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SAND2018-4065PE

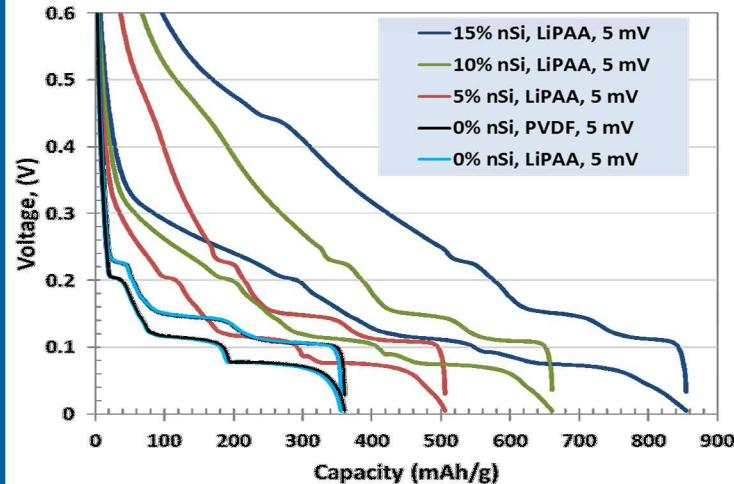
Argonne
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RESEARCH FACILITIES SUPPORT

KYLE FENTON
SANDIA NATIONAL LABORATORIES

U.S. DEPARTMENT OF ENERGY
VEHICLE TECHNOLOGIES OFFICE
2018 ANNUAL MERIT REVIEW

Silicon Deep Dive



bat349

OVERVIEW

Timeline

- Start: October 1, 2015
 - Reset: October 1, 2017
- End: September 30, 2020
- Percent Complete: 55%

Budget

- Total project funding:
 - FY18 - \$3600K
- Presentations: BAT349, BAT350, BAT351, BAT352, and BAT353

Barriers

- Development of PHEV and EV batteries that meet or exceed DOE and USABC goals
 - Cost, Performance, and Safety

Partners

- Sandia National Laboratories
- Pacific Northwest National Laboratory
- Oak Ridge National Laboratory
- National Renewable Energy Laboratory
- Lawrence Berkeley National Laboratory
- Argonne National Laboratory

RELEVANCE

- **Objectives: Stabilize the SEI - Stabilize the electrode**
- **Overall focus on insights into and advancement of silicon-based materials, electrodes, and cells.**
- **Advancements verified on life and performance of full cells using standardized testing protocols.**

Program Directly Addresses Cost and Performance Barriers and Quantifies Safety

- Elemental silicon can theoretically store >3500 mAh/g.
- Battery Performance and Cost (BatPaC) Model indicates a silicon based anode coupled with a high capacity cathode lithium-ion technology presents a pathway to less than \$125/kWh_{use}
- BatPaC also used to relate pack level benefits to program goals.
- Benefits reach diminishing returns after **1000 mAh/cm³** (electrode basis) for both cost and energy density.
- Silicon with <75 wt% graphite can achieve target.

MILESTONES AND ACTIVITIES

- The program has more than twenty milestones related to the broad range of integrated activities listed below.
- Generally, milestones are either completed or on schedule.
- Extensive electrochemical and analytical diagnostic studies.
- Facilities supporting program through a wide range of studies.
 - Battery Abuse Testing Laboratory (BATLab); Battery Manufacturing Facility (BMF); Cell Analysis, Modeling, and Prototyping (CAMP); Materials Engineering Research Facility (MERF); Post-Test Facility (PTF)
- Development and testing of coatings and additives designed to modify and stabilize the interface.
- Develop and analyze polymer binders designed to accommodate volume changes, increase conductivity, and improve adherence.
- Active material development.
 - Explore lithium inventory strategies.
 - Study alternative high-energy metals.

For reviewers, a detailed list of the milestones and progress is supplied in the reviewers only slides.

CAMP FACILITY'S CAPABILITIES:

- A wide range of mixing equipment with various mixing actions that can work with small volumes of 10 mL up to 2 L of slurry, (ex. planetary mixer with high speed disperser); high precision electrode coater with two drying zones; a hot roll press, all which enables the fabrication of high quality electrodes.
- Semi-automated equipment to make xx3450 and xx6395 lithium-ion pouch cells with a typical capacity of 200 mAh to 4 Ah.
- Semi-automated equipment to make 18650 lithium-ion cells with a typical capacity of 1 to 3 Ah.
- Most equipment located in a dry room with an area of ~45 m² that is capable of maintaining <100 PPMv (-42°C dew point) with 6 people working and 750 SCFM of exhaust ventilation.
- Currently in the process of building a new dry room that will be ~135 m²

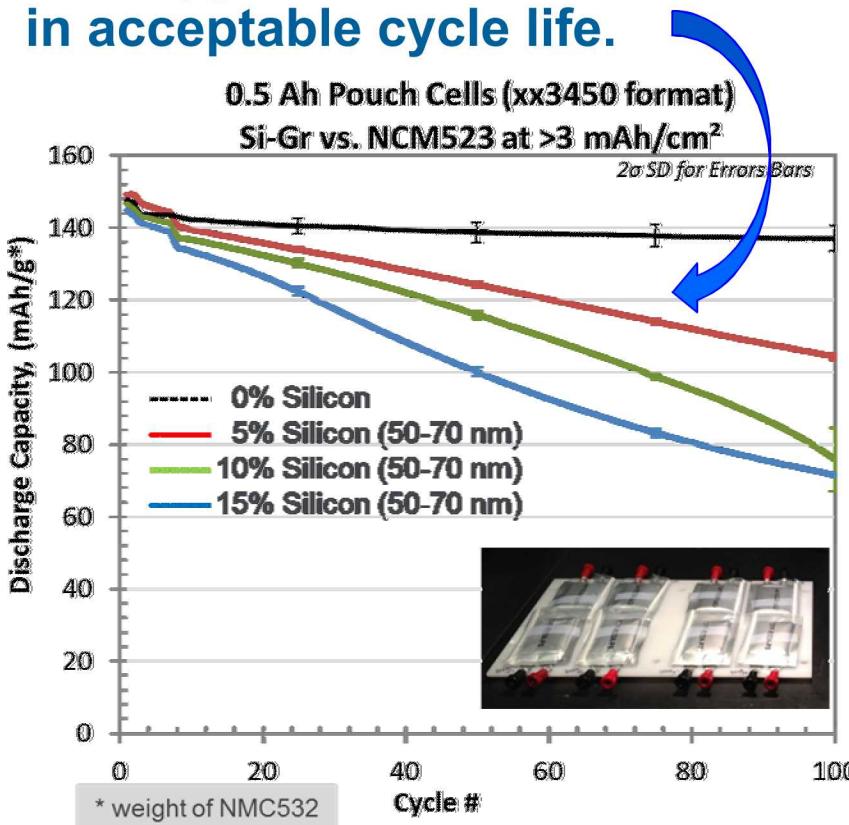


CHANGE DIRECTION OF ELECTRODE DESIGN

Previous efforts were focused on using low amounts of silicon (<15%), but with high levels of lithiation (~50 mV vs. Li⁺/Li).

This approach has not resulted in acceptable cycle life. ↗

BAT350??



This year's effort is exploring the use of higher amounts of silicon (30-70%), but with lower levels of lithiation (>100 mV vs. Li⁺/Li).

- Will lessen degree of silicon particle expansion
- Incorporating other carbon materials (e.g., hard carbon) instead of graphite because lower voltage plateau of graphite would not be utilized
- May enable other binder systems
- **Lowers cell voltage by ~200 mV**

CHARACTERIZATION OF NEW SILICON MATERIAL FROM PARACLETE ENERGY

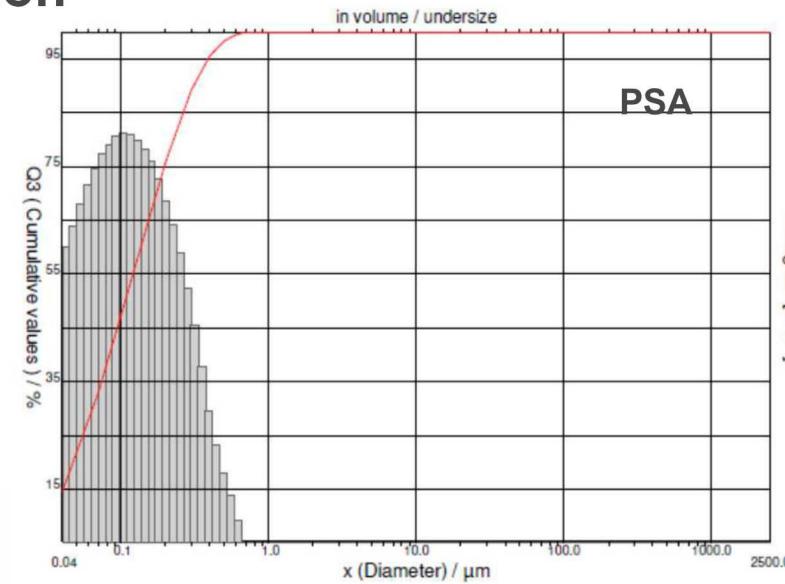
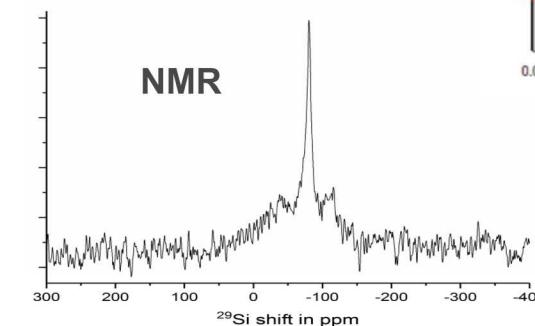
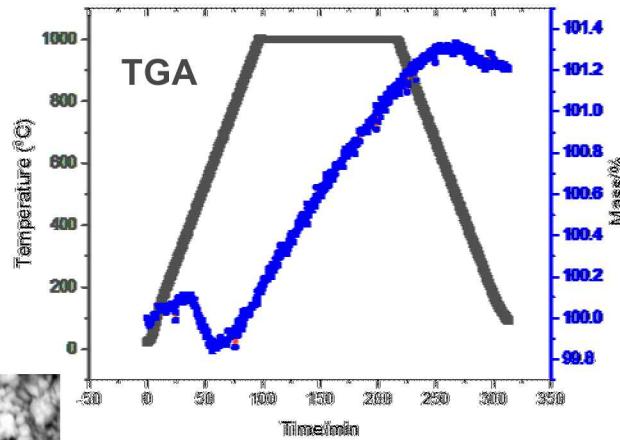
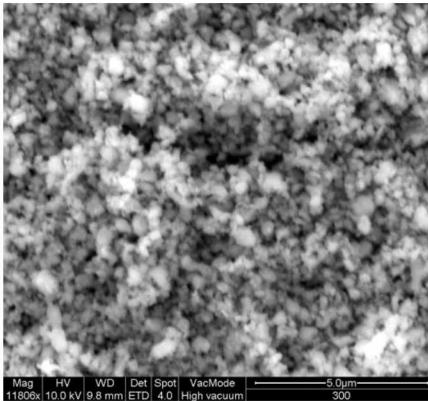
BAT351

4 kg of Si/SiO (PE “4KD” Lot – F17-021-LS) was ordered and distributed to lab partners.

This 4 kg lot was analyzed and found to be comparable to the original 500 g lot used for initial evaluation



SEM



Sample 4KD will be used after the original 500g batch of D is used up

500g D lot was used in A-A012 and A-A013 in Electrode Library



CHEMICAL ANALYSIS OF SILICON MATERIALS

A full panel ICP-MS study was performed to assess the amount of impurities in silicon materials from various sources.
 (Still working on finding a method to get accurate O₂ content)

CAMP Sample No.	F17-005-LS-AR	NA-70-130 APS	NA-50-70 APS	HQ-80 APS	4KD
	Ground	Ground	Grown	Grown	Ground
ACL Sample No.	18-0013-01	18-0017-01	18-0017-02	18-0027-01	18-0039-01
Li	< 1.08	2.51	1.58	< 0.79	< 0.47
B	< 4.04	5.31	< 1.53	5.87	NA
Na	12.6	244	247	< 0.78	0.8
Mg	< 0.37	6.73	10.2	28.8	< 1.64
Al	< 0.63	159	16.3	1040	< 2.04
Si	NA	NA	NA	NA	NA
P	< 12.4	20.5	< 8.21	< 15.9	< 19.2
K	< 10.9	< 6.82	< 7.22	23.2	< 5.0
Ca	< 25.8	122	26.4	172	< 10.5
Ti	101	1621	0.84	146	0
V	< 0.04	4.02	< 0.03	128	< 0.03
Cr	1.52	< 0.29	< 0.31	58.5	2.50
Mn	0.34	3.73	< 0.05	73.1	0.33
Fe	< 7.03	32.3	< 5.34	4427	< 13.26
Co	0.86	3.43	12.6	13.1	3.70
Ni	1.07	5.80	< 0.08	64.9	1.27
Cu	23.8	11.6	7.62	99.7	2.7
Zn	11.8	4.58	2.47	10.8	0.8
Ga	< 0.01	0.09	< 0.01	2.94	< 0.02
Ge	< 0.05	1.87	< 0.05	2.18	< 0.27
As	< 0.16	1.35	< 0.17	6.38	< 0.18
Se	< 1.33	< 0.87	< 0.92	< 2.54	< 3.29
Rb	< 0.01	< 0.01	< 0.01	0.10	< 0.01
Sr	< 0.02	0.31	< 0.08	1.13	< 0.07
Y	91.3	74.8	< 0.04	0.65	126.0
Zr	1428	1103	0.17	11.5	2001
Nb	< 0.03	0.30	< 0.01	0.95	< 0.18
Mo	< 0.15	1.00	< 0.17	5.43	< 0.40

Data Shown: PPM by weight



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BERKELEY LAB

CAMP Sample No.	F17-005-LS-AR	NA-70-130 APS	NA-50-70 APS	HQ-80 APS	4KD
	Ground	Ground	Grown	Grown	Ground
ACL Sample No.	18-0013-01	18-0017-01	18-0017-02	18-0027-01	18-0039-01
Ru	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Rh	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Pd	< 0.02	< 0.02	< 0.02	< 0.22	< 0.02
Ag	< 3.90	65.5	< 0.02	3.86	< 0.27
Cd	0.76	0.77	< 0.03	< 0.04	0.70
Sn	< 0.04	0.35	0.08	0.92	< 0.16
Sb	< 0.02	0.25	< 0.04	0.88	< 0.05
Te	< 0.24	< 0.12	< 0.11	1.43	< 0.47
Cs	< 0.01	0.47	< 0.01	< 0.01	< 0.01
Ba	< 0.05	0.83	0.84	4.80	< 0.74
La	< 0.01	0.54	< 0.02	1.17	< 0.02
Ce	< 0.01	1.21	0.32	2.31	< 0.39
Pr	< 0.01	0.14	< 0.01	0.28	< 0.01
Nd	< 0.02	0.70	< 0.03	1.12	< 0.02
Sm	< 0.01	< 0.02	< 0.01	0.14	< 0.01
Eu	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Gd	< 0.01	0.06	< 0.01	2.52	< 0.01
Tb	< 0.01	< 0.00	< 0.01	0.03	< 0.01
Dy	< 0.01	< 0.02	< 0.01	0.18	< 0.02
Ho	< 0.01	0.06	< 0.01	0.04	< 0.01
Er	< 0.01	0.03	< 0.01	0.08	< 0.02
Tm	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Yb	< 0.01	< 0.01	< 0.01	0.05	< 0.01
Lu	< 0.01	< 0.01	< 0.01	< 0.01	< 0.02
Hf	50.2	39.3	< 0.01	0.34	50.9
Ta	< 0.01	0.17	< 0.01	0.20	< 0.76
W	< 3.53	< 3.73	< 1.35	< 1.66	< 1.32
Re	< 0.01	0.03	< 0.01	< 0.02	< 0.01
Ir	< 0.12	< 0.12	< 0.01	< 0.01	< 0.12
Pt	< 0.34	< 0.70	< 0.01	< 0.02	< 0.31
Au	< 0.06	< 0.14	< 0.05	< 0.06	< 0.09
Tl	< 0.01	< 0.01	< 0.01	< 0.01	< 0.06
Pb	0.62	1.86	0.76	1.53	0.16



POST-TEST FACILITY AT ARGONNE SUPPORTS SI DEEP DIVE

- Post-test diagnostics of aged batteries can provide additional information regarding the cause of performance degradation, which, previously, could be only inferred
- Combine microscopy, spectroscopy and chromatography in a controlled-atmosphere glove box to the greatest extent possible
 - FT-IR spectroscopy
 - Raman spectroscopy
 - Optical and scanning-electron microscopy
 - Electrochemical impedance spectroscopy
 - X-ray photoelectron spectroscopy
 - High Pressure Liquid Chromatography/Gel Permeation Chromatography
 - TGA-GC/MS
 - Half-cell fabrication and test equipment



- Use capabilities to characterize pristine and aged Si-based electrode materials and Li-Si phases

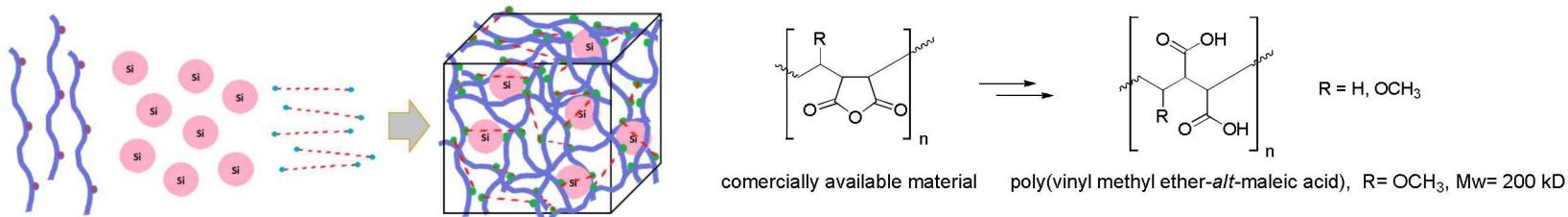
MATERIALS ENGINEERING RESEARCH FACILITY (MERF) – ANL

- General

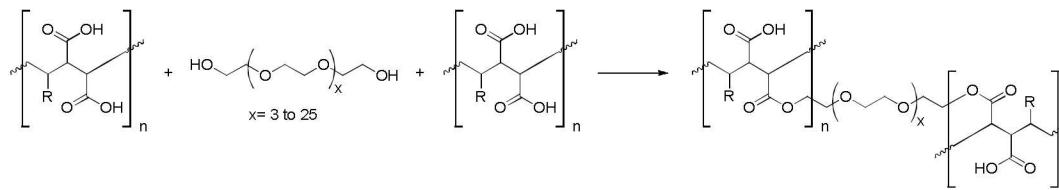
IN-SITU CROSS-LINKING BINARY POLYMER SYSTEMS AS SILICON ANODE BINDER

BAT351/3??

- The objective of this project is to develop binary polymeric system that undergoes in-situ cross-linking during laminate manufacturing to form robust 3D mesh preventing rapid mechanical degradation of the electrode.
- The binary system is based on poly(ethylene-*alt*-maleic acid) or poly(vinyl methyl ether-*alt*-maleic acid) and a short chain poly(ethylene glycol) oligomer.



- Components of the binary system are water soluble to allow for aqueous manufacturing process.



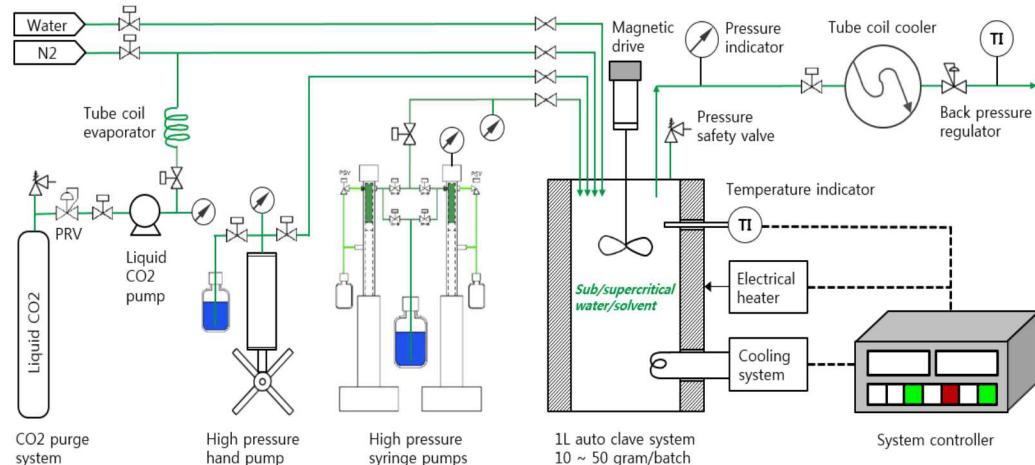
- The backbone polymer has good adhesion to copper current collector and the cross-linked structure has good cohesion to silicon and graphite particles.
- Interaction of the backbone polymer with Si surface further strengthen the structure.
- Electrode manufacturing and electrochemical evaluation in cells is pending.

HYDRO/SOLVOTHERMAL SYNTHESIS AND SCALE-UP OF SILICON AND SILICON-CONTAINING NANOPARTICLES

Milestones

Date	Description	Status
Oct-17	Project start	Done
Nov-17	System basic design	
Dec-17	Basic design review	
Jan-18	PO of equipment and parts	
Apr-18	System installation start	
May-18	WCD/ESH preparation	
Jul-18	Mechanical completion	
Jul-18	System safety review	
Aug-18	Revision and modification	
Aug-18	Operation permission	
Sep-18	Si nanoparticle synthesis	On track
Oct-18	30 g delivery to collaborator	
Nov-18	Si/C composite synthesis	
Dec-18	100 g delivery to collaborator	

Schematic diagram of bench-scale hydro/solvothermal reactor system



- A new scalable Si synthesis process will be set up.
- Si nanoparticle size and morphology will be controlled and optimized by adjusting operation pressure, temperature and reaction medium.
- Si/C composite will be synthesized to increase the uniformity of Si and graphite in laminate film.

SNL CELL PROTOTYPING FACILITY:

- The SNL cell prototyping facility dedicated R&D facility equipped to manufacture small lots of lithium-ion cells of various sizes including 2032 coin cells, 18650s, D-cells, and prismatic cells
 - 1000 sq. ft. of dry room space in two separate dry rooms**
 - Two prototype electrode coaters, 20-30 meter coating run capacity**
 - Three 18650 cell winders**
 - One multiformat cell winder for 18650, D-cell, and prismatic cell formats**
 - Electrolyte filling and associated cell hardware and packaging equipment**
 - 100 channels for battery performance testing and formation cycling**
- Experience with numerous lithium-ion chemistries including natural and synthetic graphite anodes, $\text{Li}_4\text{Ti}_5\text{O}_{12}$, LiCoO_2 , NMC, LFP, Li-S, Si, primary chemistries, and spinel cathodes (LiMn_2O_4 and $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$).



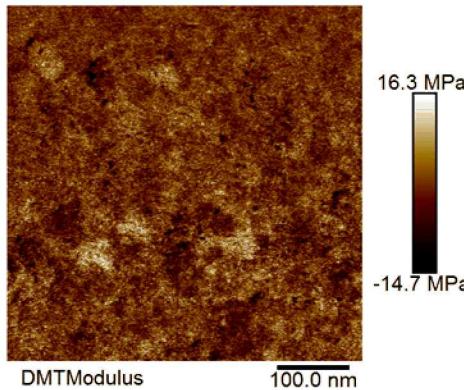
SNL BATTERY ABUSE TESTING LABORATORY (BATLAB)

- **Test/Analysis**
 - *Battery Abuse Testing Laboratory (BATLab)*
 - *Thermal Test Complex (TTC)*
 - *Burn Site (Laurence Canyon)*
- **Cell Prototype Facility**
- **Battery Calorimetry**
- **Mod/Sim**
- **Materials Development R&D (NM & CA)**



BATTERY AND MATERIALS TESTING AND DIAGNOSTICS LEVERAGE FOR SILICON

- Battery Test Facility used for Tee and cell level testing
 - Different Temperature and conditions for all cells
- Safety for scale up to 50A-hr testing per chamber
- Diagnostics (EC-AFM, XPS, XRD, EC-TEM, Auger, TOF-SIMS) available at need

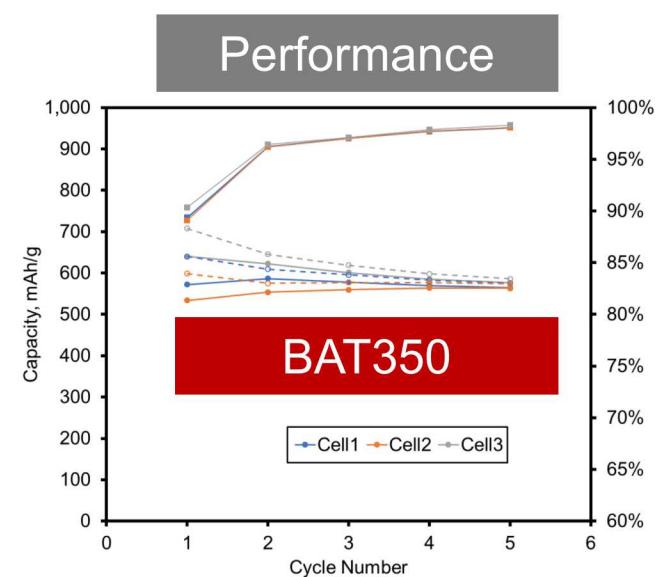
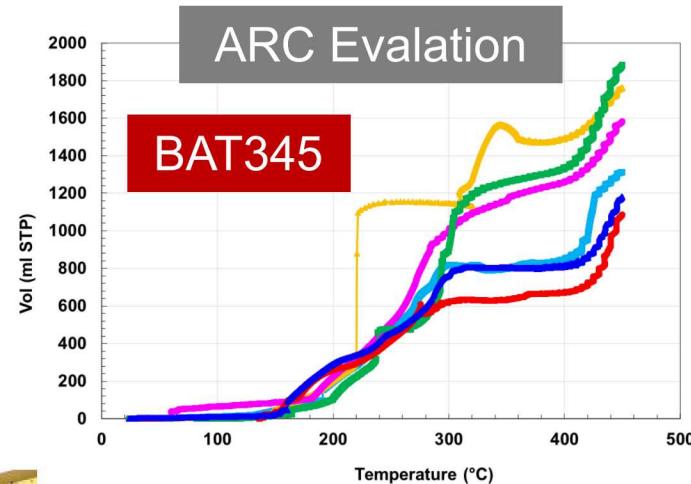


EC-AFM image of Si-substrate after electrochemical cycling and poise at lithiating potential, see talk BAT348

BAT348



Battery aging facility; ~400 channels dedicated to lifetime monitoring of batteries under environmental conditioning



BATTERY MANUFACTURING FACILITY - ORNL

Pilot-Scale Electrode Processing and Pouch Cell Evaluation

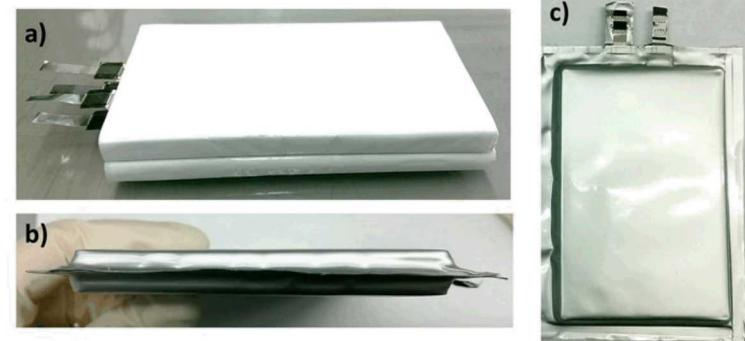


Planetary
Mixer (≤ 2 L)



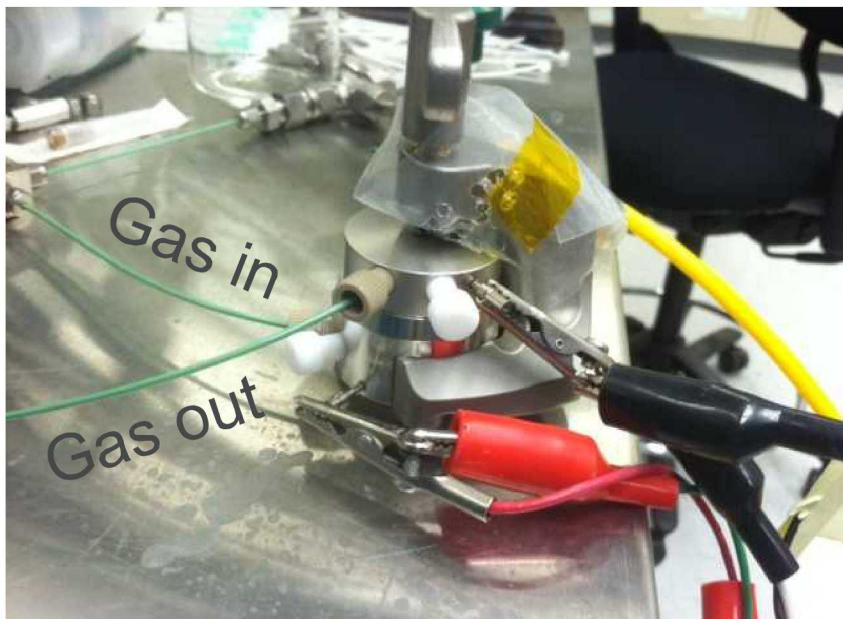
Heated Calender (80,000 lb_f)

- All assembly steps from pouch forming to electrolyte filling and wetting.
- 1400 ft² (two 700 ft² compartments).
- Humidity <0.5% (-53°C dew point maintained).
- Pouch cell capacity: 50 mAh – 7 Ah.
- Single- and double-sided coating capability.
- Current weekly production rate from powder to pouch cells is 20-25 cells.

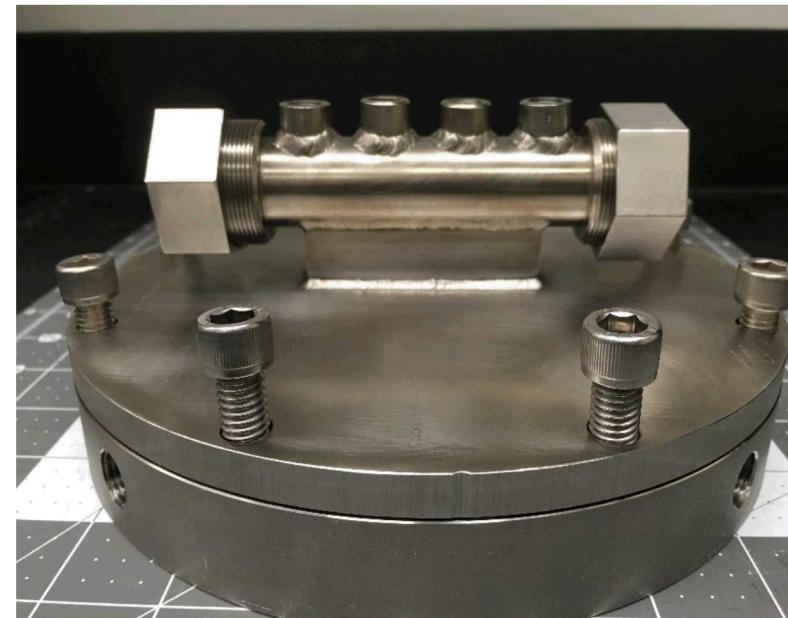


FACILITY AT ORNL TO ANALYZE GAS EVOLUTION DURING CYCLING

BAT345

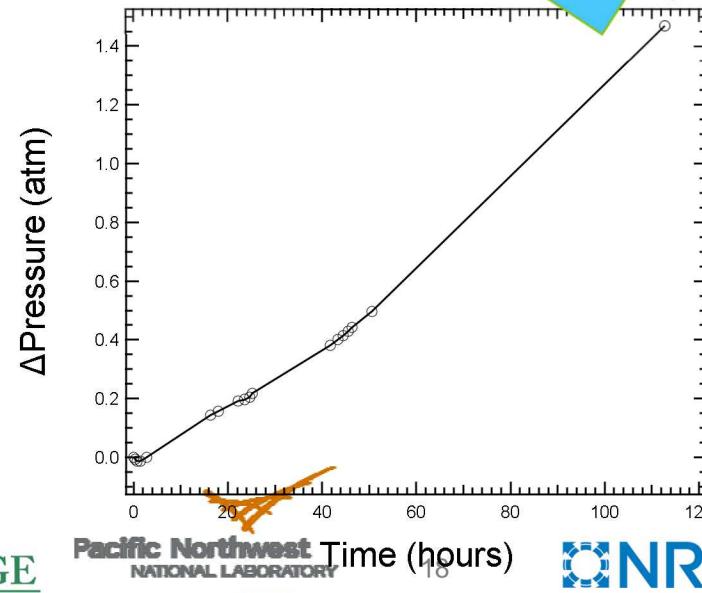
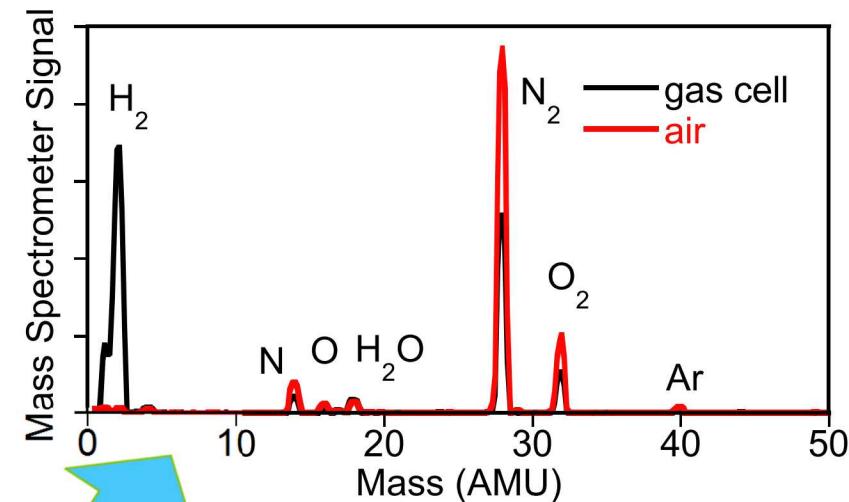


Flow reactor to monitor gassing with mass spectrometry



AEGIS System – Monitor gassing with IR spectroscopy in batch mode

FACILITY AT ORNL TO ANALYZE GAS EVOLUTION DURING PROCESSING



BAT345

TITLE – LBL

- General

LBL LEVERAGE FOR SILICON

BAT364

TITLE – NREL

- General

NREL LEVERAGE FOR SILICON

RESPONSES TO PREVIOUS YEAR REVIEWERS' COMMENTS

Last year two poster presentations covered all the project. The two posters were each reviewed by eight reviewers. We thank the reviewers for their thoughtful comments. Selected excerpts are given below.

- Many of the reviewers' comments were generally positive.
 - “applauded the excellent, thorough approach”
 - “very ambitious program to assess advantages, disadvantages and solutions for Si anode materials”
 - “very nice intra-laboratory coordination”
- One reviewer thought we could further enhance the program by bringing in experts in mechanical stresses. We conduct limited mechanical measurements and have relied on literature to establish a stable particle size, but in general we agree more in-depth studies could improve the program.
- One reviewer suggested that our commitment to openness limits our ability to examine proprietary materials. We agree totally and recognize the limitation. However, we consider that the work we are doing is addressing the fundamental issues with silicon materials and will benefit the entire community.

REMAINING CHALLENGES AND BARRIERS

- Several key challenges remain that limit integration of silicon into graphitic negative electrodes, mostly related to the large crystallographic expansion of silicon (>300%) upon lithiation.
 - SEI stability issues, which affect cycling efficiency.
 - Electrode stability issues that include particle isolation, accommodating volume changes, and adherence.

COLLABORATION AND COORDINATION WITH OTHER INSTITUTIONS

- Six National Laboratories have teamed to form this integrated effort focused on gaining insights into and advancement of silicon-based materials, electrodes, and cells.
- This effort has strong interactions with the Silicon Electrolyte Interface Stabilization (SEI-Sta) project (BAT344, BAT345, BAT346, BAT347, and BAT348).
- Paraclete Energy is supplying baseline silicon materials.

CONTRIBUTORS AND ACKNOWLEDGMENT

Research Facilities

- Post-Test Facility (PTF)
- Materials Engineering Research Facility (MERF)

- Cell Analysis, Modeling, and Prototyping (CAMP)
- Battery Manufacturing Facility (BMF)
- Battery Abuse Testing Laboratory (BATLab)

Contributors

- Daniel Abraham
- Eric Allcorn
- Seong Jin An
- Beth Armstrong
- Chunmei Ban
- Javier Barenco
- Ira Bloom
- Anthony Burrell
- Peng-Fei Cao
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- Claus Daniel
- Dennis Dees
- Fulya Dogan Key
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- Jinghua Guo
- Binghong Han
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- Haregewoin
- Kevin Hays
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- Lydia Terborg
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- Stephen Trask
- Jack Vaughey
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- Lu Zhang
- Zhengcheng Zhang
- Tianyue Zheng

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CRITICAL ASSUMPTIONS AND ISSUES

Next Generation Anodes for Lithium-Ion Batteries

- Silicon has received significant attention as a viable alternative to graphitic carbon as the negative electrode in lithium-ion batteries due to its high capacity and availability.
 - Elemental silicon can theoretically store >3500 mAh/g, nearly an order of magnitude higher than graphite (372 mAh/g and 818 mAh/mL, respectively).
- Several key issues have been identified that limit its utility, including a large crystallographic expansion ($>300\%$) upon lithiation.
 - SEI stability issues, which affect cycling efficiency.
 - Electrode stability issues that include particle isolation, accommodating volume changes, and adherence.
- The objective of this project is to overcome the challenges to the use of silicon-based anodes in high-energy density lithium-ion batteries for transportation applications, and demonstrate functional full lithium-ion cell chemistries which meet the DOE/USABC performance targets.
- The wealth of previous studies in this area is testament to the size of the challenge that must be overcome, requiring a great amount of innovation on multiple fronts.

MILESTONES

Argonne National Laboratory

- Expand strong communications with SEISta team and initiate integrated efforts. – Completed
- Fabricate and evaluate updated baseline electrodes. – Completed
- Synthesize and evaluate performance of organosilane-functionalized and polymer-functionalized nano-Si particles by the condensation and atom-transfer radical-polymerization (ATRP) reactions. – On schedule
- Chemically convert the silicon oxide surface layer on silicon particles to a stabilized lithium silicate layer and investigate its impact on performance. – On Schedule
- Continue advanced diagnostic studies on baseline and advanced materials and electrodes in half, full, and reference electrode cells, using a broad range of electrochemical and analytical studies (e.g. NMR, XRD, spectroscopy, DEMS, ...) focusing on property performance relationships, surface characterization, and establishing degradation mechanisms. – On Schedule

MILESTONES

Lawrence Berkeley National Laboratory

MILESTONES

National Renewable Energy Laboratory

REVIEWER-ONLY SLIDES

The following slides are for the use of the Peer Reviewers only and will not be shown as part of the presentation at the Review.

MILESTONES

Oak Ridge National Laboratory

- Evaluation of gas evolved during electrode processing to be used to identify solvent and mixing processes. – Complete
- Spectroscopic characterization of cycled composite electrode identifying heterogeneity. – Complete
- Evaluation of gas evolution of cycled cells with and without additives. – On Schedule
- Explore bonding of PAA binders on silicon electrodes with spectroscopic tools. – On Schedule

MILESTONES

Pacific Northwest National Laboratory

- Synthesis micron sized Si with high porosity and improve its surface property. – Complete
- Investigate the properties of the SEI formed on the electrodes within different electrolytes. – Complete
- Developing electrolyte additive to enable a stable electrolyte and SEI upon storage. – Ongoing
- Developing Si materials with the artificial coating that can have high performance using current graphite electrolyte. – Ongoing

MILESTONES

Sandia National Laboratories

- Develop understanding of silicon SEI stability and reactivity for materials to enable next generation electrochemical energy storage systems. Research will focus on interfacial reactivity of active sites and SEI stability to understand interactions with other components used in lithium ion battery systems. – On schedule

EXTRA NOT USED SLIDES

To be deleted after working group at ANL
on 4/17-18/18.

APPROACH (ORNL)

- ORNL focuses on fundamental processing studies to optimize slurry formulation and resulting electrode performance
- Specialize in controlling chemistry to optimize coating quality

ACCOMPLISHMENTS (CAMP)

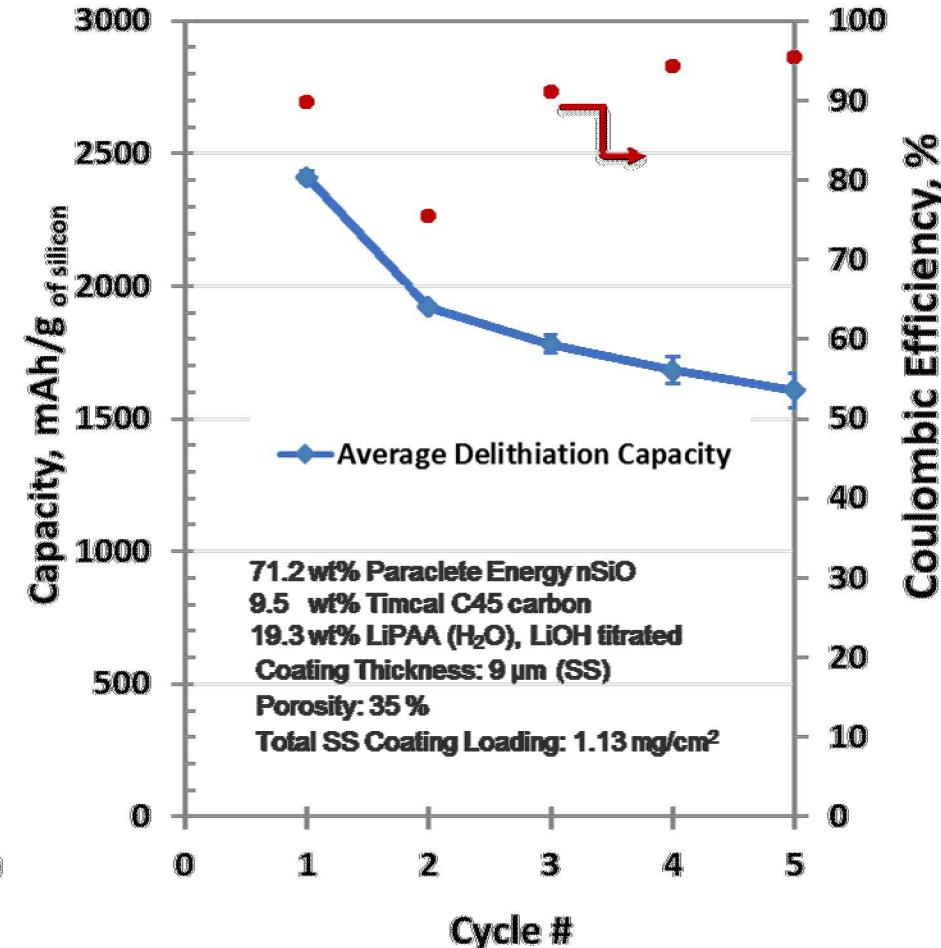
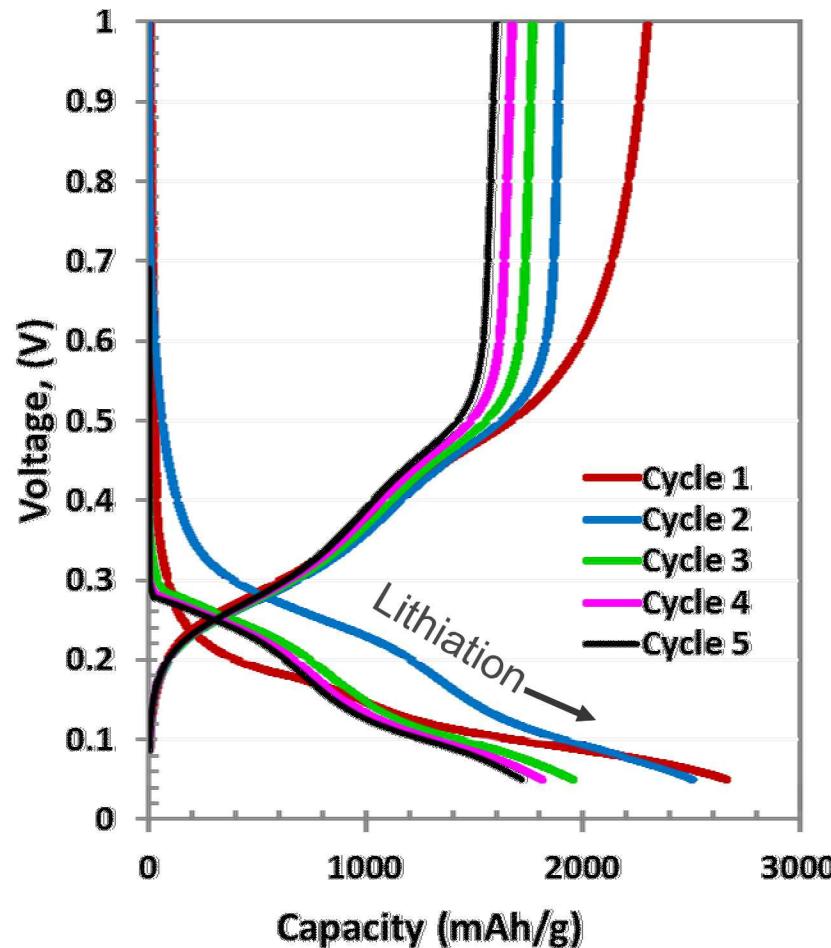
- Established relationship with Paraclete Energy as the main commercial source of silicon material, which can manufacture reproducible Si powders
 - Purchased large 4 kg batch with desired 150 nm particle size
 - Distributed to lab team
 - Characterized material properties
- Determined that source/method of silicon production yields significant differences in impurity levels
 - May have impact on formation of SEI layer and/or binder interactions

FUTURE WORK

- Use latest silicon material from Paraclete Energy in 15 wt.% Si cell build to establish comparison to original baseline material.
- Develop high Si-content (>30 wt.%) composite electrodes using lower amounts of other carbon materials (e.g., hard carbon) instead of graphite
- Continue to supply team with specialty electrodes and cells as needed

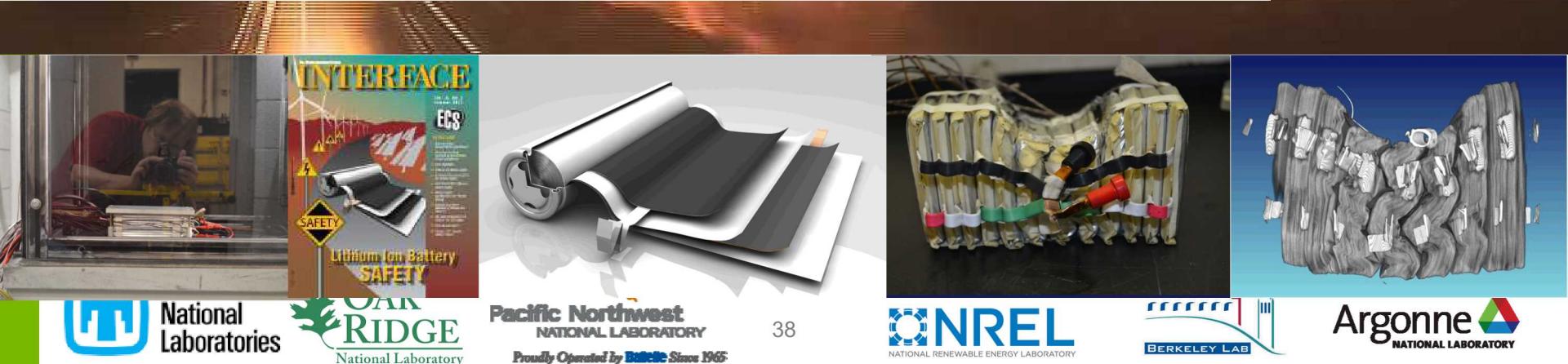
CHANGE DIRECTION OF ELECTRODE DESIGN

Initial electrochemical results of high silicon content (~70 wt.%) electrodes fabricated by CAMP Facility (at 50 mV cutoff)



SNL: NATIONAL LEADER IN BATTERY SAFETY R&D

- National leader in battery safety research and abuse testing with a focus on electric vehicle applications
 - Battery Abuse Testing Laboratory (BATLab)
 - Large scale battery calorimetry capabilities
 - Sophisticated thermal and fire modeling
 - Advanced materials development/analysis
 - Standards and testing protocol development
 - Extensive industrial partnerships
 - ARRA Award recipient in 2009



National Laboratories

OAK RIDGE
National Laboratory

Pacific Northwest
NATIONAL LABORATORY

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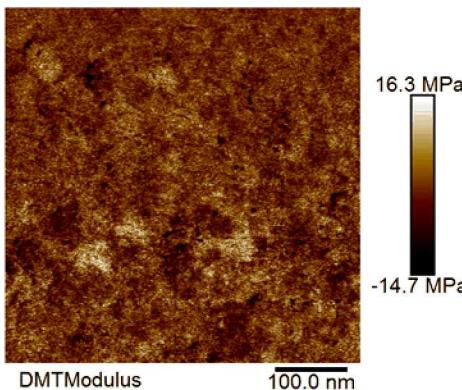
ONREL
NATIONAL RENEWABLE ENERGY LABORATORY

BERKELEY LAB

Argonne
NATIONAL LABORATORY

BATTERY AND MATERIALS TESTING AND DIAGNOSTICS LEVERAGE FOR SILICON

- Battery Test Facility used for Tee and cell level testing
 - Different Temperature and conditions for all cells
- Safety for scale up to 50A-hr testing per chamber
- Diagnostics (EC-AFM, XPS, XRD, EC-TEM, Auger, TOF-SIMS) available at need



EC-AFM image of Si-substrate after electrochemical cycling and poise at lithiating potential, see talk BAT348



Safety and scaling for up to 50A-hr capacity (boomboxes and hoods) with larger capacity at explosion pads



Battery aging facility; ~400 channels dedicated to lifetime monitoring of batteries under environmental conditioning

CALORIMETER



TAM IV

With or without Maccor cycling control

