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MATERIALS BENCHMARKING ACTIVITIES FOR The Cell Analysis, Modeling, and Prototyping (CAMP) FACILITY

Project ID: BAT028

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OVERVIEW

Timeline

- Start – Oct. 1st, 2014
- Finish – On-going

Budget

- Total project funding (as part of CAMP effort)
  - FY2025: $600K
  - FY2026: $600K
- 100% DOE

Barriers

- Development batteries that meet or exceed DOE/U.S. DRIVE goals
  - Good performance
  - Lower cost
  - Safety

Partners and Collaborators

- Coordinated effort with many DOE-CMEI-TTO projects, especially BAT167, BAT470, BAT670
- INL, LBNL, NLR, ORNL, PNNL, SLAC, SNL
- Argonne : MERF, BatPaC, APS, CNM, EADL, PTF
- See collaboration list in separate slide

RELEVANCE

- An overwhelming number of materials and technologies are being marketed/reported to improve lithium-ion batteries, which need to be validated for their impact on transportation applications.
- CAMP Facility was established at Argonne to provide a realistic and consistent evaluation of candidate materials. In order to better utilize the sophisticated equipment and expertise effect, small benchtop slurry and electrode preparation, coin cell fabrication and testing are needed to determine if the battery materials warrant further consideration.
- The benchmarking (validation) activities can also provide an objective opinion to material developers. Moreover, better understanding of active materials at the cell level will speed up material development efforts.

FY26 MILESTONES AND ACCOMPLISHMENTS

Milestone	End Date	Type	Status
Coat free-standing solid-state composite electrolyte films using reverse comma coater. (**BAT030**)	1/31/2026	Quarterly Progress	Completed
Validate advanced battery materials for transportation use ➢ Test cobalt free Lithium Manganese Rich-Nickle Manganese oxide (LMR-NM) and $LiFePO_4$ (LFP) from MERF ➢ To benchmark $NaFeMnO_2$ cathode and Hard Carbon of Na ion batteries	06/31/2026	Quarterly Progress	On schedule

Accomplishments

- Free-standing solid-state composite electrolyte (SSE) film were coated on Toray's release film, and all solid-state battery (ASSB) were fabricated and tested in both coin cells and pouch cells.
- Complete initial performance testing of lithium manganese-rich nickel manganese oxide (LMR-NM, **BAT167**) and $LiFePO_4$ (LFP, **BAT470**) scaled up by MERF. High quality electrodes were coated using reverse-comma coater in dry room. The electrodes are being further investigated.
- Sodium-ion battery cathode electrochemical performance and thermal properties were characterized.

APPROACH AND STRATEGY

- Collaborate with material developers and leverage Argonne's expertise in electrode design and cell testing.
- Cell materials that have an impact on the cell performance will be considered for testing, mainly in terms of:
  - Electrochemical performance
  - Electrode optimization
  - Thermal stability
- The electrochemical performance will be validated using 2032 coin type cells under test protocols derived from U.S. DRIVE transportation requirements. If promising, pouch cells will be fabricated and tested.

U.S. DRIVE Requirements of Energy Storage Systems for Transportation

Characteristics at EOL (30°C)	Unit	Cell level
Peak discharge Power Density (30 Sec)	W/L	1500
Peak Specific discharge Power (30 Sec)	W/kg	700
Peak Specific Regen Power (10 sec)	W/kg	300
Available Energy Density @ C/3 discharge rate	Wh/L	750

Test Protocol development

Electrochemical test protocols, such as formation, C-rate and cycle life, were established for half and full cells according to transportation requirements.

OBJECTIVES

- To identify and evaluate cell chemistries and technologies that can simultaneously meet the following criteria for transportation applications:
  - Electrochemical performance
  - Abuse tolerance
  - Cost
- To support the CAMP Facility for prototyping cells and electrode library development.
- To enhance the understanding of advanced cell components on the electrochemical performance and safety of battery system.

See BAT030

R2R SOLID-STATE ELECTROLYTE PROCESS

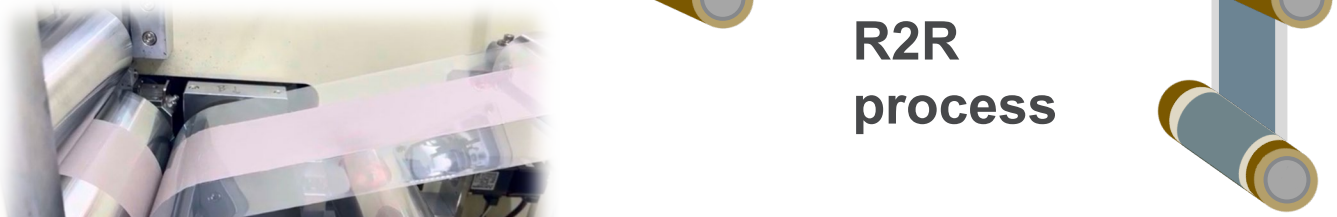
LLZO/LiTFSI/PVDF composite film

- LLZO, with high Li-ion conductivity and compatible with Li metal anode, relies on brittle, high-temperature sintered ceramics that are difficult to scale into thin, large-area films.
- Composite electrolyte design combines ceramic conductivity/mechanical strength with polymer flexibility/processability.
- Continuous, high-throughput R2R process will address the manufacturing challenge.



LLZO SSE composition

Component	Ratio (wt%)
Al-LLZO	64%
Polymer-LiTFSI	31%
PVDF	5.0%



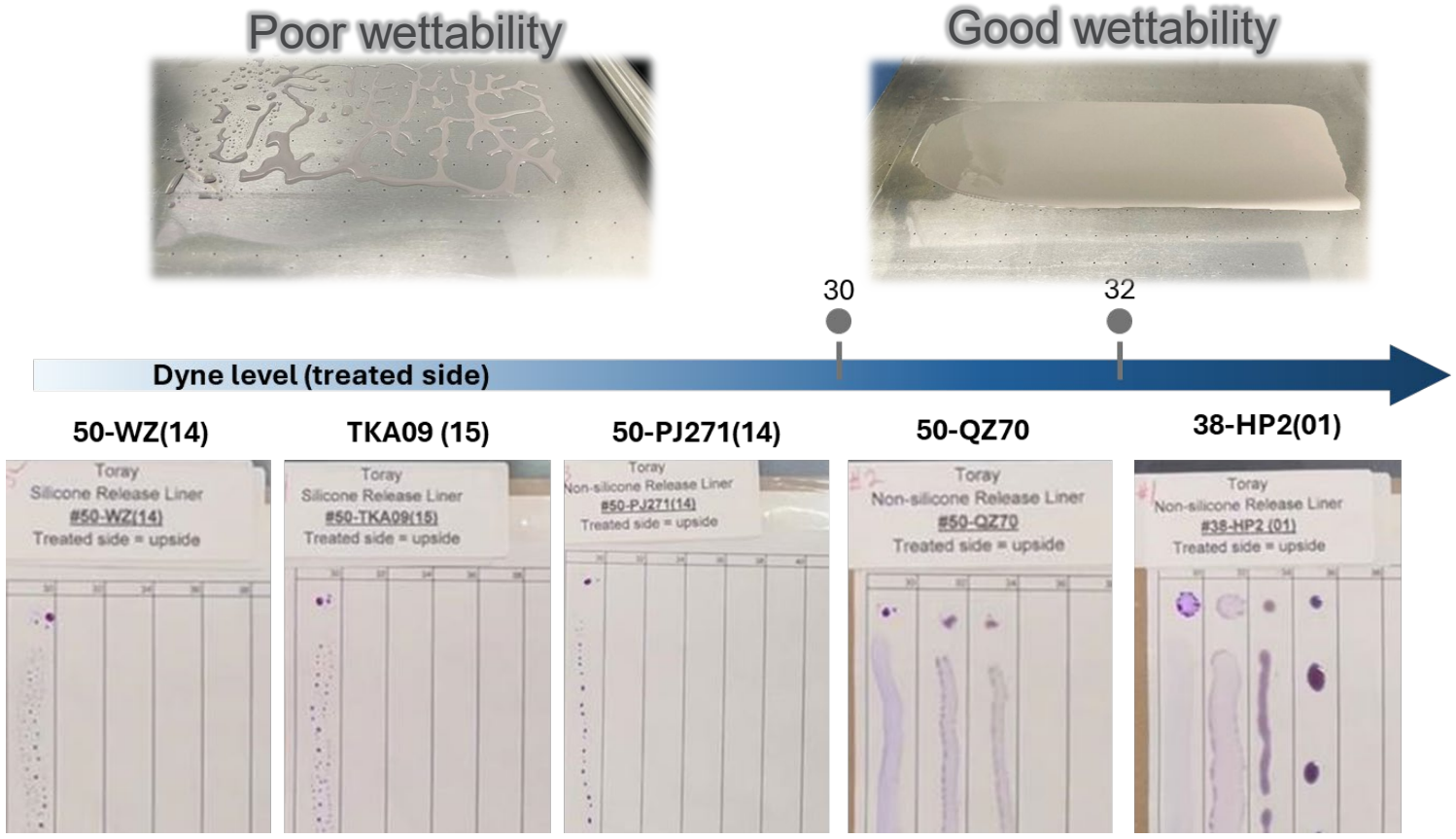
- R2R LLZO composite film is successfully coated using pilot coater at the CAMP facility.

TORAY

DYNE TEST OF RELEASE FILM

Dyne level ≥ 30 is required for good film formation

- To enable R2R process, an ideal substrate for the manufacturing free standing thin-films should have good wettability and low releasing force.
- Toray provided release films with various release forces.
- Dyne testing is conducted to determine the minimum surface tension of a specific liquid mixture that allows it to spread uniformly on the substrate.



- Slurry demonstrates good wettability on the substrate with Dyne level ≥ 30.

Courtesy to **Toray Industries, Inc.**

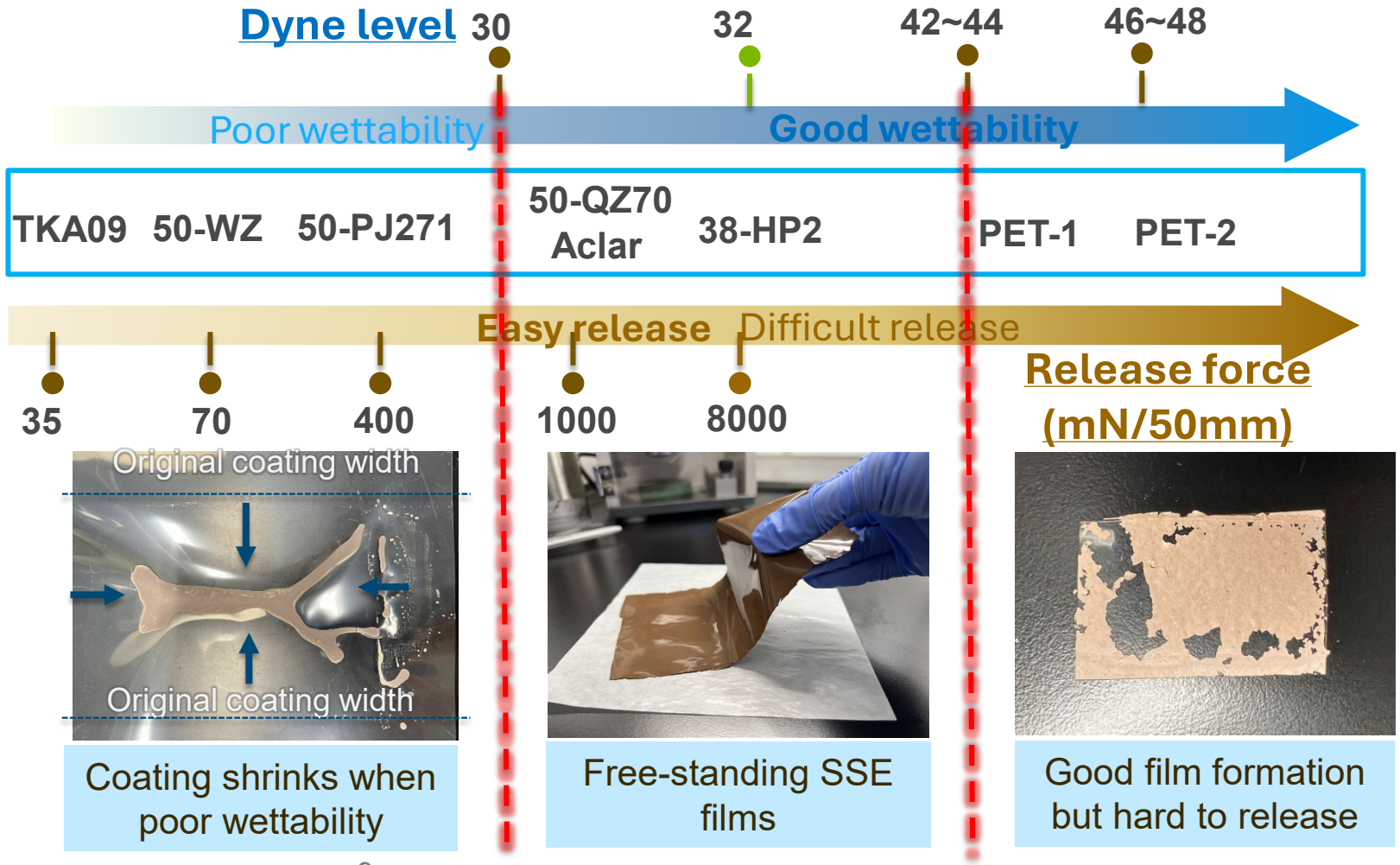
TORAY

BALANCE FILM FORMATION AND RELEASE

Free standing film obtained using 8000 mN/50mm release film

- Direct correlation between Dyne level and release force is observed.
- Good film formation with Dyne level ≥ 30.
- Nicely formed film can't be released when Dyne level is > 44.

SSE composition	
Component	**Ratio (wt%)**
Al-LLZO	64%
Polymer-LiTFSI	31%
PVDF	5%

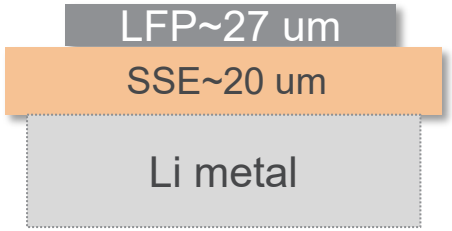


Courtesy to **Toray Industries, Inc.**

ASSB ASSEMBLY AND PERFORMANCE

Ultra thin SSE (30μm) was fabricated and tested in coin cell

Cell Architecture

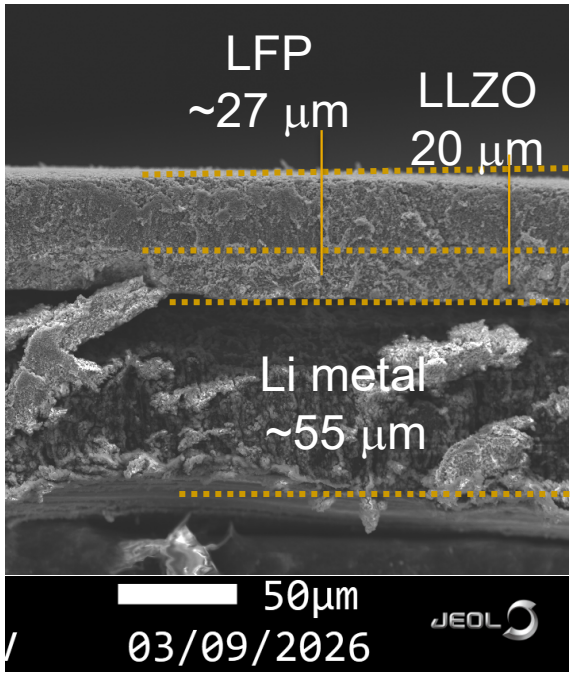


Free standing $LiFePO_4$ composite electrode containing LiTFSI salt was prepared using Toray release film.

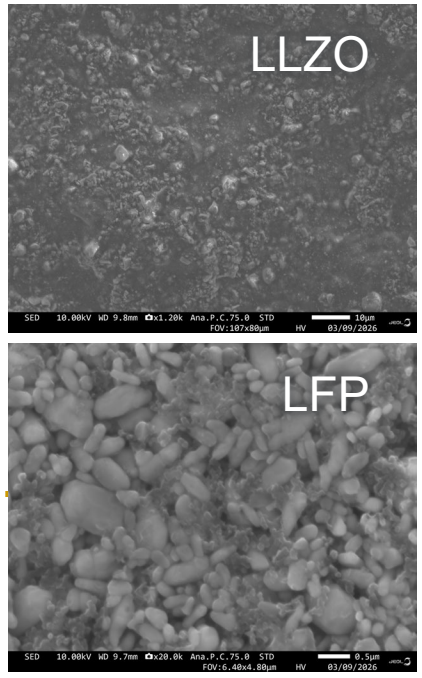
LLZO and LFP films were calendered to form bilayer.

Li metal was used as anode to fabricate Li/LLZO/LFP coin cell.

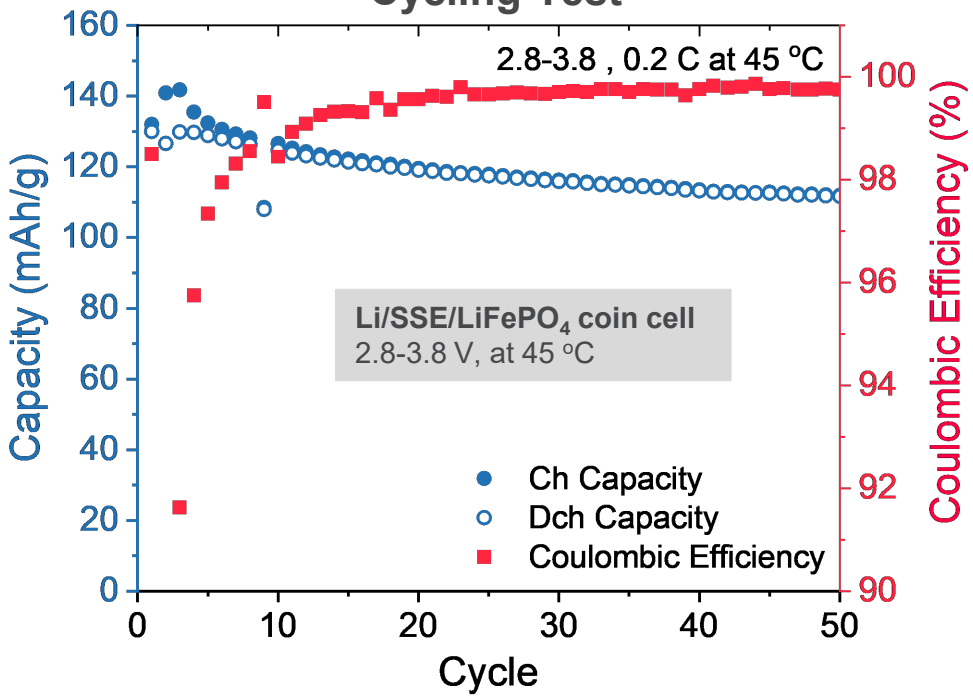
Cross-section



Top view



Cycling Test



- Good cycle performance was obtained for Li/LLZO/LFP ASSB (coin cell).
- CAMP continued pouch cell fabrication and testing. See BAT030 for more information.

MERF AND CAMP COLLABORATION

From Material Scale-Up to Pouch Cell Testing via Benchmarking

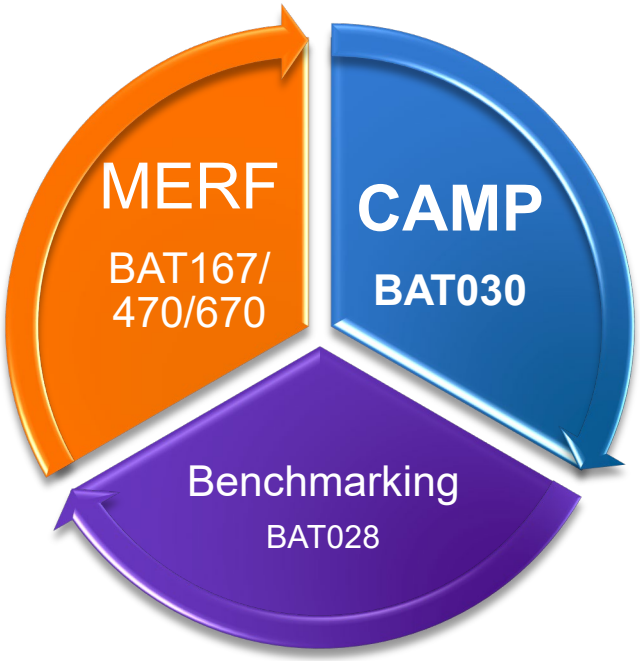
Materials Engineering Research Facility (MERF)

- Scale-up various battery materials from grams to kilograms
- Process development for larger-scale production

BAT167, Youngho Shin

BAT470, Ozge Kahvecioglu

BAT670, Feng Wang/Joseph Libera



- Before high quality electrode and pouch cell fabrication at CAMP, a bench-scale electrode will be casted and benchmarked to evaluate the scaled-up material performance.

Cell Analysis Modeling and Prototyping (CAMP) Facility

- Dry room environment
- Various mixing equipment
- Pilot coater for high quality electrode
- Pouch cell fabrication/testing

BAT030, Steve Trask

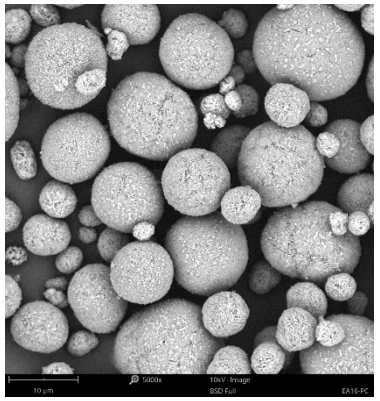
See BAT167 & BAT030

MERF AND CAMP COLLABORATION ON LMR-NM

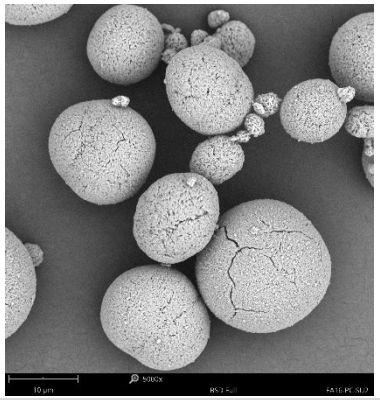
Pre-pilot electrode coating

- Around 100g well controlled dense cobalt free Lithium and Manganese Rich Nickel Manganese Oxide (LMR-NM) particles were produced by MERF using TVR reactor and LiOH salt (see **BAT167**).
- High loading (2.7 mAh/cm^2) and dense (2.8 g/cm^3) electrode was made using bench-scale coater and was tested using coin cells.
- The information was transferred to the CAMP facility for pilot electrode coating and further investigated (see **BAT030**).

P-CAM



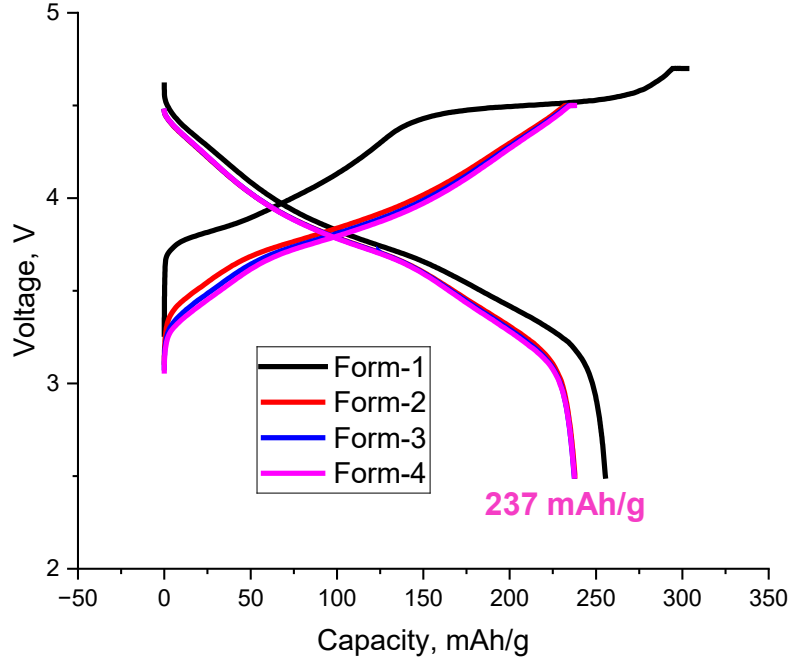
CAM



$Li_{1.35}Mn_{0.63}Ni_{0.37}O_{2+x}$

LMR/CB/PVDF = 90/5/5
Active loading: 11.2 mg/cm^2
Coating: 43 μm
Total density: 2.9 mg/cm^3

Cha/Dis: 0.2 mA (C/20)
Form 1: 2.5 – 4.7V
Form 2-4: 2.5 – 4.5V
Last cycle: 2.66 mAh/cm^2



- The LMR-NM half cell delivers 237 mAh/g reversible capacity between 2.5 – 4.5V, with initial Coulombic efficiency of 84.5%

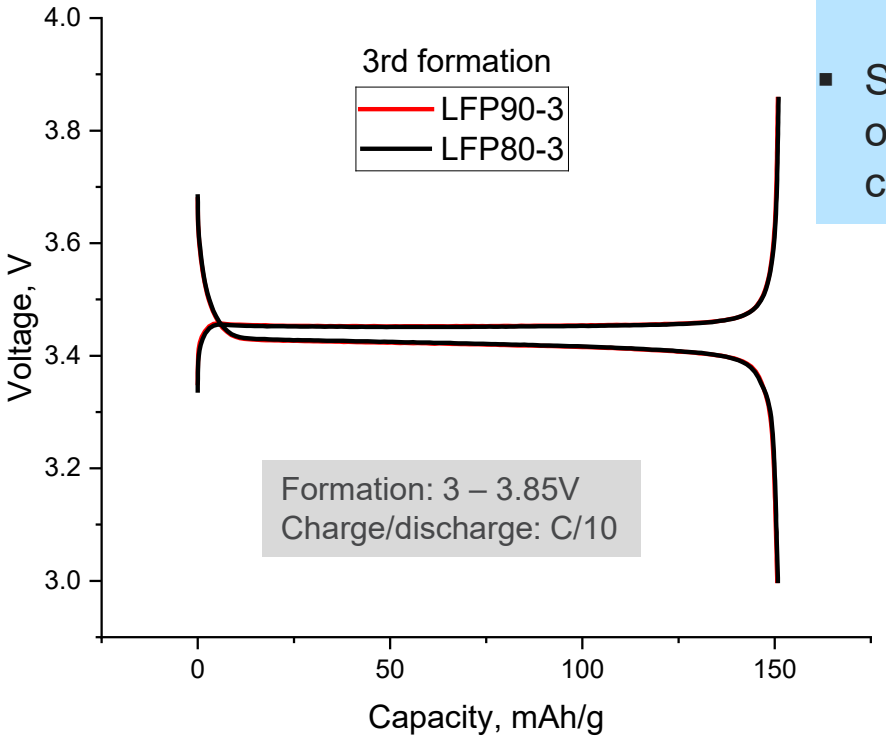
See BAT470

MERF AND CAMP COLLABORATION ON $LiFePO_4$

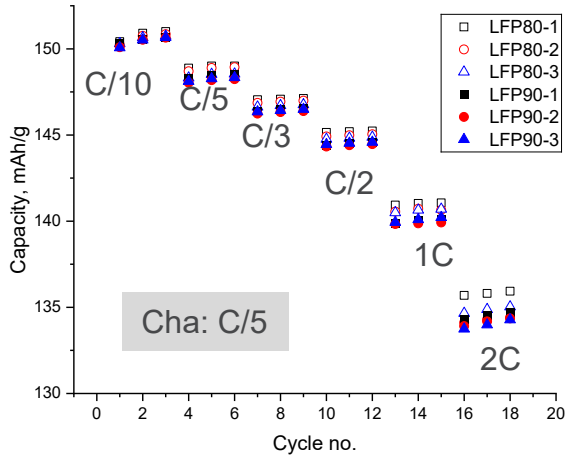
LFP electrode composition investigation

- Around 100g carbon coated $LiFePO_4$ (LFP) produced by MERF(see **BAT470**) using hydrothermal reactor.
- A small controlled bench-scale electrode coating is conducted to make sure the slurry and coating process are stable enough to scale.
- The resulting LFP electrode was tested using coin cells.

Electrode information				
	LFP	CB	PVDF	mAh/cm^2
LFP80	80	10	10	0.84
LFP90	90	5	5	1.01



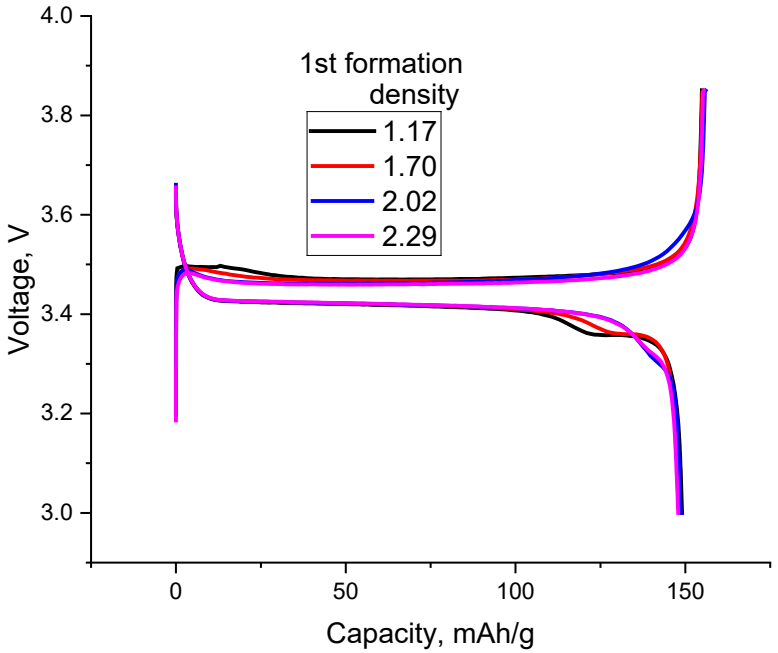
- Two electrode compositions with 80% and 90% LFP active materials show identical performance with
  - 150 mAh/g reversible capacity and
  - 96% initial Coulombic efficiency.
- Slightly better rate performance was observed for LFP electrode with more carbon black.



See BAT030

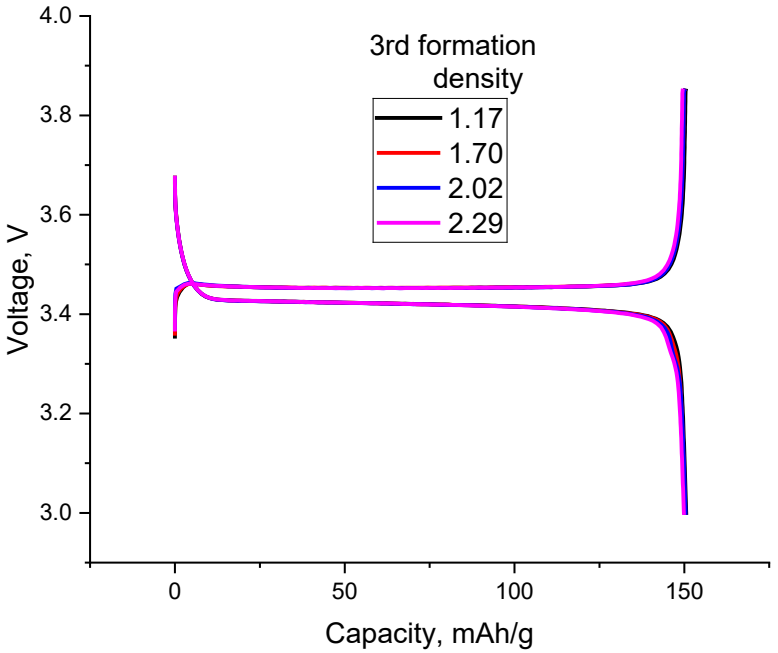
CALENDER EFFECT ON $LiFePO_4$

LFP electrode fabrication using CAMP pilot coater



CAMP electrode				
	LFP	CB	PVDF	mAh/cm^2
CAMP	92	4	4	1.9

Calendered electrode	
Thickness, μm	Density, g/cc
118	1.17
81	1.70
68	2.02
60	2.29

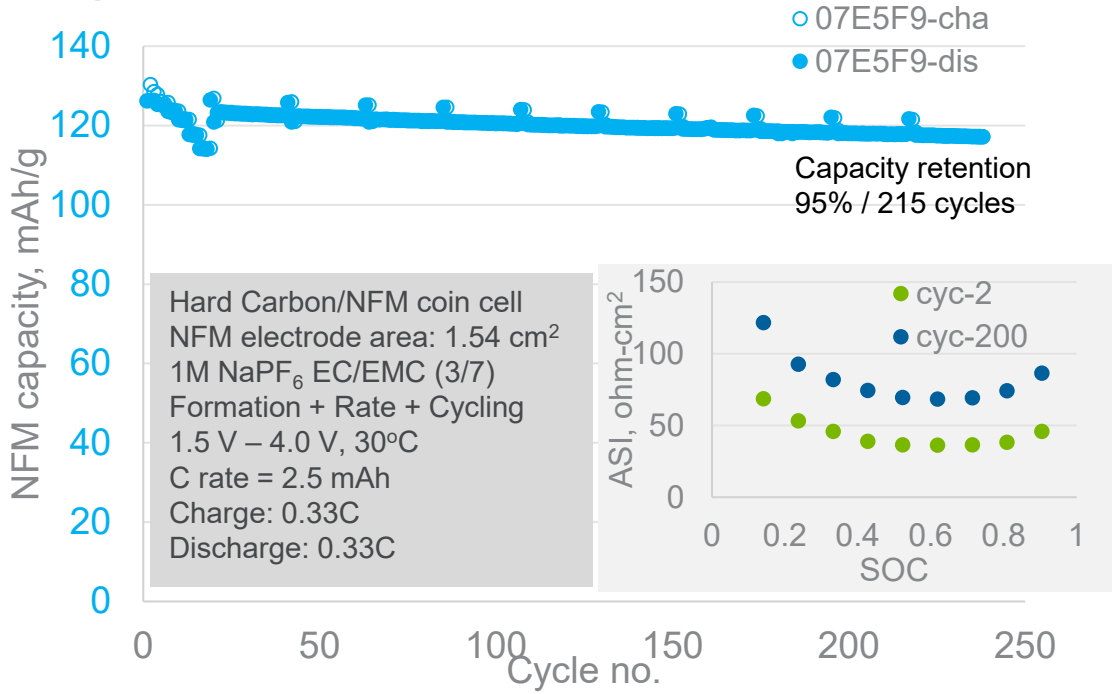


- The CAMP facility made higher LFP content and higher loading electrode (1.9 mAh/cm^2). The CAMP electrodes were calendered to various densities (see **BAT030** for more).
- The CAMP electrode shows identical reversible capacity of 150 mAh/g as hand made electrodes.
- Electrode calendering has little impact on reversible capacity. However, some voltage variations were observed at beginning of charge and end of discharge.

BENCHMARKING $NaNiFeMnO_2$ (NFM)

95% capacity retention after 200 cycles.

- NFM consisting of abundant and lower-cost elements can deliver competitive capacity with relatively high operating voltage, which makes it a practical cathode candidate for sodium ion batteries.
- Sodium-ion batteries are often viewed as a strong candidate for low-temperature and fast-charging applications, especially relative to graphite-based Li-ion limitations.
- This work will benchmark NFM performance, which is necessary to identify bottlenecks and accelerate sodium-ion battery development.



- HC/NFM full cell shows excellent cycling performance, with 95% capacity retention after 215 cycles.
- The lower reversible capacity of NFM (125 mAh/g) is due to low initial Coulombic efficiency of hard carbon anode.
- HC/NFM full cell has impedance of >36 ohm-cm², which is higher than that of Gr/NMC cells. After 200 cycles, the impedance almost doubled.

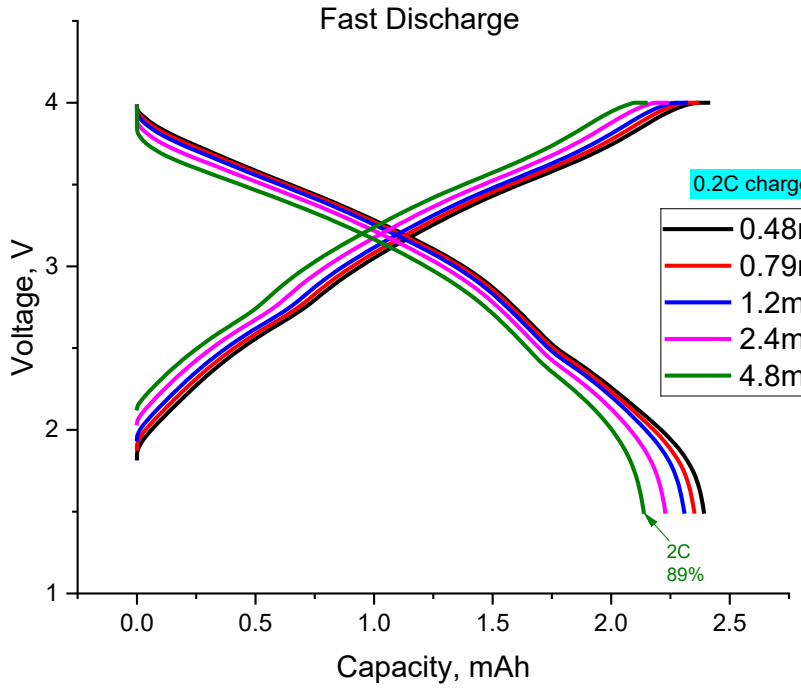
RATE CAPABILITY OF NFM

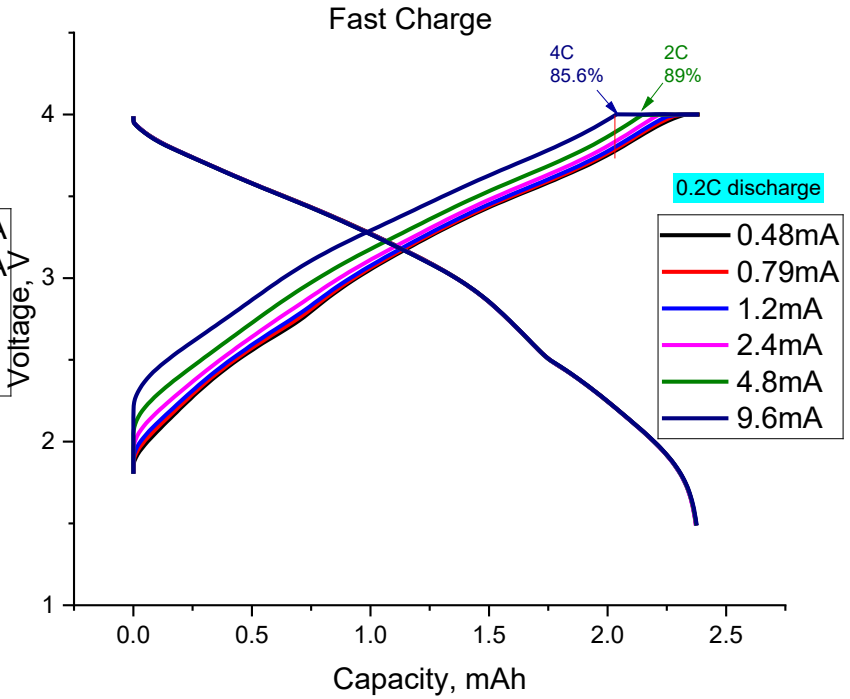
90% capacity retention with 2C discharge, and 85% capacity can be charged in 12 min

HC/NFM cell,
1.0 M $NaPF_6$ EC/EMC/DMC
1.5V – 4.0V
30°C

Current, mA	C rate
0.48	C/5
0.79	C/3
1.2	C/2
2.4	1C
4.8	2C
9.6	4C

- Good discharge rate performance, 90% capacity retention at 2C rate, was observed for HC/NFM cells.
- HC/NFM cell shows excellent fast charging capability (85% at 4C).



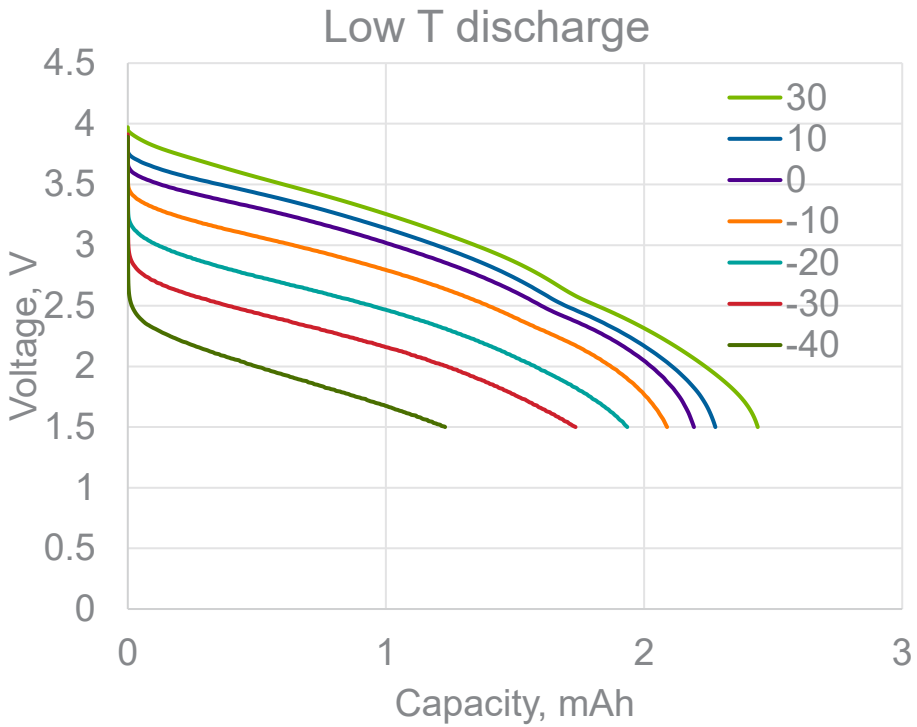


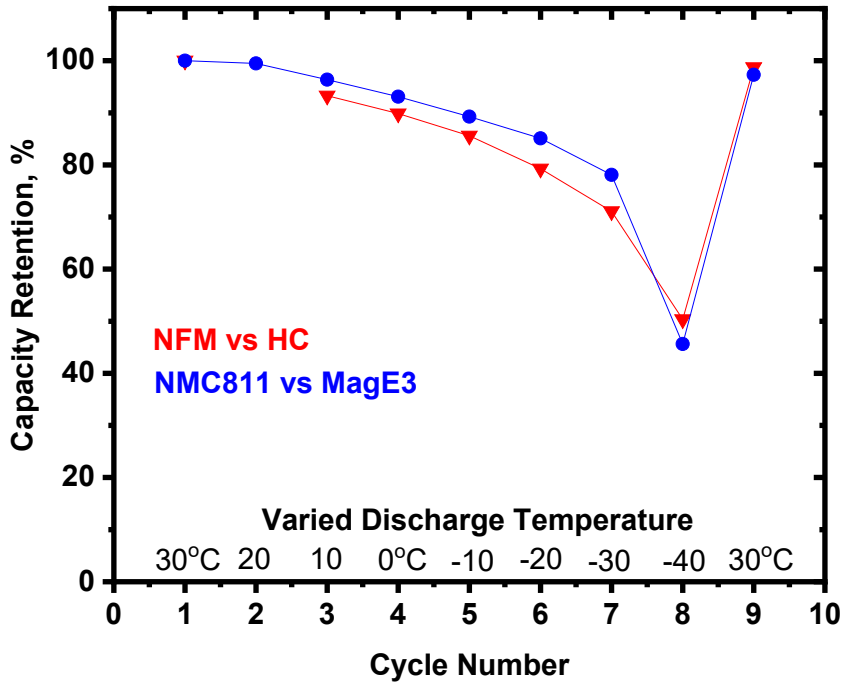
LOW TEMPERATURE PERFORMANCE

Similar low temperature discharge performance as LIB

HC/NFM cell,
1.0 M $NaPF_6$ EC/EMC/DMC
C rate: 2.4 mAh
Charg: C/3 at 30°C
Disch: C/3 at various Temp.

- Around 80% discharge capacity can be delivered at -20°C for HC/NFM cell.
- When discharged at -40°C, There is a 50% capacity drop.





THERMAL SAFETY STUDY OF Na ION BATTERY

Mixed thermal behavior of NFM and hard carbon

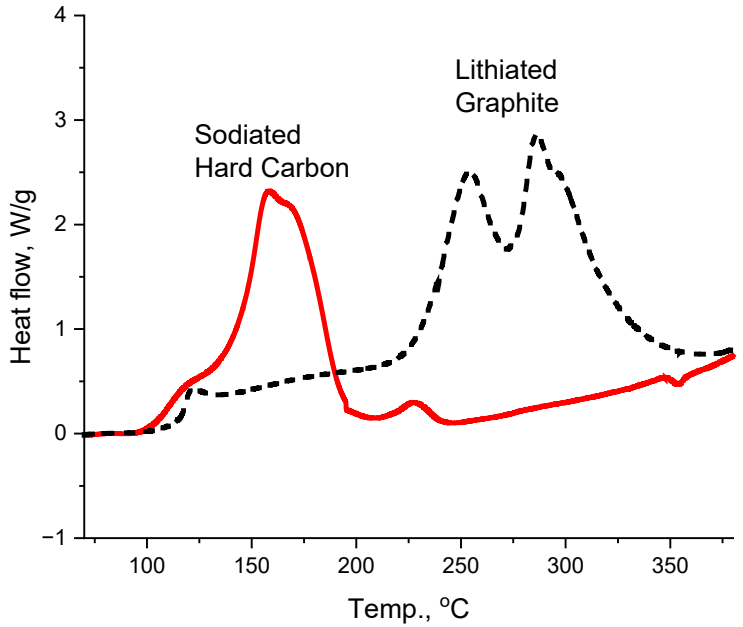
Sodiated Hard Carbon: 5 mV
Charged NFM: 4.0V
Electrolyte: 1M $NaPF_6$ in EC/EMC/DMC

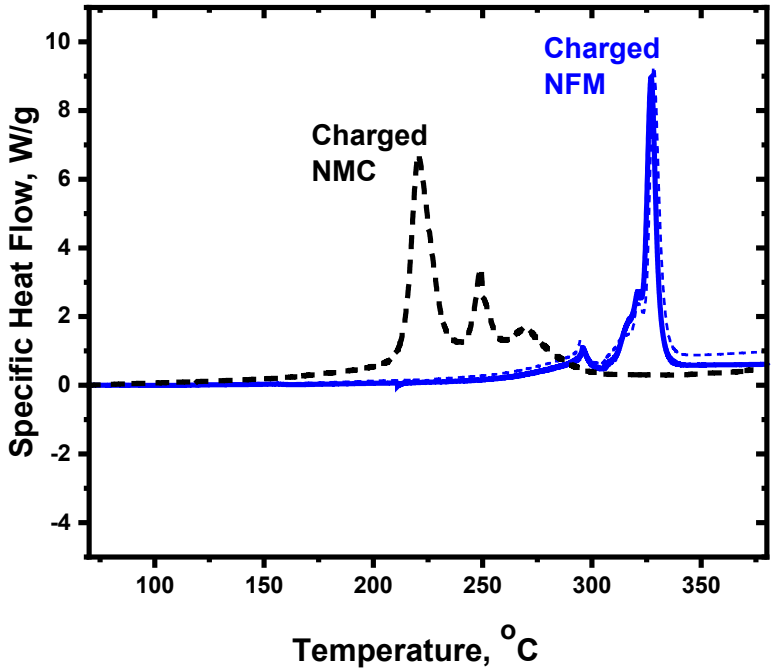
Lithiated graphite: 10 mV
Charged NMC: 4.3V
Electrolyte: 1.2M $LiPF_6$ EC/EMC

DSC scan rate: 5°C/min

DSC 6000 (Perkin-Elmer)







- Charged HC and graphite have similar on-set temperature. However, sodiated HC has more heat generation than graphite before 200°C.
- Charged NFM has much higher on-set temperature of >270°C than NMC of ~200°C.

RESPONSE TO PREVIOUS YEAR REVIEWERS' COMMENTS

All reviewers felt that the project was relevant to current DOE objectives but had split opinion on resources.

Answer: To mitigate potential constraints, we will work with the sponsor to prioritize tasks and allocate resources to the most critical activities, ensuring that key project objectives remain achievable.

The reviewers would like to see more collaboration activities.

Answer: We have an established and active collaboration with MERF on material scale-up. In this workflow, MERF scales up the material, our team evaluates electrochemical performance and provides feedback to improve particle properties for slurry and electrode preparation, and CAMP conducts performance testing in large-format pouch cells. This coordinated process supports efficient iteration and strong integration across the project.

FY25 – FY26 COLLABORATIONS

- The partners and collaborators include
  - National labs: ANL, INL, LBNL, NLR, ORNL, PNNL, SLAC, SNL, SRNL
  - Universities: Northern Illinois University, University of Arkansas, University of Delaware, Western Michigan University, The George Washington University
  - Industries: Ashland, Automat Solutions, Blue Ocean and Black Stone, Chasm Advanced Materials, Conovate, Greenchem Holdings, Orbia, OCSiAl, Oxbow, Superior Graphite Co., Targray, Toda Kogyo, Toray Industries, Inc., Zeta Energy
- Work with MERF on cathode material scale up (BAT167, BAT470, BAT670)
- The CAMP Facility is open to work with industries to advance the LIB technologies for transportation application.

REMAINING CHALLENGES AND BARRIERS

- Cathode development of ASSB remains a major challenge, as achieving stable solid-solid interfacial contact, high ionic/electronic conductivity, and compatibility with the electrolyte is difficult during repeated cycling.
- As for advanced battery materials, variability in material quality, availability, and preparation methods can affect benchmarking results and make reproducibility harder.

FUTURE RESEARCH

- Continue research on all solid-state batteries (ASSB), focusing on composite electrolyte optimization, and cathode electrode fabrication.
  - Dry processing will be explored to mitigate solvent compatibility issues. This approach may help preserve solid electrolyte integrity.
  - LFP and NMC cathode with SSE will be optimized and fabricated.
  - Optimize solid polymer electrolyte composition and fabrication process
- Work with MERF on material scale up:
  - $LiMnFePO_4$ (**BAT470**) and doped LMR-NM (**BAT670/BAT167**).
- Continue to acquire and characterize battery materials to enable high-energy lithium and sodium batteries.
  - Advanced active materials, electrolytes/additives, binders, are of interest.
- Continue to work closely with research institutions and industrial suppliers to enable the battery technology for transportation applications.

Any proposed future work is subject to change based on funding levels.

SUMMARY

- Release film with optimum release force and Dyne level enabled free-standing R2R solid state composite electrolyte film fabrication.
- Collaboration with MERF supported validation of their $LiFePO_4$ and LMR-NM cathode materials.
  - CAMP continued work on higher-loading, higher-quality electrode fabrication and testing.
- Sodium-ion battery NFM materials were benchmarked for rate capability, fast charging, low-temperature, and thermal stability.
  - HC/NFM cell shows improved fast charging capability than lithium-ion batteries.
  - NFM showed much better thermal stability, while hard carbon remained a limitation.

ADDITIONAL CONTRIBUTORS AND ACKNOWLEDGMENTS

Argonne

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Outside Argonne

- Wang, Jason (Automat Solutions)
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- Xu, Jun (UD)
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- Wu, Qingliu (WMU)
- Superior Graphite Co (SGC)
- Bo Liu (Toray, Japan)

Facilities

- Advanced Photon Sources (APS)
- Battery Manufacturing Facility (ORNL)
- Center for Nanoscale Materials (CNM)
- Material Engineering Research Facility (MERF)
- Post Test Facility (PTF)

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