

Office of Energy Efficiency and Renewable Energy

VEHICLE TECHNOLOGIES INCUBATOR

DE-FOA-0000988

Final Technical Report

Award No.: DE-EE0006830

Cooperative Agreement

24M Technologies, Inc.
130 Brookline St.
Suite 200
Cambridge, MA 02139
info@24-m.com
617-553-1012

Private Company

Project Title: Low Cost, Structurally Advanced Novel Electrode and Cell Manufacturing

Principal Investigator:
William Woodford, Ph.D.
wwoodford@24-m.com
617-553-1012 x163

DUNS number: 963160317

Award number: DE-EE0006830

January 10, 2017

Period of Performance: October 1, 2014 – September 30, 2016

Executive Summary

This document is the final technical report from 24M Technologies on the project titled: Low Cost, Structurally Advanced Novel Electrode and Cell Manufacturing. All of the program milestones and deliverables were completed during the performance of the award. Specific accomplishments are 1) 24M demonstrated the processability and electrochemical performance of semi-solid electrodes with active volume contents increased by 10% relative to the program baseline; 2) electrode-level metrics, quality, and yield were demonstrated at an 80 cm² electrode footprint; 3) these electrodes were integrated into cells with consistent capacities and impedances, including cells delivered to Argonne National Laboratory for independent testing; 4) those processes were scaled to a large-format (> 260 cm²) electrode footprint and quality and yield were demonstrated; 5) a high-volume manufacturing approach for large-format electrode fabrication was demonstrated; and 6) large-format cells (> 100 Ah capacity) were prototyped with consistent capacity and impedance, including cells which were delivered to Argonne National Laboratory for independent testing.

Section I. Accomplishments & Milestone Update

All project milestones and deliverables were successfully completed. The full set of milestones and deliverables accomplished during the performance of the award are summarized in Table 1 on the subsequent page.

Table 1 - Summary of Project Milestones and Deliverables

Task Number	Task Title	Milestone Type	Milestone Number
1	Increase anode volume loading percentage	Milestone	M1.1
1	Increase anode volume loading percentage	Milestone	M1.2
1	Increase anode volume loading percentage	Milestone	M1.3
2	Increase cathode volume loading percentage	Milestone	M2.1
2	Increase cathode volume loading percentage	Milestone	M2.2
3	F80 - Meet electrode quality metrics for production quality	Milestone	M3.1
4	F80 - Electrode yield	Milestone	M4.1
5	F80 - Cell manufacturing quality	Milestone	M5.1
	F80 - Cell manufacturing quality	Go/No-Go Decision Point	D5.2
6	Increase electrode footprint	Milestone	M6.1
6	Increase electrode footprint	Milestone	M6.2
7	F260 - Meet electrode quality metrics for production quality	Milestone	M7.1
7	F260 - Meet electrode quality metrics for production quality	Milestone	M7.2
8	F260 - Electrode yield	Milestone	M8.1
8	F260 - Electrode yield	Milestone	M8.2
9	F260 - Cell manufacturing quality	Milestone	M9.1
9	F260 - Cell manufacturing quality	Go/No-Go Decision Point	D9.2

Section II. Summary of Project Activities

The activities in the project were divided into 9 distinct tasks, as follows:

1. Increase anode volume loading percentage
2. Increase cathode volume loading percentage

Tasks 1-2 involved development at the level of the semi-solid electrode formulation.

3. Meet electrode quality metrics for production
4. Electrode yield
5. Cell manufacturing quality

Tasks 2-5 involved development of electrode forming and cell manufacturing with 80 cm² footprint prototypes.

6. Increase electrode footprint
7. Meet electrode quality metrics for production quality
8. Electrode yield
9. Cell manufacturing quality (large format cell)

Tasks 6-9 involved scale-up and development of electrode forming processes for electrodes with footprints > 260 cm², which is 24M's production-intent scale and subsequent cell assembly and prototyping with those large-format electrodes.

The activities and accomplished in each of these tasks is summarized briefly in this section.

Task 1: Increase anode volume loading percentage

The volumetric percentage of active material in a semi-solid formulation is a key driver of cell energy density and bill-of-materials (BOM) cost. This activity focused on developing anode semi-solid formulations with increased active volume loadings while retaining slurry rheology sufficient for high-quality electrode forming. The key metrics for evaluating slurry formulations identified through 24M's development activities are slurry mechanical properties (as a proxy for process robustness) and electrochemical performance. A new anode active material and new mixing methods were identified that enabled higher loading semisolid anode slurries with processability and performance comparable to the starting formulation.

Task 2: Increase cathode volume loading percentage

In parallel with the Task 1 activity to increase the volumetric loading of anode semi-solid formulations, Task 2 focused on increasing the cathode active volume loadings while retaining slurry rheology sufficient for high-quality electrode forming.

To achieve this goal, a number of LiFePO_4 suppliers and grades were screened to determine the best material to move forward with to achieve higher loadings. A total of 10 different LFP materials were evaluated from six suppliers. The material that was chosen to proceed with for higher cathode loading was is available in high volume and the manufacturer has the license to produce LiFePO_4 from the $\text{LiFePO}_4 + \text{C}$ Licensing consortium.

After this downselection, the conductive additive type, amount, and mixing method were all optimized to achieve a high loading semisolid cathode formulation with good electrochemical performance, comparable to the starting formulation

Task 3: Meet electrode quality metrics for production

In this task, 24M focused on establishing the basic process conditions and quality control metrics for semi-solid electrode forming (both anode and cathode) at an 80 cm^2 electrode footprint. Just before the kick-off of this program, 24M completed the full installation of its first pilot line manufacturing equipment, and began the process of optimizing the various conditions and processes. Figure 1 and Figure 2 are drawings and photos of the installed equipment. This equipment was used as the primary R&D vehicle for this task.

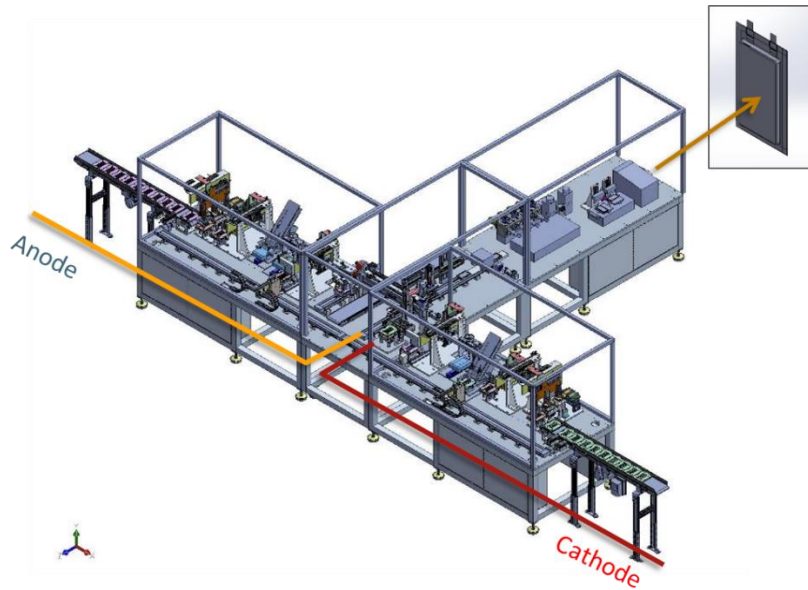


Figure 1. Isometric drawing of 24M's first pilot line for automated electrode formation and electrode stacking.

Task 3 activity initially focused on the installation and verification of all processes involved in the electrode fabrication and stacking steps. The pilot equipment was equipped with cameras for each processing step, laser metrics to measure thicknesses of each electrode made, and balances to record the weight of each electrode. Using these metrics and the approach described above, 24M optimized the electrode forming conditions at the 80 cm² electrode footprint and achieved the milestone tolerances for both weight and thickness. The methods and measurement techniques developed on this 80 cm² prototype line were later scaled and applied to the larger electrode fabrication equipment.



Figure 2. Photo of the actual pilot line.

Task 4: Electrode yield

Task 4 focused on the application of learnings and methods from Task 3 to achieve a high yield (> 90 %) of passing electrodes at the 80 cm² electrode scale.

In order to reduce the development cycle time for the evaluation of components for dispensing and shaping, 24M used 3D printed components, particularly for the evaluation of new nozzle and die designs. More rapid design iterations are possible with the 3D printed components.

24M used the in-line metric capability of the Gen2 Pilot Line to obtain electrode weights and thickness for the electrodes which were used in the Phase 1 deliverable cells and demonstrated good prototype yield levels for these deliverable cells.

Task 5: Cell manufacturing quality

Task 5 focused on the quality of cells manufactured at the 80 cm² footprint. The test vehicles for this activity were conventional pouch cells which used aluminized pouch material and heat sealed pass-through tabs.

In the second quarter of the program, 24M produced 115 cells across a period of seven days. Each cell had a target capacity of 4.676 Ah and was comprised of six unit cells (six cathode electrodes and six anode electrodes). The first milestone for Task 5 was to achieve a 75% yield from a sample size of 25 cells. The criteria used was $\pm 5\%$ for capacity and $\pm 10\%$ for impedance. 24M was able to achieve a yield of 89% based on capacity and 80% based on impedance.

Larger variations were observed in impedance than for the capacity of the cell. Based on these results, further optimizations were undertaken to improve the processing and handling of the electrodes during cell fabrication in order to reduce the variation of impedance.

At the conclusion of Phase I of the project, 24M produced 28 cells during a two day campaign of cell builds. 25 cells from the build were used for evaluation of the Milestone 5.1 criteria. The criteria for meeting 85% yield was $\pm 5\%$ capacity tolerance and $\pm 10\%$ impedance tolerance. According to the proposed criteria, the yield was 96%. Only one cell of the 25 cells was out of the impedance tolerance band. The average capacity of the cells was 4.35 Ah. All cells produced were within $\pm 5\%$ of the capacity. 10 cells from this build campaign were shipped to Argonne National Laboratory on October 7, 2015 for testing to satisfy the Go/No Go milestone, D5.2.

Task 6: Increase electrode footprint

Task 6 focused on scaling up the electrode forming methods demonstrated and optimized at the 80 cm² footprint scale to production-intent large footprint scale of > 260 cm². Activity in this task focused on using the same process concept to demonstrate scalability of the electrode forming

method. After demonstrating that the process could be scaled to larger footprints, focus shifted to demonstrating a high-volume manufacturing approach to forming large format electrodes.

The equipment shown in Figure 3 was installed at 24M just before the program kick-off and was used to demonstrate the capability of forming electrodes of a larger footprint ($>260\text{ cm}^2$) using the same processes used at the 80 cm^2 scale.

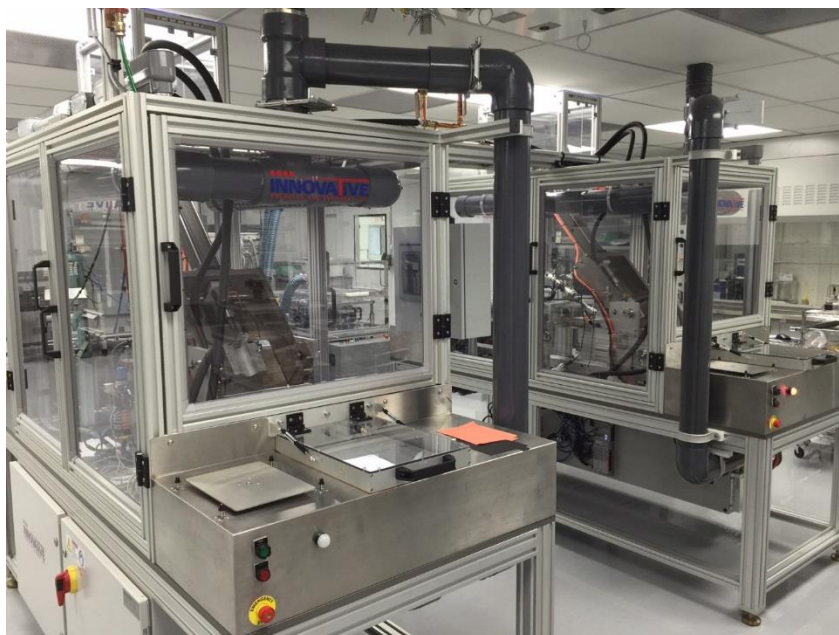


Figure 3. Electrode dispensing and forming machines for cathode and anode electrodes.

24M evaluated two different machine concepts for the large format electrode forming process. In the second half of 2015, 24M completed multiple experiments to compare the performance of the two machines in order to determine which method to use for high volume manufacturing. In most categories of comparison, the two machines are equal. However, a machine using a different forming concept than what was developed at the 80 cm^2 was selected as the high volume manufacturing approach.



Figure 4. High volume manufacturing method equipment to produce large footprint electrodes.

Task 7: Meet electrode quality metrics for production quality

Task 7 focused on demonstrating sufficient electrode quality in large format electrodes for cell production. Electrode quality was assessed and optimized for both large-format electrode forming methods.

Initial activity focused on the installation of equipment for electrode manufacturing of large format electrodes using both forming methods. The equipment allowed for hands-off processing of the electrodes to minimize deformation and defects of the electrodes during the cell assembly process. These processes are similar to what has already been installed in for the 80 cm² pilot line system shown in Figure 2.

In order to improve the quality of the larger electrodes, 24M utilized modeling and 3D printing equipment in order to quickly evaluate nozzle dispense designs. The simulations provide a quick method of evaluating new designs. This ability coupled with fast turnaround from 3D printed nozzles allowed 24M to quickly evaluate and improve the electrodes fabricated on the larger footprint equipment.

Task 8: Electrode yield

Task 8 focused on demonstrating a high yield for large-footprint (>260 cm²) anode and cathode semi-solid electrodes. As described previously, 24M demonstrated the capability of producing larger format electrodes that meet the tolerances for weight and thickness. With continued process optimization, high yields were achieved with the process concept demonstrated at the 80 cm² scale.

Task 9: Cell manufacturing quality (large format cell)

Task 9 focused on demonstrating cell manufacturing quality with the large format ($>260\text{ cm}^2$ footprint) electrodes. Ultimately, the program milestones were satisfied with large capacity ($>100\text{ Ah}$) can cells.

In Q8, the team completed a large format cell prototyping campaign to complete the remaining milestones for this program. With the improved product design and processes, the yield was 100% (8/8 good cells) according to the established program yielding criteria of $\pm 5\%$ capacity and $\pm 10\%$ impedance. The total variation in capacity (at both C/10 and C/4 rates) is 3.4% and the total variation in 10 kHz impedance is 2.8%. These data demonstrate completion of Milestone 9.1.

These prototype cells also satisfy the final program milestone, Deliverable 9.2. Four of these prototype cells were shipped to Argonne National Laboratory on September 28, 2016; four additional cells remained at 24M for parallel testing. These eight cells also achieved a yield of 100% yield according to the Deliverable 9.2 criteria. This yield and the shipment of these cells demonstrates completion of Deliverable 9.2.

Figure 5 shows a photo of two of the prototype cells shipped to Argonne National Laboratory for testing; both cells are in compression plates for testing. The compression plates are used to simulate the stack pressure which is applied when the cells are grouped into a module.



Figure 5 - Photo of two of the prototype 100+ Ah can cells in compression plates for testing. These cells were shipped to Argonne National Laboratory for testing to complete Deliverable 9.2. The yield of this cell prototyping campaign was 100% (8/8 cells good) according to the established program yielding criteria.

These eight deliverable cells are divided into two groups which are undergoing parallel test plans. All cells at Argonne National Laboratory are undergoing an initial capacity check and then three cells will be cycled at C/4 100% DOD at 30°C; the fourth cell is held as a reserve. The four cells at 24M are all cycled at C/4 100% DOD across a range of temperatures. Table 2 summarizes the detailed test conditions for the four cells which remained at 24M for testing.

Table 2 - Summary of test conditions for cells which remained at 24M for testing.

Cell Index	Cycling Parameters	Temperature (°C)
1	C/4 100% DOD (2.0-3.6V)	25
2	C/4 100% DOD (2.0-3.6V)	15
3	C/4 100% DOD (2.0-3.6V)	45
8	C/4 100% DOD (2.0-3.6V)	45

Section III. Products Developed Under Award

This project has led to several inventions, which have been disclosed to DOE. The titles of these inventions are

- Overcharge protection device
- Processes for Semisolid Battery Electrode Forming