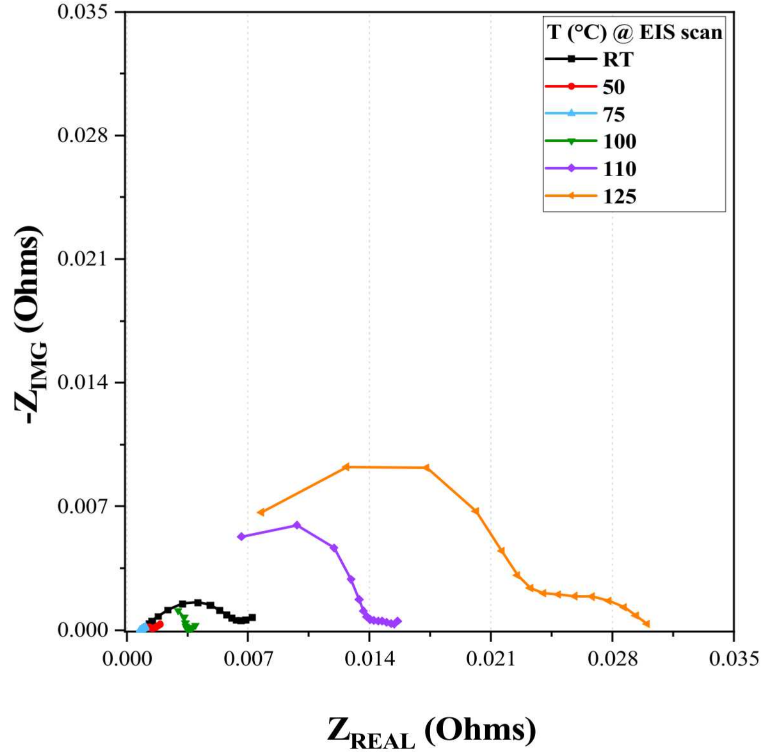
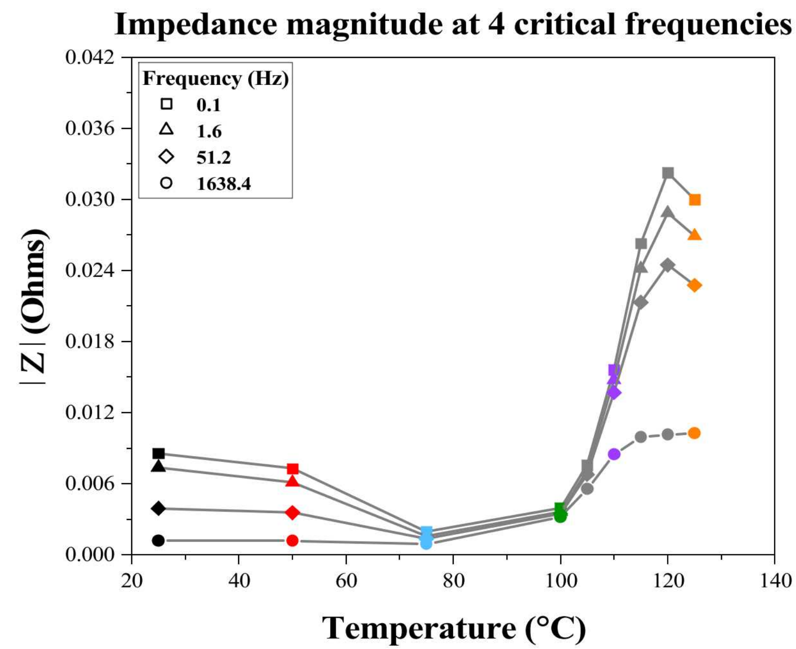


Test parameters	
Heating rate	2 ˚C/min

- There is minimal voltage loss as the cell reaches temperatures > 160 ˚C.
- Identifying these minor changes may be difficult if it occurs within a localized area of a battery pack.

In-operando EIS





$$|Z| = \sqrt{Z_{REAL}^2 + Z_{IMG}^2}$$

- The complex impedance (left) shows that significant changes are observed between 75 ˚C and 100 °C, and past that point the damage is evident with the rise of the solid electrolyte interface resistance, and charge transfer resistance .
- The scalar impedance (right) shows that all frequencies start exhibiting changes at temperatures above 80 °C, and past 100 °C the IMB tool delivered a strong detection signal.

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Summary

- Overtemperature procedures were applied to 10 Ah NMC and 10 Ah LFP single cells.
- The electrochemical and structural characterization exhibited major changes for temperatures >100 ˚C.
- EIS, dQ/dV and XRD results suggest that NMC cells will be operational with minimal changes observed up to 75 ˚C. LFP cells displayed significant degradation for temperature > 80 ˚C.
- In the near future, we will investigate further the microstructural and structural changes of the electrodes.

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