BAT456

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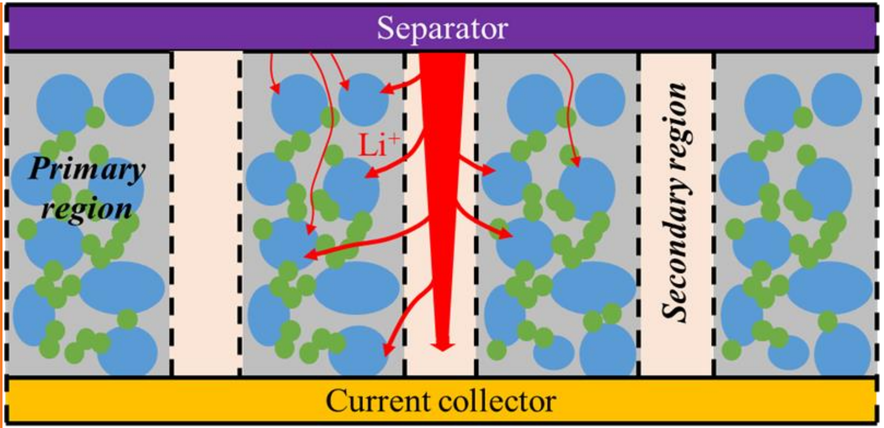
XCEL
eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

ELECTRODE STRUCTURES AND ELECTROLYTES THAT ENHANCE FAST CHARGE



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U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy
VEHICLE TECHNOLOGIES OFFICE
Argonne NATIONAL LABORATORY · SLAC · NREL · BERKELEY LAB · INL Idaho National Laboratory · OAK RIDGE National Laboratory

OVERVIEW

Timeline

- Start: October 1, 2017
- End: September 30, 2021
- Percent Complete: 94%

Barriers

- Cell degradation during fast charge
- Low energy density and high cost of fast charge cells

Budget

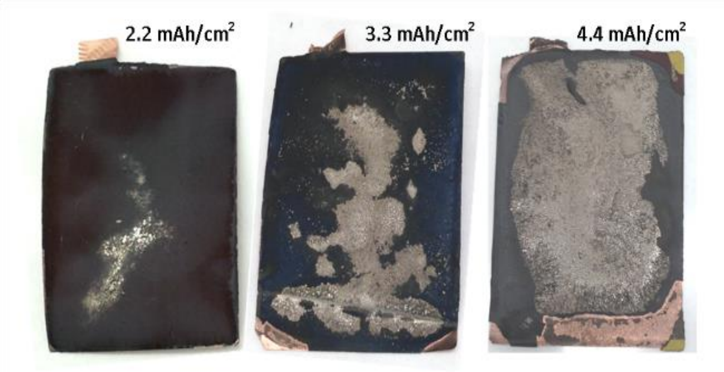
- Funding for FY20 – $5.6M

Partners

- Argonne National Laboratory
- Idaho National Laboratory
- Lawrence Berkeley National Lab
- National Renewable Energy Laboratory
- SLAC National Accelerator Lab
- Oak Ridge National Lab

RELEVANCE

- Fast charging at rates over 2C can result in lithium plating on typical negative electrodes, which is more likely with thicker electrodes and lower temperature.
- High tortuosity in the electrodes and low electrolyte conductivity are likely to favor lithium plating.
- Carefully selected design of experiments that include modeling, fabrication and testing of prototype cells, and post-test diagnostics are needed to develop a cell system that minimizes the possibility of lithium plating in EV batteries.



Gallagher, *et al., JES* 2016

2020-2021 MILESTONES

Related milestones in XCEL – Electrode & Electrolyte Thrust

Milestone	End Date	Status
Identify & optimize best anode composition and architecture	6/30/2020	Completed
Identify & optimize best electrolyte composition and accompanying formation process	6/30/2020	Completed
Fabricate 24 pouch cells using best anode and electrolyte	8/30/2020	Completed
Estimate cost of fast charge designs using BatPaC	9/30/2020	Completed
Produce 10 meters of advanced design electrodes	12/30/2020	Completed
Fabricate 24 Midterm Pouch Cells with NMC811	3/30/2021	Completed
Provide advanced electrolyte compatible with NMC811 through modeling & testing	6/30/2021	On-Track
Fabricate 24 Final Pouch Cells with advanced design graphite-NMC811 electrodes & advanced electrolyte	7/30/2021	On-Track

OBJECTIVE

- The objective of the XCEL-Electrode & Electrolyte Thrust effort is to design and fabricate electrode architectures and advanced electrolytes that minimize the possibility of lithium plating under fast charge conditions.

APPROACH

- Modeling team (NREL) will predict ideal anode and cathode architectures that prevent anode from going below lithium potentials, and determine effect on energy density. BatPaC Model (Argonne) will be used to estimate impact on cost.
- CAMP Facility (Argonne) and LBNL will fabricate electrodes that best approximate the electrode architectures predicted by modeling effort and assemble cells to validate electrochemical performance. Cells will be made with graphite vs. NMC electrodes with loadings between 2 and 4 mAh/cm^2. Latest advanced electrolytes (INL & NREL) will be utilized and compared to baseline.
- Post-Test Facility (Argonne) will post-mortem cells for presence of lithium plating.

SUMMARY OF RECENT MAJOR POUCH CELL BUILDS

Build Reference	Composition	Thrusts	Comments
FY20 Q4	90% NMC811 92% SLC1506T 2 mAh/cm²	Cathode	Gen2 electrolyte Celgard 2320
FY20 Q4	90% NMC532 92% SLC1506T 3 mAh/cm²	Charge, Heat	Gen2 electrolyte Celgard 2320
FY20 Q4 – FY21 Q1 “Hero”	96% NMC532 96% SLC1506T 3 mAh/cm²	Charge	Gen2 and B26 electrolyte Celgard 2320 & 2500
FY21 Q2	90% NMC811 92% SLC1506T 3 mAh/cm²	Charge, Cathode, Heat	Gen2 and B26 electrolyte Celgard 2320
FY21 Q2 “March Midterm”	97% NMC811 (new) 92% SLC1506T 3 mAh/cm²	Charge, Heat, Cathode	B26 electrolyte Celgard 2500
FY21 Q4 “July Final”	96% NMC811 92% Dual Layer >3 mAh/cm²	Charge, Cathode, Heat	SLC1506T/AET LM2803 Improved electrolyte Celgard 2500

BASELINE ELECTRODES USED IN 2019-2020

Referred to as “Round 2” (3 mAh/cm² anode loading)

Anode: LN3107-190-4A

91.83 wt% Superior Graphite SLC1506T
2 wt% Timcal C45 carbon
6 wt% Kureha 9300 PVDF Binder
0.17 wt% Oxalic Acid

Lot#: 573-824, received 03/11/2016
Single-sided coating, CFF-B36 anode

Cu Foil Thickness: 10 µm
Total Electrode Thickness: 80 µm
Total Coating Thickness: 70 µm
Porosity: 34.5 %
Total SS Coating Loading: 9.94 mg/cm^2
Total SS Coating Density: 1.42 g/cm^3

Made by CAMP Facility

Cathode: LN3107-189-3

90 wt% Toda NMC532
5 wt% Timcal C45
5 wt% Solvay 5130 PVDF

Matched for 4.1V full cell cycling
Prod:NCM-04ST, Lot#:7720301
Single-sided coating, CFF-B36 cathode

Al Foil Thickness: 20 µm
Al Foil Loading: 5.39 mg/cm^2
Total Electrode Thickness: 91 µm
Coating Thickness: 71 µm
Porosity: 35.4 %
Total Coating Loading: 18.63 mg/cm^2
Total Coating Density: 2.62 g/cm^3

Made by CAMP Facility

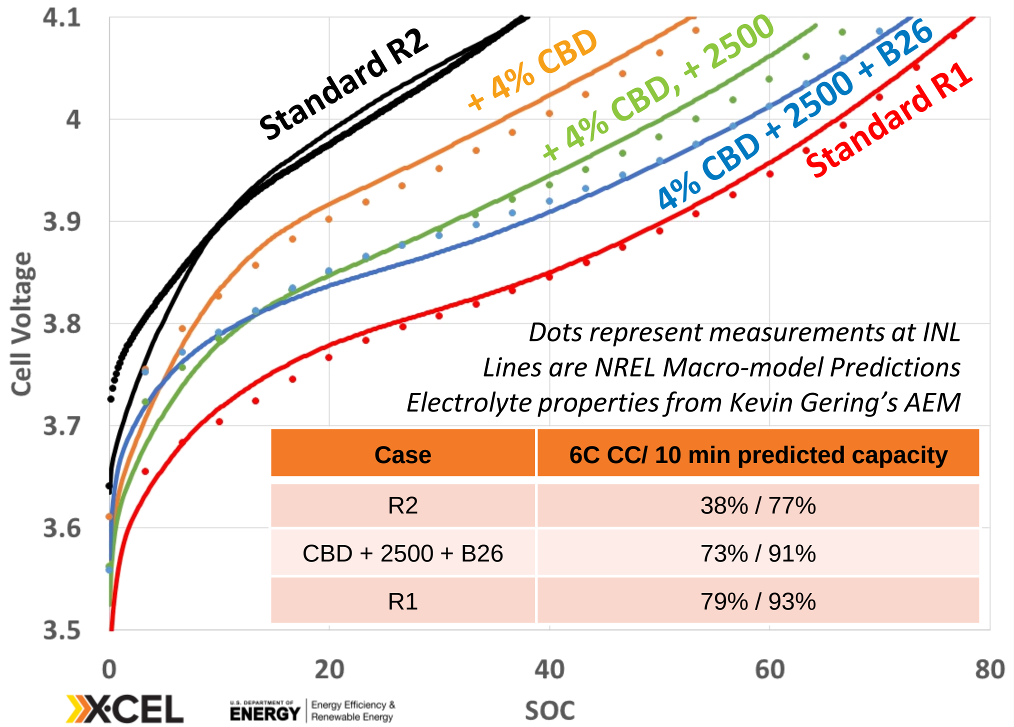
“Round 1” has same composition, but 2 mAh/cm² anode loading

Round “1” & “2” nomenclature for other systems refer to loading only

COMBINED APPROACH ALLOWS FOR HIGHER LOADING

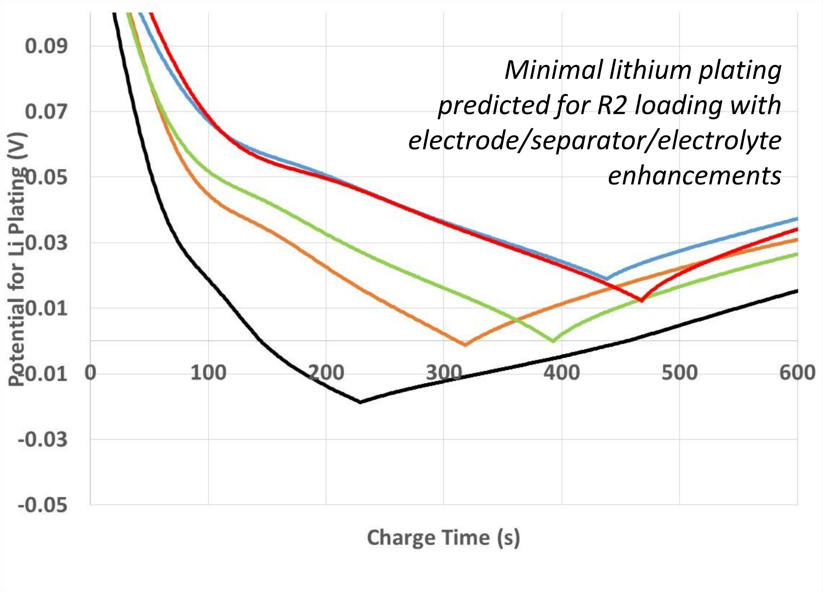
Reduced CBD loading + Celgard separator 2500 + enhanced electrolyte

6C CC Charging of Single Layer Pouch Cells



Case	6C CC/ 10 min predicted capacity
R2	38% / 77%
CBD + 2500 + B26	73% / 91%
R1	79% / 93%

6C CCCV to 4.1 V (10 minutes cutoff)

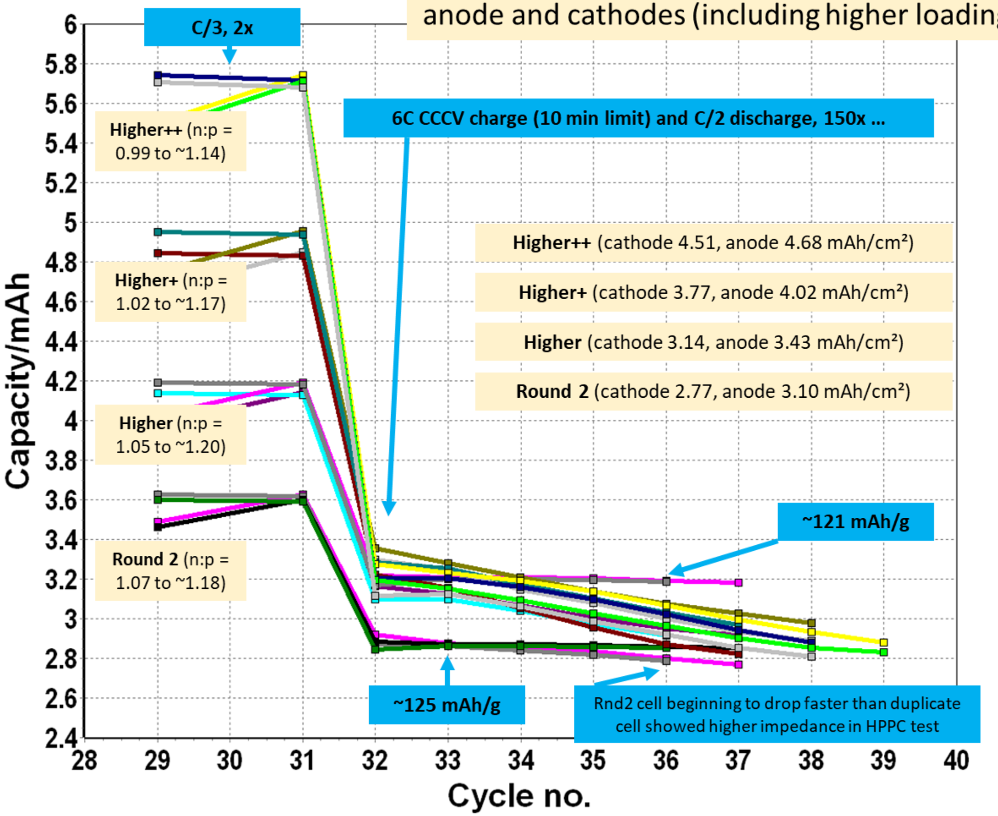


- R2 loading now almost behaving like R1
- Model predictions match exp. data

ANL

NMC811 ELECTRODES WITH LOWER CARBON AND BINDER

Full cell (coin cell) data using XCEL initial mid-term anode and cathodes (including higher loading pairs)



Cathode: LN3237-100-_

(single-sided)

97 wt% Targray NMC811

1.5 wt% Timcal C45

1.5 wt% Solvay 5130 PVDF Binder

XCEL, 2021 Mid-term electrode

Targeted **Round 2** areal capacity, Prod: SNMC03008 (NMC811 "unwashed"), Lot#: LT-200280164, MFG. Date: 2020.02.04

*"SS" = single sided -> **CALENDERED***

Anode: LN3237-101-_

(single-sided)

95.83 wt% Superior Graphite SLC1506T

0.5 wt% Timcal C45 carbon

3.5 wt% Kureha 9300 PVDF Binder

0.17 wt% Oxalic Acid

XCEL, 2021 Mid-term electrode

*Targeted **Round 2** areal capacity. SLC1506T Lot#: 573-824*

*"SS" = single sided -> **CALENDERED***

Notes:

-Gen2, Celgard 2500, 30°C, cathode 1.54 cm², anode 1.77 cm²

-Each loading was tested in duplicate (plots here showing charge and discharge data for each cell)

-Prior to this test, cells did formation, rate study, and HPPC

-Data in plot is showing 2x C/3 cycles, then beginning of 150x 6C CCCV charge, C/2 discharge

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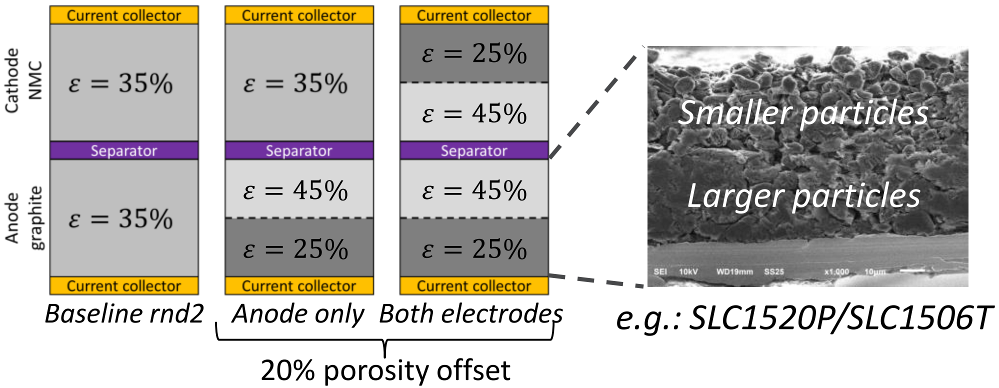
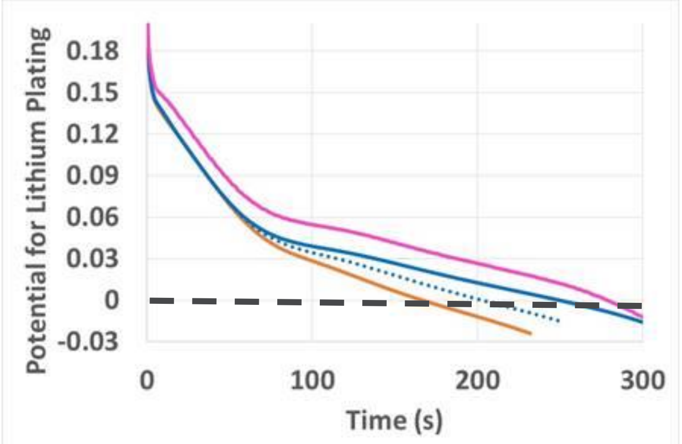
BILAYER ARCHITECTURE

Model predicts particle size and porosity graded electrodes delays lithium plating



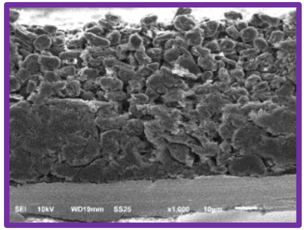


Case	Plating onset
R2 (baseline)	166s
Graded porosity (anode)	200s
Graded porosity (anode and cathode)	244s
Graded porosity (anode and cathode) + graded particle size (anode: 2.35µm/8µm)	280s

- Front of electrodes is optimized for transport (~power cell) while back of electrodes is optimized for storage (~energy cell)
- Architecture gains are conditioned with tortuosity T decreasing with porosity

OBTAINED NEWLY DEVELOPED SMALL PARTICLE GRAPHITE FOR DUAL-LAYER ANODES

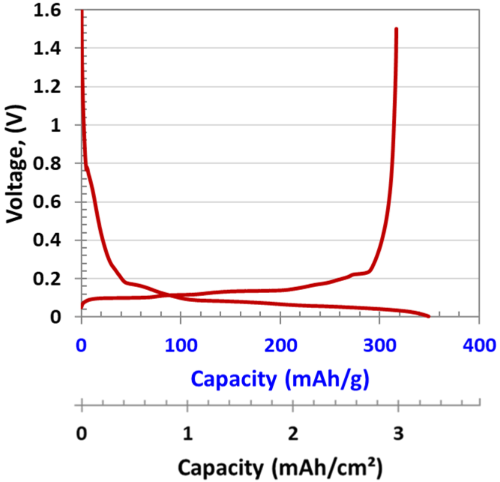
Synthetic graphite developed by American Energy Technologies Co.



#2 – Cu : SLC 1520P : 1506T

Last year showed dual layers of 20/8 μm showed Li plating – needed smaller particles for both layers

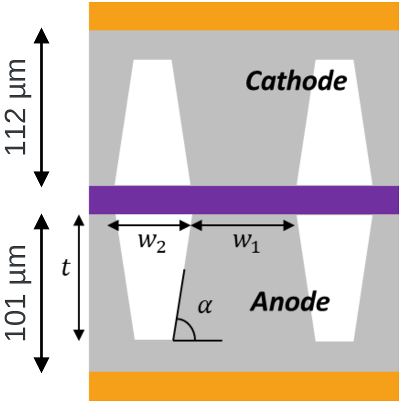
	De-lithiation			CE, %
Cycle#	mAh/g	mAh/cm²	Time, hr	
1	316.3	2.98	24.8	90.54
2	316.8	2.98	24.8	99.00
3	319.9	3.01	12.5	99.48
4	320.7	3.02	12.6	99.65



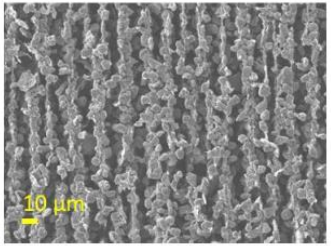
- AETC synthetic graphite LM2803 has a D50 of 3.1 μm, BET 2.09 m²/g, and good capacity, which should be ideal for fast charge
- Plan to use in dual layer- Cu: SLC1506T: AETC LM2803

SECONDARY PORE NETWORK ARCHITECTURE

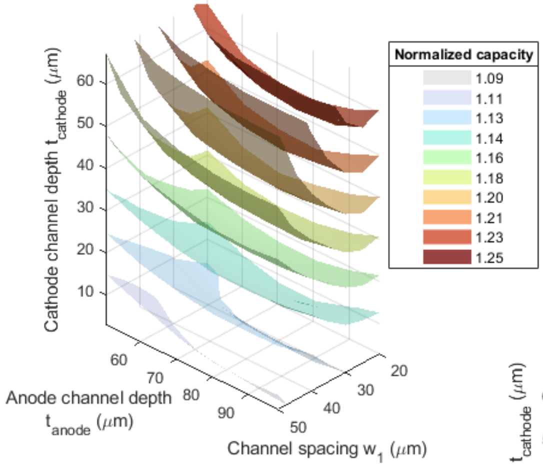
Lithium plating nearly nullified with significant capacity improvement



Channel width w_2 = 10 µm
Channel spacing w_1 (var)
Channel depth t (var)
Channel slope $\alpha = 90°$
Design space investigated compatible with technique limitations

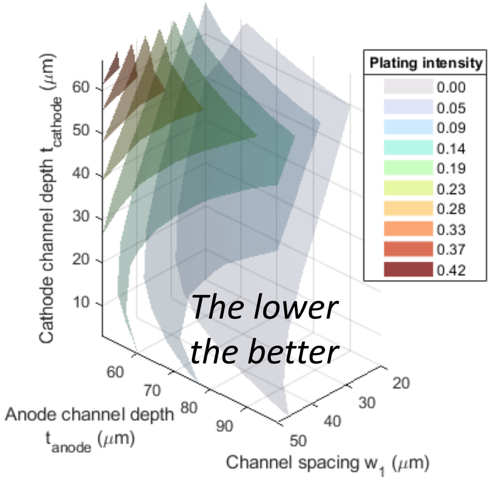


Freeze-tape-casting



- **Objective**: minimize lithium plating (10x reduction), minimize th. capacity loss (10% maximum), and maximize reached capacity @6C

- **Design recommendation**: aligned channels, channel spacing 40 µm, anode channel depth 70.7 µm, cathode channel depth 56 µm (N/P ratio from 1.048 to 1.002)
- 6C CC-CV (10min) capacity +20.4rel%, from 41.3% to 49.7%
- Capacity improvements starts @1.6C



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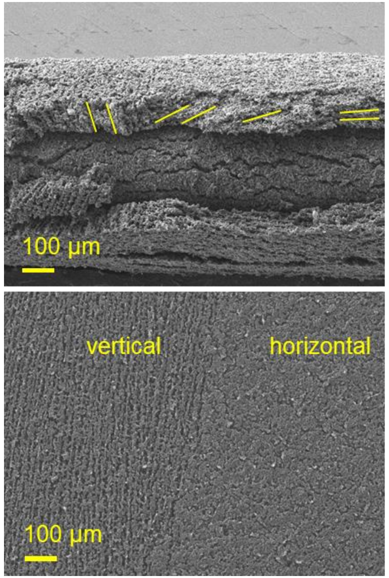
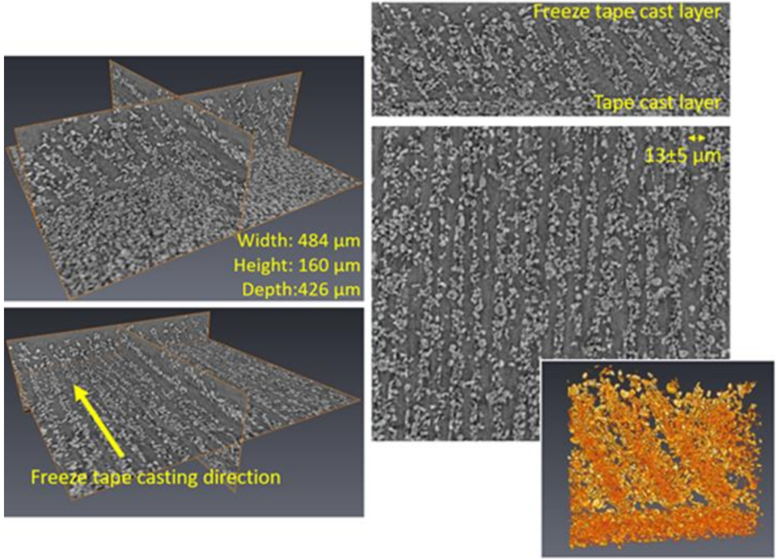
FREEZE-TAPE CAST FOR GRAPHITE

Free-standing Freeze-tape Cast Applied Onto Thin Graphite Layer

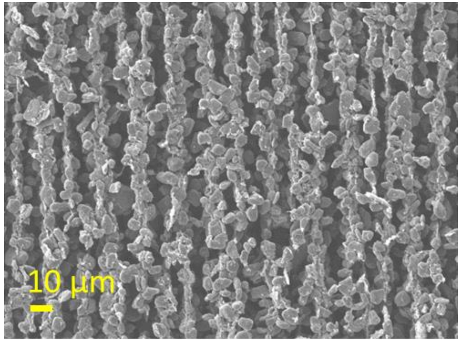


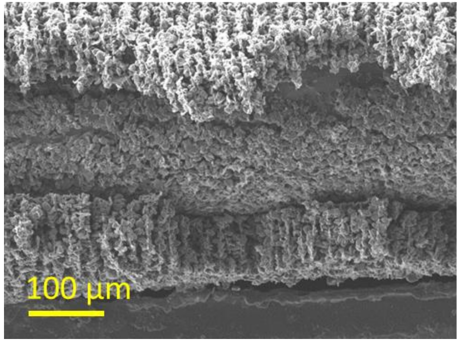
Direct freeze-tape-casting on tape-cast graphite electrodes continued to cause ice misalignment issues in various processing conditions tested.

Micro-tomography images of dual electrode



- Freeze-tape-cast electrodes can be detached from Mylar substrate prior to freeze drying.
- The free-standing electrodes are placed on tape cast electrode on casting (prior to drying NMP) for attachment.



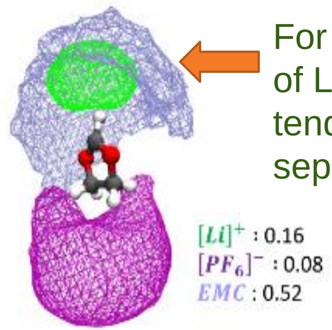


Pore structures are maintained when free-standing freeze-tape-cast electrodes are attached to tape-cast electrodes.

UNDERSTANDING TRANSPORT LIMITATIONS

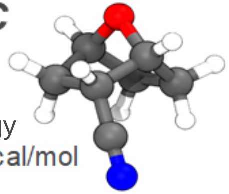
Using target specifications developed from continuum level models and local solvation structures for different solvents, four electrolyte formulations were compared.

Lower values of binding energies between Solvent C and Li^+ infer a lower cost of lithium desolvation.



For a given concentration of $LiPF_6$, EC-like solvents tend to solvate Li^+ ions separately from PF_6^-

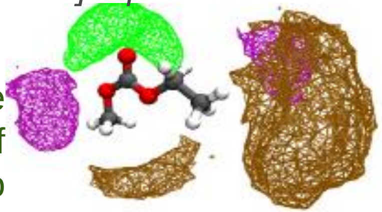
Solvent C



Solvation Energy
-36.9 kcal/mol

7-Oxabicyclo[2.2.1]heptane-2-carbonitrile

With EMC, $LiPF_6$ does not dissociate (due to low dielectric constants of EMC) and thus, the salt tends to accumulate locally within the electrolyte resulting in wide range of local conductivities.



Probability densities for isosurface

Salt Conc.	Gen-2	20% C + Gen-2
0.5M	6.96E-10	5.05e-11
1.1 M	5.2E-10	1.07e-10
1.7 M	7.12E-11	1.17e-10
2.3 M	5.02E-11	1.24e-10

Li^+ Diffusivities from GITT Measurements (m^2/s)

Based on measured diffusivities and conductivities, formulations containing Solvent C were used for evaluating cycling performance using CAMP electrodes and standard XCEL formation protocols, in coin cells.

A. Mallarapu, V. Bharadwaj and S. Santhanagopalan, "Understanding extreme fast charge limitations in carbonate mixtures", *J. Mater. Chem. A*, 2021,9, 4858-4869.

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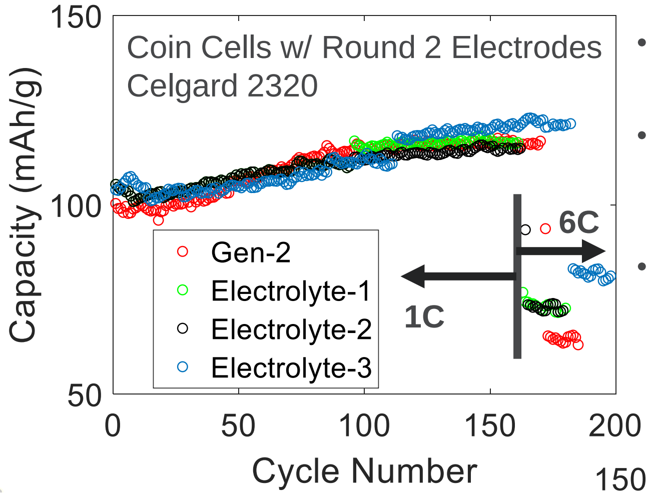
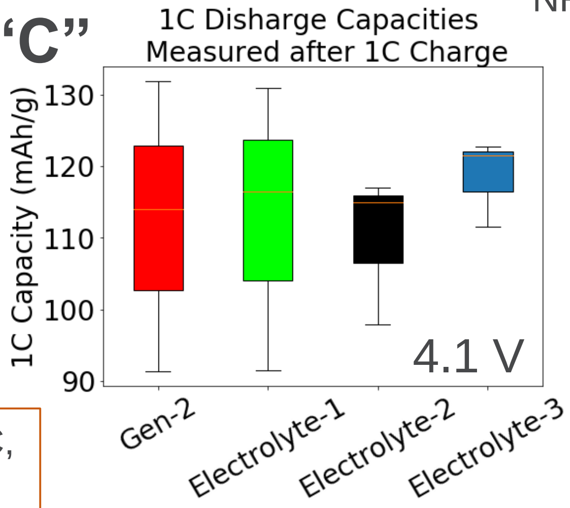
NREL

CYCLING PERFORMANCE OF SOLVENT "C"

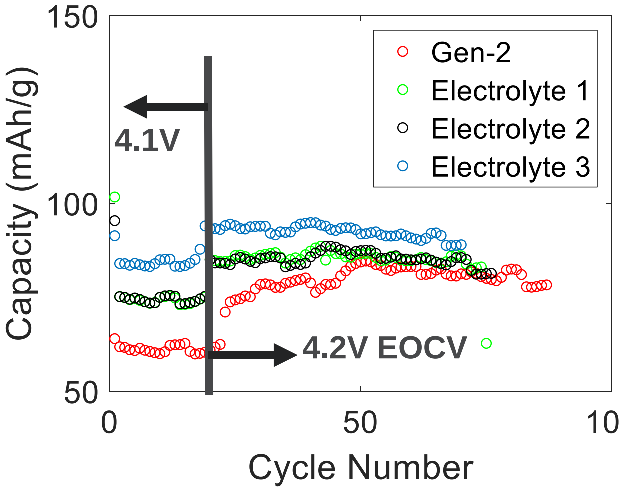


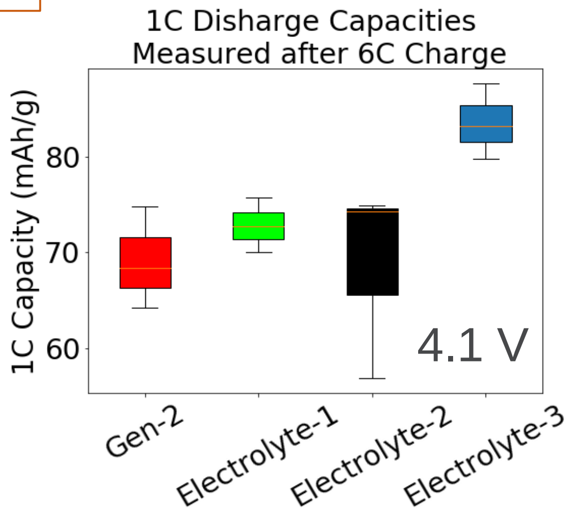
- CC-CV with time limit (determined by the C-rate)
- Current cut-off for CV step was set to 10% but not reached within the set time limit.
- DPAs of the cells will be performed after cycling.

Electrolyte 3 = Solvent C:EC:EMC, 1.2M $LiPF_6$ + 1% FEC + 1% VC



- Formulations stable at 4.2V full cell.
- Electrolyte-3 retained ~ 70% of the 1C capacity when charged at the 6C rate at 4.1V and ~77% at 4.2V.
- The next build will include 2500 for the membrane.





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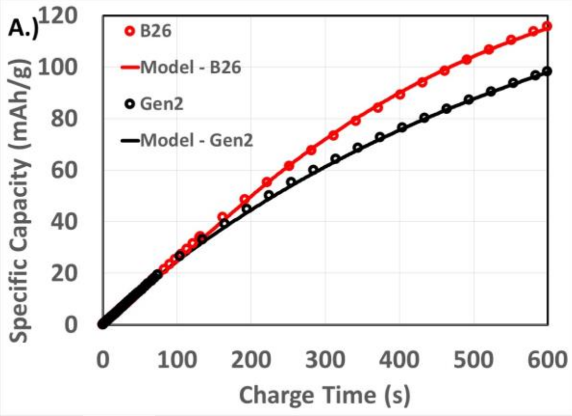
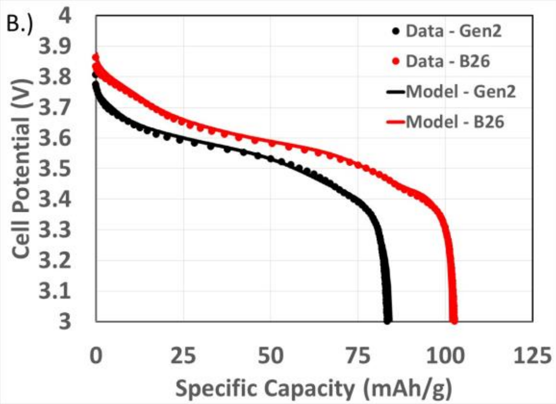
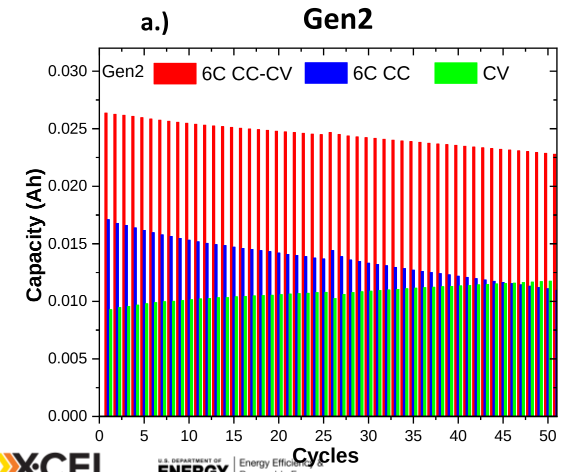
AEM ADVISED ELECTROLYTE

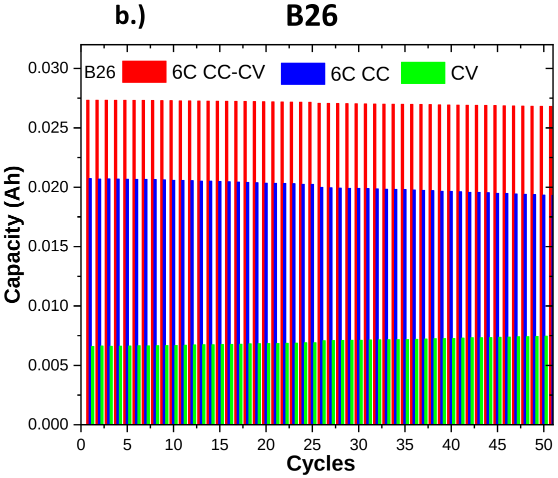
Cell testing and modeling (INL, ANL, NREL, UCB) has validated B26 as a viable candidate for XFC with NMC/Gr, providing performance and life benefits past Gen2. B26 testing continues in hero cell trials.

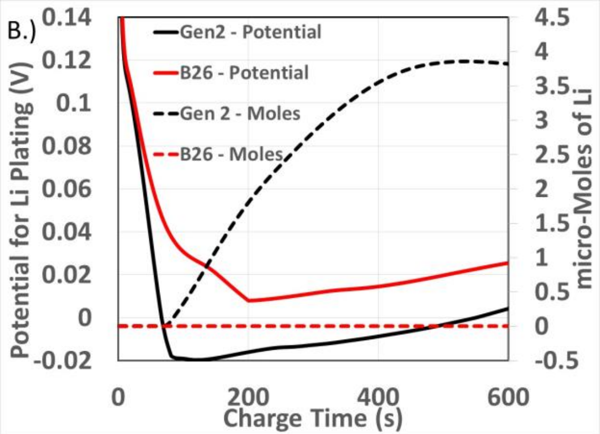
B26: EC-DMC-DEC-EP-PN (20:40:10:15:15 mass) plus $LiPF_6$ with 3% VC and 3% FEC.











AEM ADVISED ELECTROLYTE REDUCES LI PLATING

ANL
UC-B

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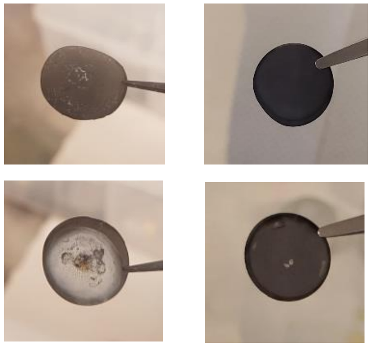
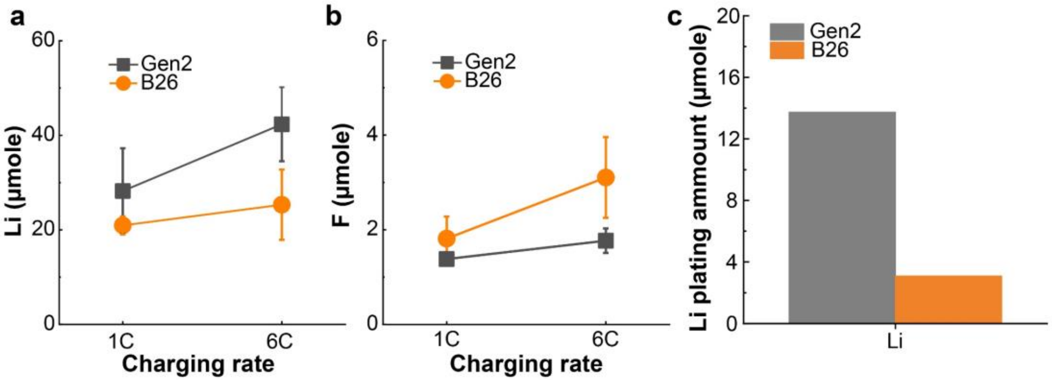
	Gen2	B26
1C charging		
6C charging		



Anode images after 50 cycles.

B26 greatly reduces the incidence of lithium plating in cells that undergo fast charging (6C). Modeling (NREL) and lab results are in good agreement on these trends.



MS Titration Results (UC-Berkeley)

B26 Anode	Carbonates (nmol/cm^2)
RT Full	982
40 Full	875

Gen2 Anode	Carbonates (nmol/cm^2)
RT Full	1010
40 Full	1332

B26 Cathode	Carbonates (nmol/cm^2)
RT Full	758
40 Full	760

Gen2 Cathode	Carbonates (nmol/cm^2)
RT Full	1743
40 Full	1765

FY2020 Q4 – FY2021 Q1 --- “HERO” CELLS

- *Fabricate 24 pouch cells using best anode and electrolyte*
- Two separator were selected: baseline Celgard 2320 (PP:PE:PP trilayer) and Celgard 2500 (PP), which has higher porosity & larger pore sizes (lower tortuosity).
- Two electrolytes were selected: baseline Gen2 and B26 ($LiPF_6$ in EC:DMC:DEC:EP:PN (20:40:10:15:15) with 3% VC & 3% FEC), which INL provided >100 mL.
- Cells made with low carbon & binder content electrodes divided as such:
  - 8 cells with Gen2 electrolyte and Celgard 2320
  - 8 cells with B26 electrolyte and Celgard 2320
  - 4 cells with Gen2 electrolyte and Celgard 2500
  - 4 cells with B26 electrolyte and Celgard 2500
- Two dozen pouch cells fabricated at the end of September 2020 and filled with the selected electrolytes.
- They were formed using a modified formation protocol that included longer rest times between cycles.
- These cells were shipped to INL and began testing in November 2020.

Anode: LN3237-70-2

(single-sided)

95.83 wt% Superior Graphite SLC1506T
0.5 wt% Timcal C45 carbon
3.5 wt% Kureha 9300 PVDF Binder
0.17 wt% Oxalic Acid

XCEL, Trial coating as part of electrode compositional study
Targeted Round 2 areal capacity. SLC1506T Lot#: 573-824
“SS” = single sided -> ***CALENDERED***

Cu Foil Thickness: 10 µm
Total Electrode Thickness: 80 µm
SS Coating Thickness: 70 µm
Porosity: 37.4 %
Total SS Coating Loading: 9.57 mg/cm^2
Total SS Coating Density: 1.37 g/cm^3
Estimated SS Areal Capacity: 3.03 mAh/cm^2
[Based on rev. C/10 of 330 mAh/g for 0.005 to 1.5 V vs. Li]

Made by CAMP Facility

Cathode: LN3237-78-4

(single-sided)

96 wt% Toda NMC532
2 wt% Timcal C45
2 wt% Solvay 5130 PVDF Binder

XCEL, Coating used in FY20 Q4 SLP Hero Cells
Targeted Round 2 areal capacity, Prod:NCM-04ST, Lot#:7720301
“SS” = single sided -> ***CALENDERED***

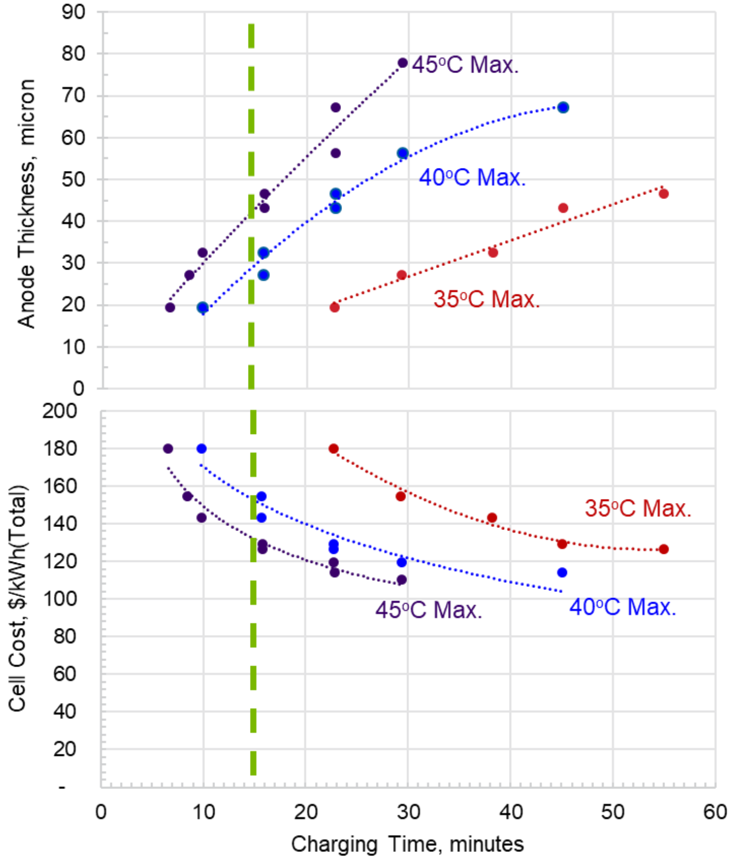
Al Foil Thickness: 20 µm
Total Electrode Thickness: 80 µm
SS Coating Thickness: 60 µm
Porosity: 34.9 %
Total SS Coating Loading: 17.24 mg/cm^2
Total SS Coating Density: 2.87 g/cm^3
Estimated SS Areal Capacity: 2.65 mAh/cm^2
[Based on rev. C/10 of 160 mAh/g for 3.0 to 4.2 V vs. Li]

Made by CAMP Facility

BATPAC ESTIMATES BASED ON HERO CELL

FY20 Hero cell costs are lower if allowed to reach 45°C after adiabatic charging

- Hero Cell properties defined from BatPaC, CAMP, XCEL partner(s) [Colclasure], and literature
- Fast Charge = 80% capacity (15 to 95% SOC) recharged in **15 min**.
- Adiabatic operation during charging
- Charging Protocol
  - Initially, constant power
  - C-rate adjusted to avoid lithium plating potential
  - C-rate adjusted to avoid maximum allowable temperature (35, 40, 45 °C) at end of charge
  - Constant voltage hold till SOC limit is reached
- Hero Cell with fast charge costs $130/kWh*, if allowed to heat up to 45°C during adiabatic charging
  - 70 micron negative electrode without fast charging (60 min) costs $111/kWh



*Total Energy
Cost estimated with BatPaC 4.0

RESPONSES TO PREVIOUS YEAR REVIEWERS' COMMENTS

Project not reviewed in 2020

COLLABORATION ACROSS LABS AND UNIVERSITIES

Argonne National Laboratory: Cell and electrode design and building, performance characterization, post-test, cell and atomistic modeling, cost modeling

Idaho National Laboratory: Performance characterization, failure analysis, electrolyte modeling and characterization, Li detection, charging protocols

Berkeley Lab: Li detection, electrode architecture, diagnostics

NREL (National Renewable Energy Laboratory): Thermal characterization, life modeling, micro and macro scale modeling, electrolyte modeling and characterization

Oak Ridge National Laboratory: Detailed Li plating kinetic models, SEI modeling

SLAC National Accelerator Laboratory: Li detection, novel separators, diagnostics

REMAINING CHALLENGES AND BARRIERS

- While the freeze-tape cast method is able to make an electrode with the lowest tortuosity, it may be difficult to densify the primary region required in an ideal secondary pore network in a scalable manner

PROPOSED FUTURE RESEARCH

- Develop bilayer anode with new smaller graphite particles near separator
- Validate bilayer model predictions with ANL bilayer electrodes
- Update design parameter for higher loading cell (4 mAh/cm^2, 100-µm electrodes)
- Develop bilayer anode & cathode with split porosity in each electrode
- Incorporate pore formers (salt, volatile solids, etc.) to create secondary pores
- Compare effect of formation conditions with advanced electrolytes
- Increase initial salt concentration to explore degradation products

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

SUMMARY

- Reduced the amount of binder and carbon additives to increase energy density and increase charge rate as advised by modeling effort
- Used AEM to predict electrolyte compositions that minimize lithium plating
- Built “Hero” pouch cells based on model predicted improvements: lower carbon & binder, advanced electrolyte, high-porosity low-tortuosity separator
- Identified “Solvent C” as new solvent with lower binding energies
- Switched to NMC811 and exploring >3 mAh/cm^2 loadings
- Developed model to design two electrode architectures that reduce lithium plating
- Fine tuned model for secondary pore network (SPN) to predict ideal architecture
- Worked with company to make small graphite particles for dual-layer anodes
- Developed new method to fabricate dual-layer graphite electrodes via freeze-tape cast to mimic SPN
- Multiple pouch cell builds to support XCEL Thrust activities

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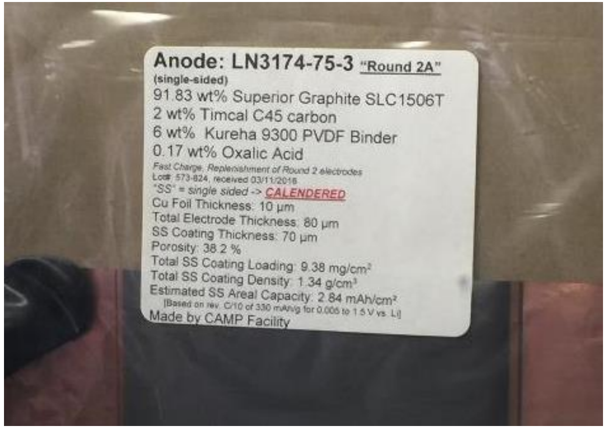
TECHNICAL BACK-UP SLIDES

ELECTROLYTE FORMULATIONS WITH SOLVENT "C"



- **Baseline:** Gen-2 electrolyte with 1.2 M $LiPF_6$ salt concentration
- **Electrolyte 1:** *Chosen based on solvation shell calculations showing easier dissociation of Li^+ at higher local salt concentrations (which are bound to happen under ultra high fast charging rates)*

  Ethylene Carbonate:Ethyl Methyl Carbonate 3:7

  1.2M $LiPF_6$ with 10% of the EC replaced by Solvent C
- **Electrolyte 2:** *Addition of FEC was based on initial cycling-stability results comparing baseline electrolyte and Electrolyte 1 above. It is not clear yet, if Solvent C can be fluorinated instead of adding FEC separately.*

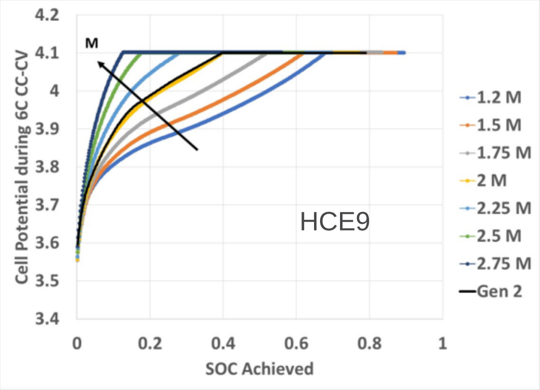
  EC:EMC 3:7, 1.2M $LiPF_6$ with 10% of the EC replaced by Solvent C, 2% FEC
- **Electrolyte 3:** *Cells with FEC (Electrolyte 2) showed higher interfacial resistance; VC is a common additive to lower the surface impedance.*

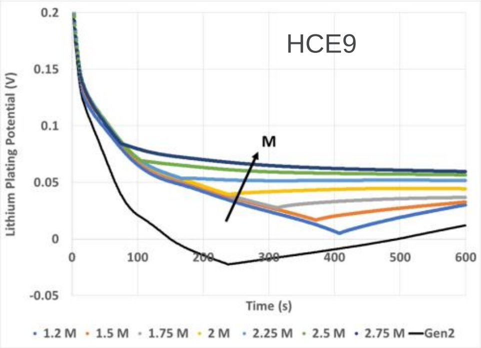
  EC:EMC 3:7, 1.2M $LiPF_6$ with 10% of the EC replaced by Solvent C + 1% FEC + 1% VC

HIGHLY CONCENTRATED ELECTROLYTES

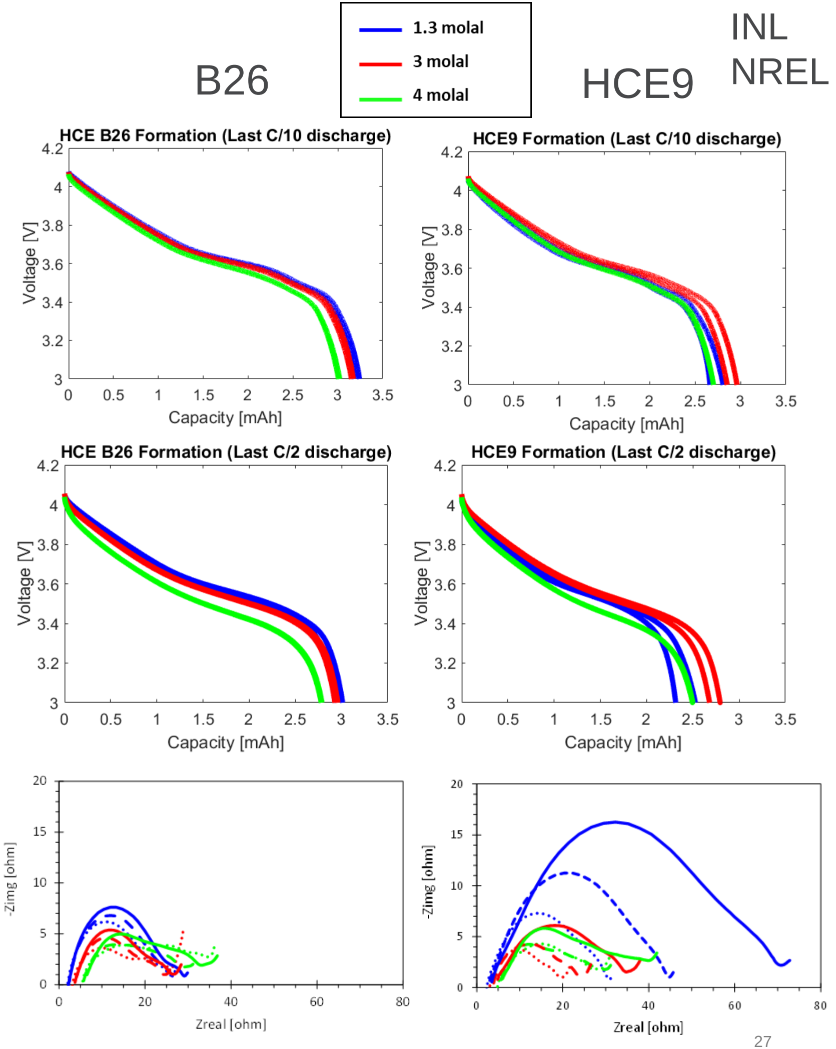
Highly concentrated electrolytes (HCE) are being investigated as candidates for XFC applications. AEM identified HCE systems for XFC, and best candidates have undergone early testing (Tier 1) with newer systems currently on test (Tier 2).

HCEs may facilitate transition to 811 cathodes as well as electrodes with unique porous architectures.





NREL Model doesn't (yet) consider changes in interface properties from different SEI/CEI formed during formation as a function of salt conc.



XCEL eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

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