



Silicon Electrolyte Interface Stabilization Update with Question and Answer Session

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Project ID # **bat387**

This presentation does not contain any proprietary, confidential, or otherwise restricted information.

Timeline

- October 1st 2016 - September 30st 2019.
- Percent complete: 40%

Budget

- Funding for FY19: \$3900K

Barriers

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

Partners

- Five Laboratory Team lead by NREL:
 - Sandia National Laboratory
 - Argonne National Laboratory
 - Oak Ridge National Laboratory
 - Lawrence Berkeley National Laboratory
- UC Berkeley, Colorado University Boulder, Colorado School of Mines, University of Rhode Island



Si anodes are ~10x higher capacity than graphite anodes

1. Si anodes have three major challenges to commercialization

- High Capacity Fade
- Poor Shelf Life
- Electrode formulation/stability

2. SEI formation in Si much more complex than in graphite, and seems to be dependent on initial state and history

- Large volume expansion on alloying
- Extensive gas formation upon

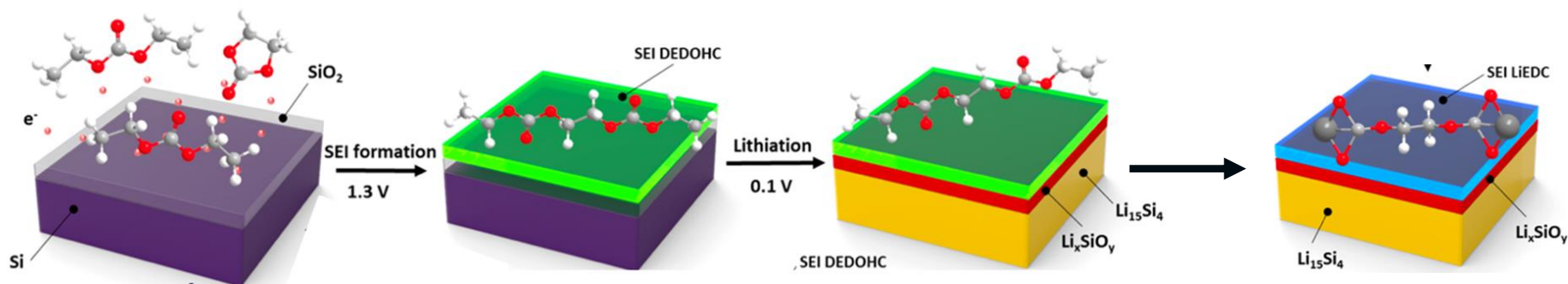
Objective:

Improve calendar life and understand initial stages of SEI formation by understanding intrinsic chemical reactivity of Si electrodes

FY19 Milestones

- Have determined if the pristine surface of the silicon influences the composition and the function of the SEI after 1, 10 and 50 cycles (XPS, SIMS, IR, and Raman, STEM, SSRM).
- Have determine that the nature of the silicon surface can affect the composition, function and the thickness of the SEI.
- Have determine how water concentration (as a function of water content up to 100 ppm) in the electrolyte affects SEI thickness and composition (electrochemistry, spectroscopy, impedance) of the SEI formed at 1.5, 1.0, 0.7, 0.4, 0.15 and 0.05V vs Li/Li+”
- Have determine the nature of the soluble SEI components, that are formed over 10 cycles, that are soluble in the gen 2 electrolyte.

SEISta – Approach and Overview



[J. Phys. Chem. C 2017, 121, 14476–14483](#)

Overarching Mission: Develop a stable SEI layer for Silicon Anodes to enable the use of intermetallic anodes for lithium ion batteries.

This is not a new challenge but we believe that the difficulties working with silicon have precluded a “quick fix” to long term stability of silicon electrodes.

We require a more foundational understanding of the formation and evolution of the SEI on silicon.

Understand first, fix later!

Approach

Critical Questions:

- What are the properties of the lithiated silicon electrolyte interface?
- What is the Silicon SEI actually made of and what reactions are contributing to it?
- How fast does the Silicon SEI grow?
- Does it stop growing?
- Is it soluble?
- Can it be stabilized?

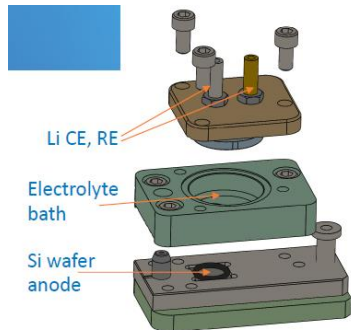


Team work

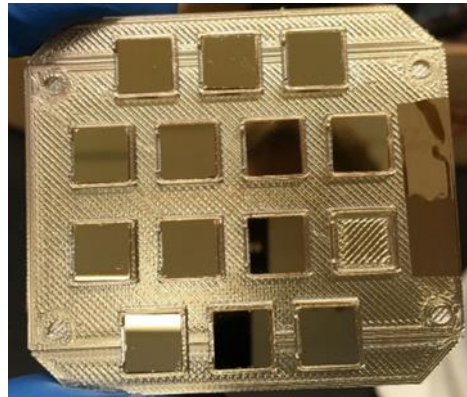
- Coordination with the Deep Dive
- Understanding is critical
- Multiple characterization tools on the same problem
- Well characterized samples
- Standardized protocols
- Reproducibility across the team (multiple labs)
- Communication !!!!
 - All information is stored on BOX
 - Weekly team meetings (video)
 - Quarterly face to face
 - Multiple side phone meetings
 - Site visits by researchers to other labs

Standardized model electrodes applied across the program for reproducibility

Well defined electrodes and cells are shared across the whole project.



O-ring 3-electrode cell to control surface exposure



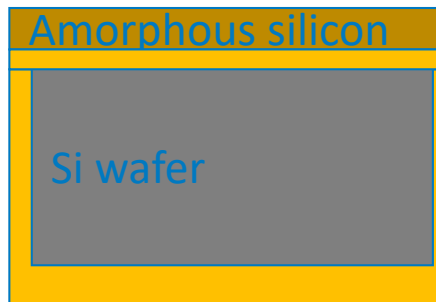
Sputter deposited Si heterostructures



Polished Cu rod



- Custom “x-wing” cell

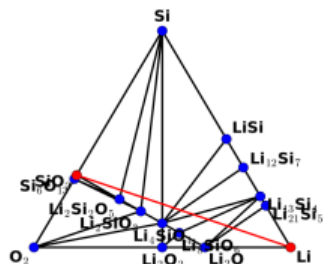


Copper layer



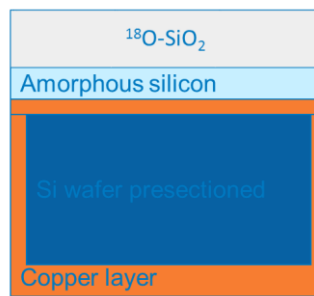
Baselines must be consistent across all partners. The life history of the silicon sample may play a critical role in the formation and evolution of the SEI. If each partner is not “making the same measurements on the same sample” then progress is going to be slow.

High Priority Thrusts : Lithium Silicate and SEI Stability



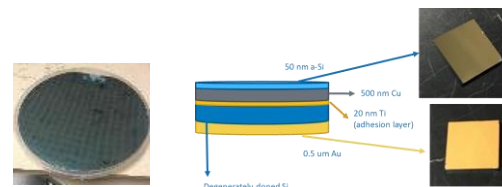
“Equilibrium” pathway for lithiation of crystalline SiO_2 can be followed along the red line shown in the bulk Li-Si-O phase diagram obtained from the Materials Project database.

Si^{18}O_2 samples grown by Gabe V.

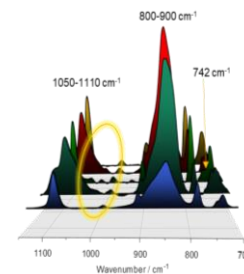
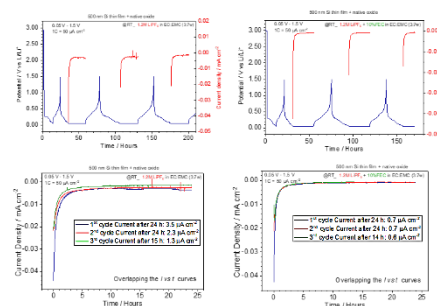


We will focus a significant experimental effort to understand the different mechanisms that are present in the long term stability of the SEI:

Careful electrochemistry on model electrodes



Precision electrochemistry coupled with detailed spectroscopy

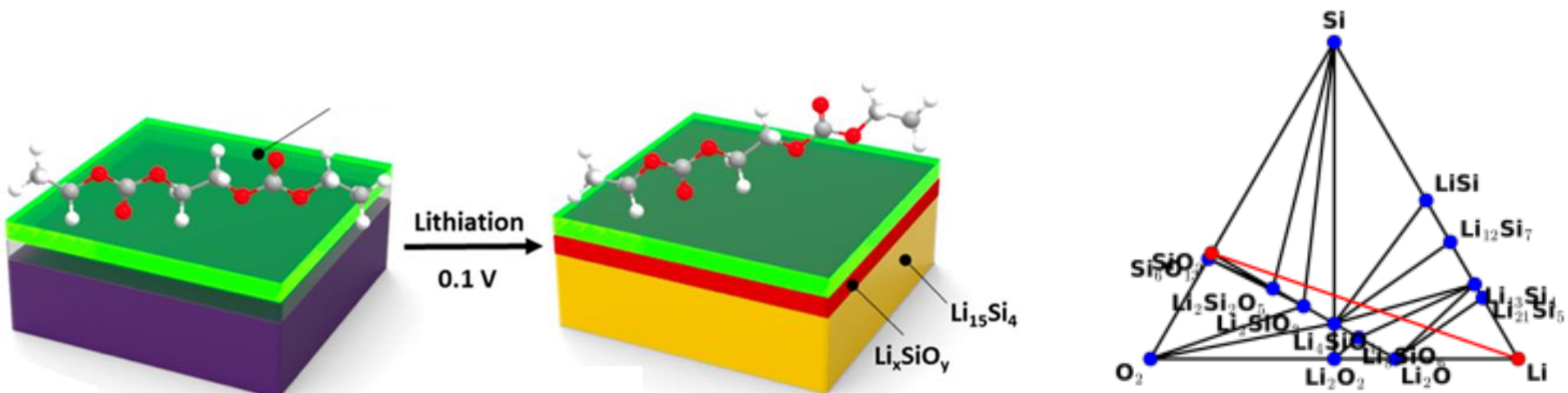


Using model lithium silicate thin film layers on silicon thin film anodes to determine effect of lithium silicate formation on evolution of silicon SEI

Do Lithium silicate films have an effect on the lithiation kinetics and SEI formation and evolution?

Does the SEI on Silicon ever passivate?

Priority Thrust 1: What Role Does Silicon Dioxide Play ?

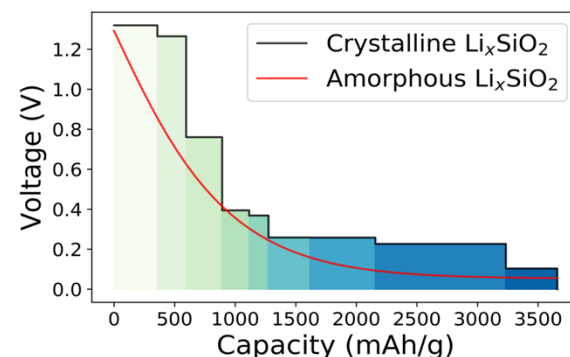
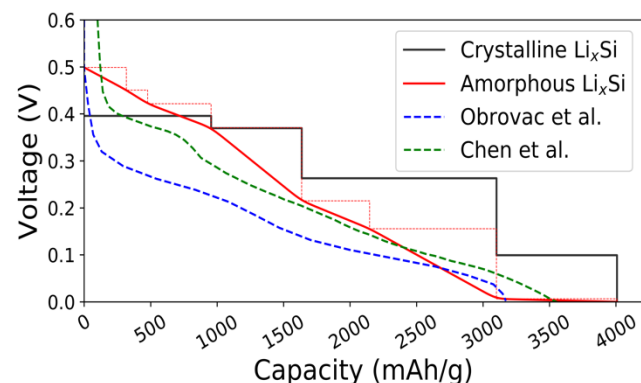
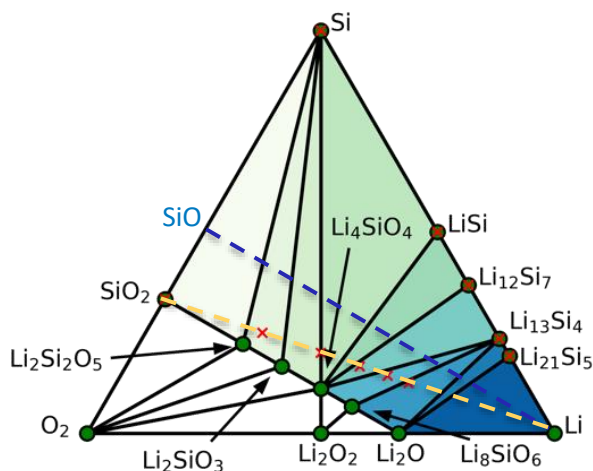


SiO₂ is a wide bang gap material how does it allow for any electrochemistry?

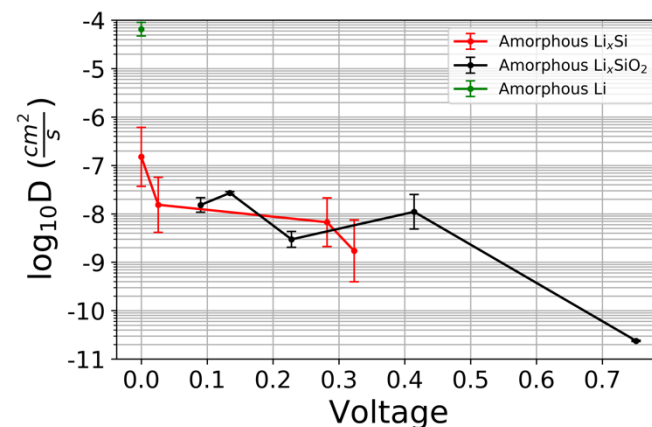
Thermodynamics suggest that conversion of the SiO₂ to Li₂O and Si will occur does it?

How does the evolution of the SiO₂, lithium silicate, effect the stability of the SEI?

Priority Thrust 1: Phase diagram (Si-Li-O) including amorphous phases

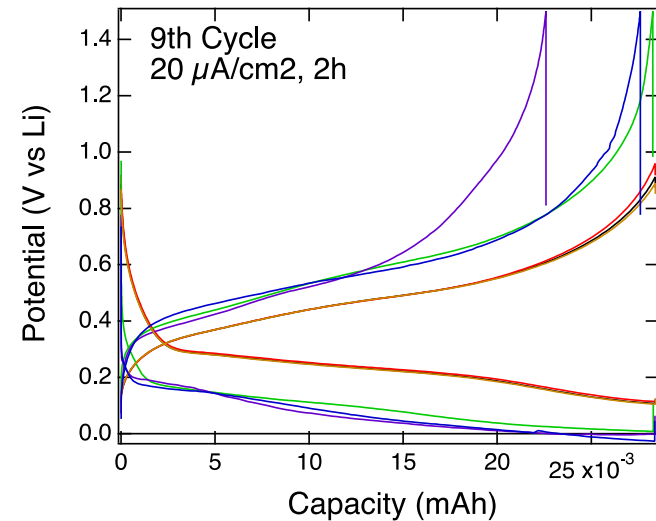
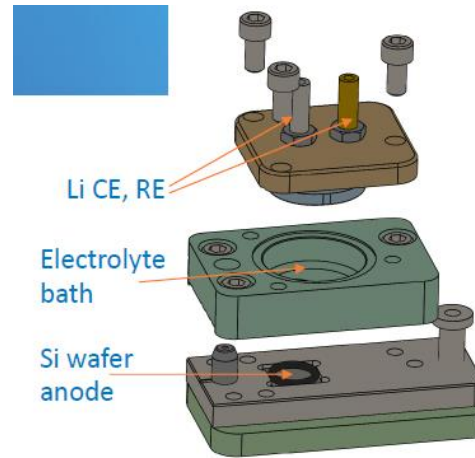
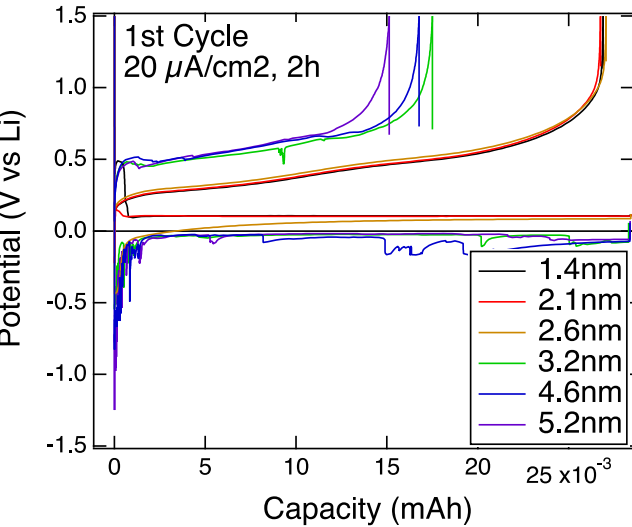


- Amorphous phases are metastable with respect to their crystalline and phase separated counterparts
- Lithiation of c-SiO₂ is thermodynamically favorable.
- Structural evolution of amorphous lithiated SiO_x agrees with phase diagram
 - Forms regions of Li₂O and Li_xSi
- The voltage profile of amorphous SiO₂ is 0.8 V higher than that of amorphous Si, providing a clear signature when SiO₂ is lithiated
- Li diffusion in amorphous SiO₂ starts out 2 orders of magnitude lower than in Si, for low Li content, which presents a kinetic bottleneck.

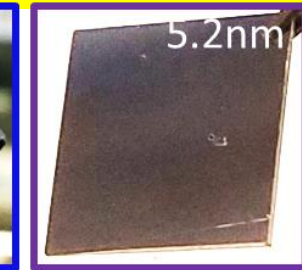
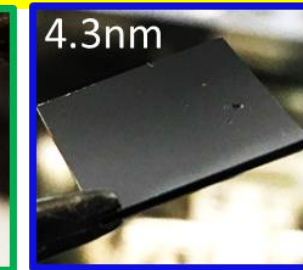
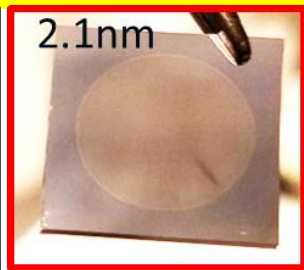
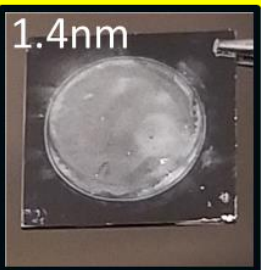


Priority Thrust 1: SiO₂ on Si: Effect of Thickness on Cycling

Control thickness SiO₂ on silicon wafers were used to determine the affect of SiO₂ on lithiation using the O-ring cell to ensure that only the wafer surface can participate in electrochemistry



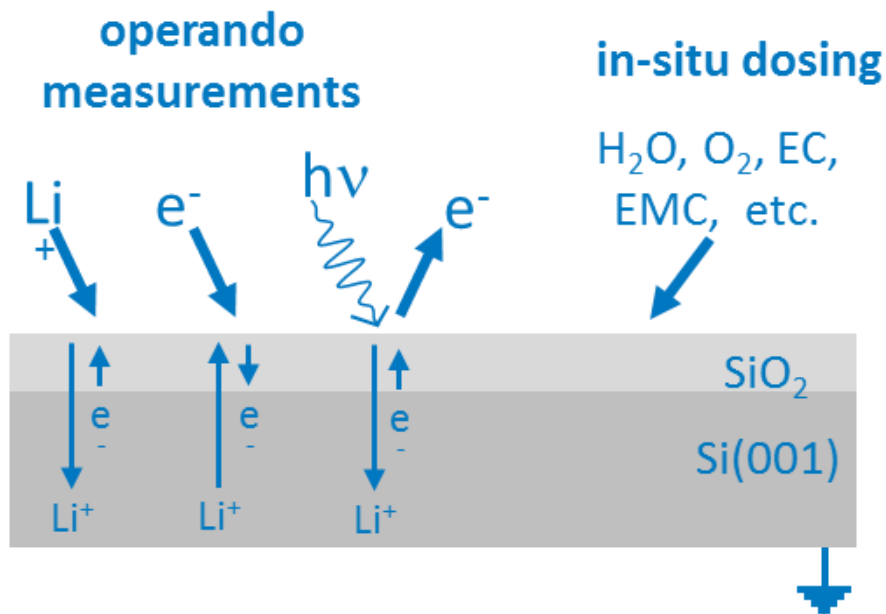
There is a strong dependence of $20\mu\text{Acm}^{-2}$ cycling on thickness of SiO₂ on Si, with no lithiation observed above 3.2 nm SiO₂. However, long term cycling resulted in breakthrough even in SiO₂ coatings above 4 nm. Thick SiO₂ prevents lithiation of Si unless pinholes are present.



Virtual-electrode approach for *operando* and *in situ* studies of battery materials

Virtual-electrode capabilities developed at NREL

- Li^+ ion gun
 - 1–1000 eV incident energies
 - isotopically enriched ^6Li cathode
 - typical current densities $\sim 200\text{--}500 \text{ nA/cm}^2$
 - raster and pulsing capabilities
- e^- gun
 - 1–1000 eV incident energies
 - typical current densities $\sim 1\text{--}5 \text{ }\mu\text{A/cm}^2$
 - raster and pulsing capabilities
- light source
 - 365 – 850 nm high-power LEDs (CW-500 kHz)
 - can drive photoelectron current ($\phi < 3 \text{ eV}$)
 - e^- - h^+ pair generation



Virtual electrode approach

- Can drive Li^+ and e^- currents at **exposed interfaces** in battery materials and devices
- Concurrent XPS measurements reveal **compositional changes and phase transformations**
- XPS binding-energy shifts reveal **chemically resolved overpotentials**

Priority Thrust 1: XPS *in-situ* lithiation: 5-nm SiO₂/Si(001)

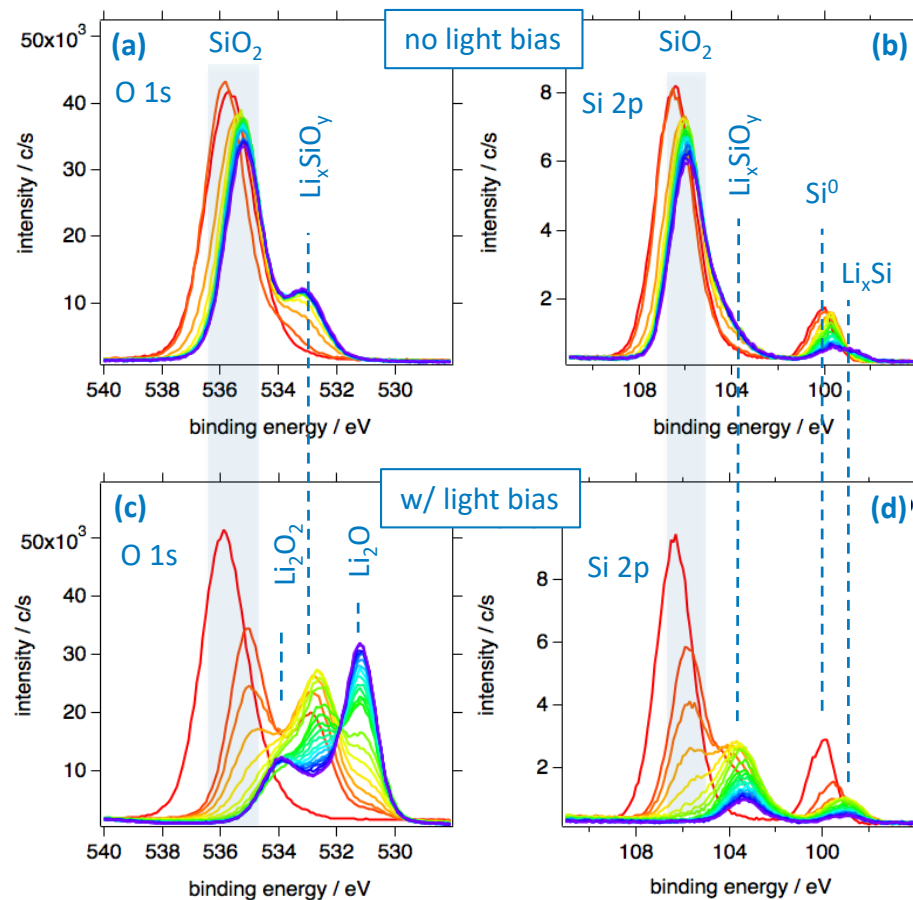
In-situ lithiation experiments

- Model-system studies on Si(001) wafer samples probe formation and evolution of inorganic SEI constituents
- Light bias controls e⁻ population at interface

Results for in situ lithiation of 5-nm SiO₂/Si(001)

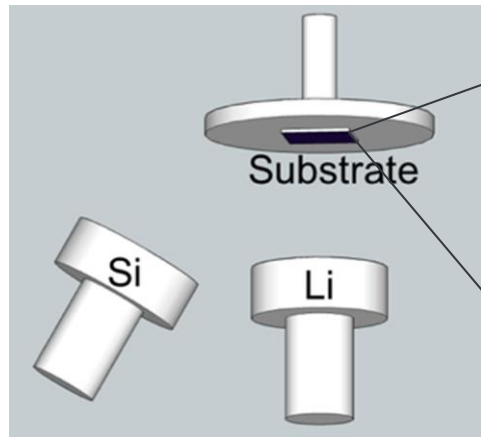
- XPS chemical states associated with **Li_xSiO_y** and **Li_xSi** phases are clearly resolved
- Net overpotential required to drive Li⁺ ion current ~500 nA/cm² is ~2.4 eV
- SiO₂ layer remains partially intact**; only ~20% of SiO₂ is converted to Li_xSiO_y phase
- Results show that **formation of silicates is controlled by availability of electrons** at SiO₂/Si(001) interface according to:

$$x \text{ Li}^+ + x \text{ e}^- + \text{SiO}_y \rightarrow \text{Li}_x\text{SiO}_y$$

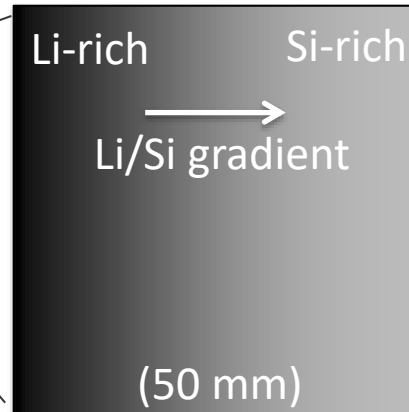


Priority Thrust 1: Li_xSiO_y films is as model system for lithiated native oxide on Si

Li/Si co-sputtering



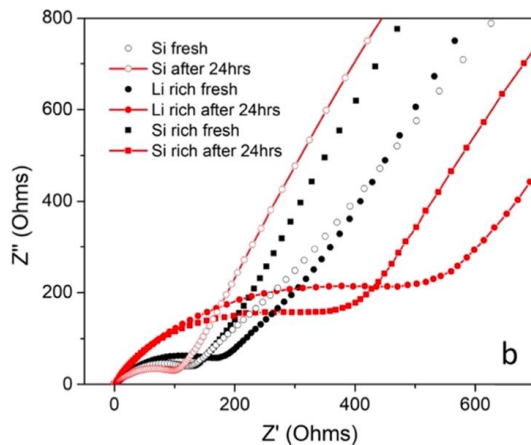
Li/Si composition spread



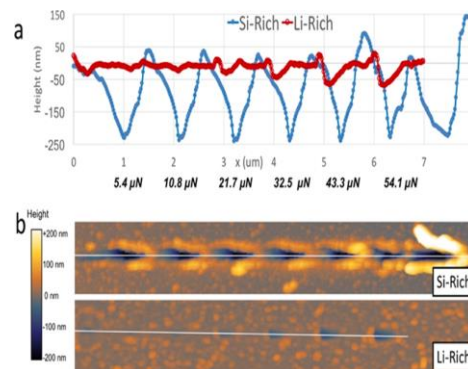
Experimental methods

- Li_xSiO_y films with Li/Si gradient by co-sputtering from Li and Si targets
- Li_xSiO_y electrical & mechanical properties measured by EIS & AFM
- Cycling of $\text{Li}_x\text{SiO}_y/\text{Si}/\text{Cu}$ -foil stacks compared to Si/Cu-foil reference

Electric properties



Mechanical properties



Experimental results

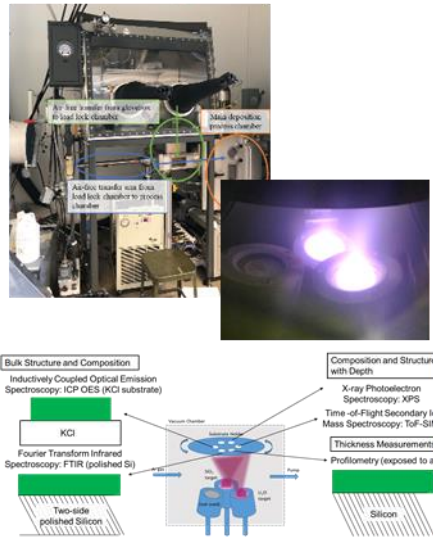
- Li_xSiO_y films have ductile mechanical properties and low impedance
- $\text{Li}_x\text{SiO}_y/\text{Si}$ have worse cycling performance compared to Si
- Consistent with results of first principles calculations

Conclusion: Li_xSiO_y is not beneficial in stabilizing the Si anode surface during battery operation

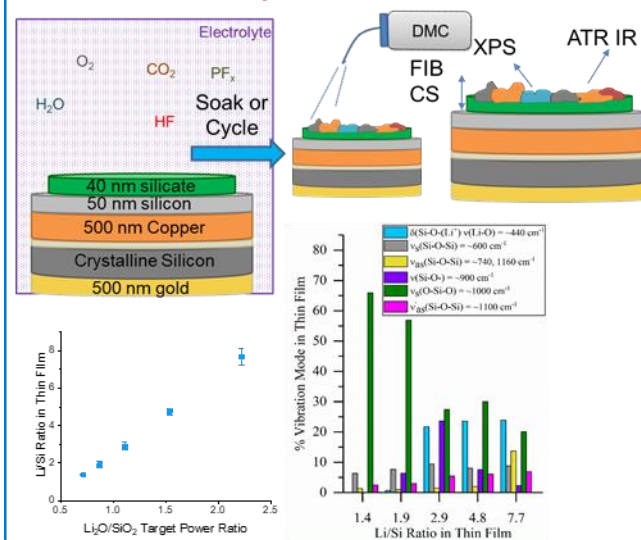
Xu, Y., Stetson, C., Wood, K., Sivonxay, E., Jiang, C.S., Teeter, G., Pylypenko, S., Han, S.D., Persson, K., Burrell, A.K. and Zakutayev, A., "Mechanical Properties and Chemical Reactivity of Li_xSiO_y Thin Films, ACS Applied Materials & Interfaces 10, 44 (2018)

Priority Thrust 1: Modeling SEI formation Through Engineered Interfaces: Lithium Silicate Studies

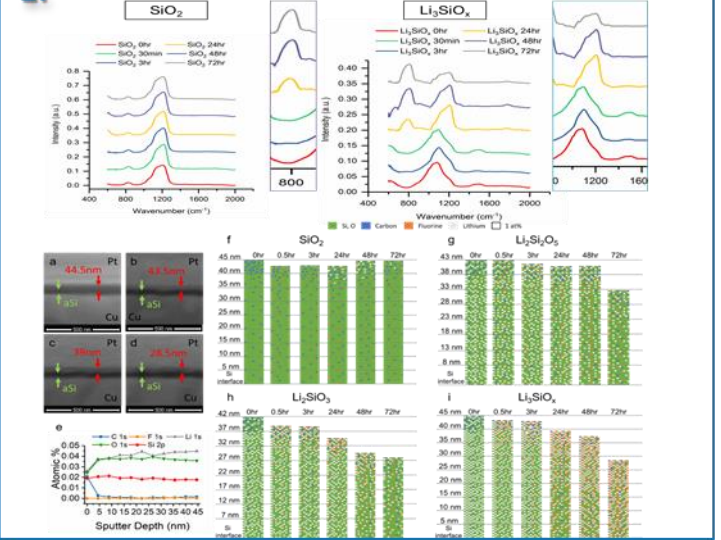
Sample Creation



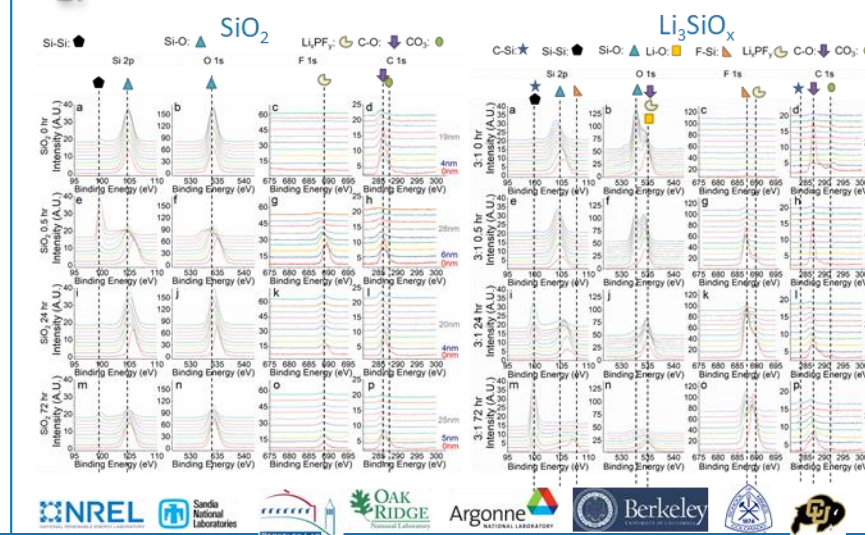
Sample Treatments



1. Soaking Evolution: FTIR, XPS, FIB



2. XPS Binding Energy Shifts Show Significant Role of F



- Ex-Situ FTIR (no exposure to atmosphere) after soaking in gen-2 electrolyte shows composition evolution over time on exposure for high order silicates; combined XPS/thickness graphs show increasing role of F with disordered structure as electrolyte decomposes existing film over time
- XPS composition analysis shows very different surface and depth speciation between un lithiated oxide (SiO₂) and highly lithiated oxide (Li₃SiO₃)
- For SiO₂, slow formation of LiPF₆ on surface over time, but little interaction of the fluorine with Si and little penetration of Li into bulk
- In Li₃SiO₃, significant fluorine penetration through the bulk of the film starting very early in exposure and continuing
- More C-O products on surface may indicate more solvent decomposition, as well as more salt decomposition (as a source of F)

Even if lithium silicates form they are not stable in traditional electrolytes

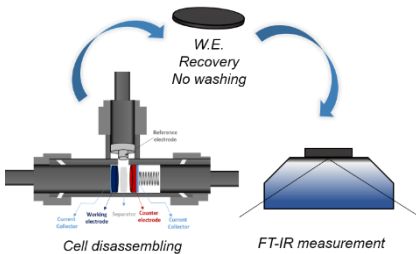
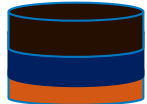
Priority Thrust 1: Understanding processes occurring at the Silicon/electrolyte interphase: SEI stability and Effect of SiO_2

Investigated systems

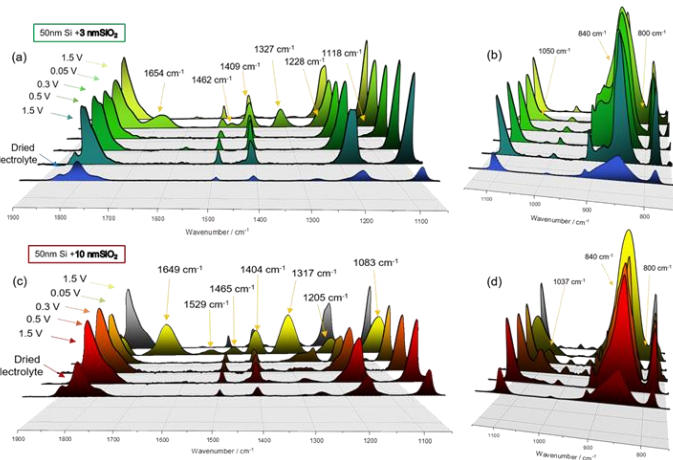
50 nm Si +
3nm SiO_2



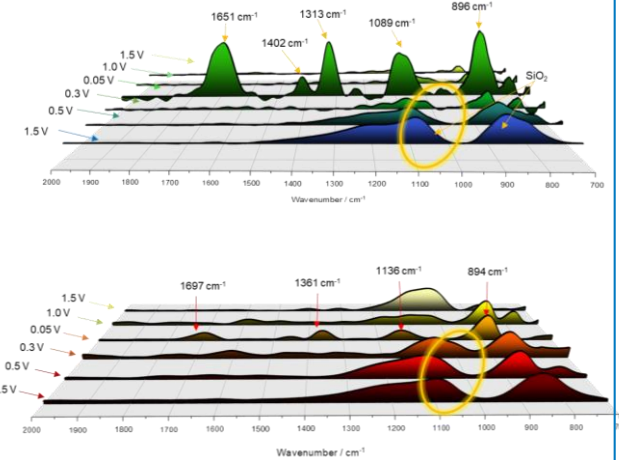
50 nm Si +
10nm SiO_2



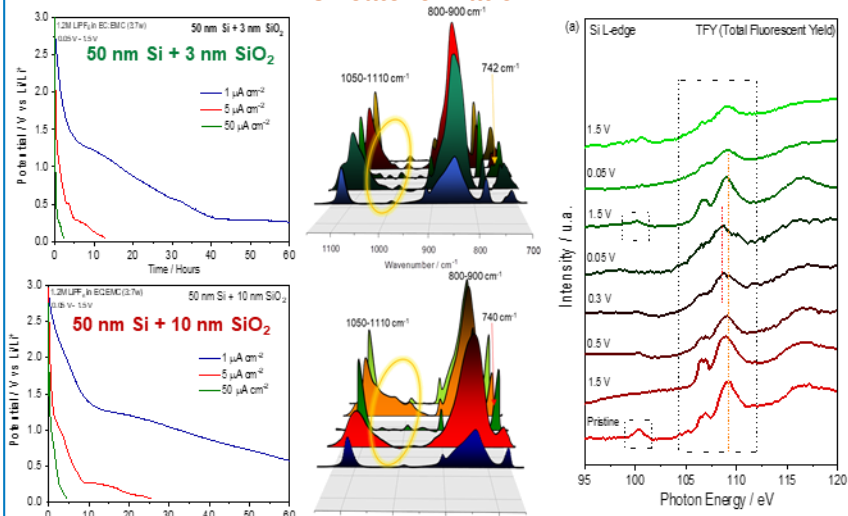
1. SEI on un-washed Silicon thin film electrodes: Visualization of the SEI breathing



2. SEI on washed Silicon thin film electrodes: Detection of the insoluble SEI components



3. Accumulation of SEI and conversion reaction products: silicate formation



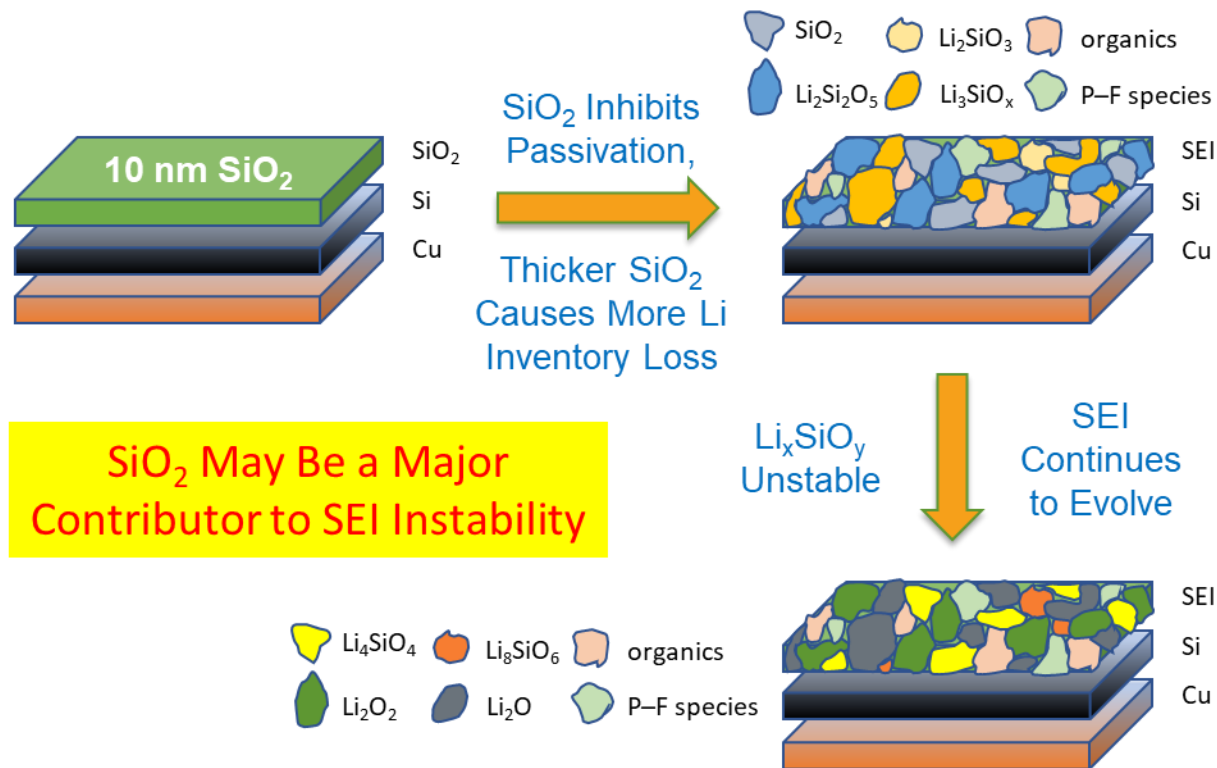
- Ex-situ FTIR on the two model electrodes investigated reveals the instability of the passivating layer formed on silicon.
 - LiEDC and P-F containing compounds form and disappear upon cycling for both model electrodes.
- Ex situ FTIR on washed electrodes enable the detection of the insoluble products of the SEI. LiEDC is an insoluble product of SEI. P-F containing compounds are soluble.
 - By rinsing the electrodes, new features appear at low frequency region and are most likely related to the electrochemical activity of SiO_2 .
- More current is involved during the charging process with a thicker SiO_2 layer. SiO_2 enhances electrolyte decomposition and delays the lithiation process.
 - XAS analysis at the silicon L-edge indicates modification of the SiO_2 double peak feature, indicating activity of the SiO_2 layer.
 - Ex-situ investigation upon cycling enables the detection of accumulation of species most likely related to the formation of non-stoichiometric silicates. Suggested reversibility of the process, which links the silicates to the breathing effect of the SEI together with LiEDC and P-F containing compounds.

The SEI on SiO_2 continually changes on cycling.

Summary Priority Thrust 1: : SiO₂: The Good, The Bad, and The Ugly

Key Issues:

- Si nanoparticles (NPs) often contain silicon dioxide (SiO₂).
- Thick SiO₂ prevents electron and lithium conductivity, but changes over time to enable lithiation.
- Nature of the SiO₂ (crystalline, amorphous, Si/O ratio, etc.) affects its chemical and electrochemical stability.
- SiO₂ is a key component of the early-stage SEI and evolves to lithium silicates (Li_xSiO_y) during cycling.
- Li_xSiO_y also unstable and evolves during cycling.



Priority Thrust 2: Parasitic reaction and SEI instability

Two different protocols have been developed for the characterization of the parasitic currents

STEP 1: Carry out 1 galvanostatic cycle at 1C from OCV to 0.05 V and back to 1.5 V

STEP 2: Charge the electrode galvanostatically at 1C until the electrode reaches $E_t = 0.5, 0.3$ or 0.05 V vs. Li/Li^+

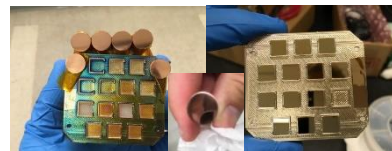
STEP 3A (PROTOCOL 1: GCPL+CA)

- Hold the potential at E_t and record the current/charge for at least 24 hours.
- Perform another galvanostatic cycle at 1C from E_t to 1.5 V and back to E_t . Hold the potential at E_t and record the current/charge for at least 24 hours.
- Continue the cycling and record the current/charge at E_t for at least 24 hours...

STEP 3B (PROTOCOL 2: GCPL+OCV)

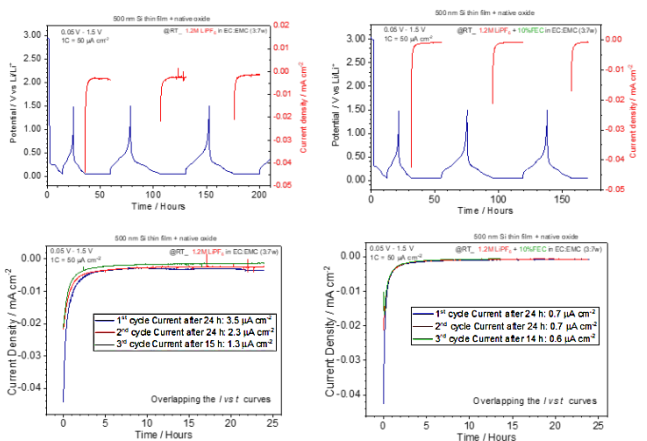
- After reaching E_t open the circuit voltage and record the electrode potential for at least 24 hours.
- Perform another galvanostatic cycle at 1C from E_t to 1.5 V and back to E_t . Open the circuit voltage and record the electrode potential for at least 24 hours.
- Continue the cycling to E_t and record the electrode potential at OPC for at least 24 hours...

Several model electrodes have been screened before selecting the 500 nm Si thin films as standard platform.



Substrate	Cu piston	Cu piston + protective plastic	Cu thick disk	Cu foil
Residual Current after 1 st cycle	2.7 $\mu\text{A cm}^{-2}$	1.8 $\mu\text{A cm}^{-2}$	2.6 $\mu\text{A cm}^{-2}$	1.0 $\mu\text{A cm}^{-2}$

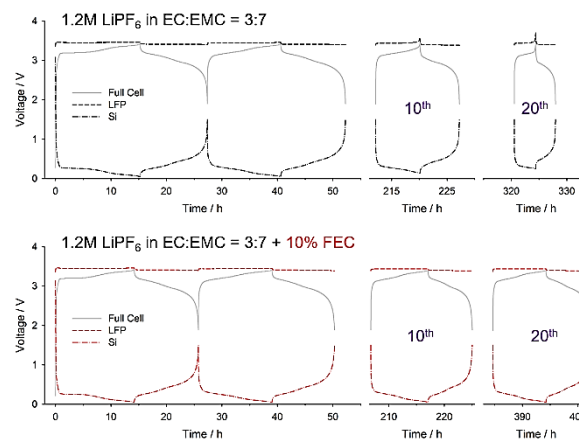
Effect of FEC: LiPF_6 vs LiPF_6 + FEC in half cell configuration



Using protocol 1 to investigate the passivating properties of silicon in half cell configuration. The residual current related to parasitic reaction with the standard Gen 2 electrolyte are 5 times higher than the one obtained by adding FEC.

FEC addition greatly improved the passivating properties of silicon.

Effect of FEC: LiPF_6 vs LiPF_6 + FEC in full cell configuration



Using LFP as source of limited amount of lithium to investigate silicon SEI evolution and lithium consumption.

As the cycle goes on, lithium in the system is consumed due to the reactivity of silicon. This directly affects the cycle performance of full cells. Adding FEC improved the cycle retention, implying FEC can decelerate lithium consumption on the silicon surface.

Other effects have been investigated with different model electrodes. The list of effects and the corresponding conclusive observations are reported in the Table.

Investigated Effects:

Effect of the salt : LiPF_6 vs LiTFSI

Effect of additives: FEC

Effect of the state of charge on corrosion current

Effect of Silicon thickness: decoupling cracking effect from catalytic activity of Si

Effect of SiO_2 on passivating behavior

Conclusions:

LiTFSI based electrolytes exhibit lower residual currents. Improved passivation.

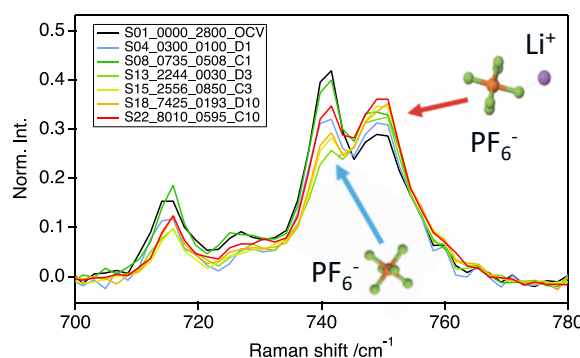
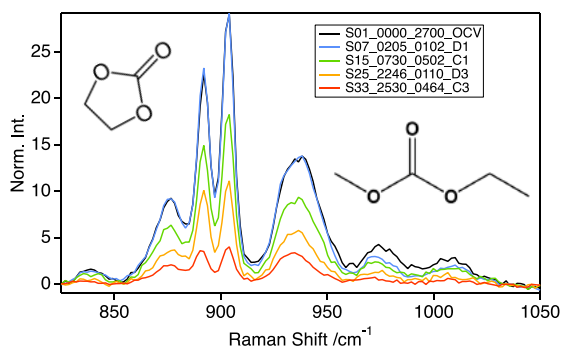
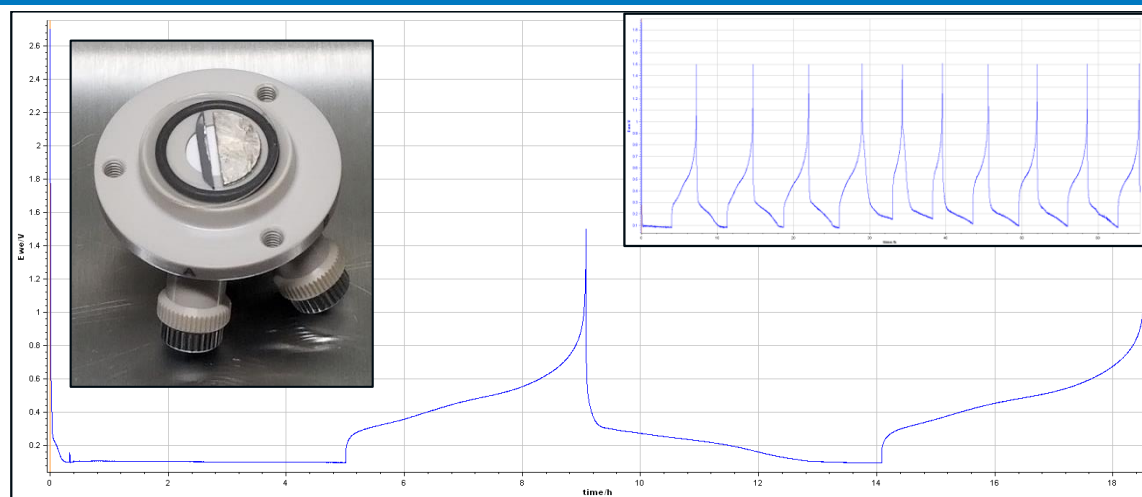
FEC improves the passivation properties of Si in half and full cell configuration.

Larger currents are observed at fully lithiated state (0.05 V) rather than at 0.5 V.

The cracking effect is responsible of about 60 % of the parasitic currents

SiO_2 inhibits passivation of silicon surface

Priority Thrust 2: Reactivity and Evolution of SEI during Cycling using *in-situ* Raman

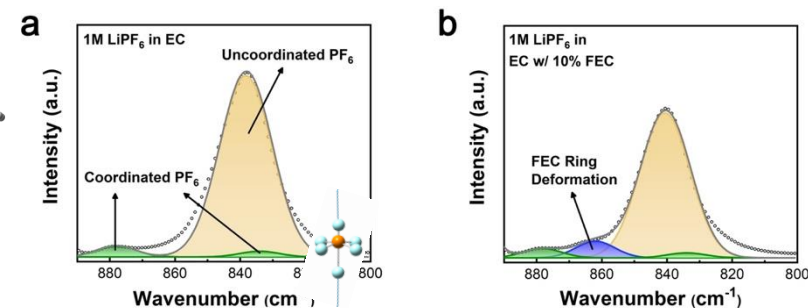
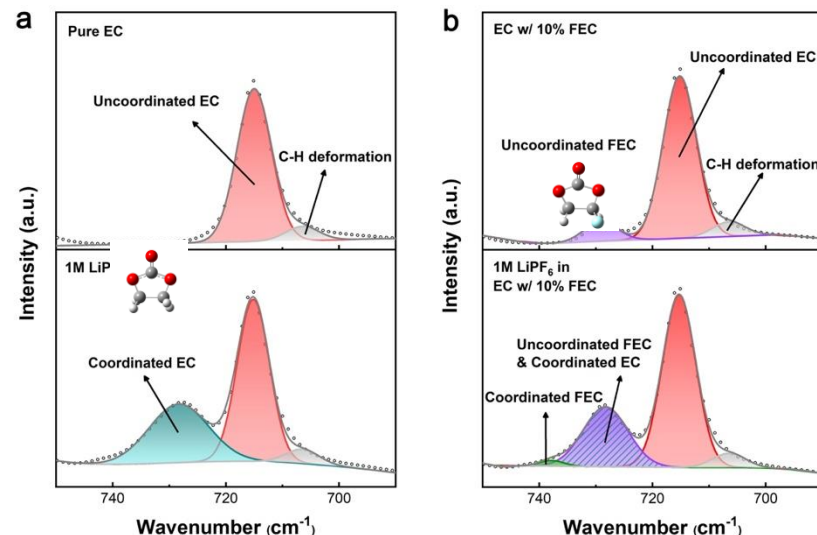
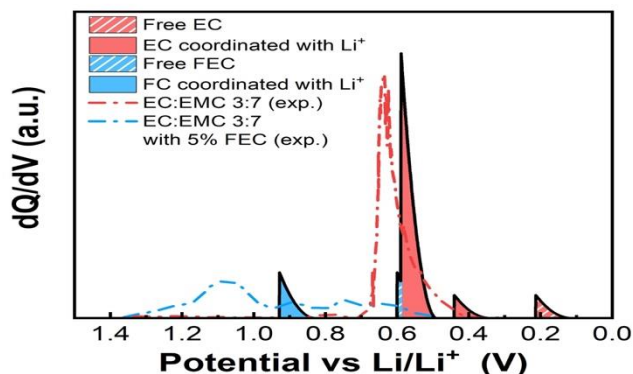
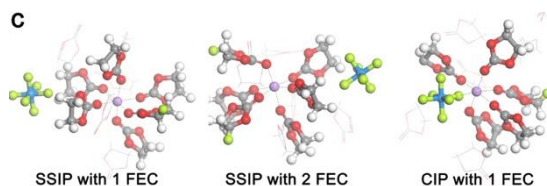
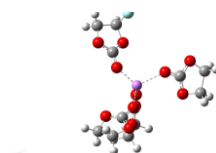
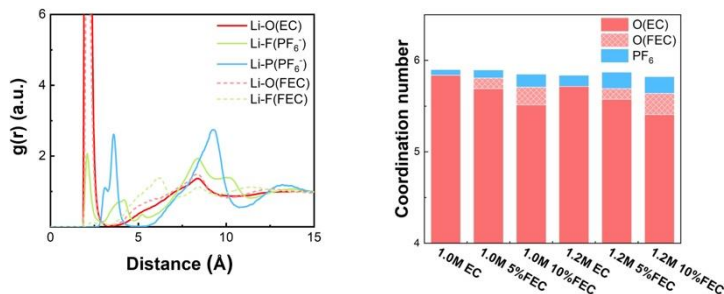


Developing *in-situ* spectroscopic analysis can provide new insight and help in elucidating the mechanism of interfacial failure in battery systems.

Raman Band Center	Assignment	Species of Origin
380	E_g	Al_2O_3 (from sapphire window)
417	A_{1g}	Al_2O_3
431	E_g	Al_2O_3
450	E_g	Al_2O_3
472		PF_6^-
480 (very broad)		a-Si (amorphous silicon)
513		?
520	1TO	c-Si (crystalline silicon)
530		?
556		PF_6^-
570		PF_6^-
578	E_g	Al_2O_3
645	A_{1g}	Al_2O_3
720	o, O-C-O	EC (ethylene carbonate)
728	o, O-C-O	FEC (fluoroethylene carbonate)
733	o, O-C-O	$EC \cdots Li^+$
740	ν , P-F	PF_6^-
742	ν , P-F	$PF_6^- \cdots Li^+$
748	E_g	Al_2O_3
868	ν , C-F	FEC
897	β , C-C	EC
903		EMC (ethyl methyl carbonate)
905		$EMC \cdots Li^+$
908	β , C-C	FEC
932	δ , O-C-O	EMC
945	δ , O-C-O	$EMC \cdots Li^+$
970		EC
1455		?
1488	δ , CH_3	EC
1705-1730	ν , C=O	$EMC \cdots Li^+$
1748	ν , C=O	EMC
1778	ν , C=O	EC
1804	ν , C=O	EC

ν = stretching, δ = bending, o = ring breathing; β = ring deformation

Impact of FEC on Solvation structure of LiPF_6 EC/FEC bulk electrolytes



The solution chemistry of the electrolyte directly influences the formation and stability of the SEI

- After FEC inclusion into EC and EC/EMC electrolytes we find:
 - Contact ion pairs (CIP) ratio increased from 6% to 14%
 - FEC appears in the first solvation sheath with a coordination number of 0.2
- Experimental and calculated FTIR confirmed the solvation structure obtained from MD simulations
- Simulated reduction potentials compare well with experimental one; in terms of both population statistics as well as magnitude. Specifically, we find that FEC contributes to an early onset of SEI formation and anode passivation through the promotion of contact ion pairs (CIPs) which facilitate the formation of LiF ;

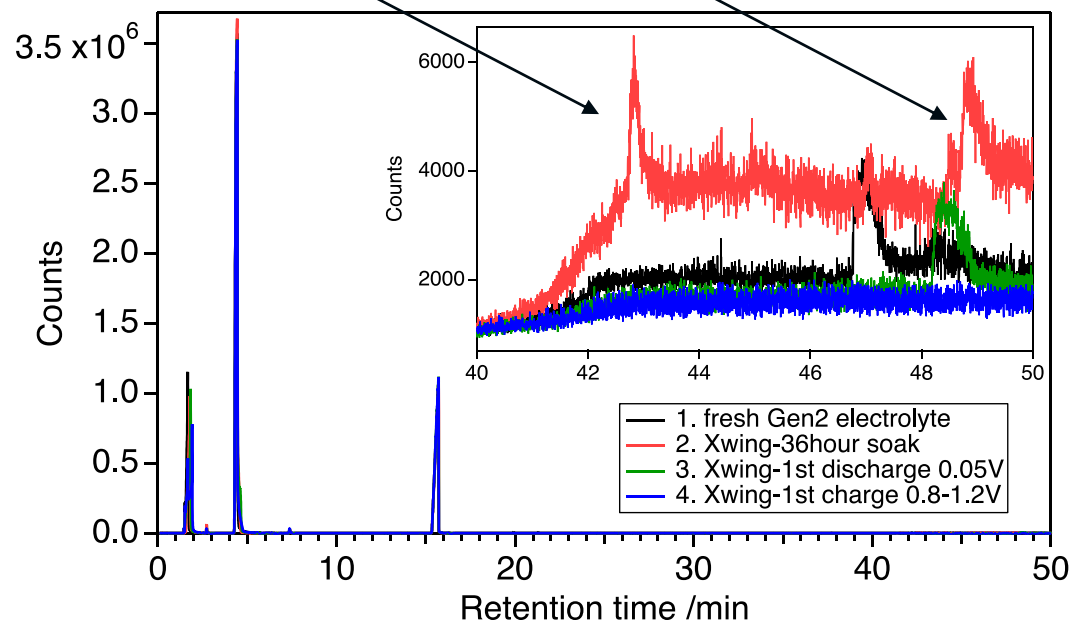
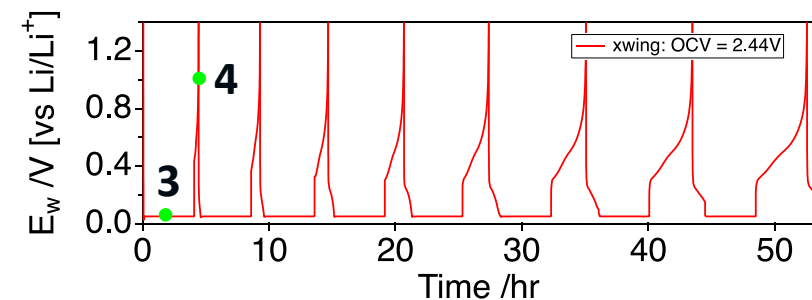
Nature of the Soluble SEI Components during Cycling : GC-MS with a X-Wing Cell



- Custom “x-wing” cell to sample electrolyte during cycling
- Electrolyte samples run through GC-MS to detect soluble decomposition products

Chemical degradation
of electrolyte

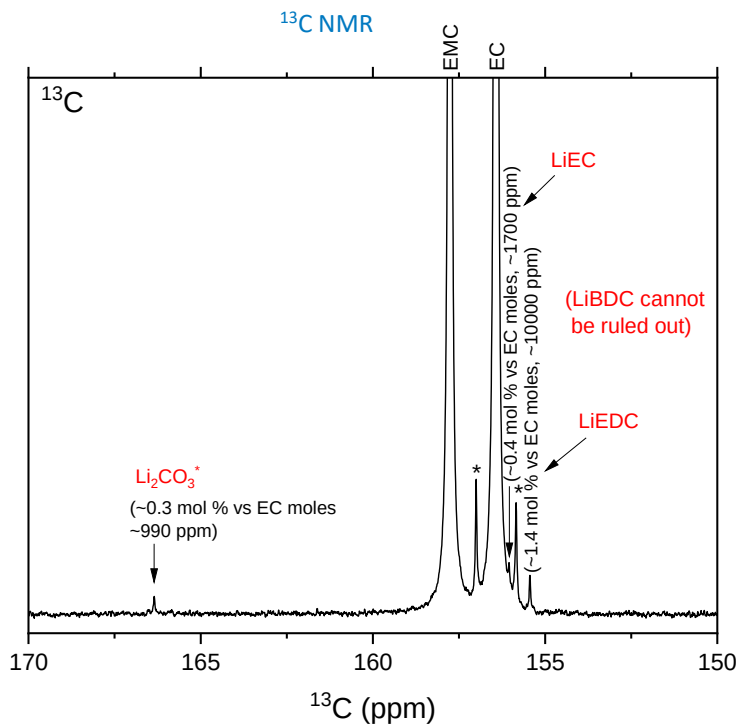
Possible species from chemical reaction
of electrolyte with Si anode



Using GC-MS to detect electrolyte decomposition and soluble SEI components before, during and after battery cycling

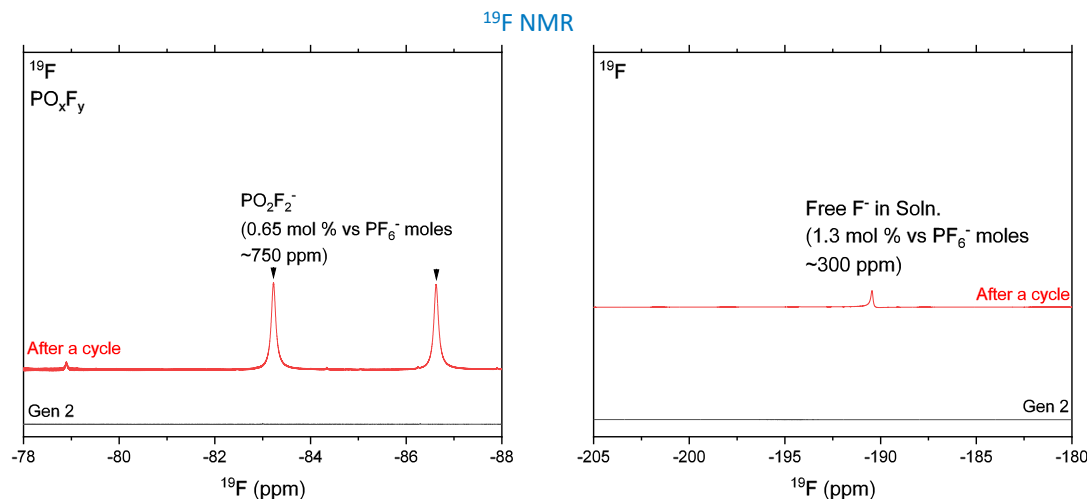
NMR: Quantification of Soluble SEI species

SEISta Half cells post 1 cycle NMR characterization – Gen 2 electrolyte – Wafers with amorphous silicon



* Lithium carbonate (along with other carbonates) solubility is expected to be very low therefore internal standards are required in future experiments for accurate quantification

A protocol for quantitatively measuring soluble SEI products in standard SEISta research samples is established using NMR spectroscopy. The findings suggest significant electrolyte decomposition and lithium bearing SEI species solubilization in the electrolyte. For accurate quantification, internal standards are planned to be used in the future. Extended cycled standard cells will be analyzed to monitor the buildup of the identified and/or new species.



Jin et al., *J. Am. Chem. Soc.*, 2018, **140** (31), 9854.

Li et al., *J. Phys. Chem. C* 2008, **112**, 12550.

Michan, Leskes and Grey, *Chem. Mater.* 2016, **28** (1), 385.

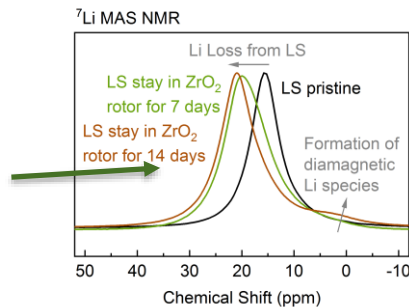
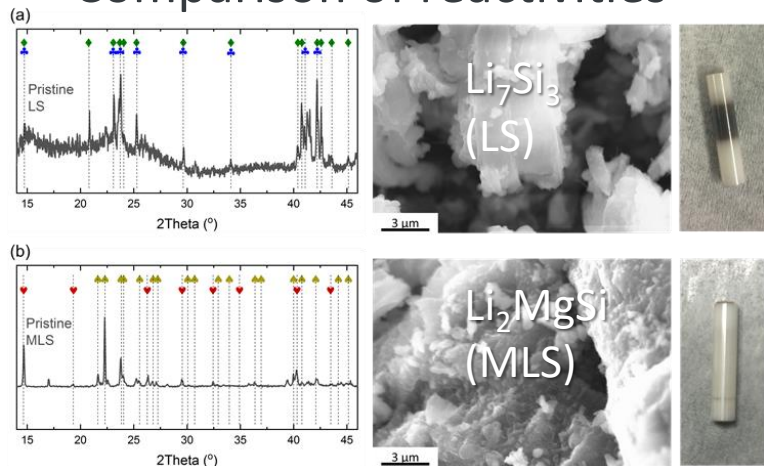
Tasaki et al., *J. Electrochem. Soc.*, 2009, **156** (12), A1019.

<http://citeseerx.ist.psu.edu/viewdoc/download?doi=10.1.1.826.1172&rep=rep1&type=pdf>

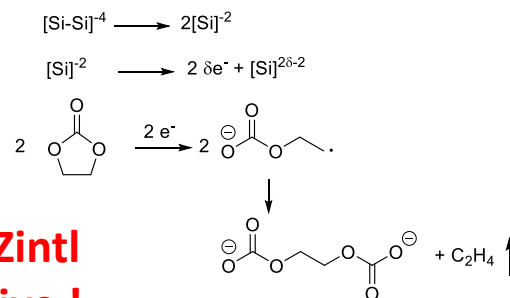
Using NMR to detect electrolyte decomposition and soluble SEI components provides quantitate data

Chemical stability of Li-Mg-Si TERNARY ZINTL SYSTEM

Comparison of reactivities



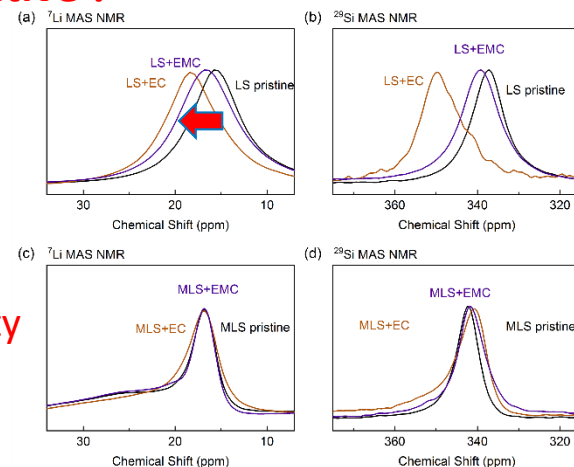
Proposed Si-anion reactions



Si^{-2/-4} anions in Li-Si Zintl phases are too reactive !

Direct evidence of Li loss from the bulk

Direct evidence of bulk stability

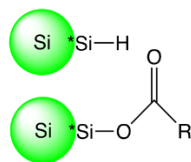


Binghong Han, Chen Liao, Fulya Dogan, Stephen E. Trask, Saul H. Lapidus, John T. Vaughey, Baris Key, *Energy & Environmental Science*, 2019, submitted

Using understanding gained in previous work suggests that stabilizing the lithium silicide may be a route to longer life silicon anodes.

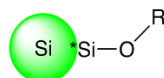
Instability of lithium silicides (identified last year) bulk and surface can be largely mitigated by incorporation of M^{+2/+3} which provide coulombic stabilization in the Zintl phase. See BAT388 for applications in half and full cells

Chemical reactivity studies lead to predictions of stable surfaces and processes to prepare stabilized silicon powders at 1-50g scale for all teams



(Silicon Hydride) **Reactive**

(Silyl Ester) **Reactive**



(Silyl Ether) **Stable**



(Geminal OH) **Reactive**

(Linear OH) **Stable**



(Li₂SiO₃) **Reactive**

(Li₄SiO₄) **Stable**

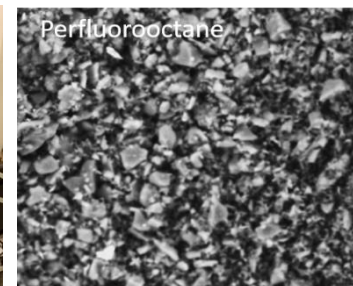
From SEISta collaboration we identified stable chemical bonds with respect to chemical activity and gassing.

Aid in electrode fabrication and initial SEI formation reaction

Top Down (ORNL)

Milling silicon to <200 nm particles in a sacrificial reactant

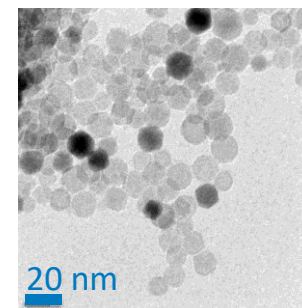
As mill expose surfaces which react with solvent forming functionalized passivation



Bottom Up (NREL)

Fabricating 20–50 nm Si (1–5 g) nanoparticles with PECVD

Passivating with chemical species by post-growth chemical reactions with surface hydrides



Understanding obtained from SEISta will be used to prepare reproducible surface modified silicon for both SEISta (model electrodes) and the Silicon Deep Dive (full cell).
Moving from understanding to mitigation.

Conclusions

- The nature and life history of the silicon has a dramatic effect on the formation and evolution of the SEI on silicon.
- Understanding the SEI on silicon will require multiple characterization techniques but if the samples and experiments are not “the same” then confusing results are obtained.
- A team approach with rigorous sample and experimental control is at the heart of this project.
- Understanding is the key to enabling silicon anodes and clear experimental data that is universally applicable is the goal.
- Control and reproducibility of silicon synthesis and surface chemistry is critical.
- Understanding of how changes affect the SEI chemistry is fundamental to progress.



Future work

- Understand the nature of the evolution of the SEI under both static and cycling conditions.
- Identify and understand the soluble species generated at the negative electrode.
- Produce reliable and reproducible research silicon samples where the surface can be modified and controlled.
- Identify solutions to SEI stability based upon SEISta understanding and transfer these to the Deep Dive full cell approach.



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