



# 2012 DOE Vehicle Technologies Program Review Presentation



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

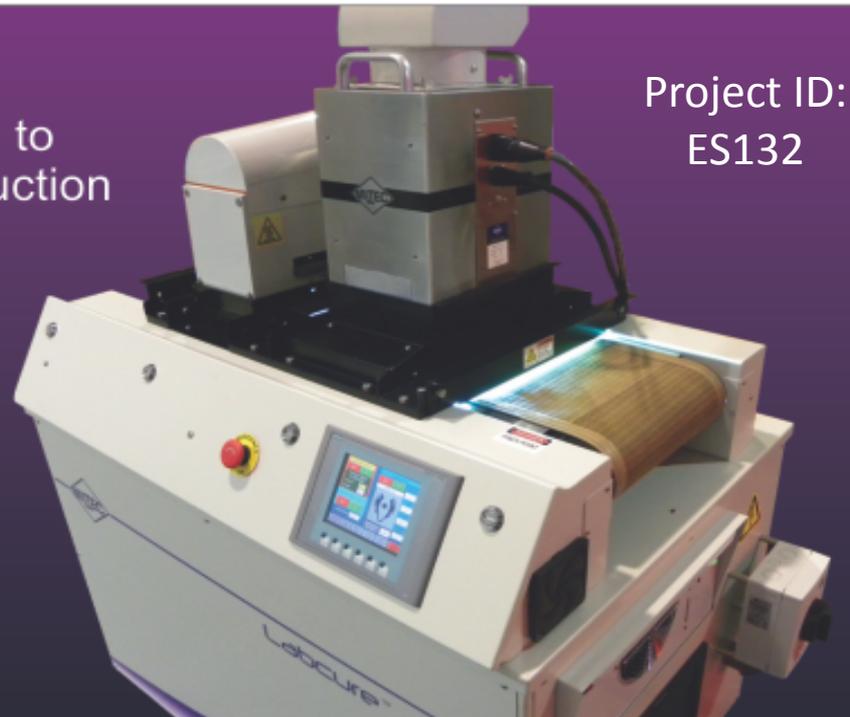
A revolutionary  
high speed approach to  
manufacturing cost reduction

Project ID:  
ES132

May 14<sup>th</sup>, 2012

Principal Investigator:  
Dr. John Arnold

Presented by:  
Gary Voelker,  
Project Director





# Overview



## 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

DOE Share:	\$4,572,709.00
Miltec Share:	<u>\$1,143,299.00</u>
Total Project Funding:	\$5,716,008.00
FY11 Funding:	\$381,086.80
FY12 Funding:	\$1,524,347.20

## Partners:





# Project Objectives



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:**

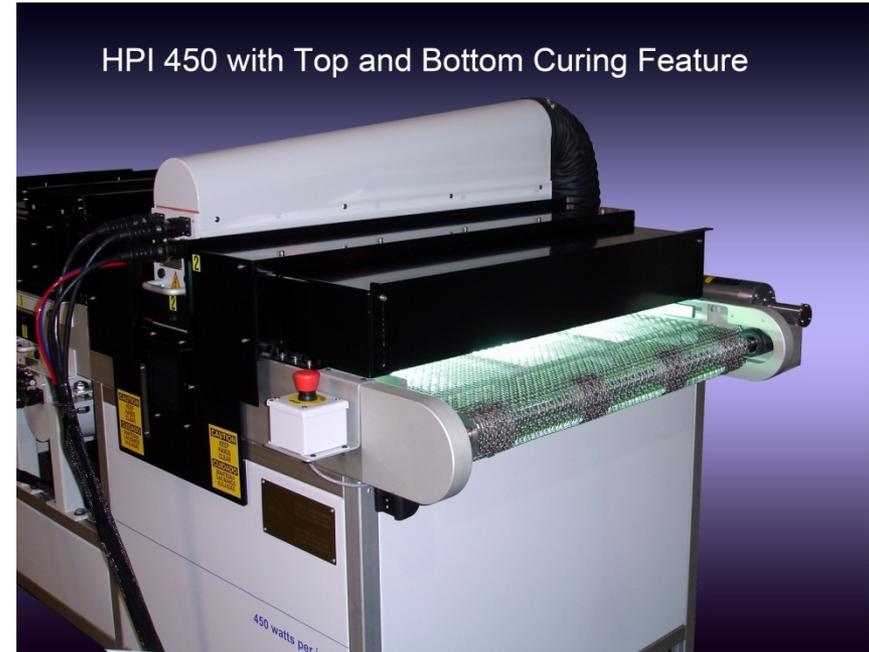
- Increase Process Speeds*
- Reduce Energy Requirements*
- Eliminate Solvent Recovery and VOCs*
- Double-sided Coating*



# Increase Process Speeds & Double Sided Curing

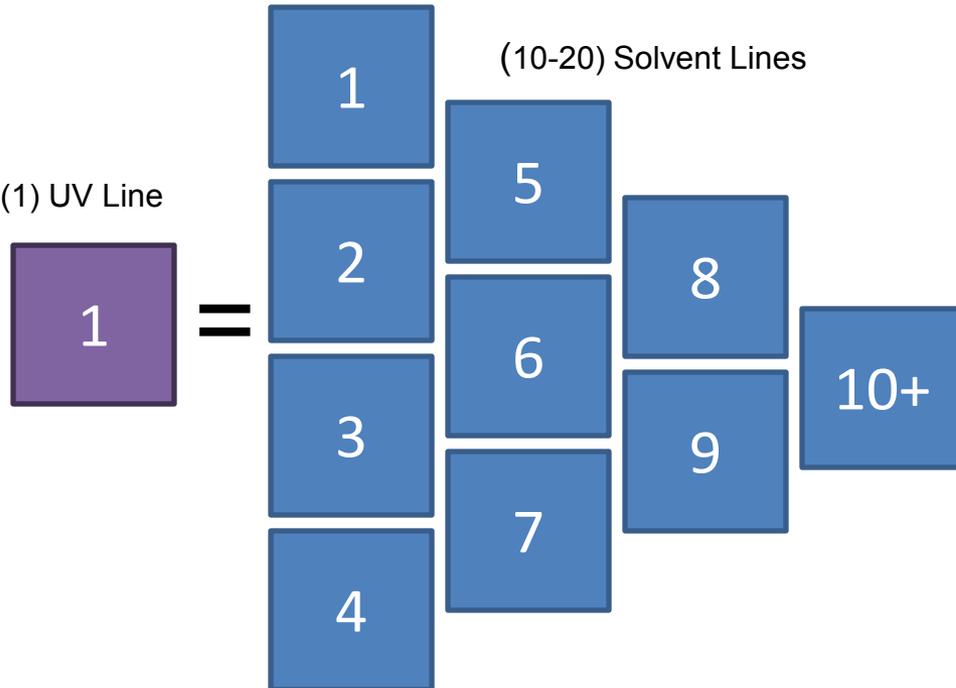


*Current curing speeds for solvent based systems are approximately 15-25 fpm. With the introduction of a UV Curing System, these speeds would increase 10-20 times, to 100-200 fpm*



HPI 450 with Top and Bottom Curing Feature

*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.*





# Reducing Capital, Energy, VOC and Space Requirements



*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 1/20<sup>th</sup> to 1/100<sup>th</sup> a solvent based system.*

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

*A UV System essentially eliminates VOCs.*





# Project Milestones

(FY11 & FY12)



<b>FY2011</b>	<b>Milestone or Go/No-Go Decision</b>
Oct-11	Milestone: Initiate formulation of cathode binder for Interim Cells using ANL and Miltec/ACTEGA binder and Lithium iron phosphate and NMC
Oct-11	Milestone: Initiate formulation of anode binder for Interim Cells using ANL and Miltec/ACTEGA binder
Oct-11	Milestone: Initiate Baseline Cell Design

<b>FY2012</b>	<b>Milestone or Go/No-Go Decision</b>
Feb-12	Milestone: Initiate Baseline Cell Fabrication
Feb-12	Milestone: Initiate Baseline Cell Test Plan
Mar-12	Milestone: Complete Baseline Cell Test Plan (Deliverable)
Mar-12	Milestone: Complete Baseline Cell Design (Deliverable)
Mar-12	Milestone: Complete Fabrication Baseline Cells for DOE Testing (Deliverable)
Aug-12	Milestone: Finalize UV Curable Binder for Cathode, Interim Cells



# Qualifying UV Curable Binders



## Binder Constituents

- Monomers
- Oligomers
- Photoinitiators
- Dispersants

## Miltec UV & ACTEGA Kelstar

- Qualify = Testing
- Electrolyte Immersion
  - Cyclic Voltammetry

### **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) :**

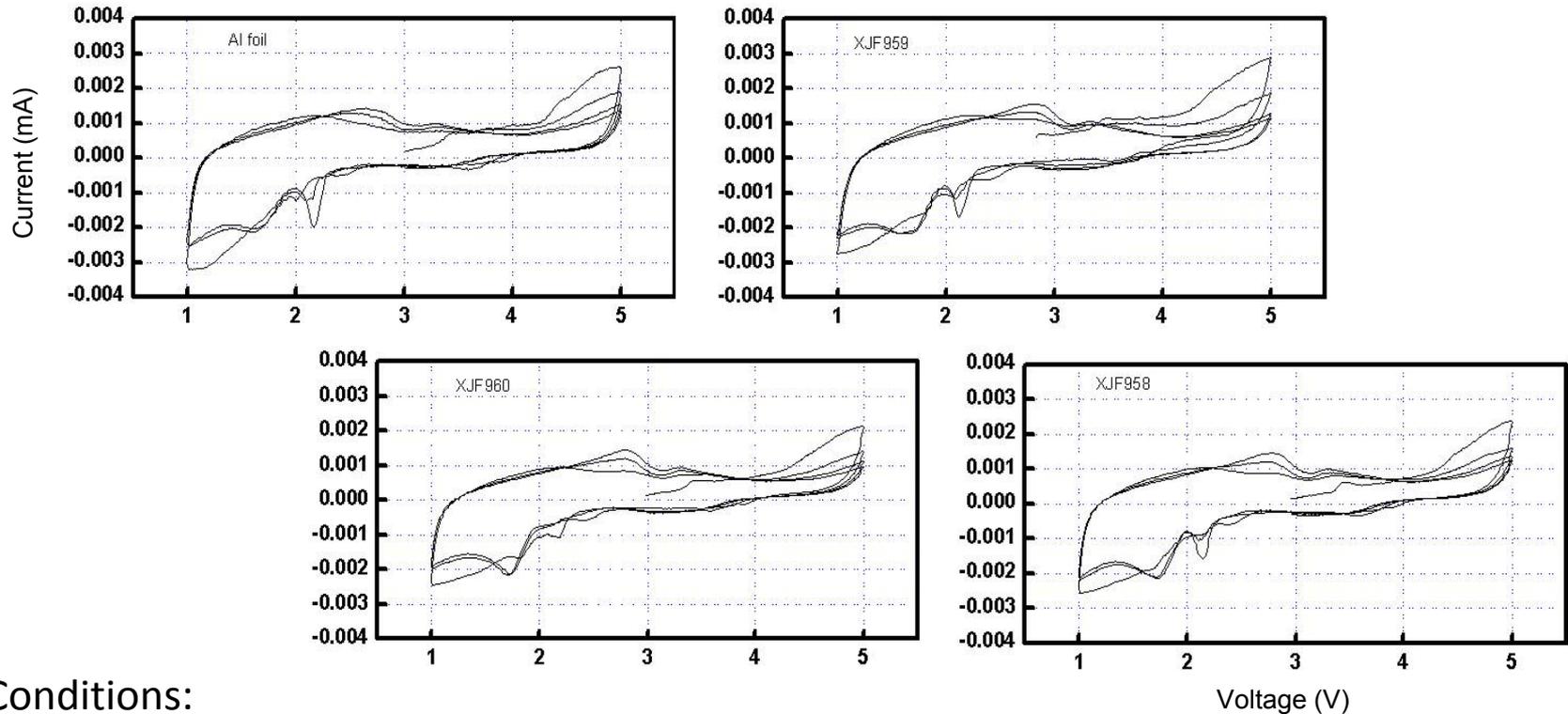
An electrochemical technique which measures the current that develops in an electrochemical cell under conditions where voltage is in excess of an electrodes predicted potential. CV is performed by cycling the potential of a working electrode, and measuring the resulting current.



# 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  $\text{LiPF}_6$  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



# Mix, Cure & Fabricate



## Miltec UV

Mixing and Curing

- Baseline Loading Parameters
- Adhesion
- High Conductivity

## A123 Systems & ANL

Baseline Cells

- Fabricate
- Test
- Submit

### Baseline Loading:

87% NMC  
8% Binder  
5% Carbon

### Fabrication

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

### Adhesion:

Tape/Pull Test  
Poor = Moderate Flaking  
Good = Minimal Flaking  
Excellent = No Flaking

### Conductivity/Resistance:

Measured in Ohms:  
0.1 = Achieved  
0.2 = Target  
0.35 = Acceptable



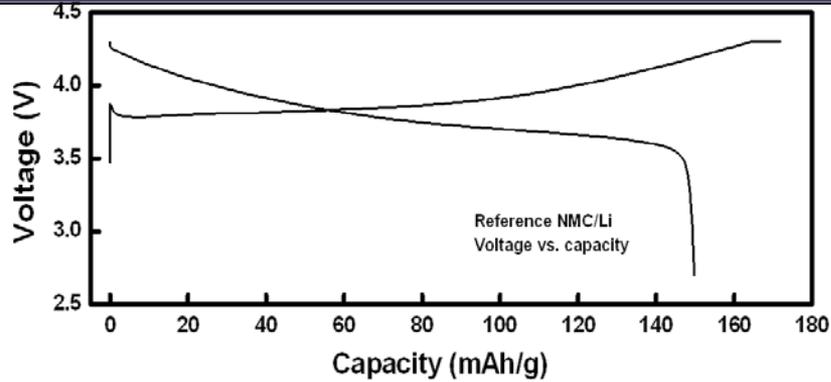
# Coin Cell Test Conditions



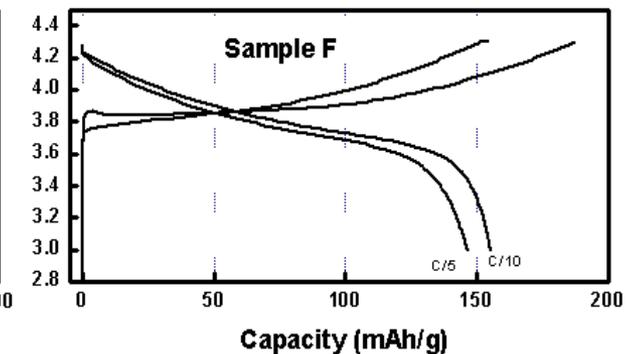
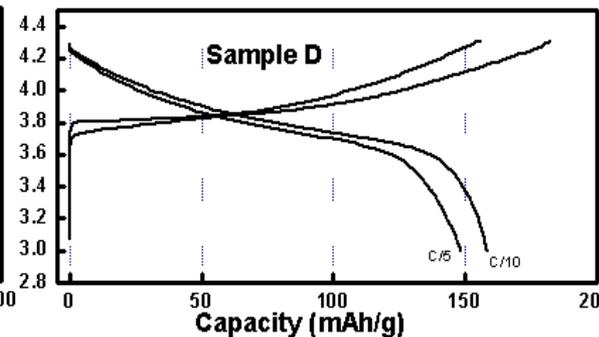
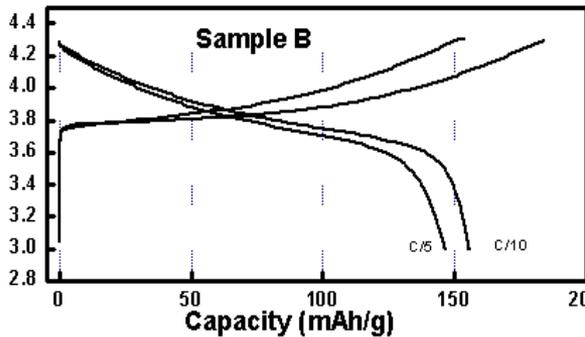
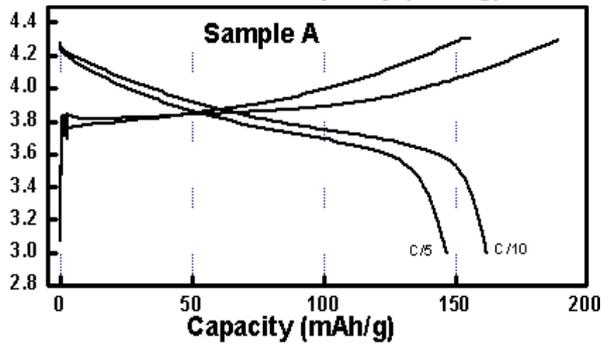
- Room temperature (about 20°C)
- Half cell (Li metal as anode) Coin cell 2032
- Electrolyte: 1.2 M LiPF<sub>6</sub> in EC:EMC (3:7 in weight)
- Separator: Celgard 2325
- Assemble in He-glove box
- Electrochemical equipment: Maccor 4400



# 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/cm <sup>2</sup>
Sample B	184	155	145	14	1.94 mg/cm <sup>2</sup>
Sample D	183	158	149	17	2.41 mg/cm <sup>2</sup>
Sample F	187	155	147	15	2.01 mg/cm <sup>2</sup>

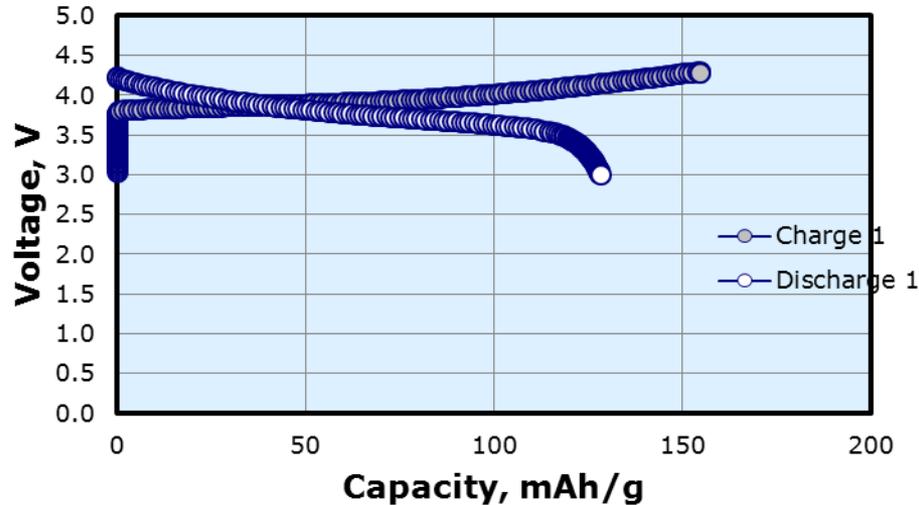




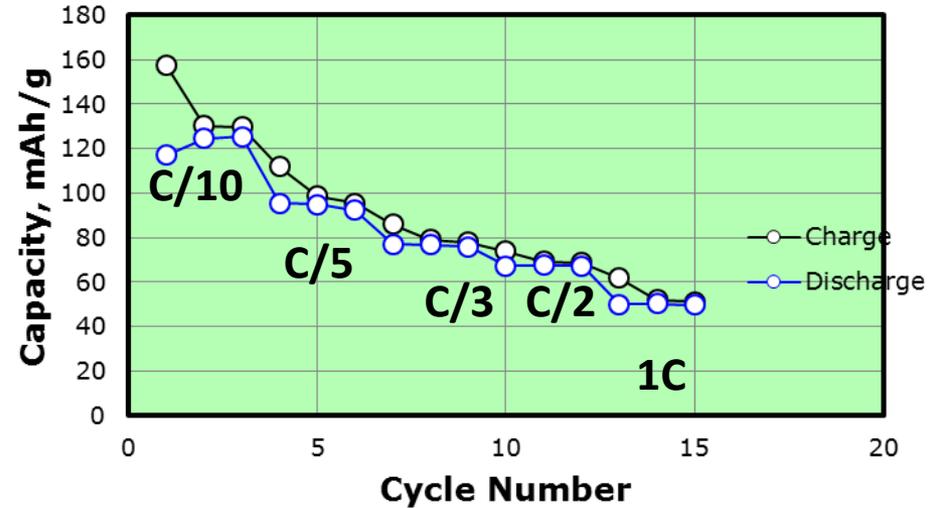
# Profiles, Performance and Curves – Single Layer



### VOLTAGE PROFILE

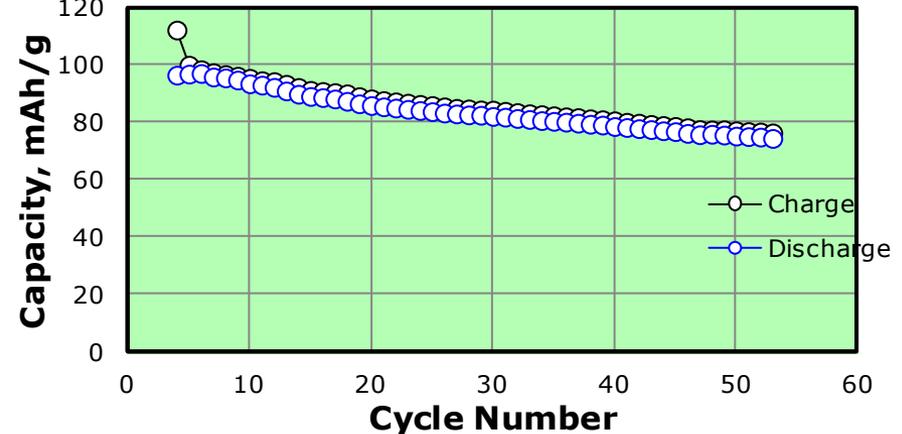


### CYCLE PERFORMANCE



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%

### CYCLE PERFORMANCE

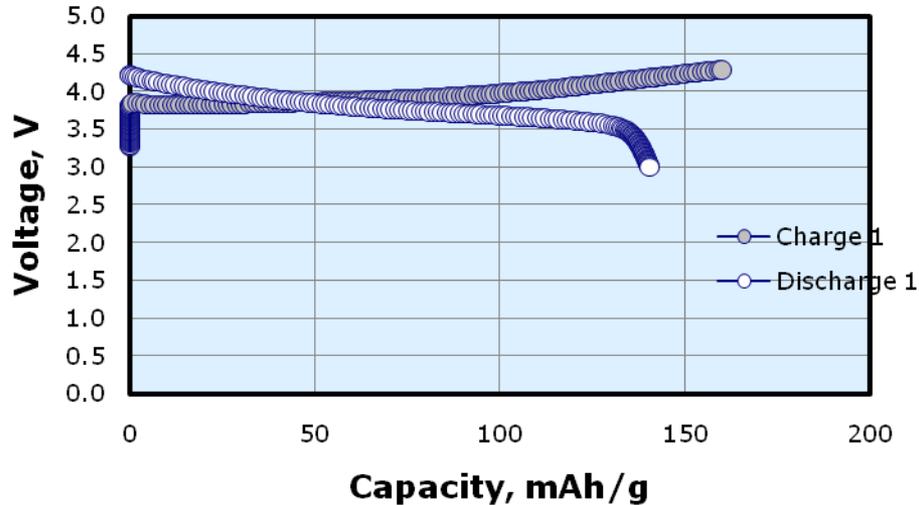




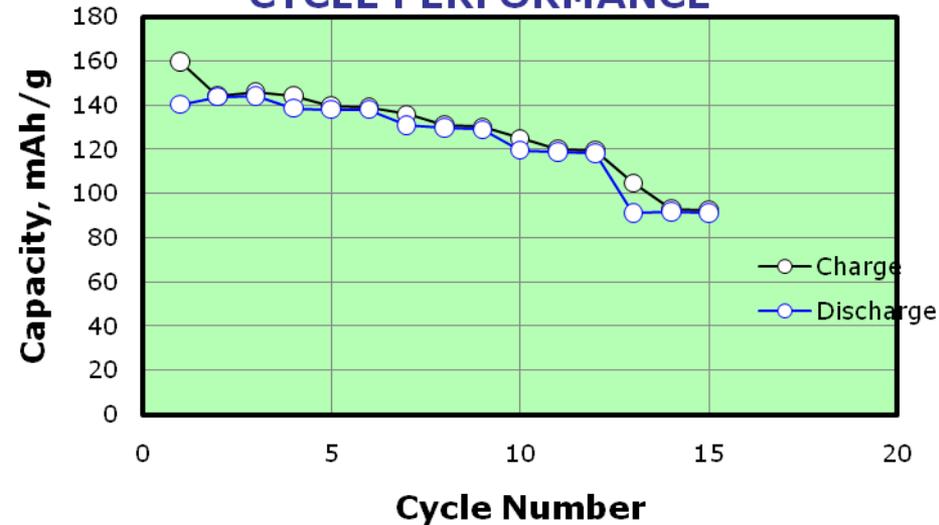
# Profiles, Performance and Curves – Double Layer



### VOLTAGE PROFILE

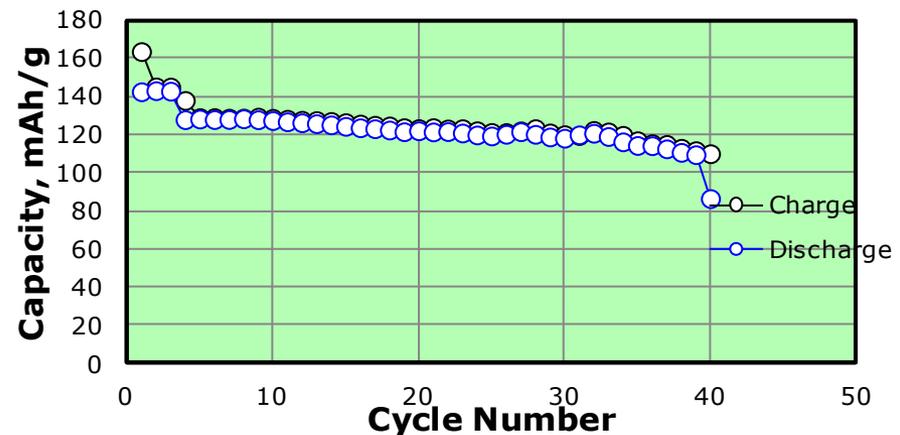


### CYCLE PERFORMANCE



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%

### CYCLE PERFORMANCE





# Technical Results Summary



## **Overall Results:**

- Successfully qualified 5 oligomers, 4 monomers, 4 photoinitiators and 4 dispersants
- Successfully mixed 87% NMC, 8% UV binder, 5% Carbon
  - Coated, cured at 150 fpm
  - Excellent adhesion per scratch test
- Nominal 35% voids
- 14 mg/cm<sup>2</sup> NMC loading
- 0.15-0.36 ohm resistance
- Initial charge discharge nominal 150/135 mAh/g



# Technical Results Summary



## **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



# Collaboration and Coordination



## **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



# Summary



- 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