Miltec UV International, LLC

Utilization of UV or EB Curing Technology to Significantly Reduce Costs and VOCs in the Manufacture of Lithium-ion Battery Electrodes

A revolutionary high speed approach to manufacturing cost reduction

May 14th, 2012

Principal Investigator: Dr. John Arnold

Presented by: Gary Voelker, Project Director

This presentation does not contain any proprietary, confidential, or otherwise restricted information
### Project Timeline:
- **Start Date:** 10/01/2011
- **End Date:** 09/30/2014
- **Percent Complete:** 20%

### Project Barriers:
- Battery Manufacturing Knowledge
- Coating optimization
- Calendering optimization

### Project Budget

<table>
<thead>
<tr>
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<th>Amount</th>
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<tbody>
<tr>
<td>DOE Share:</td>
<td>$4,572,709.00</td>
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<td>Miltec Share:</td>
<td>$1,143,299.00</td>
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<tr>
<td>Total Project Funding:</td>
<td>$5,716,008.00</td>
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<tr>
<td>FY11 Funding:</td>
<td>$381,086.80</td>
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<tr>
<td>FY12 Funding:</td>
<td>$1,524,347.20</td>
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### Partners:
- A123 Systems
- Argonne National Laboratory
- Oak Ridge National Laboratory
- Actega Coatings & Sealants
The objective of this project is to further develop and demonstrate the use of Ultraviolet (UV) and Electron Beam (EB) curing technology to significantly reduce the cost of manufacturing Lithium-ion battery electrodes by more than 50%.

**Additional Objectives:**

* Increase Process Speeds
* Reduce Energy Requirements
* Eliminate Solvent Recovery and VOCs
* Double-sided Coating
Current curing speeds for solvent based systems are approximately 15-25 fpm. With the introduction of a UV Curing System, these speeds would increase 10-20 times, to 100-200 fpm.

Solvent based curing systems only have the capability to cure one side. UV Curing systems, Miltec specifically, is introducing technology that will allow for double-sided curing. Increased curing speeds and double sided coating offer huge reductions in capital cost and operating expenses.
Current energy requirements for solvent based systems are large. The major contributor is fuel for the drying ovens plus Humidity Control, Heating & Cooling.

Capital cost for a UV curing system per unit of electrode area produced can be 1/20th to 1/100th a solvent based system.

Space requirements for a UV system are 1/10th to 1/20th a solvent based system.

A UV System essentially eliminates VOCs.
### FY2011

<table>
<thead>
<tr>
<th>Date</th>
<th>Milestone or Go/No-Go Decision</th>
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<tbody>
<tr>
<td>Oct-11</td>
<td>Milestone: Initiate formulation of cathode binder for Interim Cells using ANL and Miltec/ACTEGA binder and Lithium iron phosphate and NMC</td>
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<tr>
<td>Oct-11</td>
<td>Milestone: Initiate formulation of anode binder for Interim Cells using ANL and Miltec/ACTEGA binder</td>
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<tr>
<td>Oct-11</td>
<td>Milestone: Initiate Baseline Cell Design</td>
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### FY2012

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<th>Date</th>
<th>Milestone or Go/No-Go Decision</th>
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<tr>
<td>Feb-12</td>
<td>Milestone: Initiate Baseline Cell Fabrication</td>
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<tr>
<td>Feb-12</td>
<td>Milestone: Initiate Baseline Cell Test Plan</td>
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<tr>
<td>Mar-12</td>
<td>Milestone: Complete Baseline Cell Test Plan (Deliverable)</td>
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<td>Mar-12</td>
<td>Milestone: Complete Baseline Cell Design (Deliverable)</td>
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<td>Mar-12</td>
<td>Milestone: Complete Fabrication Baseline Cells for DOE Testing (Deliverable)</td>
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<td>Aug-12</td>
<td>Milestone: Finalize UV Curable Binder for Cathode, Interim Cells</td>
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<tr>
<td><strong>Binder Constituents</strong></td>
<td><strong>Miltec UV &amp; ACTEGA Kelstar</strong></td>
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<tr>
<td>• Monomers</td>
<td>Qualify = Testing</td>
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<tr>
<td>• Oligomers</td>
<td>• Electrolyte Immersion</td>
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<tr>
<td>• Photoinitiators</td>
<td>• Cyclic Voltammetry</td>
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<td>• Dispersants</td>
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**Electrolyte Immersion:**

Two grams, of varying formulas, are cured under a standard UV lamp system at 100 (fpm). After curing, the two gram ‘pucks’ are placed in dishes filled with a mixture of 60% dimethyl carbonate and 40% propylene carbonate, and placed in oven at 140°F for two weeks.

**Cyclic Voltammetry (CV):**

An electrochemical technique which measures the current that develops in an electrochemical cell under conditions where voltage is in excess of an electrodes predicted potential. CV is performed by cycling the potential of a working electrode, and measuring the resulting current.
Cyclic Voltammetry: Passing the Test

The CV tests shows UV binder chemistry can resist electrochemical degradation

Test Conditions:
Coin cell: CR 2032
Lithium metal as counter electrode
Electrolyte: 1.2 M LiPF₆ in EC: EMC (3:7 in weight)
CV: 0.1 mV/s between 1-5 V at room temperature for 3 cycles
Instrument: Solartron Analytical 1400 cell test system
**Miltec UV**
Mixing and Curing
- Baseline Loading Parameters
- Adhesion
- High Conductivity

**Baseline Loading:**
- 87% NMC
- 8% Binder
- 5% Carbon

**Adhesion:**
- Tape/Pull Test
  - Poor = Moderate Flaking
  - Good = Minimal Flaking
  - Excellent = No Flaking

**A123 Systems & ANL**
Baseline Cells
- Fabricate
- Test
- Submit

**Fabrication**
- 18 Baseline Cells
  - 9 NCM (ANL)
- 9 Nano-phosphate (A123)

**Conductivity/Resistance:**
- Measured in Ohms:
  - 0.1 = Achieved
  - 0.2 = Target
  - 0.35 = Acceptable
• Room temperature (about 20°C)
• Half cell (Li metal as anode) Coin cell 2032
• Electrolyte: 1.2 M LiPF6 in EC:EMC (3:7 in weight)
• Separator: Celgard 2325
• Assemble in He-glove box
• Electrochemical equipment: Maccor 4400
Electro-chemical Testing: Charge and Discharge

Sample | Charge (C/10) | Discharge (C/10) | C/5 | Thickness (Al foil 25 um) | Loading (87%:5% carbon:8%binder)
---|---|---|---|---|---
Sample A | 189 | 161 | 146 | 15 | 1.91 mg/cm²
Sample B | 184 | 155 | 145 | 14 | 1.94 mg/cm²
Sample D | 183 | 158 | 149 | 17 | 2.41 mg/cm²
Sample F | 187 | 155 | 147 | 15 | 2.01 mg/cm²
Coating: Single Layer
Thickness: 50 microns, Not calendered
Composition: 87% NMC, 8% Binder, 5% Carbon
Curing Speed: 100 fpm
UV Lamps: HPI Lamps, 2 @ 550 wpi
Initial Charge/Discharge: 154/128 mAh /g
Void = 32%
Profiles, Performance and Curves – Double Layer Coating: Two Layers

- Thickness: 20 microns, calendered,
- Composition: 87% NMC, 8% Binder, 5% Carbon
- Curing Speed: 100 fpm
- UV Lamps: HPI Lamps, 2 @ 550 wpi
- Initial Charge/Discharge: 163/142 mAh /g
- Void = 30%

**Voltage Profile**

**Cycle Performance**

- Voltage, V
- Capacity, mAh/g
- Cycle Number

**Cycle Performance**

- Capacity, mAh/g
- Cycle Number
Overall Results:
• Successfully qualified 5 oligomers, 4 monomers, 4 photoinitiators and 4 dispersants
• Successfully mixed 87% NMC, 8% UV binder, 5% Carbon
  o Coated, cured at 150 fpm
  o Excellent adhesion per scratch test
• Nominal 35% voids
• 14 mg/cm² NMC loading
• 0.15-0.36 ohm resistance
• Initial charge discharge nominal 150/135 mAh/g
Coin Cell Test Results Significance:

• Coin cell tests show thin coatings with UV binders and NMC show promise for handling charge and discharge for power applications

• Coin cell tests show thicker coatings with UV binders and NMC show promise for handling charge and discharge for energy applications

• Coin cell tests show thin and thick coatings with UV binders and NMC show promise for multiple charge and discharge cycles
**ACTEGA Kelstar, Inc. – Innovation Laboratory**

- Subcontractor to Miltec UV
- 30% cost share in all efforts
- ACTEGA Kelstar provides a representative to the Advisory Team and will provide input to the Cell Design Reports with regard to binder description and testing.
- ACTEGA effort managed by Jim Wittig, VP Innovation Laboratory

**A123 Systems - Advanced Research & Government Solutions Group**

- Cost Sharing Partner
- The total A123 systems effort includes 44% cost share.
- A123 efforts are conducted at A123’s headquarters and Lithium-ion battery research facilities in Watertown, MA, and Ann Arbor, MI.
- A123 Systems serves a consultant role to Miltec UV as a member of the Advisory Team ensuring Miltec UV approaches to electrode and cell design and manufacturing will be compatible with a typical battery manufacturing process in either a new or retrofit application
- A123 effort managed by Mike Wixom, Senior Technical Director
Collaboration and Coordination

Argonne National Laboratory – Chemical Sciences and Engineering Division

• Subcontractor to Miltec UV
• Performs analytical and electrochemical testing efforts
• Cell fabrication efforts
• ANL has world class laboratory facilities for the fabrication and testing of Lithium-ion cells.
• The ANL effort is managed by Dr. Khalil Amine, Senior Scientist

Oak Ridge National Laboratory – Materials Science and Technology Division

• Subcontractor to Miltec UV
• Acts in a consultant role to Miltec UV in the technical areas of electrochemical performance and coating technology
• Provides specific analytical testing of selected electrode samples prepared by Miltec UV
• Provides technical review support as needed to Miltec UV for Cell Test Plans and Cell Design Reports
• ORNL effort is managed by Dr. Claus Daniel, Staff Scientist
Proposed Future Work – FY12 and FY13

FY12:
• Baseline Cell Test Plan
• Baseline Cell Design
• 18 Baseline Cells for DOE validation testing
  • (9) NCM Cells prepared by ANL
  • (9) Nano-Phosphate cell prepared by A123
• Finalize UV Curable Binder for Cathode, Interim Cells

FY13:
• Initiate Interim Cell Fabrication
• Initiate Interim Cell Test Plan
• Complete Interim Cell Test Plan (Deliverable)
• Complete Fabrication Interim Cells for DOE Testing (Deliverable)
• Initiate UV/EB Curable Binder Formulation for final Cells
• Complete Interim Cell Performance Milestone Report
• Go-No-Go Decision Point Based on Interim Cell Performance (Deliverable)
• Initiate UV/EB Curable Binder Formulation for Final Cells
Foundations in place: During the first quarter of the contract award, Miltec UV acquired personnel, equipment, materials, and a new facility to initiate the program.

Since October 2011, Miltec UV has successfully qualified candidate UV Curable constituents including: 5 Oligomers, 4 Monomers, 4 Photoinitiators, and 4 Dispersants.

ANL and A123 Systems continue to report successful CV and Electrochemical testing on cathode samples.

In March 2012, Miltec UV delivered 18 Baseline Cells prepared by ANL & A123.

Anode Preparation Work for Budgetary Period 2 Initiated for Interim Cells.

With initial success in only the first two quarters, the Interim Cell milestones are ahead of schedule with a Go/No-Go decision due 11/22/2013.