

Silicon Consortium Project Calendar Aging Electrochemical Screening Protocol 1.2.

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Executive Summary: The purpose of this document is to provide a framework for the development of silicon (Si) electrodes for high capacity lithium ion batteries by providing test protocols that researchers and early stage developers can use to assess the progress of silicon modifications, cell designs, electrolytes, or additives. Overall while full-cell cycling for silicon anodes has progressed significantly over the past several years, similar progress in the area of calendar life remains a major challenge. The procedures detailed below have been developed out of a major scientific effort, funded by the Vehicles Technology Office, to understand the formation and evolution of the silicon solid electrolyte interphase. This first generation of procedures is designed to enable a reasonably well-equipped research laboratory to assess early research progress towards improving calendar aging issues in silicon cells in a reasonable timeframe. These generation-one procedures will be updated, modified and expanded as the research team obtains feedback from stakeholders and as our understanding of the SEI evolves.

OVERVIEW

Traditional approaches to investigate calendar aging often involve experiments that take as long as the period they investigate; the aging inflicted by months of storage is evaluated by exposing the charged cell to controlled conditions for that same number of months. More specifically, such tests age the cells under open-circuit conditions (no current or load applied), with periodic reference performance tests (RPTs) that measure the cell degradation as a function of aging time. The RPTs directly measure the metrics of interest, such as reversible capacity and energy/power retention. While providing extremely useful information, such approaches are clearly time-consuming and are likely to slow down the development of Si-based cells with robust SEI layers.¹

The testing protocol described in this document is designed to provide semi-quantitative insights on the quality of the SEI in under ~2 weeks. Rather than tracking cell capacity losses over very long times, the protocol presented relies on measuring, in real time, the currents associated with reactions at the SEI as the anode is held at a constant state-of-charge (SOC). The evolution of these currents over time provides a basis to understand how the state of passivation of the anode evolves over longer time scales. We note that calendar aging is a complex process that involves not only loss of Li⁺ inventory, but also active material loss, electrolyte degradation and power fade. While the testing protocol being developed by our team will *not* predict all these aspects of calendar aging, it will nonetheless provide information about the rates of Li⁺ inventory losses to the SEI, which is a main factor limiting the calendar life of silicon-based anodes.

The testing protocol described herein departs from traditional calendar aging tests in that *it does not provide absolute calendar lifetime predictions*. Instead, the results should be compared against those of standard electrodes like graphite with calendar lifetimes well characterized by other methods. Future versions of this protocol may provide the ability to quantitatively predict calendar lifetime as the analysis methods are further developed and the lifetime predictions are validated by independent tests. Until such capabilities are available, this protocol can be used to quickly and easily screen electrolyte compositions and silicon electrodes. After this initial investigation, the most promising systems can be studied under more resource intensive experiments, such as traditional calendar life tests that use long rests with intermittent RPTs.

CALENDAR LIFE SCREENING PROTOCOL

The procedure to test the calendar life of Si anode materials *is extremely sensitive to the test cell setup, especially the geometric size and areal capacities of both the test electrode and the counter electrode*. Therefore, for reliable comparisons between tests, the testing procedures should adhere to the following requirements, and for best comparisons, cell tests conducted in an environmental chamber held at 30 °C:

Cell Design

Table 1: Requirements of test and counter electrodes

| Electrodes | Chemistry | Capacity | Diameter |
|-------------------------------|------------------------------|--------------------------|----------------------------------|
| Test Anode: | Si material | <1.3 mAh/cm ² | 15 mm diameter (14 mm vs. Li) |
| Preferable cathode: | Lithium iron phosphate (LFP) | >2.5 mAh/cm ² | 14 mm diameter |
| Acceptable counter electrode: | Lithium metal | >2.5 mAh/cm ² | 15 mm diameter |

Size 2032 stainless steel coin cells are used as the electrochemical test vehicle. The electrodes consist of the Si anode material (the test electrode) against a thick high-capacity lithium iron phosphate (LFP) cathode. When an LFP electrode of sufficiently high areal capacity is not available a Li counter electrode can be used instead; we warn that the chemical reactivity of lithium can alter the electrolyte composition, and can affect the generality of observations obtained for some systems. The diameters are 14 mm for the cathode (LFP) when it is paired with the 15 mm diameter Si-containing test anode. When the Si-containing electrode is paired with a Li counter electrode then the Si electrode is 14 mm diameter with a 15 mm diameter Li foil (less than 1 mm thick).

The areal capacities of the electrodes are chosen to ensure that there is enough Li⁺ inventory to supply the Si-containing test electrode with capacity during the aging protocol. The LFP electrode is greater than or equal to 2.5 mAh/cm², while the Si-containing test electrode is less than or equal to 1.3 mAh/cm² at the state-of-charge (SOC) at which aging will be assessed. This additional Li⁺ inventory is meant to guarantee that the cathode would still retain excess capacity even after the large irreversibility that is typical of Si electrodes in the first few cycles. If electrodes with loadings other than those designated are used, it is

imperative that the total utilized capacity of the Si electrode (reversible capacity + accumulated irreversible capacity) not exceed the capacity of the counter electrode during the test. This design ensures that the counter electrode can effectively supply the Li^+ needed to (re)form the SEI at the Si test electrode.

Note that the Si test material can have been previously broken-in according to any pre-testing regime prior to the capacity check with LFP electrode in a coin cell (see below). Examples could include deep discharge (eg. formation down to 10 mV), if necessary, or pre-lithiation treatment to the Si test material or other formation protocol. Following this Si material formation testing, the below *Electrochemical Protocol* (calendar life test), can proceed forward as long as the total capacity values within Table 1 are followed for coin cell

Cell assembly

The 2032 stainless steel coin cell is built following an assembly process that has been well established at Argonne National Laboratory² and is summarized below:

1. Electrodes should be dried under dynamic vacuum for at least 14 h at 120 °C for PVDF binder (LFP cathode) containing electrodes, and 150 °C for PAA or LiPAA binder containing electrodes. Other binders that are used should be dried appropriately so as not to affect the cell chemistry.
2.
 - a. Note that thick single sided LFP electrodes can curl when wet with the electrolyte. Thus, we recommend assembling LFP containing cells from the anode side up in the following order: cell cap with attached polypropylene gasket, 0.5 mm thick stainless steel spacer, 15 mm Si test electrode, 20 μL electrolyte, 19 mm diameter layer of 2325 Celgard separator or equivalent, 20 μL electrolyte, 14 mm LFP counter electrode, 0.5 mm thick stainless steel spacer, stainless steel wave spring, cell case.
 - b. Li half-cells should be assembled in the following order: cell case, 14 mm Si test electrode, 20 μL electrolyte, 19 mm diameter separator layer, polypropylene gasket (oriented to properly mate with cell cap), 20 μL electrolyte, 15 mm diameter Li metal foil (< 1 mm thick), 0.5 mm thick stainless steel spacer, stainless steel wave spring, cell cap.
3. The assembled cell stack should then be crimped together with a hydraulic crimper, preferably an automatic crimper for consistency.

Electrochemical Protocol

C-rate determination: The C-rate is defined as the current necessary to drive the Si test electrode from one voltage cut-off to the other in 1 hour (a C/10 rates takes 10 hours to drive the electrode from one voltage cutoff to the other). This value should be determined using half-cell data of the second cycle of the Si test electrode. Because the LFP electrode delithiates at ~ 3.45 V vs. Li/Li^+ , and relithiates at ~ 3.40 V vs. Li/Li^+ , the cutoff potentials correlate as follows:

2.7 V vs. LFP (discharge) = 0.7 V vs. Li/Li^+

3.35 V vs. LFP (charge) = 0.1 V vs. Li/Li⁺

LFP full-cell:

1. Rest cell at OCV for 4 hours to allow electrolyte wetting
2. At a C/10 rate, charge to 3.35 V, discharge to 2.7 V, repeat 3 times, data acquisition should be $\Delta V = 5$ mV
3. Hold cell at 2.7 V until $i < C/100$ to fully delithiate test electrode, data acquisition should be $\Delta t = 1$ min, $\Delta i = 10$ μ A
4. Charge to 3.35 V at a C/10 rate, data acquisition should be $\Delta V = 5$ mV
5. Hold potential at 3.35 V for 180 hours, data acquisition should be $\Delta t = 1$ min, $\Delta i = 10$ μ A
6. Discharge to 2.7 V at a C/10 rate, data acquisition should be $\Delta V = 5$ mV
7. Hold cell at 2.7 V until $i < C/100$ to fully delithiate test electrode, data acquisition should be $\Delta t = 1$ min, $\Delta i = 10$ μ A
8. At a C/10 rate, charge to 3.35 V, discharge to 2.7 V, repeat 2 times, data acquisition should be $\Delta V = 5$ mV

Li Half-cell (same procedure as LFP full-cell with potential cutoffs substituted as follows):

- 0.1 V instead of 3.35 V
- 0.7 V instead of 2.7 V

The preliminary efforts described in this report are limited to studying SEI passivation at the SOC achieved by the anode at ~ 0.1 V vs. Li/Li⁺; for a graphite electrode, this would correspond to $\sim 55\%$ of lithiation. Most Si-based electrodes can remain mechanically stable when cycled to this potential, and thus permanent losses of active material are less likely to occur during the tests discussed here. Our team recognizes that SOC is an important variable when investigating calendar aging and modeling SEI growth,³ and future studies will expand the scope of this report.

Data collection and presentation

Verifying the current response of the cell during the voltage hold is critical to guarantee that the data analysis will be meaningful. **Figure 1** shows the current that is measured during the hold, normalized to the capacity (lithiation capacity measured during the charge immediately before the voltage hold) of a test silicon electrode and a graphite baseline electrode. The expected behavior for the current measured in both systems are shown in **Figure 1a**, where the continuous decay exhibited by the traces indicates that the counter electrode is supplying sufficient Li⁺ inventory. This plot also qualitatively demonstrates that the currents that arise from parasitic reactions on the graphite electrode, seen long after the initial decay, are roughly an order of magnitude smaller than those observed for the silicon, demonstrating its better passivating behavior and longer calendar lifetimes, as expected. The results displayed in this type of plot should serve as a gate to determine if further analysis should be performed using the testing protocols described in this document. *If the residual current (i) does not approach that of a baseline electrode such as graphite (necessarily within an order of magnitude), then further analysis is not warranted, as the SEI is clearly not sufficiently stable.* It is recommended that the data acquisition parameters

discussed in the previous section are used in the voltage hold test, to ensure enough resolution.

A major pitfall of this experimental protocol occurs when the Li^+ inventory of a test cell is exhausted (counter electrode is completely delithiated). The sudden decline of the current responses to diminishingly small and indistinguishable values as seen in **Figure 1b** (after ~40h of voltage hold) indicates that the Li^+ inventory supplied by the counter electrodes has been exhausted during the V-hold. Under these conditions, the external current no longer provides a direct measure of the rate of parasitic reactions, yielding inaccurate information about the electrode's calendar lifetime. This is another critical reason why the current response of the collected data needs to be evaluated before performing the analysis described in the next section. If the current response displays a similar behavior to that shown in **Figure 1b**, the test cell should be remade with a higher capacity counter electrode or a lower capacity test electrode.

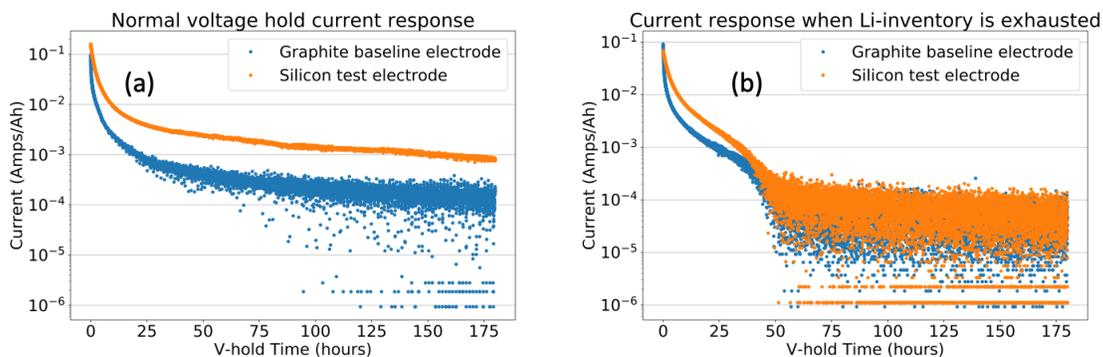


Figure 1. Current decay versus time during voltage holds of (blue) graphite baseline anode and (orange) 80 wt% Si test anode (performed at 30 °C). The current has been normalized to the capacity of the test electrode measured during the lithiation immediately before the voltage hold. Panel (a) shows the current response where there is an excess of Li^+ inventory supplied by a counter electrode with a flat voltage profile, as is the scenario marked (a) in Figure 3. Panel (b) shows how the current suddenly declines as the Li^+ inventory supplied by the counter electrode is exhausted, polarizing the electrodes to high potentials. Under these conditions, the measured current underestimates the actual rate of parasitic reactions experienced by the test electrode.

Data Analysis and Result Reporting

Analysis of the data should first be done by qualitative visual inspection and comparison between the test electrodes and a baseline electrode. **Figure 2** shows the current decays of 3 different Si test electrodes and a graphite baseline electrode. The normalization of the current data to the reversible capacity of each electrode is important because the resulting units of Amps/Ah indicate the rate at which each electrode is losing reversible capacity due to Li^+ consumption at the SEI. If the normalized current measured from a Si test electrode at the end of the 180 hour voltage hold is distinguishably higher than the baseline electrode, further analysis is not needed, as the electrode's SEI is clearly not sufficiently stable. This is the case for each of the Si test electrodes shown in **Figure 2**, thus showing the effectiveness of this simple qualitative comparison for the initial screening of Si test electrodes.

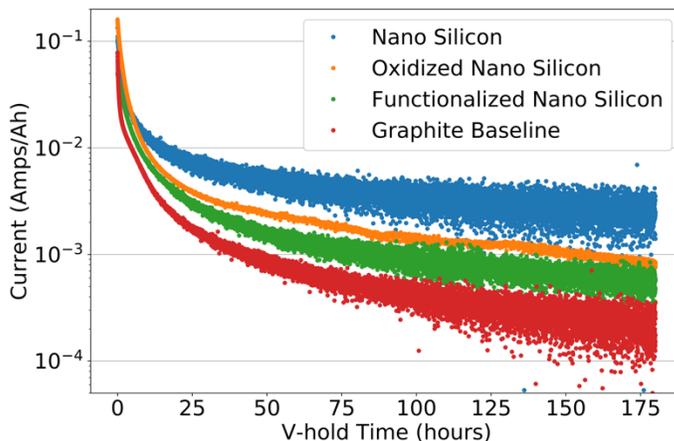


Figure 2. Current decay versus time during voltage holds of several different Si test electrodes and a graphite baseline electrode (test performed at 30 °C). The rate of parasitic reactions of the Si test electrodes can be qualitatively distinguished by visual inspection, and all exhibit greater aging rates than the graphite baseline electrode.

The average current after 180 hours of voltage hold can be used as a metric to rank the rate of parasitic reactions at each electrode, and is reported below for the examples shown in **Figure 2**. This information should be complemented by details about electrode composition, areal capacity, and electrolyte used in the tests. Additionally, because there can be variability between tests for a given electrode and electrolyte pair, we recommend that a sample size of at least three test cells for each test electrode type be reported.

Example reporting:

Graphite baseline electrode

1.17 mAh/cm²

91.83 wt% Superior Graphite SLC1520P

2 wt% Timcal C45 carbon

6 wt% Kureha 9300 PVDF Binder

0.17 wt% Oxalic Acid

Electrolyte: 1.2 M LiPF₆ in ethylene-carbonate:ethyl-methyl-carbonate 3:7 + 10 wt% FEC

Mean terminal current = 0.23 mA/Ah

Nano Silicon

0.3 mAh/cm²

20 wt% 3.9 nm diameter Si nanoparticles

65 wt% Timcal C65 carbon

15 wt% PAA Binder

Electrolyte: 1.2 M LiPF₆ in ethylene-carbonate:ethyl-methyl-carbonate 3:7 + 10 wt% FEC

Mean terminal current = 2.62 mA/Ah

Oxidized Nano Silicon

1.24 mAh/cm²

80 wt% Surface oxidized 150 nm diameter Si nanoparticles

10 wt% Timcal C45 carbon

10 wt% LiPAA Binder

Electrolyte: 1.2 M LiPF₆ in ethylene-carbonate:ethyl-methyl-carbonate 3:7 + 10 wt% FEC

Mean terminal current = 0.92 mA/Ah

Functionalized Nano Silicon

0.7 mAh/cm²

40 wt% 4-phenylphenol surface functionalized 30 nm diameter Si nanoparticles

40 wt% Timcal C65 carbon

20wt% PAA Binder

Electrolyte: 1.2 M LiPF₆ in ethylene-carbonate:ethyl-methyl-carbonate 3:7 + 10 wt% FEC

Mean terminal current = 0.59 mA/Ah

These examples highlight the capabilities of the screening protocol described in the current version of this document. Because it is currently limited to qualitative analysis, it is especially important to use a baseline electrode with a calendar life well characterized by other methods under the conditions of interest. Future versions of this report may include a semi-quantitative model to describe the rate of parasitic reactions in the electrodes of interest, which is currently under validation by long-term experiments; this model is outlined in general terms at the end of the discussion section.

DISCUSSION

Our methodology is based on using *voltage holds* to measure the rate of Li⁺ trapping at the SEI in Si-containing cells. *This approach has the advantage of recording real-time rates of side reactions, providing information about the time-dependence of such processes and potentially enabling extrapolation of behaviors observed in relatively short duration experiments.* Its working principle is based on the fact that parasitic *reduction* reactions (such as the ones involved in SEI formation) effectively consume electrons from the anode, which decreases the anode SOC. The instantaneous voltage of a cell is the difference between the potentials of the positive and negative electrodes, so a change in the anode SOC yields a change in cell voltage. If the anode SOC is “pinned” by holding the cell voltage constant, electrons must flow from the cathode to the anode to replenish the charge consumed by side reactions, in order to keep the anode SOC invariant; i.e., the *current* flowing through the cell and measured by an external circuit is equal to the rate of these parasitic processes. This ideal equivalence between the parasitic and measured currents is achieved when the anode has a sloped potential and is most sensitive to SOC and the cathode is insensitive to SOC (the voltage profile is “flat”). We can approximate these conditions by using LiFePO₄ (LFP) as a cathode (which can provide most of its capacity while at ~3.45 V vs. Li/Li⁺), and by performing the voltage hold at potentials in which the anode cycling profiles are notably sloped to maximize sensitivity.

For these tests, it is important that the LFP counter-electrode have a capacity in excess of what is used by the reversible and irreversible reactions occurring at the Si test electrode.

The importance of this condition is demonstrated by the simulated voltage profiles shown in **Figure 3**, where the LFP voltage profiles are shown in blue. Profiles for pristine (solid line) and aged (dashed line) Si anodes are displayed in black with the corresponding full-cell voltage profiles displayed in red. The intersecting lines marked **(a)** in **Figure 3** represent the pristine full-cell being held at 3.35 V, where the potential at the pristine Si anode would be roughly equivalent to ~ 100 mV vs. Li/Li⁺. In this pristine cell at 3.35 V, the LFP voltage profile is insensitive to SOC (voltage profile is flat), and thus any changes of the Si SOC would require a change in cell voltage i.e., the Si SOC is “pinned”. As electrons and Li-ions are consumed at the Si electrode during aging, they are replenished by the LFP cathode, as additional Li⁺ extraction is possible at that same electrode potential. Nevertheless, if enough capacity is lost, the aged cell may no longer be able to keep the Si SOC constant. This Li⁺ inventory exhaustion scenario is represented by the intersecting lines marked **(b)** in **Figure 3**, where the aged cell is still held at 3.35 V. In the aged cell with an exhausted Li⁺ inventory, the LFP voltage profile is polarized above its plateau potential and is sensitive to SOC (highly sloped), resulting in the Si electrode no longer being held at the desired potential of 100 mV vs. Li/Li⁺. When that happens, additional Li⁺ extraction from LFP to counter parasitic reactions at the anode would cause an increase in the potential of both electrodes; this would involve a net decrease in anode SOC, and thus the currents measured by an external circuit would underestimate the extent of side-reactions in the cell.

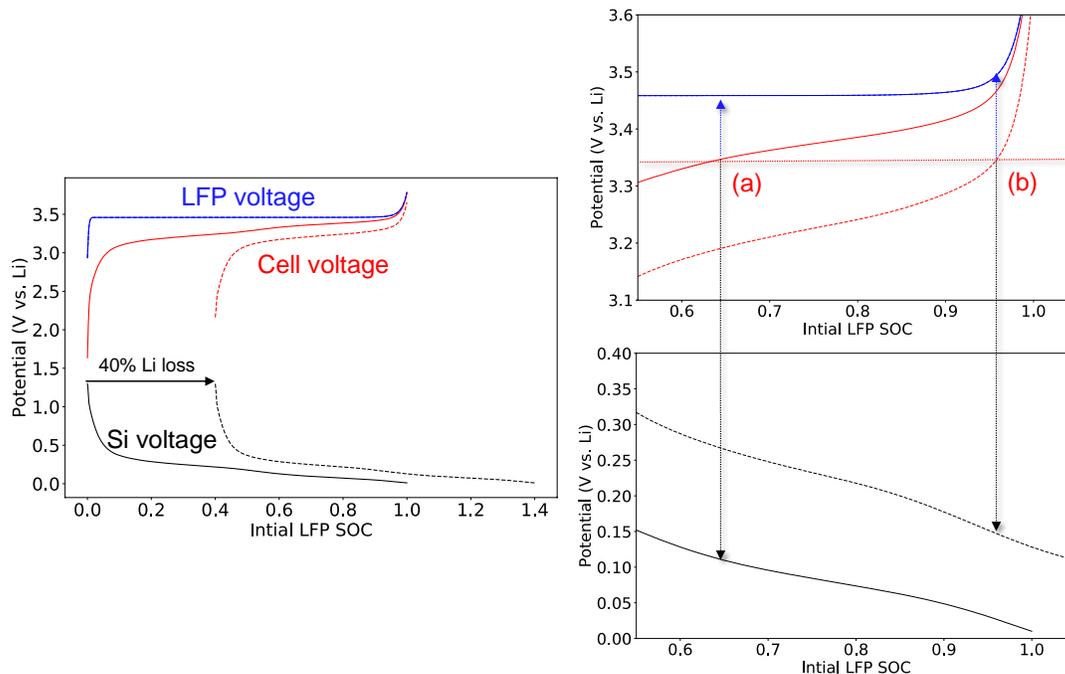


Figure 3. (left) Simulated voltage profiles for an LFP cathode (blue), Si anode (black), and the 2-electrode cell combining those two electrodes (red). The dashed black line represents Si electrode aging by the loss of Li⁺ inventory, which effectively shifts the Si voltage profile relative to the LFP voltage profile, and results in the aged cell having the dashed red voltage profile. (right) Zoomed in portions of the voltage profiles where the dotted horizontal red line represents a voltage hold potential of 3.35 V for pristine (a) and aged (b) Si electrode containing cells.

The use of LFP as a counter-electrode has additional advantages. One is that the cathode can be assumed to be an essentially inert electrode, since its low potentials will lead to little changes in electrolyte composition and are unlikely to generate oxidative currents that could affect the analysis. Additionally, the extremely flat voltage plateau of LFP over its entire capacity range allows it to be used as a reliable pseudo-reference electrode in a two-electrode coin cell. Thus, the absolute potential of the anode can be well controlled, facilitating studies on the effect of SOC on passivation. The test protocol specifics and an example voltage profile are shown in **Figure 4**, where a 180-hour long voltage hold is preceded by three formation cycles and followed by two additional diagnostic cycles. The total test time is ~290 hours (less than two weeks).

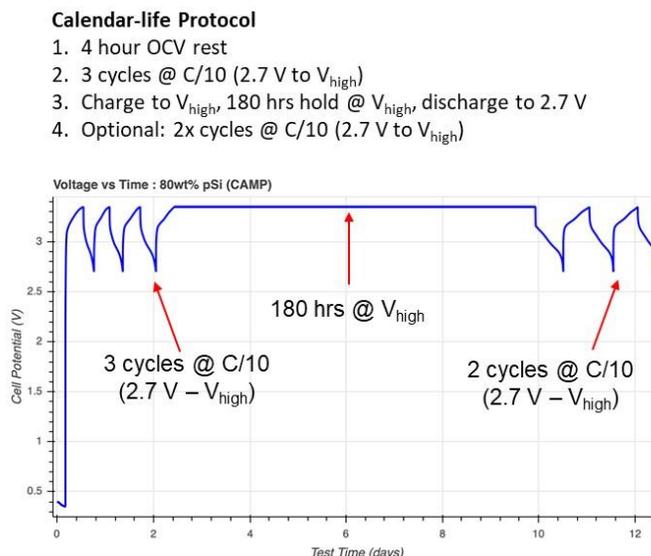


Figure 4. The test protocol to estimate the rate of parasitic reactions of Si-based electrodes. The example voltage profile is from a cell coupling a 80% Si electrode vs. an LFP cathode.

Although this experimental approach is a logical pathway to derive information about electrode passivation from short term tests, it also presents challenges. In practice, electrodes present a finite impedance and lithiation processes are incomplete by the time the voltage hold initiates. Hence, the current measured during the early periods of voltage hold is a combination of reversible lithiation processes (lithiation of Si) and irreversible electrochemical reactions (SEI formation and concomitant Li^+ consumption). As the voltage is held for longer and longer times, the proportion of the current driving reversible reactions diminishes relative to the current driving the irreversible reactions, and the measured current becomes a more reliable descriptor of parasitic processes. For these component contributors, *the challenge exists to validate that the analyses can differentiate between both these contributions to the measured current*. The reversible capacity that is passed during the voltage hold can be experimentally quantified by inspecting cell discharge after the hold, and these values are used to help differentiate the time dependent contributions from the reversible and irreversible processes.

The purpose of the 180-hour long voltage hold is to determine the rate of Li^+ inventory consumption, as measured by the current, due to irreversible side reactions at the anode surface. Using this method, the current measured during the voltage hold is due to a

combination of the reversible lithiation reactions and the irreversible side reactions. **Figure 1a** shows the typical current responses during the voltage hold of example electrodes. The current densities decrease more than an order of magnitude during the first ~20 hours of the voltage hold as the reversible lithiation reactions near completion. In contrast, towards the end of the voltage hold, the current densities stabilize at diminishingly small values that are primarily due to irreversible Li^+ consuming reactions. The lower current density of the graphite electrode at the end of the voltage hold compared to the Si electrode is good confirmation that the measurement is qualitatively capturing the expectedly better SEI stability (and lower Li^+ inventory consumption rate) of the graphite electrode. **Figure 1b** shows the current response during the V-hold where the Li^+ inventory of the cell becomes exhausted. This occurs when the cathode is unable to supply additional electrons and Li^+ ions to keep the anode pinned at a fixed SOC, as outlined by marker **(b)** in **Figure 3**. In this case, the full-cell is maintained at a fixed voltage with a net decrease in anode SOC, resulting in the measured currents underestimating the parasitic reaction rates. This can be seen in **Figure 1b** by the sudden decrease in current to vanishingly small values, and highlights the importance of performing these experiments in cells with excess counter electrode capacity.

Integrating the measured current over time during the voltage hold and normalizing to the electrode's reversible capacity yields time dependent capacity, $Q_{\text{hold}}(t)$. In order to deconvolute the irreversible and reversible contributions to the capacity, the following function can be fit to the normalized capacity data:

$$Q_{\text{hold}}(t) = Q_{\text{irrev}}(t) + Q_{\text{rev}}(t)$$

The numerical forms of these functions are still being determined using long term voltage hold validation experiments. As the validation data becomes available, future versions of this report may include numerical models that can be used to semi-quantitatively describe the rate of SEI-related side-reactions. Preliminary results suggest that $Q_{\text{rev}}(t)$ has an asymptotic time dependency as the reversible capacity approaches a maximum value at long times, whereas $Q_{\text{irrev}}(t)$ may be proportional to the square-root of time. The square-root of time dependency of $Q_{\text{irrev}}(t)$ is well supported by empirical observations in the literature, and is believed to capture the general behavior of SEI growth.^{3,4} This particular functional form arises from the hypothesis that the irreversible side reactions occurring at the electrode surface due to SEI formation are diffusion limited, and thus their rates are inversely proportional to the square root of time.

Notwithstanding, the Si SEI stability will naturally fluctuate as a function of a plethora of internal and external factors. The present report is just the beginning of an exploration of the Si calendar life behavior embedded in a current response, the measurement probe of choice. In an electrochemical process, electrode kinetics and their reaction rates are tied to a current value. The parasitic reactions in Si electrodes are exceedingly complex and, as a first pass, it is affiliated with (1) the Li ion conductivity in the SEI, (2) the mechanical stability of SEI, (3) chemical stability (solubility) of the SEI, and (4) the electronic conductivity within the SEI. What factors dominate thus begs the question: how can one slow down this degradation phenomena and create 'immunity'?

CONCLUSIONS

A method to qualitatively compare the calendar aging rates of silicon anodes has been developed using full-cells with an excess amount of electrolyte and of Li⁺ supplied by a LFP counter electrode. After formation cycles, the voltage of a test cell is held for 180 h, and the current response measured during the voltage hold can be used to qualitatively compare the rates of Li⁺ consumption at the SEI of silicon test electrodes to a baseline electrode. Long term voltage hold aging experiments are underway to determine numerical models that can be used to derive aging parameters for silicon test electrodes. Once the numerical models developed by our team are fully validated, they can be used to support developments of cell, material, and interfacial designs that minimize calendar aging rates of Si electrodes.

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