NATIONAL ENERGY TECHNOLOGY LABORATORY (NETL)

UNITED STATES DEPARTMENT OF ENERGY

Final Technical Report

High-Voltage Solid Polymer Batteries for Electric Drive Vehicles

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Disclaimer

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Executive Summary

The purpose of this project was for Seeo to develop a high energy lithium based technology with targets of over 500 Wh/l and 325 Wh/kg. Seeo would leverage the work already achieved with its unique proprietary solid polymer DryLyte^{TM} technology in cells which had a specific energy density of 220 Wh/kg.

The development work was focused on establishing a dual electrolyte system, coated cathode particle techniques, various types of additives, and different conductive salts. The program had a duration of three years, with Seeo delivering the final cells at the end of 2014 for evaluation by a DOE laboratory.

A fundamental advantage of the solid polymer system that Seeo had developed was that it allowed the company to work with a dual polymer electrolyte system in the cell. A traditional liquid electrolyte must and will permeate throughout the cell in order to create the necessary conductivity and transportation of the lithium-ions. This also means that in order to achieve higher voltage, and thereby higher energy cells the traditional liquid electrolytes need to be stable over a wide voltage window, which has been proven to be a challenge that has yet to be solved. Due to the inherent nature of solid polymer electrolytes being immiscible one can adopt a dual electrolyte system. One solid polymer can then be optimized and stable against the lower voltage electrode, the anode, while the other solid polymer electrolyte can be developed to be stable against the higher voltage electrode, the cathode. **Figure 1** illustrates this with the single electrolyte system on the left and the dual electrolyte system on the right.

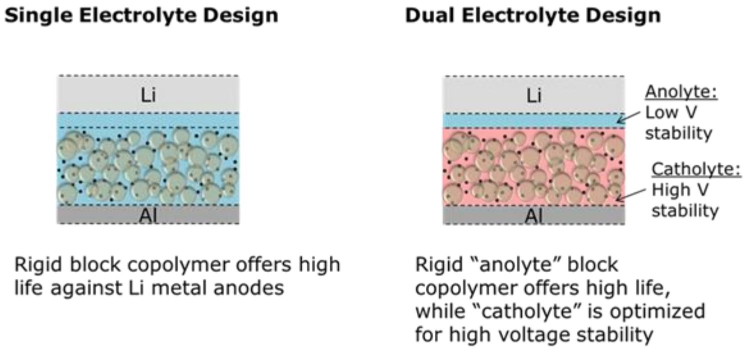


Figure 1: Differences between single and dual electrolyte designs based on solid-state polymers.

Seeo had already developed its proprietary solid polymer DryLyte electrolyte, which was stable against the lithium anode as well as towards a lower voltage cathode material such as the $LiFePO_4$. This system had been demonstrated to work well and meet the industry accepted performance characteristics.

The focus of this Vehicle Technologies Office funded program was therefore to implement a high voltage and high energy cathode achieving unparalleled energy density levels. Seeo had a solid polymer electrolyte, the DryLyte system, which is stable against the lithium metal anode. Work under this program would therefore be focused on the development of achieving a high voltage composite cathode that could be paired to the DryLyte system. This would entail work on finding the optimal polymer, cathode material, and any materials that could contribute to and enhance the performance of this new cathode combination. It was also envisaged that different types of conductive salts and additives to enhance the performance characteristics and manufacturability of these high energy and high voltage cells would be investigated.

All tasks were achieved over the duration of the program, though some modifications were made to the final deliverables. A timeline over the duration of the program is shown in **Figure 2**.

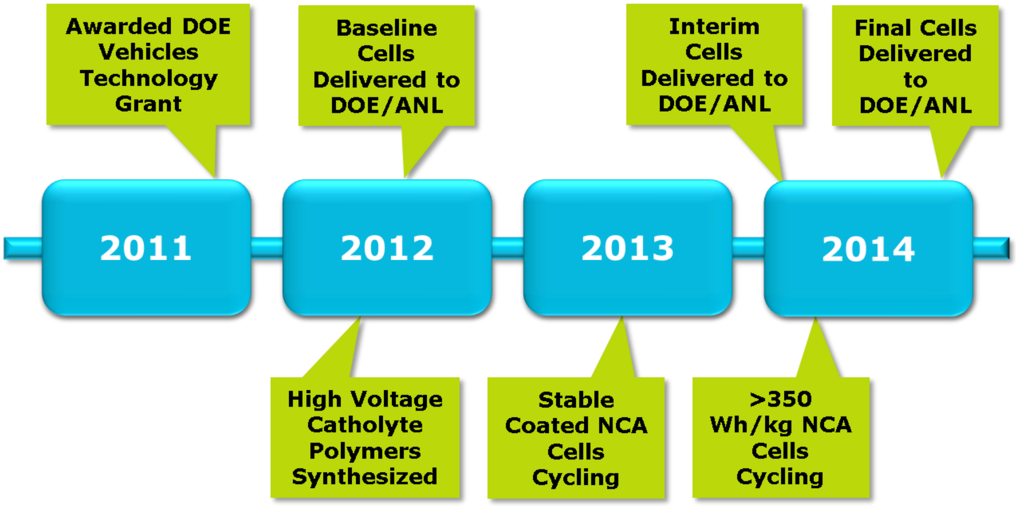


Figure 2: Timeline of program and major achievements

Source: Seeo, Inc.

The following are the main achievements achieved under this project.

Stable cycling at 4.2 V in a polymer system was demonstrated at various energy densities. In an all solid-state Li metal / NCA cell, 200 cycles at C/3 discharge rate were achieved at a specific energy of 210 Wh/kg. At a higher loading in an improved cell design (300 Wh/kg), 1C discharge rate was achieved with 20s pulses up to 3C. At 350 Wh/kg, 75 cycles were achieved to 80% DOD. In conclusion, these achievements were demonstrated and can be summarized as follows:

- Specific energy of 350 Wh/kg
- Energy density of 600 Wh/l
- Power density of 790 W/l

Seeo continues to develop its technology, is aggressively pursuing market opportunities, sees strong and growing interest from potential customers and strategic partners for its technology, and is committed to commercializing its proprietary technology. Seeo expects to further to expand its activities in the US and California.

Introduction

Background

Seeo was founded in 2007 with the goal of creating a new class of high-energy rechargeable lithium-ion batteries based on a nanostructured polymer electrolyte initially developed at the Lawrence Berkeley National Laboratory under funding from the Department of Energy's Batteries for Advanced Transportation Technology (BATT) program. Seeo has continued the development of solid-state lithium-ion batteries based on this proprietary dry polymer electrolyte technology, which is non-flammable and non-volatile, and offers superior energy density and lifetime performance. The company is headquartered in and has its pilot line manufacturing facility in Hayward, California.

Lithium-ion batteries in today's markets face challenges with combining the requirements demanded by the market for superior safety, lifetime, energy density, and low cost. Chemical degradation leads to premature failure in existing applications, and poor lifetimes prevent lithium-ion cells from addressing new key markets. The effort to increase energy density to provide longer operating duration while at the same time minimizing cost have exposed the vulnerability of this system to safety issues and potential catastrophic failures, as has been witnessed in the recent Boeing 787 Dreamliner incidents. The safety performance and high cost of lithium-ion cells are major concerns, and this becomes especially critical for larger energy storage capacities required for electric vehicles and grid-connected applications. Wide scale deployment of lithium-ion technology and other energy storage technologies faces obstacles due to such concerns.

Seeo has developed a proprietary polymer electrolyte platform that enables a new generation of rechargeable lithium-ion batteries that achieves high levels of safety, lifetime, and energy density, as well as offering potential cost advantages relative to conventional systems. Performance results are included in this report. Seeo's technology, which was developed and demonstrated here in California, represents a unique solution for addressing critical national needs for electric vehicles and large-scale renewable energy storage.

Seeo's Solid-State Battery Technology

Seeo's solid-state battery technology offers specific energies of 220 Wh/kg in the first generation, and is already demonstrating specific energies of 300-400 Wh/kg in cells developed in this program. The technology also offers safer batteries, with long reliability and the promise of lower costs. The key to these products lies in Seeo's solid DryLyte^{TM} electrolyte which replaces the flammable liquid electrolyte typically found in other lithium-ion batteries. A battery, such as a lithium-ion battery, consists of an anode (negative electrode), a cathode (positive electrode), a separator in between the anode and cathode that insulates the anode and cathode from each other, and an electrolyte that provides the transport of ions from one side of the battery to the other. The direction of the transport of the ions depends on whether one is charging or discharging the battery.

The unique characteristics of Seeo's DryLyte solid polymer electrolyte is that it functions both as a separator and an electrolyte, and allows Seeo to safely use the lightest and most energy efficient anode material, lithium metal. In this way, Seeo cells use standard cathode materials and manufacturing processes and achieve at least 50 percent higher gravimetric energy density than other existing lithium-ion technologies. The structure of Seeo's unique technology is shown in **Figure 3**.

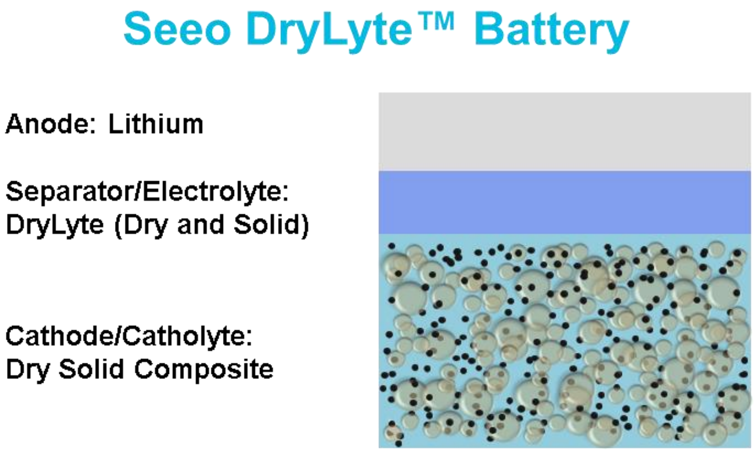


Figure 3: Structure of Seeo DryLyte™ Cell

Source: Seeo, Inc.

Department of Energy Program

The funding for this project was received as a cooperative research agreement under the Department of Energy's Program of FOA DE-FOE-0000239.

CHAPTER 1: Project Objectives and Phases

1.1 Project Objectives

The main objectives to be achieved over the three year duration of the project (October 2011 through September 2014) was to develop and demonstrate new materials and cells that are capable of achieving the performance characteristics listed in **Table 1** below. Seeo's achievements towards meeting these objectives have been included in the table as well.

Table 1: Program Objectives and Achievements

Project Objectives	Demonstrated / Achieved
Energy density of at least 500 Wh/l	600 Wh/l
Power density of at least 500 W/l	790 W/l
Specific energy of at least 325 Wh/kg	350 Wh/kg
Cycle life of at least 300-1000 cycles at 80% depth of discharge	75 cycles at 80% DOD
Calendar life of at least 5-10 years	Projected to >20 years
Durable cell construction and design capable of being affordably mass produced	Cells produced on standard equipment and constructed from commodity feed stocks

Source: Seeo, Inc.

1.2 Project Phases

Based on the goal of achieving the objectives over the three year duration of the program the project work was split into three phases as follows:

Phase I: Baseline Evaluation and Material Synthesis

This phase included activities required to establish a baseline level for project evaluation and to commence major research activities. The recipient delivered a set of baseline cells to establish performance and reliability standards for final cell prototypes. Additionally, the project team identified and developed high-voltage polymer and cathode materials, which were monitored for mechanical stability and electrochemical properties.

- Update project definition, scope and resource plan with DOE contract officers
    - Submitted to and approved by DOE in Q4-2011
- Deliver single electrolyte Li-$LiFePO_4$ cells to set baseline performance and safety parameters
    - Baseline cells (four of approximately 1.5 Ah each) delivered to ANL/DOE early Q3

    2012
  - Cells established baseline for rate capability and ESR
- Cathode sourcing and characterization
  - Source small scale batches of HV cathode active materials
  - Evaluate materials properties: specific capacity, particle size, tap density, surface area
  - Mix and coat HV cathode slurries and establish processing parameters
  - Finalize choice of initial two cathode active material candidates
- Initial synthesis, characterization and design iterations for polymer and cathode materials
  - Mechanical stabilization of catholyte polymers
  - Anolyte-catholyte interfacial stability

Phase II: Material Formulation and Scale-Up

In this phase, polymer and cathode mechanical and electrochemical properties were optimized to improve voltage stability and cycling performance in small-area cells. Additionally, the project team developed volume synthetic techniques, comparing cost and performance to help determine the material formulation chosen for prototype cell deliverables.

- Iterative synthesis and characterization for polymer and cathode materials
  - Set target molecular weights of polymers
  - Set target block copolymer structures
- Cycling of small-area cells to choose 2 polymer-cathode couples for Phase III development
  - Build small area single-stack cells to validate materials
  - Perform initial QC testing
- Polymer Scale-up
  - Design process for large scale synthesis
  - Synthesize and characterize >10kg polymer batch
- Deliver bilayer interim design cells
  - Interim cells (six of 5 mAh capacity) were delivered to ANL/DOE in January 2014
  - Cells met predicted rate capability and ESR

Phase III: Cell Fabrication and Testing

This phase consisted of optimizing the design, testing, and constructing of prototype cells. Validating cell design and construction of interim cells lead to final specification for advanced cells. Data collected from baseline cells was combined with the final design cell specifications to deliver a robust commercialization plan that accompanied the advanced cell prototypes delivered to the DOE at the conclusion of the project.

- Monitor stability and performance of large-area, stacked cell prototypes
    - Coat large area current collectors
    - Build single-stack large area cells and monitor for performance
    - Build multi-stack cells and monitor for performance
    - Optimize processes
- Execute safety and performance test plan on final design cells
    - Develop internal test protocols
    - Perform abuse testing on final cell design
- Deliver bilayer final design cells for independent verification by DOE
    - Final cells (six of 250 mAh capacity) were delivered to ANL/DOE in Dec 2014
    - Cells met predicted rate capability and ESR
- Document commercialization plan, including high-volume production costs and risks
    - Accomplished and submitted to DOE in December 2014

1.3 Milestones

The milestones as originally defined and scheduled are illustrated in **Table 2**. The actual completion date, verification method, and reasons for any delays or differences from the original milestones and schedule have also been noted. The original objectives and timeline of the program were aggressive, but Seeo achieved all of the milestones with some minor delays and adjustments to the milestone based on experiences gained during the project.

Table 2: Project Milestones

Milestone	Planned Completion Date	Actual Completion Date	Verification Method	Comments
Baseline Cells Delivered to DOE	6/30/2012	7/12/2012	Received by Argonne National Lab	Delay in receiving shipping clearance for cells
Active Material Structure Specified	1/15/2013	1/15/2013	Internal	
Cathode Batches to Specification	6/30/2013	6/30/2013	Internal	
Catholyte Polymer to Specification	12/31/2013	12/31/2013	Internal	
Interim Cells Delivered to DOE	1/15/2014	1/15/2014	Received by Argonne National Lab	
Final Cells Testing Completed	9/29/2014	12/23/2014	Internal	Delayed by 3 months
Final Cells Delivered to DOE	9/29/2014	12/23/2014	Received by Argonne National Lab	Delayed by 3 months
Commercialization Plan Completed	9/29/2014	12/26/2014	Received by DOE	Delayed by 3 months

Source: Seeo, Inc.

1.4 Budget and Funding

The total budget for this DOE Cooperative Agreement was $6,927,880, with the federal government’s funding of $4,874,391. This was a cost share project and Seeo has provided funding of $2,053,489 as the company’s share.

CHAPTER 2: Phase I – Baseline Evaluation and Material Synthesis

2.1 Project Definition and Resource Plan

Activities in Phase I were focused on completing all proposal documents and finalize the grant agreement, delivery of baseline cells, and starting the development work. Actual development work began on the project on the scheduled start date of October 1, 2011, and the work under Phase I was expected take until the end of 2012.

The Project Management Plan, including the project schedule, budget and resource allocation plan was submitted and approved by DOE contract officers as the initial action taken by Seeo before any development work started.

Seeo had earlier been awarded a grant by the DOE under the Smart Grid Program and Seeo was simultaneously finalizing the establishment of Seeo's pilot production facility in Hayward, CA as part of this program. Though separate from the VTO program this was instrumental in achieving the first project milestone of delivery of baseline cells in mid-2012 and allowed Seeo to gain valuable knowledge in the fabrication of uniform and high quality cells. This experience was leveraged in the project work performed in this program.

2.2 Baseline Cell Delivery

The first milestone of this program was the delivery of the baseline cells for testing by Argonne National Laboratory. In preparation of the delivery Seeo initiated discussions with Argonne National Laboratory regarding the testing early in 2012 in advance of the delivery. Quantity, capacity, resource commitment, and basic test plan for characterization of the baseline cells were agreed upon. Delivery of baseline cells was accomplished at the end of Q2-2012. Baseline cells were based on Seeo's current generation technology which consisted of a lithium metal anode, DryLyte polymer separator, and LFP cathode. Four cells were delivered to Argonne National Laboratory for testing. **Table 3** below shows the details of the cells tested and verified by DOE.

Table 3: Baseline Cell Characteristics

CELL PARAMETER	VALUE
Voltage Range	2.5 – 3.6 V
0.2C Discharge Capacity	1.41 ± 0.01 Ah
0.5C Depth of Discharge	89 ± 1%
Rated Internal Resistance (ESR), 5.25%SOC, 100s	76.4 ± 3.1 mOhm

2.3 Initial Polymer Synthesis

The initial technical activities focused on developing high-voltage stable polymer electrolytes and sourcing of high-voltage cathode materials. Synthetic work was started on new chemistries for high-voltage stable polymers that were to be interfaced with high-voltage cathode materials. As an example of the initial work performed **Figure 4** shows voltage stability test data for a novel pendant group that could be attached to a polymer backbone. Seeo was following the approach of using a backbone polymer with functional pendant groups attached to it. Through this approach, novel pendant groups could be rapidly screened for desired stability. In **Figure 4**, the pendant shows stability to a voltage of 4.6 V, ensuring that after attachment to a polymer, this functionality would be stable for use with $LiMnPO_4$, one of the candidate high-voltage cathode materials Seeo was considering.

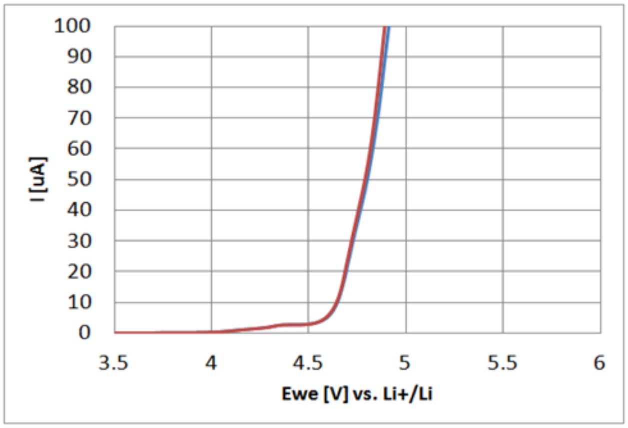


Figure 4: Voltage stability of a novel pendant group against carbon working electrode.

During Q1-2012, this pendant group was attached to a polymer backbone and tested for process ability and stability using inert platinum electrodes. The polymer was then tested against low-voltage $LiFePO_4$ cathodes, which mimic the surface properties of select high-voltage active material candidates, to evaluate electrochemical stability and mechanical properties. During subsequent quarters Seeo focused on the iterative development of the initial high-voltage stable polymer and synthesis of an alternative pendant group to exhibit improved stability and conductivity.

2.4 Cathode Sourcing and Characterization

Early in Phase I Seeo received samples of high-voltage cathode materials and basic characterization analyses were performed. These materials comprised standard metal oxide cathode materials, experimental phosphate based materials, as well as experimental mixed metal oxide cathode materials. As an example **Figure 5** shows a single charge/discharge curve of a cell consisting of a lithium anode and a promising high voltage cathode based on $LiMnPO_4$. In order to isolate polymer degradation issues from cathode degradation issues, initial tests were performed using 1M $LiPF_6$ in EC:PC as the electrolyte. The capacity of the cathode material was measured to be 150 mAh/g with an upper voltage cutoff of 4.4 V.

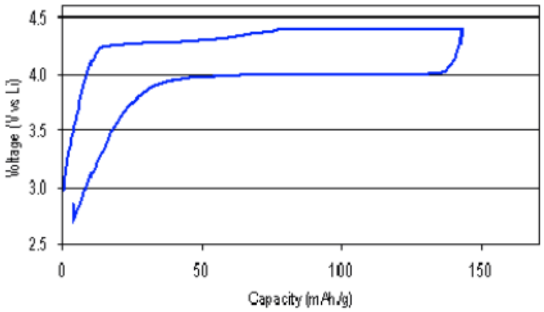


Figure 5: Capacity evaluation of $LiMnPO_4$ using EC/PC LiPF6 liquid electrolyte.
Single charge/discharge curve.

Based on this early data Seeo continued the evaluation of $LiMnPO_4$ from two different commercial suppliers using liquid electrolyte (EC:PC) cells. Both sources show adequate capacity (>150 mAh/g) with an upper voltage cutoff of 4.4 V. In addition, a source for high-capacity NCM and NCA cathode materials was identified and initial samples were sourced. Materials showed expected capacity and voltage characteristics.

2.5 Synthesis and Design Iterations for Polymer and Cathode Materials

In addition to capacity evaluation using traditional liquid electrolytes, cycle life testing with Seeo's first generation solid polymer electrolyte was also performed. While Seeo's solid polymer DryLyte electrolyte is stable to Lithium metal potentials, the data in **Figure 6** clearly demonstrated the need for polymers that would be stable to higher voltages. It was therefore evident that the types of functional groups demonstrated in **Figure 4** above would need to be incorporated into the polymers and to be tested with candidate high-voltage cathode materials to find the optimal polymer to use in the cathode.

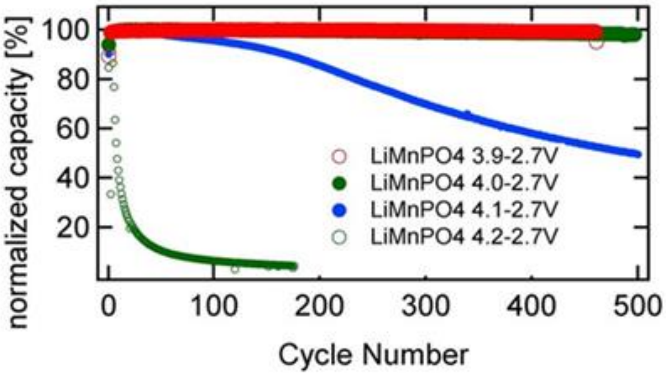


Figure 6: Cycle life data for cells made with Li metal anodes and $LiMnPO_4$ cathodes with Seeo's first generation block copolymer electrolyte, cycled to different upper cutoff voltages

With this in mind, efforts were made to increase the molecular weight of the catholyte polymer to improve mechanical properties and isolate interfacial performance with active materials and the anode facing electrolyte. Target molecular weight metrics were specified for this polymer, and subsequently Seeo was able to achieve a doubling of the molecular weight of the polymer, enabling the usage of the polymer as a binder to formulate solid polymer cathodes. As a result cathodes using the first set of phosphate-based high-voltage material were synthesized, and cycling tests were started.

During the second half of 2012 work continued on testing of two candidate initial high-voltage stable polymers with active material sourced from third-party providers. Both the first polymer and a second polymer were tested through use of these polymers as a binder against higher-voltage cathode materials. **Figure 7** shows initial results from this evaluation. Both polymer groups demonstrate significantly improved stability over the baseline, low-voltage binder polymer. The initial cycle life results were judged to be promising, but it was also apparent that further development was required to achieve the cycle life required for vehicle applications.

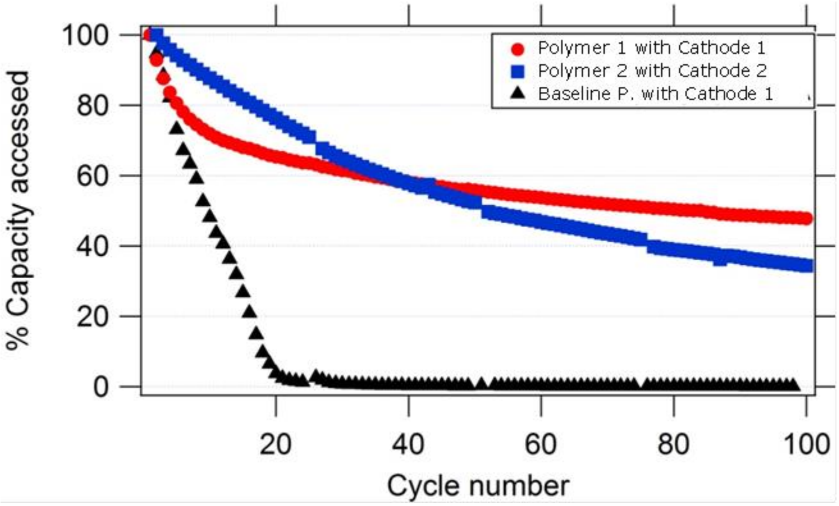


Figure 7: Stability of HV Polymer-Cathode Couples over Baseline

Seeo also identified an alternative technique to achieve voltage stability in a solid-state cell, and it was explored. Materials with different dielectric constants were coated onto high-voltage cathode materials and were tested using conventional liquid electrolytes. These coated materials showed improved stability over the baseline materials and it was decided to investigate this further, initially with liquid electrolytes and subsequently with candidate polymer materials further.

During the latter part of Phase I Seeo also started investigations and experiments with conductive salts that show higher voltage stability. The purpose of reviewing these different salts was to prevent degradation of the salt at the upper voltage range. These were characterized in liquid electrolytes and then tested in solid state test cells. **Figure 8** shows the voltage stability of various lithium salts tested at Seeo's facility.

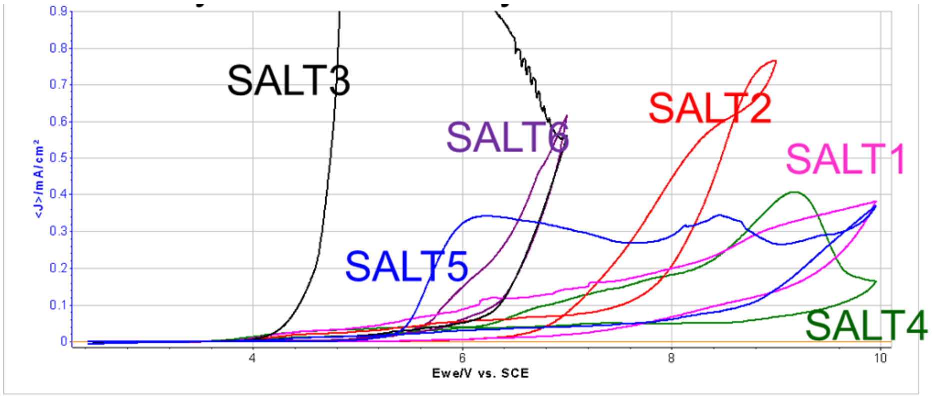


Figure 8: Stability of Various Lithium Salts Tested Using Cyclic Voltammetry

CHAPTER 3: Phase II – Material Formulation and Scale-Up

3.1 Materials Formulation – Lithium Salts

Activities in Phase II were focused in three primary areas. First, Seeo's materials team continued to develop high-voltage stable, conductive polymers, as well as work to isolate specific mechanical and electrochemical attributes that contribute to desired performance in full solid-state cells. Secondly, the team focused on determining which conductive salts to use that would result in stability in high-voltage cathodes. The third development path was the investigation of using coatings on cathode particles as an alternative technique for constructing full cells. Phase II was expected to take place in its entirety during 2013.

Early in Phase II Seeo achieved the second milestone as planned, which was to down-select the high-voltage cathode materials and vendors to use for Phase II development activities. This was based on the evaluation work performed in Phase I. The selected cathode material was NCA.

Seeo continued the evaluation of the voltage stability of various salts that had been sourced. **Figure 9** shows continuous voltage testing of Li salts at 4.3V during Q1-2013, revealing candidates with higher voltage stability than the conductive salts used in baseline cells.

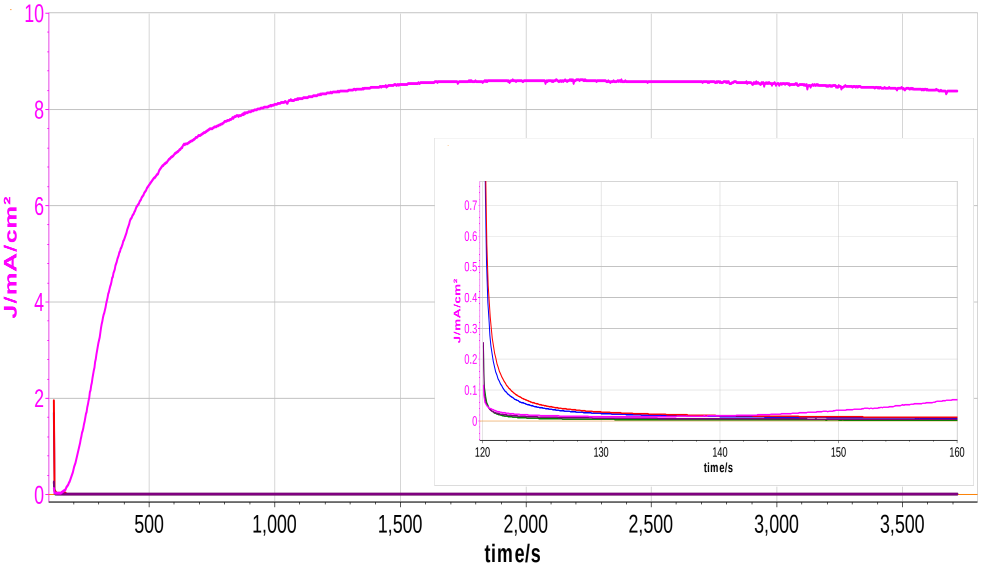
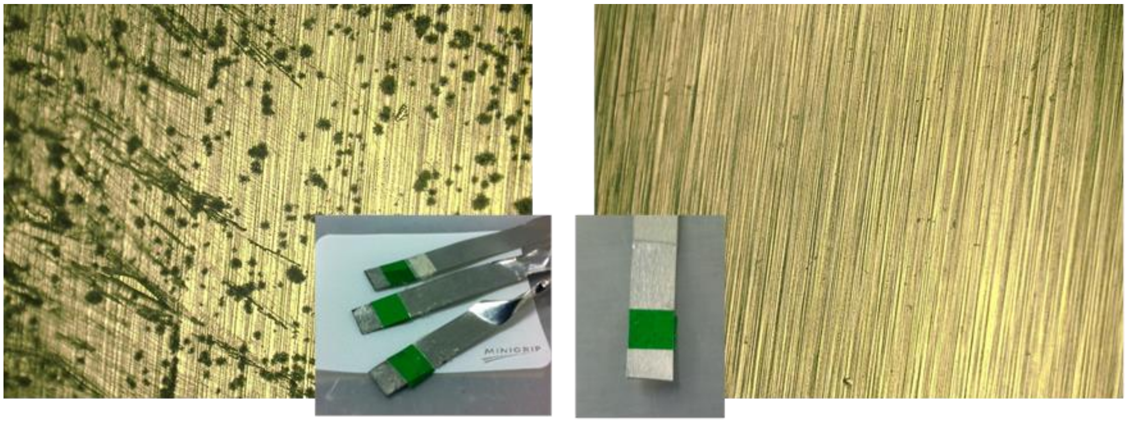


Figure 9: Stability of Various Lithium Salts

Experiments testing high-voltage stable salts against reference electrodes confirmed that the stability of the chosen salt held up when incorporated into test cells; **Figure 10** shows the lack of corrosion of the current collector in the test cell with the high-voltage salt when compared to that of the baseline salt.



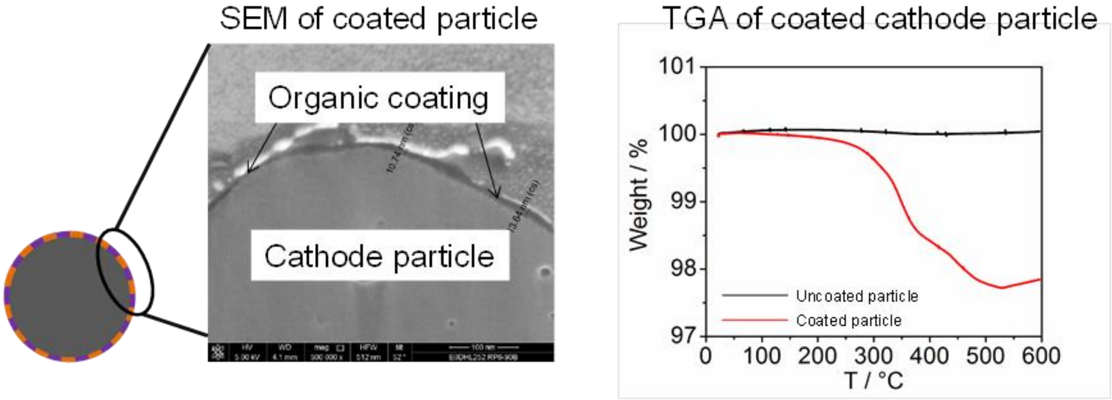
Corroded Al electrode using Baseline salt

Pristine Al electrode using HV salt

Figure 10: Salt solutions exposed to 4.3V (Li counter electrode, EC/DMC electrolyte)

3.2 Materials Formulation – Coatings on Cathode Particles

The work on coating techniques that was started in Phase I was continued as an alternative direction to using high-voltage polymers alone, and they were shown to have initial promising results. Organic coatings were demonstrated to be both conformal and adherent to the coated particle surface, suggesting that this technique may have the required properties to both impart high-voltage stability in the full cell and withstand the required deep cycling requirements of electric vehicle applications. **Figure 11** shows an SEM of a conformal organic coating on an active material particle alongside a TGA of a then washed coated particle, which confirms a successfully adherent coating:

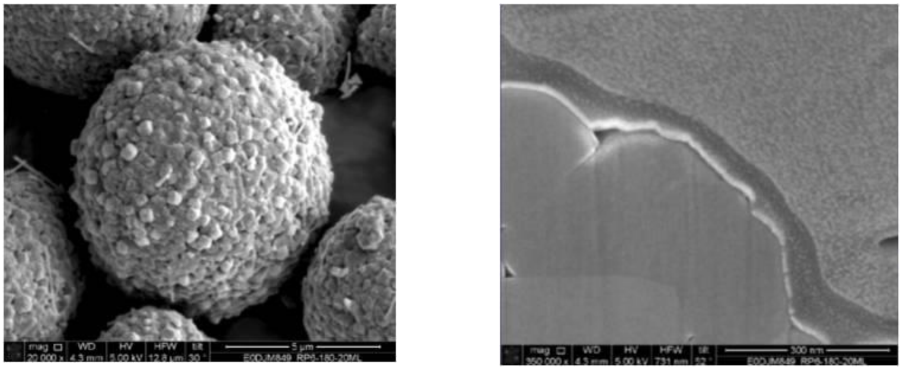


SEM of coated particles shows conformal polymer-coating

TGA of “coated and washed” particles shows weight loss, indicating adherent polymer coatings

Figure 11: Organic coatings on HV cathode particles

Subsequently further data was gathered with improved coating techniques and **Figure 12** shows a conformal surface coating of NCA particles with the high-voltage stable solid-state material at two different resolutions:



- Coated NCA, 5µm zoom
- Coated NCA, 300µm zoom

Figure 12: Organic coatings on HV cathode particles

3.3 Small Area Cell Cycling

As a result of this promising data, cycling of coated NCA cathodes was started. Cathode specific capacity of 200mAh/g was accessed at 4.2V in solid-state cells, using high-voltage stable salts that had previously been selected. Initial cycling data in **Figure 13** shows a 3-4x improvement in cycling stability of coated cathodes versus uncoated cathodes, with additional cycle life improvement being a primary focus during Phase II and II in order to achieve the project goals.

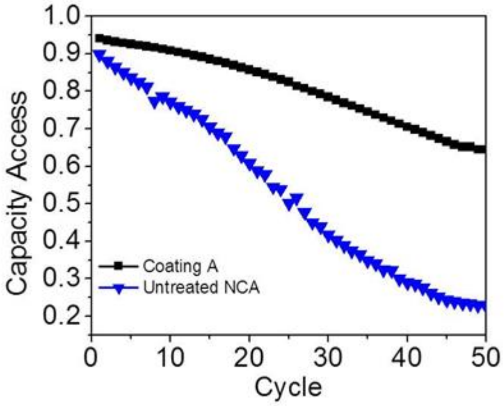


Figure 13: Cycle life of coated vs. untreated NCA-based cells

At the end of Q2-2013 Seeo achieved the third milestone of the program, in which the cathode batches were meeting the specifications. This was confirmed through the measured capacity in full solid state cells.

Additional test cells were constructed and cycled using coated NCA particles. **Figure 14** shows initial cycling results of these test cells, where cells at the target specific energy show distinct improvement over the control cells. Continued cycling was required to determine stability long term.

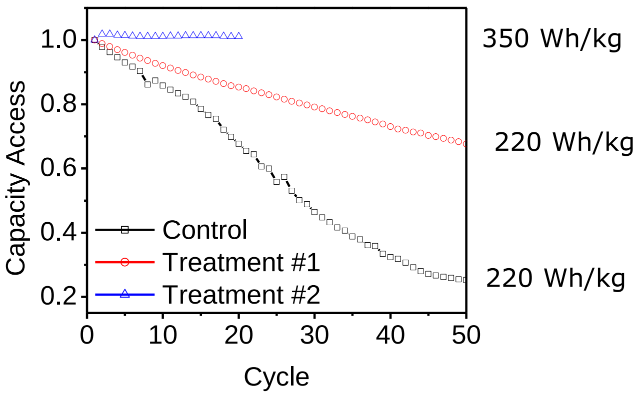


Figure 14: Initial cycling results at 350 Wh/kg using coated NCA

3.4 Alternative High Capacity Cathode Active Material

It was also suggested by DOE that Seeo's research team should begin evaluation of a low-voltage, high-capacity anode material, V_2O_5, in a solid-state cell design. This was thought as a promising material that would offer alternatives. Cell design parameters, including cathode/catholyte composition, loading, porosity and separator thickness needed to be optimized to evaluate additional cycling performance at high specific energies. **Figure 15** shows initial cycling results from test cells constructed with vanadium-based cathodes:

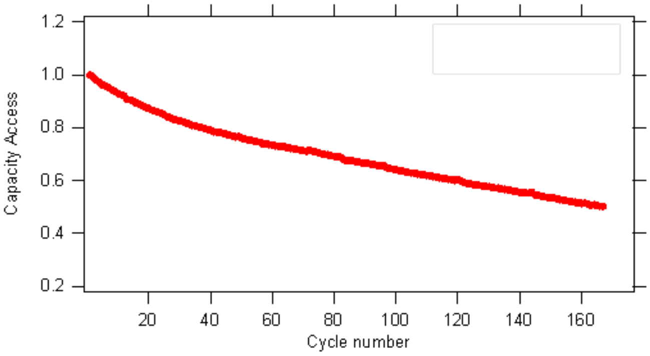


Figure 15: Cycling of 270 Wh/kg V_2O_5 test cells

Seeo continued this evaluation of the vanadium-based cathode, including other cell design parameters, but ultimately the cycling results did not match previous achievements with the selected high voltage cathode material.

3.5 In-Situ Coatings through Additives

Seeo therefore turned its focus to developing formulations, processes, and cell designs to further evaluate high-energy coated NCA cells in a solid-state cell. In addition to the pre-formed coatings approach, Seeo also investigated the use of additives which formed coatings in-situ upon cycling the cell. **Figure 16** shows results from cycling of these test cells – two items are clear from the figure. First, the approach using the combined coating demonstrates the most stability with NCA, and second, that there is an abrupt capacity fade possibly indicative of a mechanical break in the coating.

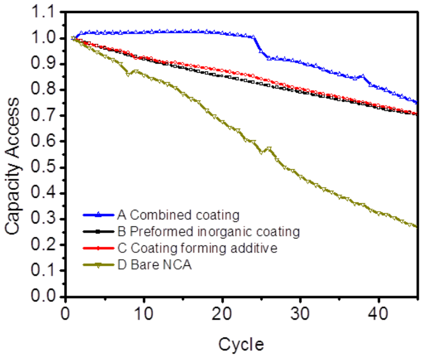


Figure 16: Updated cycling results using coated NCA

With the relative success and abrupt failure of this coating treatment, Seeo concluded that further development of thicker and thinner coatings on NCA would be required in order to optimize for stability and durability.

3.6 Construction of the Interim Cells for Delivery to ANL

Seeo ultimately decided to pursue the coated NCA design as the cathode material for interim and final cell deliveries. By the end of Q4-2013 and the end of Phase II Seeo accomplished the fourth milestone. This was to finalize the catholyte polymer and determine the material composition of the interim deliverable cells to be delivered to Argonne National Labs at the beginning of Phase III. Interim test cells were constructed, tested, and prepared to be shipped to DOE in mid-January 2014 for evaluation.

3.7 Lithium Foil Development and Fabrication (Hydro-Quebec)

As part of the program Hydro-Québec had been selected as a partner. Hydro-Québec was tasked to work on the development and fabrication of treated Lithium metal foils. During Phase II, Hydro-Québec, completed the installation and equipment upgrade work necessary for development and supply of treated Li foil samples. Process development on Li foil was commenced and Hydro-Québec delivered initial samples of treated Li foil for evaluation by Seeo's cell development team. Feedback on the thickness and surface properties of the sample was provided to Hydro-Québec (see figures below), and Hydro-Québec incorporated these recommendations into the development of future Li foil samples.

Analysis of treated Li foil samples provided by Hydro-Québec demonstrated significantly lower surface level roughness and AC impedance than a majority of commercial suppliers evaluated. Results from Li foil samples provided by HQ offered promising results. **Figure 17** shows the AC impedance of the HQ Li foil relative to 8 other foil samples; the interfacial impedance of the HQ sample was significantly lower than the average of the 8 samples from commercial suppliers.

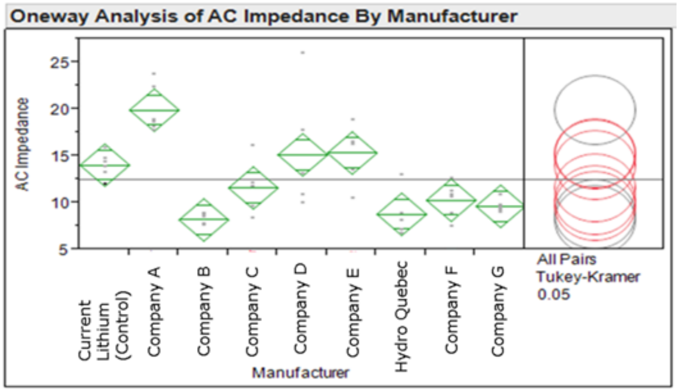
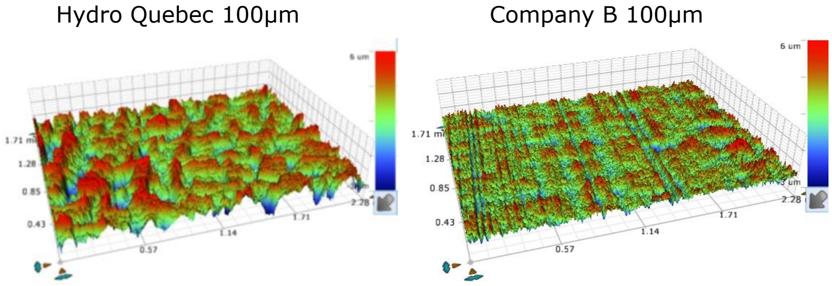


Figure 17: Interfacial Impedance of R&D Li Foil vs. Commercial Samples

A further look into the surface characteristics of the HQ Li Foil and that of the best performing commercial supplier reveals a similar low level of roughness. **Figure 18** presents a side-by-side comparison of white light interferometry of these two samples:


Hydro Quebec 100µm

Company B 100µm

Figure 18: Surface Profiles of R&D Li Foil vs. Best performing commercial supplier

A key goal of Li foil development in subsequent quarters was to replicate this performance using thinner foils. Seeo was committed to a continued collaboration with its project partner Hydro-Québec to develop thin Li foil samples and narrow down on the specification for final deliverable cells. But as the project progressed through Phase III in 2014, agreed upon and contracted delivery expectations were not met. Due to this reason, Seeo's subcontract with HQ lapsed. In order to move forward with the project as planned, sufficient quantities of thinner lithium foil were required and additional suppliers were investigated and finally selected. To continue the project in a timely manner, Seeo procured the necessary thinner lithium foil from alternative commercial suppliers.

CHAPTER 4: Phase III – Cell Fabrication and Testing

4.1 Interim Cells for Delivery to ANL

Initial activities in Phase III were focused on shipping of the high-voltage solid state test cells for interim testing at Argonne National Labs. Upon meeting this fifth program milestone, the research team continued worked on evaluating techniques and materials for the formation of stabilizing coatings on cathode particles. Materials research, process development, cell construction, testing, and cycling were the main development activities for the majority of Phase III. One of the major achievements was success in the combined cathode coating approach that demonstrated a significant improvement in capacity retention during cycle testing. Finally, the delivery of the final cells demonstrating the cumulative achievements during the program was accomplished. A commercialization report was also delivered at the end of this phase and end of the program.

The duration of Phase III was originally from the start of 2014 through September 30, 2014, which was also the contractual end of the project. The deliveries of the final cells was accomplished in Q4 of 2014 due to some process issues when scaling the technology up to larger form factor cells. DOE was informed of the delay and delivery was made in agreement with DOE.

At the beginning of Phase III Seeo delivered six interim cells with a capacity of 5 mAh each to Argonne National Labs for testing in January 2014. These cells had a lithium metal anode, an NCA cathode, and a dual electrolyte design. The tests performed on these deliverable cells were comparable to those on the baseline solid polymer electrolyte cells that were delivered to and evaluated by Argonne National Laboratory in mid-2012. **Table 4** below shows the details of the interim cells tested and verified by DOE.

Table 4: Interim Cell Characteristics

CELL PARAMETER	VALUE
Voltage Range	3.0 - 4.2 V
0.1C Discharge Capacity	5.64 ± 0.20 mAh
0.17C Depth of Discharge	94 ± 5%
Rated Internal Resistance (ESR), 5.25%SOC, 100s	72.8 ± 3.3 mOhm

4.2 Stability Monitoring of Cells and Continued Development

At the start of Phase III Seeo continued the development work from end of Phase II where it had been determined that stability of test cells that used pre-formed coatings on cathode particles were improved when combined with additives that form a coating in-situ during cycling. Based on this approach Seeo evaluated additional materials that could form coatings on high-voltage particles, initially with bare NCA and then with pre-formed coated NCA particles.

Results shown in **Figure 19** demonstrate cycling improvement over bare NCA with three out of four candidate additives. These three additive were then used in cells with pre-formed coatings on NCA. Two of the selected three additives showed significantly improved stabillity when tested with the precoted NCA particles, as can be seen in **Figure 20**. It was thought that this combined cathode particle coating approach was likely to be utilized in the final test cell design.

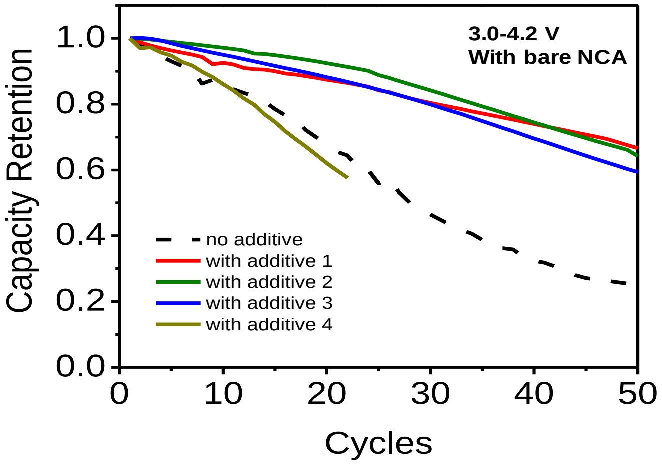


Figure 19: Cycling using additives with bare NCA

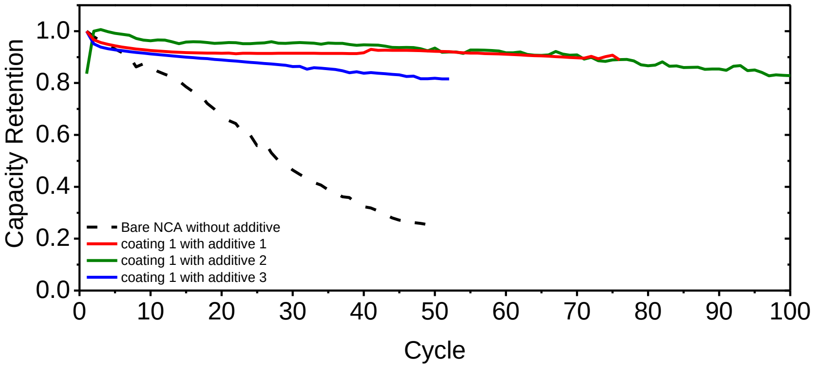


Figure 20: Cycling using additives with coated NCA

Based on the above results Seeo performed a series of optimization studies with this approach and after down-selecting the additive formulation, created test cells to study performance at various energy and power densities.

Seeo settled in on a final cell design based on results shown in **Figure 21** that demonstrate cycling improvement over using NCA with the Gen 1 catholyte, achieving several hundred cycles without a steep reduction in capacity. This was a significant improvement and the improved cycle life results are based on a cell design at 210 Wh/kg and a C/3 discharge – **Figure 22** shows the rate performance of test cells using the chosen combined additive and pre-formed coating technique at various energy densities, exceeding the project's goal of 350 Wh/kg cells. The rate performance of the test cells is much lower for higher energy densities, as expected, and the development focus during the remainder of the project was on improving the rate performance of the higher energy density cells while minimizing impact to cyclability.

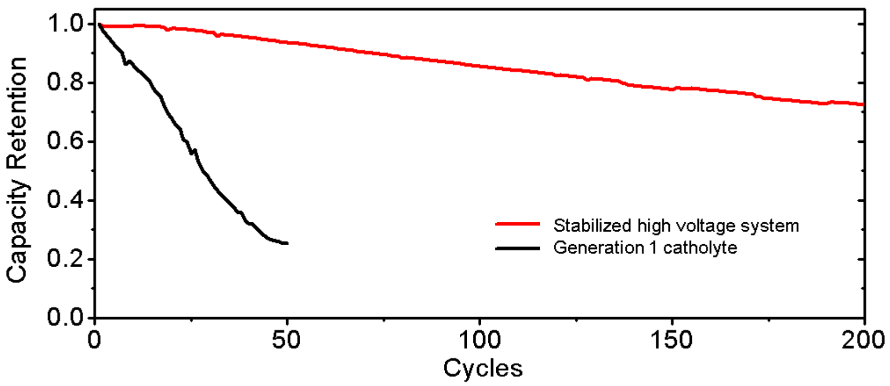


Figure 21: Stabilized high-voltage cell cycling vs. Gen 1 catholyte (with NCA)

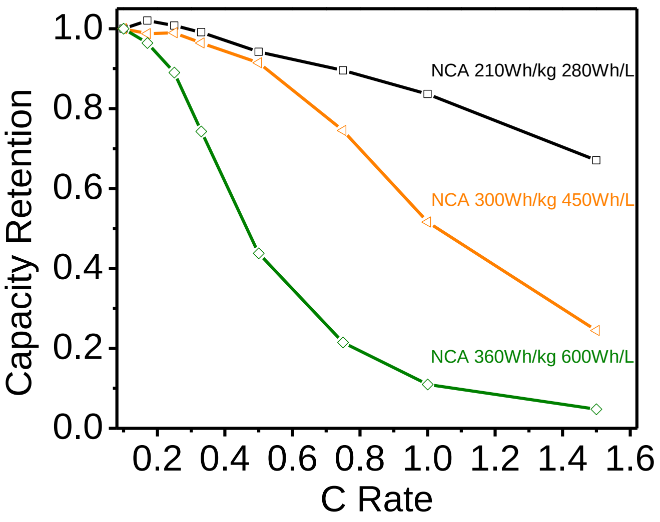
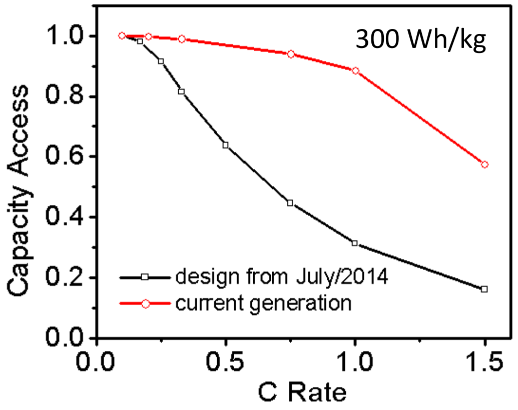


Figure 22: Cycling of stabilized cell at increasing energy densities

Seeo continued the optimization studies with the technique of combining pre-formed coatings on NCA cathode particles with additives to form a coating in-situ during cycling, with a focus on improving rate performance of high-voltage cells.

The effort on this approach was demonstrated in the results shown in **Figure 23** that show significant power improvements using this final cell design versus that achieved earlier. The final cell design enables 1C continues discharge and 3C pulse with only a mild reduction in available specific energy; this power/energy density profile can be improved with further cathode optimization but in its current state, represents about 50% performance improvement over Seeo's LFP based solid state cells currently in pilot production.



3C Pulse Testing

SOC at Start of Pulse	Pulse Duration
100%	108s
50%	20s

Figure 23: 1C continuous discharge (left) and 3C pulse capability (right) enabled through cathode optimization

4.3 Safety & Abuse Testing

As part of the development of the high energy cells, Seeo evaluated the safety performance of the Li metal/polymer/NCA system at the cell operating temperature of 80C. Tests were conducted by a third party and focused on four primary abuse tests: short circuit, overcharge, nail penetration, and thermal ramp. Three cells were studied for each abuse test. The capacity of the cells was 3mAh. While this is a relatively low capacity and testing will need to be repeated on larger capacity cells, these tests serve as a starting point and demonstrate the inherent materials safety of the solid state cells. The results of the abuse tests are summarized in **Table 5** below.

Table 5: Safety Test Results

4.4 Large Area Cell Processing Development

As part of the task to deliver final larger capacity cells achieving the R&D cell performance for high-voltage cells, Seeo's researchers focused on tuning coating and assembly processes to scale up cells from R&D size (5-25mAh) to manufacturing prototypes without any decline in performance. **Figure 24** shows results from this optimization effort, with large format cells (1 Ah) demonstrating identical charge/discharge profiles to that of R&D cells.

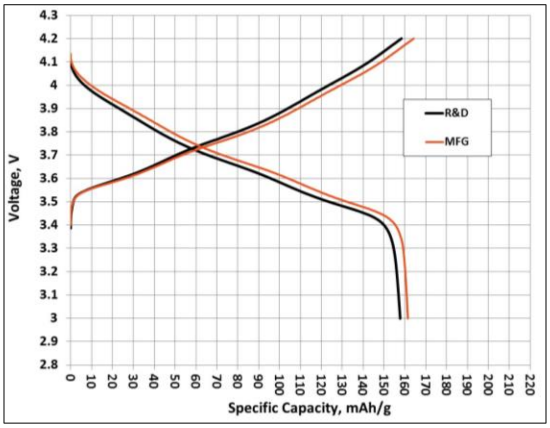


Figure 24: Charge/discharge curves of R&D and Manufacturing scale cells

4.5 Final Cells for Delivery to ANL

For the final cell deliverable, Seeo managed to fabricate cells with a capacity of 250mAh each, a significant increase from the interim cells that were delivered in January 2014 that had a capacity of 5 mAh each. Though the original objective was to deliver 2Ah cells, which turned out to be impossible in the time frame due to an unforeseen process related issue, Seeo achieved cells with 50 times the capacity of the cells delivered at the beginning of Phase III. Six cells were delivered to Argonne National Laboratory at the end of December for verification testing. These cells had a lithium metal anode, a coated NCA cathode, and a dual electrolyte design. The tests performed on these final deliverable cells were comparable to those performed on the previous cells that were delivered to and evaluated by Argonne National Laboratory. **Table 6** below shows the details of the final cells tested and verified by DOE.

Table 6: Final Cell Characteristics

CELL PARAMETER	VALUE
Voltage Range	3.0 - 4.2 V
0.1C Discharge Capacity	242 ± 24 mAh
0.17C Depth of Discharge	92 ± 3%
Rated Internal Resistance (ESR), 5.25%SOC, 100s	2.58 ± 0.4 Ohm

The final three milestones for the program were all completed in December of 2014. These were final cell testing, final cell delivery to DOE, and submission of a commercialization plan. The Commercialization Report was based on Seeo's experience with the lower voltage solid polymer cells manufactured on the pilot line in Hayward, customer interactions, and industry knowledge. Part of the Commercialization Report is included in the Final Technical Report.

CHAPTER 5: Commercialization Plan

5.1 Seeo Business Plan and Scale-Up

Through the work performed under this project, which was made possible by the support of the Department of Energy, Seeo has been able to scale-up both its core solid polymer electrolyte technology and cell technology, as well as demonstrating that Seeo's technology can be mass-manufactured. Seeo is committed to commercializing the technology here in the United States and in California and intends to further scale up its manufacturing capability to be able to more widely sample and qualify its technology with potential customers.

The funding provided by the DOE for this project has involved the participation and engagement of over 20 full-time employees. Seeo expects to expand its work force as required in connection with continued expansion of its customer opportunities in both grid energy storage projects and electric vehicle programs. Seeo's technology is equally well suited to both of these applications.

Seeo is targeting market opportunities in both the grid energy storage segment and the electric vehicles as these markets will be utilizing the same cells as the automotive industry. The battery packs will be different for the various applications but the base cell is expected to be the same to leverage the cost advantages offered by high volume demand from both of these markets. There are many projections for the potential size of these market segments over the coming years, and Seeo has made the following projections in **Figure 25** based on various market projections:

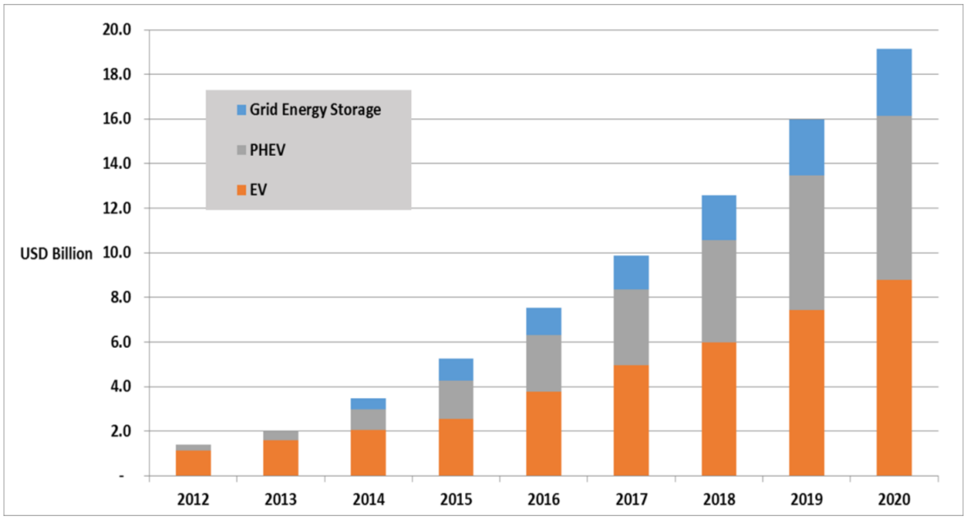


Figure 25: Market forecast for Plug-in Electric Vehicles and Grid Energy Storage

Source: Seeo, Inc. estimates based on data from B3 (2012), AABC (2013); Pike Research (2012)

As can be seen these market segments expect to see significant market growth over the coming years, with the market reaching over $20 billion early in the next decade. At that time it would be double the size of the existing lithium-ion market for portable electronics. Seeo's technology is ideally suited for these applications that require light and high energy density batteries.

Seeo's first step in addressing the market is to further scale up its manufacturing capability to be able to more widely sample and qualify its technology with potential customers. Seeo already has established testing and evaluation of its present technology with customers. The initial step will be to expand its existing pilot line in Hayward and has plans to increase the capacity ten-fold from its present 200-300 kWh production capacity to 2-3 MWh of cell production. Seeo expects to expand its work force as required in connection with continued expansion of its customer opportunities in both grid energy storage projects and electric vehicle programs.

From there Seeo expects to build a high volume manufacturing plant with one or more strategic partners. Seeo will leverage the manufacturing expertise and capital base of these potential strategic partners to most efficiently build out and continually expand the manufacturing capacity. Seeo will bring its unique technology and process knowhow and experience with our technology to the partnership. Seeo expects to have a significant ownership stake in these cooperation's, which likely would take the form of joint ventures.

Seeo has already made significant progress with a couple of strategic partners and is therefore well prepared to take this step once the technology is deemed mature and customers have been signed up to enter high volume production. Seeo has adopted this partnership strategy to allow it to go to market in a measured manner with experienced partners, which also builds the confidence of potential customers.

The following **Figure 26** illustrates the anticipated scale up and commercialization timeline that Seeo expects to achieve. This schedule assumes that one strategic partnership and manufacturing plant will be set up starting from late 2016.

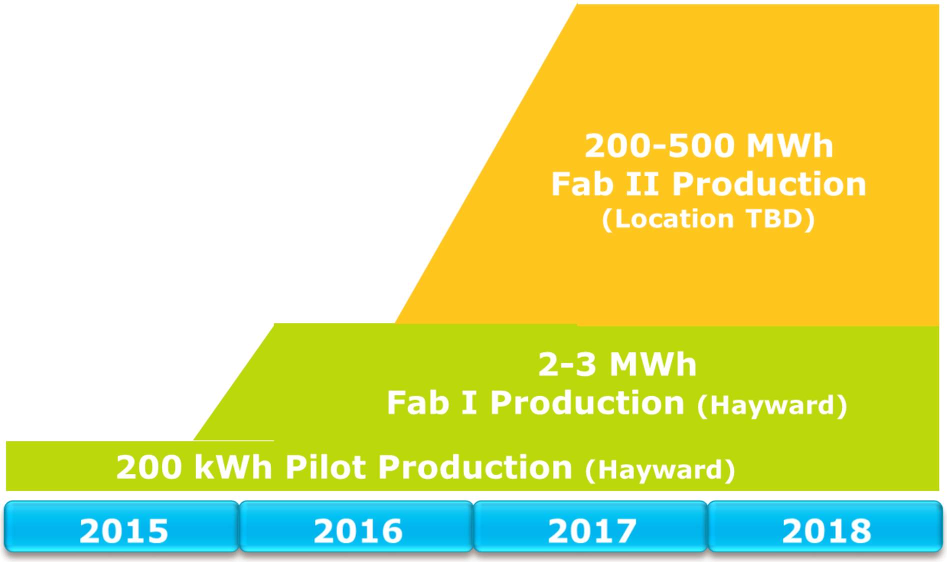


Figure 26: Seeo forecasted Manufacturing Capacity Expansion

Source: Seeo, Inc.

Seeo has actively been pursuing and engaging with customers in the United States and globally in both the automotive and grid storage market segments. Seeo has and continues to receive strong interests from a broad range of well-recognized companies in both of these market segments. This has been witnessed on various testing and evaluation of Seeo's technology by these companies. The activity is expected to increase in 2015 as the company continues to improve its technology. Seeo will use the increased capacity of its line in Hayward to sample and proceed through the qualification process with several of these customers. The expanded pilot line would have an output of up to 200 battery systems per year depending on the size of the systems, which should initially be sufficient for the anticipated qualification process.

The large scale manufacturing coming online in late 2016 and 2017 should match requirements from customers for production quality units, and achieving high volume manufacturing later in 2017 and into 2018.

5.2 Cost Model and Estimate

Cost is an important competitive parameter in the battery industry. Cost is typically defined in \$/kWh, especially for batteries for electric and plug-in hybrid electric vehicles, while \$/kW is a more relevant comparison for batteries for hybrid electric vehicles where the power capability is the competitive parameter. As Seeo's technology is a high energy technology the $/kWh value is discussed. Seeo has developed cost models for its technology based on the knowledge of present market prices of materials and projections for future development. An important contributor to the projected decrease in the cost is the achievement of higher energy density cells. Even though these higher energy density materials may have a slightly higher projected cost, the significant jump in energy density they provide lead to substantial improvements in the cost per kilowatt-hour.

The following **Figure 27** illustrates the cost development that Seeo is forecasting for its high energy technology for the period 2016 to 2020. Two curves are shown, one for a yearly production capacity of 400 MWh (equivalent to about 6 million cells with a capacity of 17Ah each) and the other for a capacity of 1GWh (1000 MWh). A continuous cost improvement is expected with the significant decreases in early 2017 and early 2018 coming from increases in energy density from 300Wh/kg to 350 Wh/kg (2017) and 350 Wh/kg to 400 Wh/kg (2018).

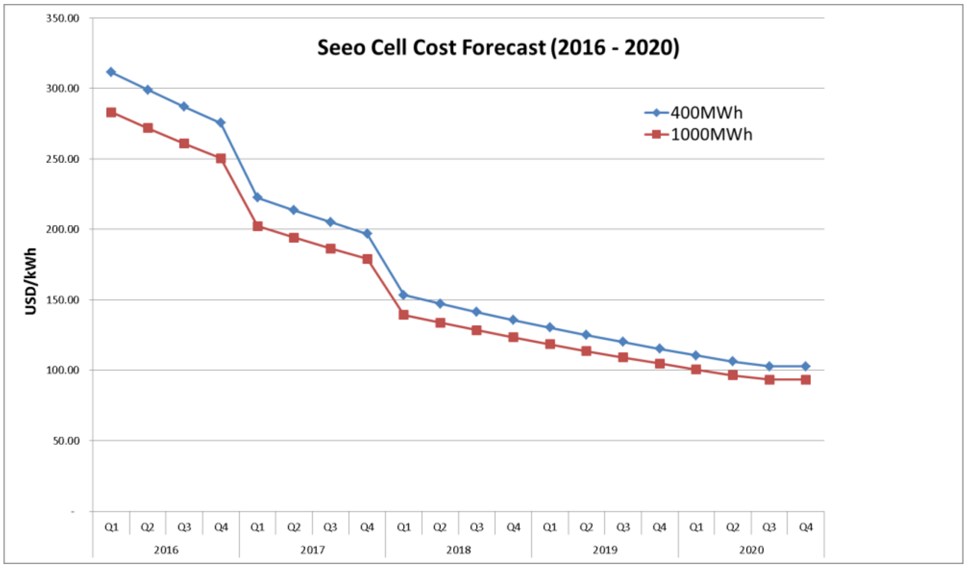


Figure 27: Seeo Forecasted Cell Price 2016 to 2020

Source: Seeo, Inc.

The cost projections for 2020 meet the long term targets set by the United States Automotive Battery Consortium and should prove highly competitive.

5.3 Intellectual Property

As part of the work performed during the duration of this project a number of invention disclosures and patent applications were filed, further strengthening Seeo's competitive position. Eleven invention disclosures were filed and are listed as Appendix B. The inventions encompass a wide range of technological contributions including novel materials, processing, cell design, and battery monitoring/control techniques.

5.4 Conclusions – Seeo Business Plan

Seeo's business plan and interest from potential customers and strategic partners demonstrates the strong interest for Seeo's technology. Seeo intends to commercialize its technology both in California and globally through these opportunities. Seeo is confident the materials and manufacturing of the technology can be achieved in highly cost competitive manner allowing for the mass adoption of Seeo's technology for electric vehicles, as well as for grid energy storage, applications. The partnership strategy the company is pursuing will also allow the company to leverage both its own expertise and the manufacturing and industry experience of its partners allowing for a lower risk and more market driven entry into the chosen markets for Seeo's technology.

Appendix A: Risks

At the beginning of the project a number of potential risks were identified. These are listed in **Table 7** below, with dates when the risks were retired and relevant comments tied to the findings in addressing the risks.

Table 7: Identified Risks Associated with Project and Specific Actions and Outcomes Taken to Eliminate Identified Risks

Risk	Like-lihood	Impact	Consequences	Retired or Realized	Date Retired	Comments
Phase I – Baseline Evaluation and Material Synthesis						
Difficulty in finding qualified technician resources	Low	Low	Project delays	Retired	10/2011	
Delay in availability of test facilities for baseline cell evaluation	Low	Low	Baseline cells delivered to DOE with partial performance and safety report	Retired	7/2012	
Increasing stiffness of polymer for interfacial stability compromises conductivity of electrolyte	Med	Low	Reduced power or temperature capabilities	Retired	12/2013	
Mechanically optimized polymers unstable at high potential	Med	Med	Reduced cell lifetime or add cost to manufacturing process	Realized		Secondary approach of coating particles adopted. Stability achieved in 2014.
Polymer backbone flexibility compromises mechanical performance of electrolyte.	Low	Med	Poor mechanical performance may degrade stability or add cost to manufacturing process	Retired	12/2013	
Additional impedance at polymer-polymer interface introduced	Low	Med	Reduced power capabilities	Retired	6/2013	

Phase II – Material Formulation and Scale-Up						
Cathode film processing cannot accommodate low porosity	Med	Low	Reduced energy capabilities	Retired	6/2013	
Large scale synthesis process does not achieve material specification	Low	High	Reduced cell performance	Retired	12/2013	
Large scale synthesis process proves prohibitively costly	Low	High	Compromised cost advantage	Retired	12/2013	
Delays in equipment availability for cathode formulation	Low	Med	Project delays	Retired	6/2013	
Reduced electrode uniformity in larger area coatings	Low	High	Compromised cell reproducibility, yield, and reliability	Retired	9/2014	
Phase III – Cell Fabrication and Testing						
Defects or inhomogeneity's resulting from cell stacking	Low	High	Compromised cell reproducibility, yield, and reliability	Retired	9/2014	
Large format cells do not meet expected capacity	Med	Med	Reduced energy performance	Realized		Unexpected processing difficulties resulted in smaller cells for deliverables.
Cells do not pass safety tests	Low	High	Project delays	Retired	7/2014	

Source: Seeo, Inc.

Appendix B: List of Inventions

Seeo Invention Disclosures made under US DOE Contract No DE-EE0005449

Seeo Reference	Title	Inventors	Date Reported	DOE S-Number
SE-054	Coated Particles For Lithium Battery Cathodes	Mohit Singh, Hany Basam Eitouni, Russell Clayton Pratt, Scott Allen Mullin, Xiao-Liang Wang	11/13/2014	S-140,312
SE-055	Coated Particles For Lithium Battery Cathodes	Mohit Singh, Hany Basam Eitouni, Russell C. Pratt, Scott Allen Mullin, Xiao-Liang Wang	11/13/2014	S-140,313
SE-056	Oxyphosphorus-Containing Polymers As Binders For Battery Cathodes	Russell Clayton Pratt, Scott Allen Mullin, Hany Basam Eitouni	11/13/2014	S-140,314
SE-057	Condensation Polymerization As A Route To Battery Catholyte Composites	Russell C. Pratt	11/13/2014	S-140,315
SE-059	High Density Electrodes For Lithium Metal Polymer Batteries	Xiao-Liang Wang, Scott Allen Mullin, Mohit Singh	11/13/2014	S-140,316
SE-062	Solvent Coating Directly Onto Lithium-Metal-Based Anodes	Scott Allen Mullin, Mohit Singh, Xiao-Liang Wang, Andrew Bingei	11/13/2014	S-140,317
SE-063	Nitrile-Containing Polymer Binders	Yan Li, Hany Basam Eitouni, Jin Yang	11/13/2014	S-140,318
SE-064	Relaxation Model In Real-Time Estimation Of State-Of-Charge In Lithium Polymer Batteries	Scott Allen Mullin, Albert Aumentado, Mohit Singh	11/13/2014	S-140,319
SE-066	A Method For Determining State Of Charge In Lithium Batteries Through Use Of A Novel Electrode	Mohit Singh, Xiao-Liang Wang	11/13/2014	S-140,320
SE-070	Lithium Ion Battery Catholyte And Cathodes	Russell C. Pratt, Yan Li, Xiao-Liang Wang, Hany Basam Eitouni	11/13/2014	S-140,321

SE-074	Improved Performance Block Copolymer Electrolytes Containing Polymeric Additives	Russell C. Pratt, Jin Yang, Jonathan Pistorino, Hany Basam Eitouni, Mohit Singh, Vishal Vijay	11/13/2014	S-140,322

Appendix C: List of Abbreviations

Ah	**Ampere-hour**
BMS	**battery management system**
C (rate)	**Current, rate of discharge or charge of cell or battery, e.g. C/3 is defined as a 3-hour discharge or charge of cell or battery**
DOE	**United States Department of Energy**
ESR	**equivalent series resistance**
FOA	**Funding Opportunity Announcement**
G	**Gram**
IP	**intellectual property**
Kg	**Kilogram**
kWh	**kilowatt-hour**
LBNL	**Lawrence Berkeley National Laboratory**
R&D	**research and development**
SOC	**state-of-charge**