ES-132

Miltec UV International, LLC

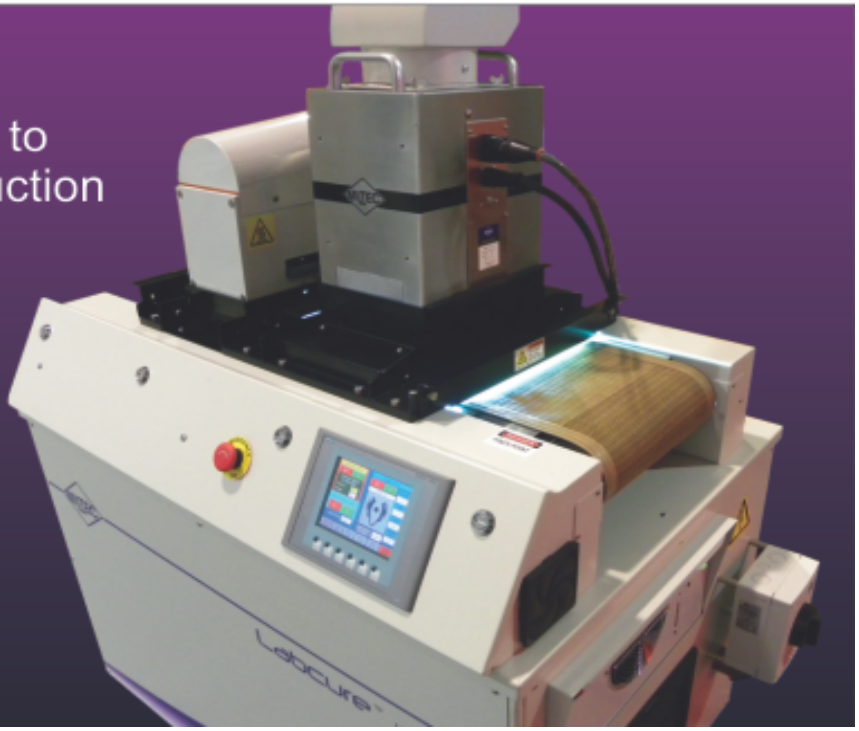
Utilization of UV or EB Curing Technology to Significantly Reduce Costs and VOCs in the Manufacture of Lithium-ion Battery Electrodes

A revolutionary high speed approach to manufacturing cost reduction

May 13th, 2013

Principal Investigator:
Dr. John Arnold

Project Director:
Gary Voelker



Overview

Project Timeline:

- Start Date:10/01/2011
- End Date: 09/30/2014
- Percent Complete: 50%

Project Goal:

Demonstrate utilization of UV curable binder to produce LIB with performance equal to or greater than PVDF baseline and reduce electrode manufacturing cost by 50%.

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

Partners:

Argonne NATIONAL LABORATORY

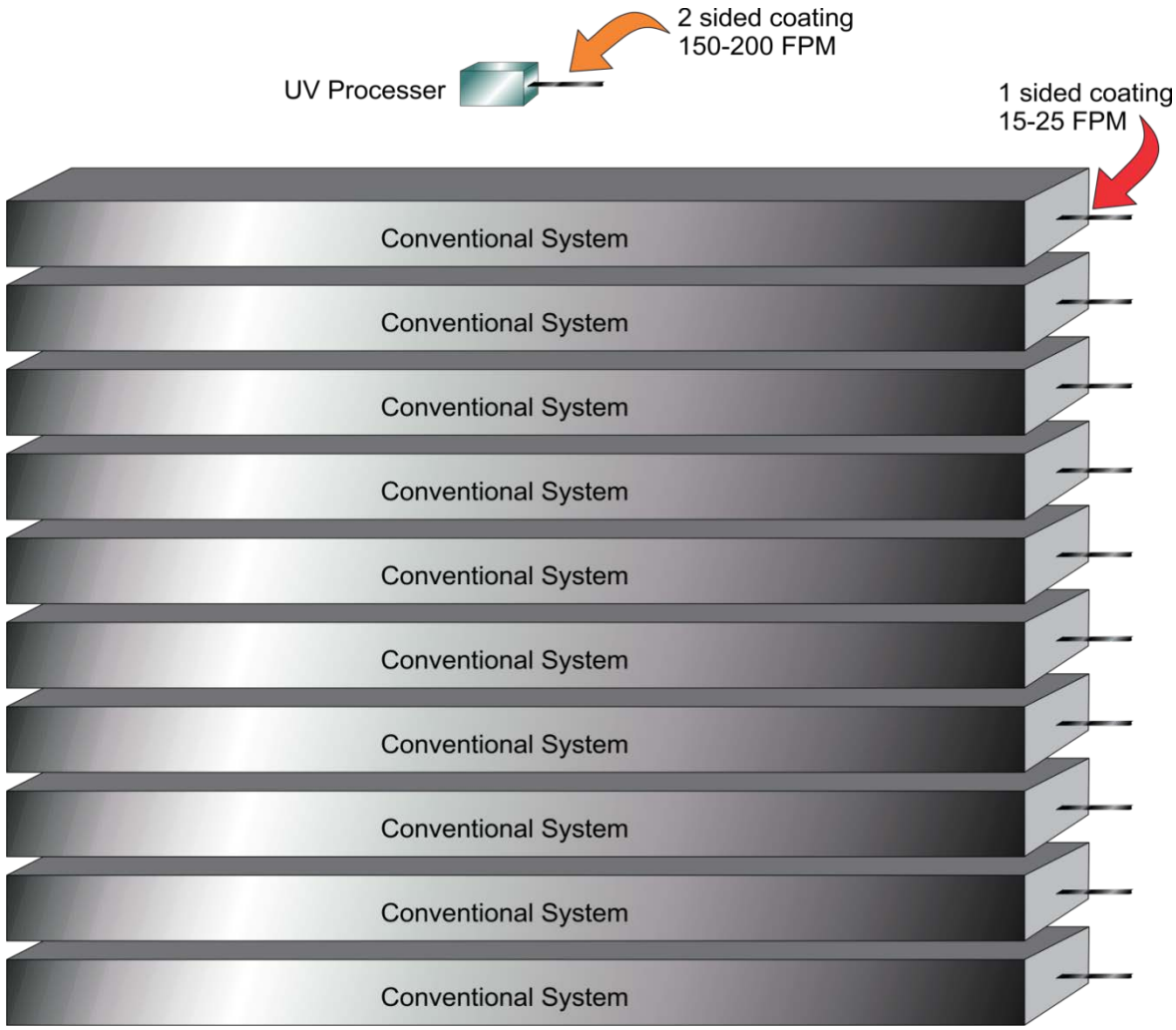
OAK RIDGE National Laboratory

ACTEGA Coatings & Sealants

Miltec UV Electrode Coating and Curing vs Conventional System

1 UV coating and curing system equivalent output of 10-20 conventional systems

- Dramatically reduced space, capital, and operating cost
- Minimized solvent recovery
- Estimate 60%+ reduced cost of electrode, including materials
- Multiple layered coatings tested



Major Milestones

Milestone	Date	Status
Deliver Baseline Cells to INL	Mar 2012	Complete
Finalize UV Binder for Interim Cell Cathodes	Aug 2012	Complete
Finalize UV Binder for Interim Cell Anodes	July 2013	On schedule
Deliver Interim Cells to INL	Sep 2013	On schedule
Go-No-Go Decision	Nov 2013	On schedule

Mix, Coat, Cure & Fabricate

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

Baseline Loading :Anode
92% Carbon/Graphite
8% Binder

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

Conductivity/Resistance: Cathode
Measured in Ohms:
0.1 = Achieved
0.2 = Target
0.35 = Acceptable

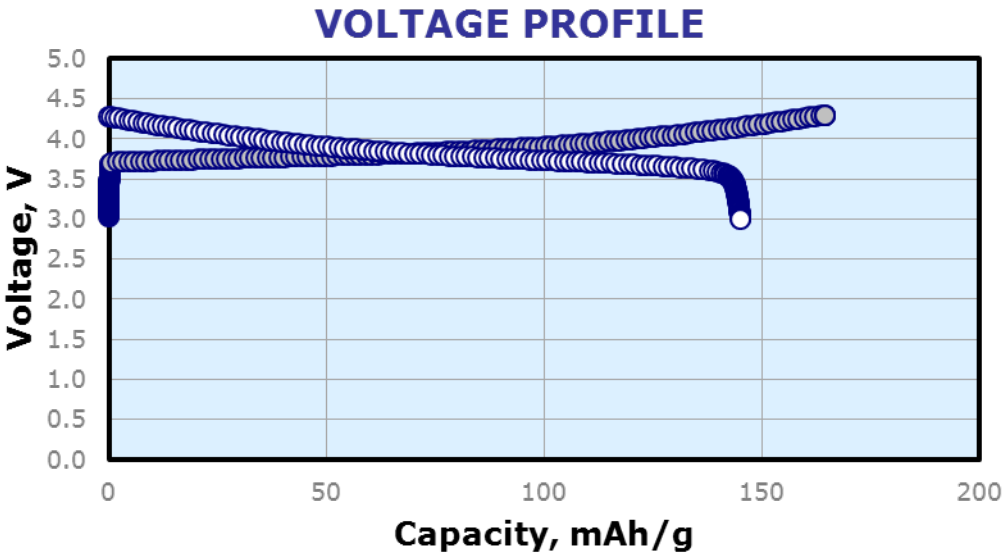
Electrochemical Testing
Recent Results

Typical Coating, Curing, and Testing Conditions

- 50-75 micron coating before curing and calendering, single layer
- 75-150 fpm, 2 lamps
- 87% NMC, 5% Carbon, 8% UV curable binder
- Nominal 2500-3500 lb/in^2 calendering
  - Nominal 40-55 micron thickness after calendering
  - Porosity calculated, nominal 20-35%
- ANL, coin cell, half and full
- Adhesion and conductivity passed before shipment to ANL

Reference cell

- **Cathode:**
- NCM/Carbon/PVDF: 87/5/8
- NCM: BASF 111
- Carbon: Timcal C-45
- 50 microns after calendering



		Ch. mAh/g	Dischar, mAh/g	Ah eff,%	
First cycle	C/10	164.56	145.11	88.179	
	C/3		141.0		
	1C		136.3		

Electrochemical Testing
Recent Results

- Sample 106, UV Cured NMC Cathode
  - 87% NMC, 5% Carbon, 8% UV curable binder
  - Nominal 3,000 lbs/in^2 calendering
    - Nominal 40-55 micron thickness after calendering
    - Porosity calculated, nominal 25-35 %
  - 2,000+ cycles full cell
- Sample 154, UV Cured $LiFePO_4$ Cathode
  - Good rate and capacity performance
  - Thin coating
- Sample 164, UV Cured MCMB Graphite + Si Anode

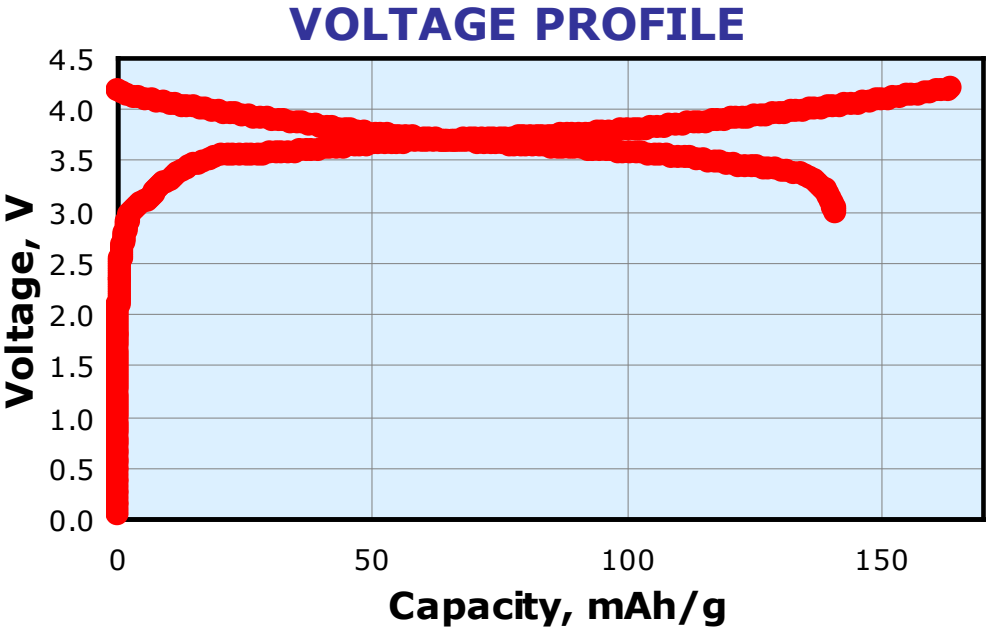
NMC (111) full cell with UV Binder: Miltec 106

Full-cell cycling conditions

-Anode: ConocoPhillips A12 graphite as anode, anode/cathode=~1.1

-Electrolyte: Gen 2, 1.2 M LiPF6 in EC:EMC (3:7 by wt.)

-Separator: Celgard 2325 -Potential window: 3.0-4.2 V



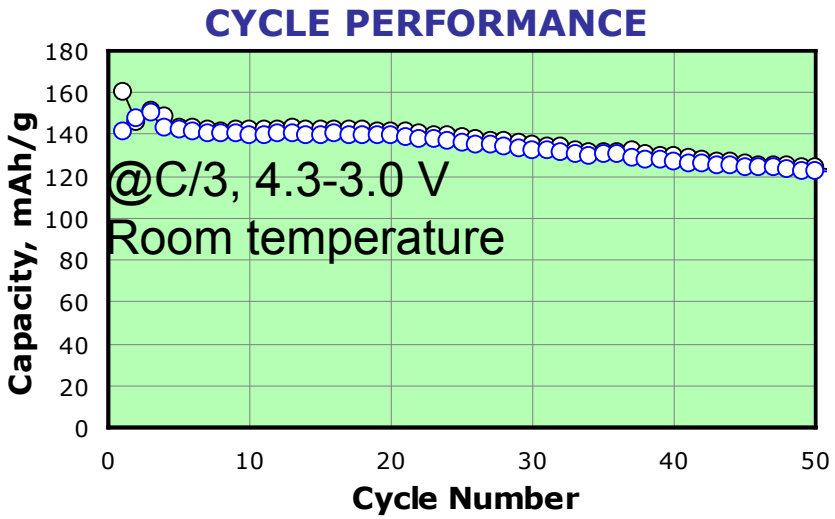
Formation cycle @C/10 between 3.0-4.2 V

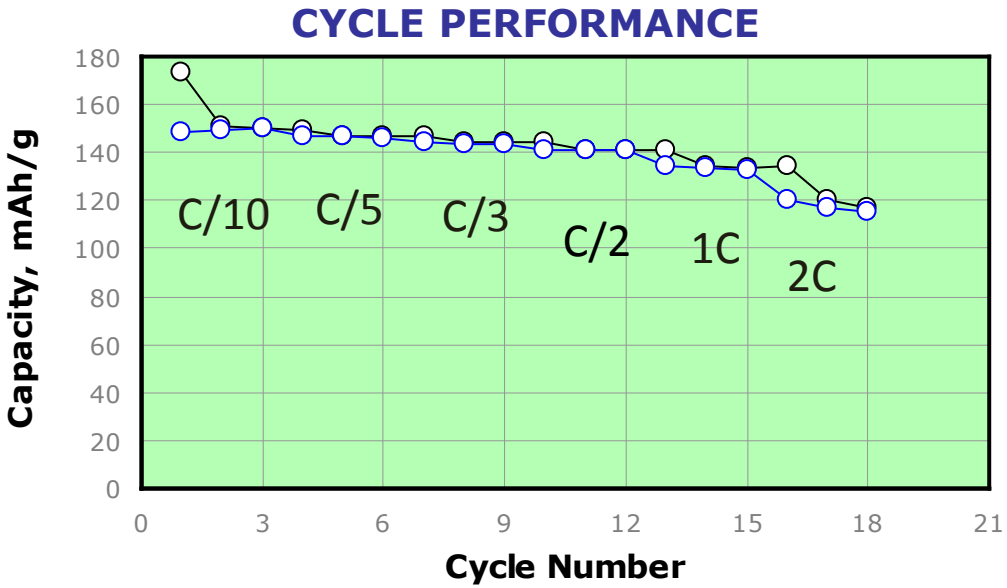
First charge capacity: 163.81 mAh/g

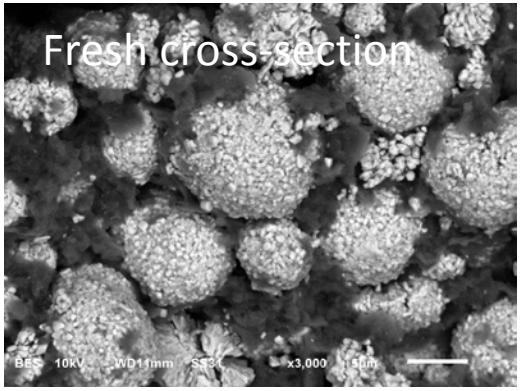
First discharge capacity: 140.93 mAh/g

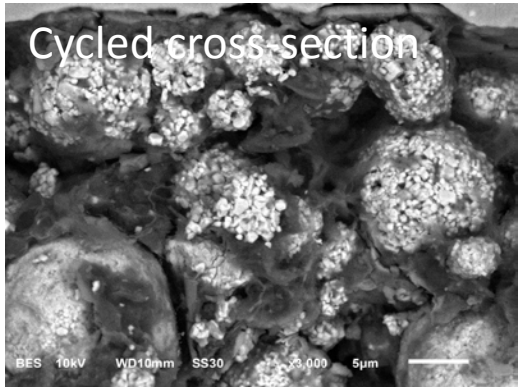
First cycle Efficiency: 86.03%

NMC (111) half cell with UV Binder: Miltec 106



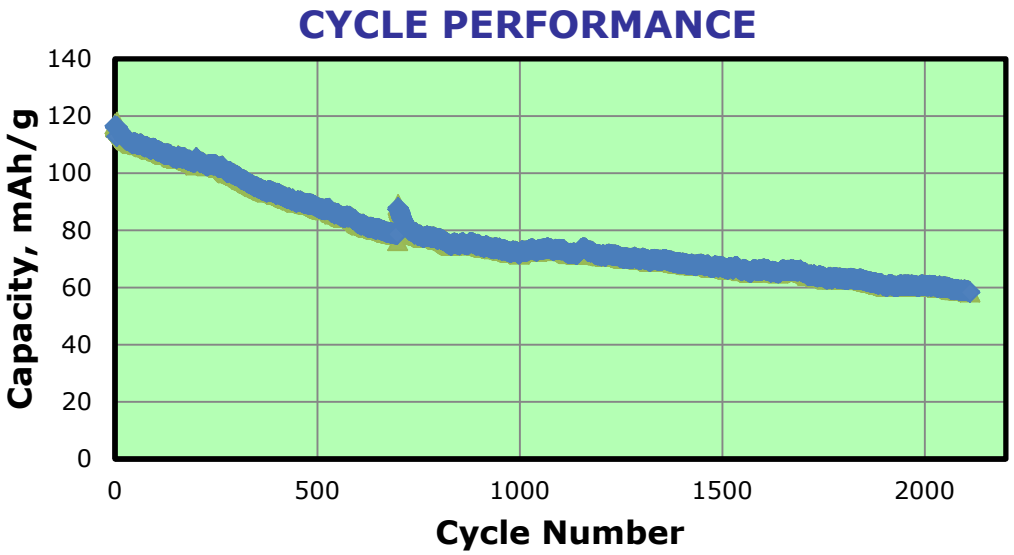


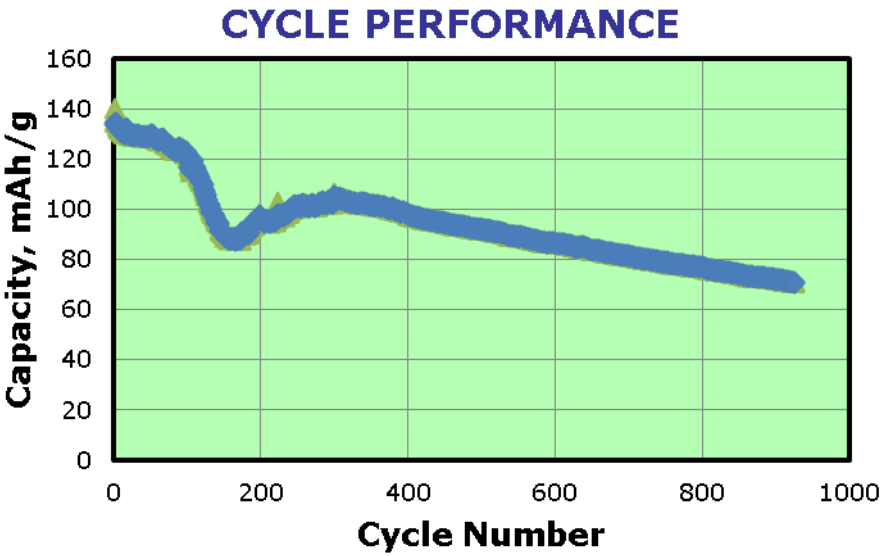




1. **Good cycle stability**
2. **Good rate performance**
3. **Very stable electrodes**

NMC (111) full cell with UV Binder: Miltec 106



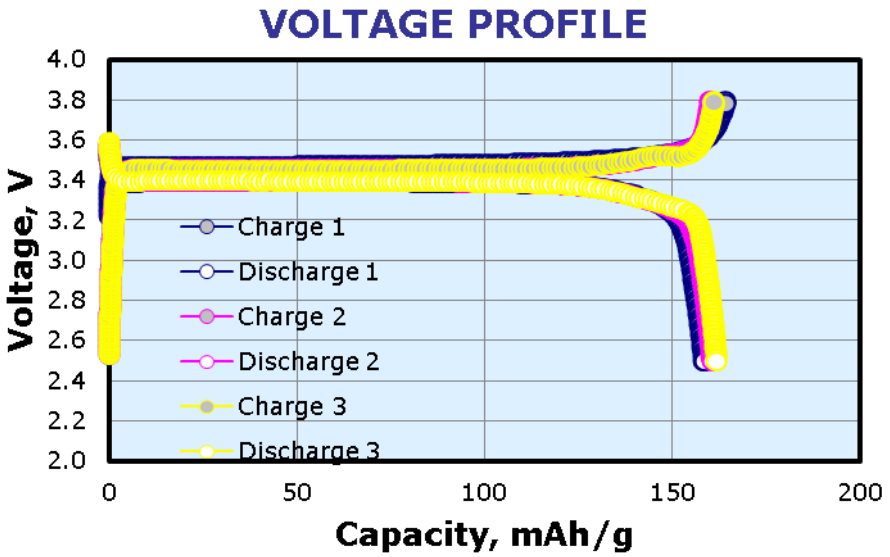


Full cell @1C, room temperature
Long cycles performance

Full cell @1/3C, room temperature
Long cycles performance

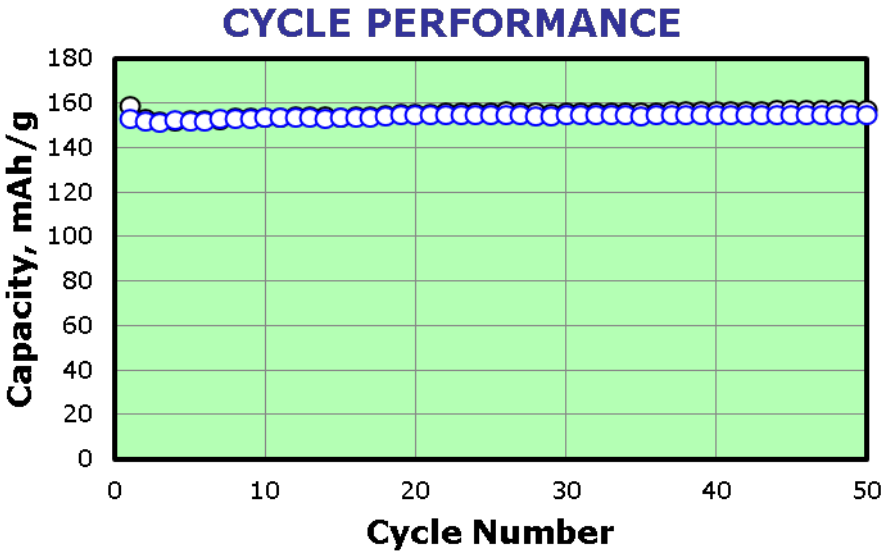
Performance of $LiFePO_4$ half cell: Miltec 154

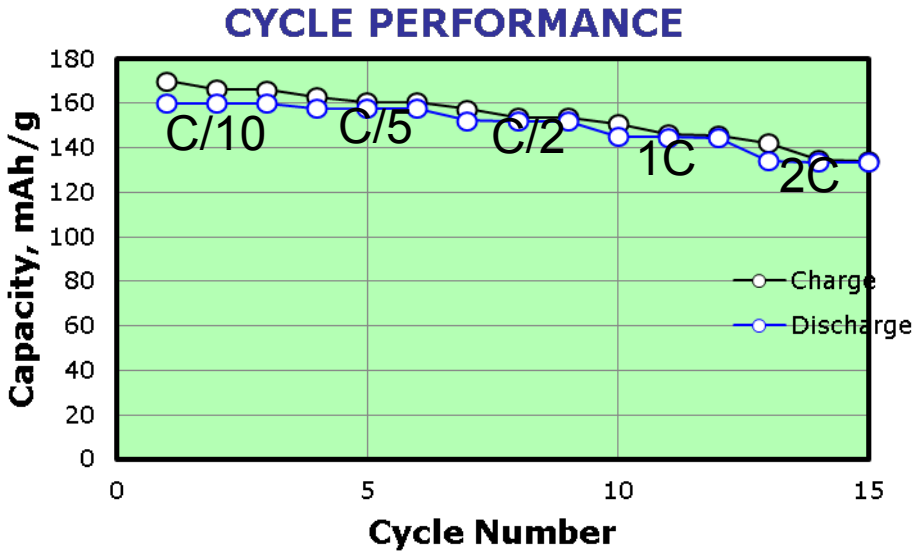
Electrode information:
77.09% active material,
Half-cell cycling conditions
-Anode: Li metal -Electrolyte: Gen 2, 1.2 M LiPF6 in EC:EMC (3:7 by wt.)
-Separator: Celgard 2325 -Potential window: 2.5-3.8 V



Formation cycle @ C/10 between 2.5-3.8 V
First charge capacity: 164.2 mAh/g
First discharge capacity: 158.5 mAh/g
First cycle Efficiency: 96.5%

$LiFePO_4$ half cell with UV Binder: Miltec 154

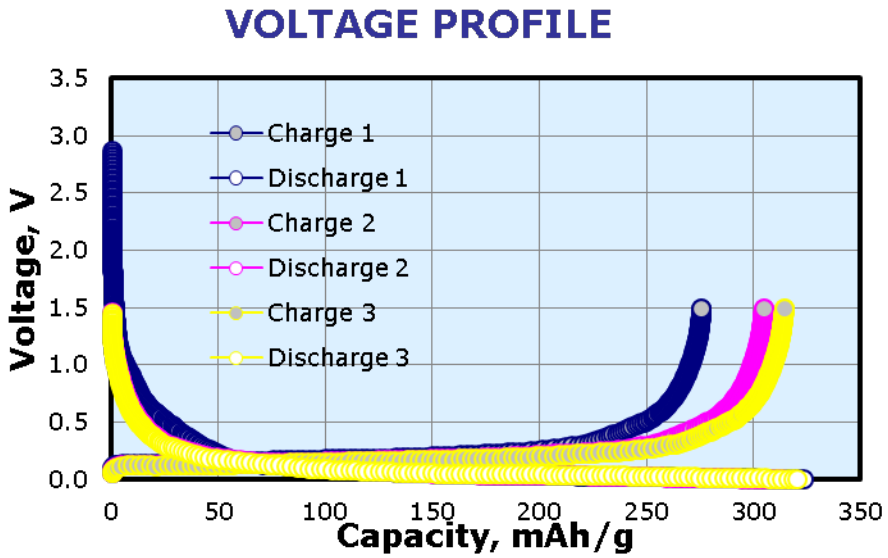


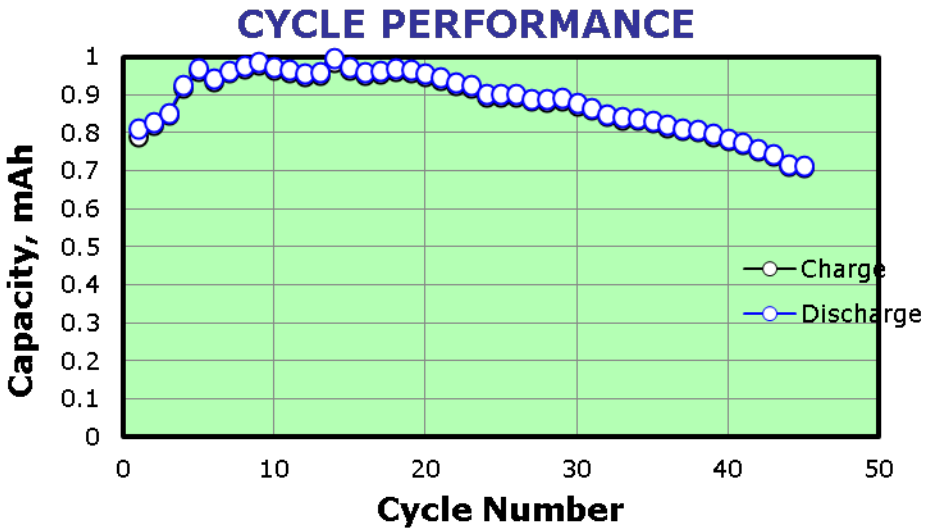


1. **Extreme good cycle stability: no capacity loss at all.**
2. **Good rate performance**

MCMB anode with UV binder in half cell: Miltec 164

Electrode information:
72% active material (62%MCMB and 10% Si)
Half-cell cycling conditions
-Anode: Li metal -Electrolyte: Gen 2, 1.2 M LiPF6 in EC:EMC (3:7 by wt.)
-Separator: Celgard 2325 -Potential window: 0.005-1.5 V





Formation cycle @C/10 between 1.5-0.005 V
First discharge capacity: 323.58 mAh/g
First charge capacity: 275.56 mAh/g
First cycle Efficiency: 85.15%

Cycle stability @C/3

PolySiloxane Alternate UV Binder Tests

- Miltec-ANL joint effort on Polysiloxane based UV curable binder
  - UV curable binder formulated
  - UV curing of binder thin coating and NMC laminate

PolySiloxane Alternate UV Binder Tests

UV-curing of PSA thin film and NMC laminate

Curing conditions

MPI (D bulb); 25 fpm; 1 run curing

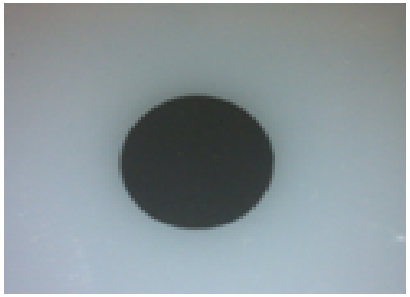
PSA Thin film

-PSA: 95 wt%
-Miltec photoinitiator: 5 wt%

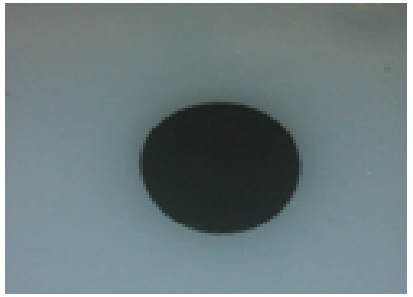
- Good adhesion to Cu and Al current collectors (example shown as Cu)
- No delamination in tape peeling test

NMC electrode with PSA binder

-NMC (85 wt%)
-C45 carbon (5 wt%)
-PSA (8.5 wt%)
-Miltec photoinitiator (1.5 wt%)



Pristine cured electrode



After calendaring

- Good adhesion to current collector
- No delamination upon calendaring

PolySiloxane Alternate UV Binder Tests

Cell data of UV-cured NMC laminate with PSA binder

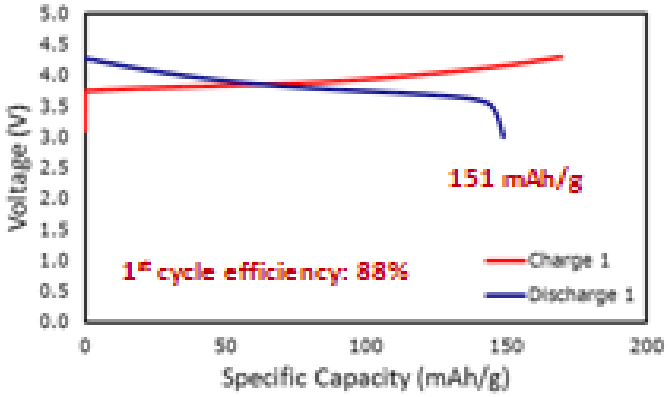
Electrode properties

-Active material: 5.9 mg/cm²
-Thickness: 40 µm
-Porosity: 45% (calendared)

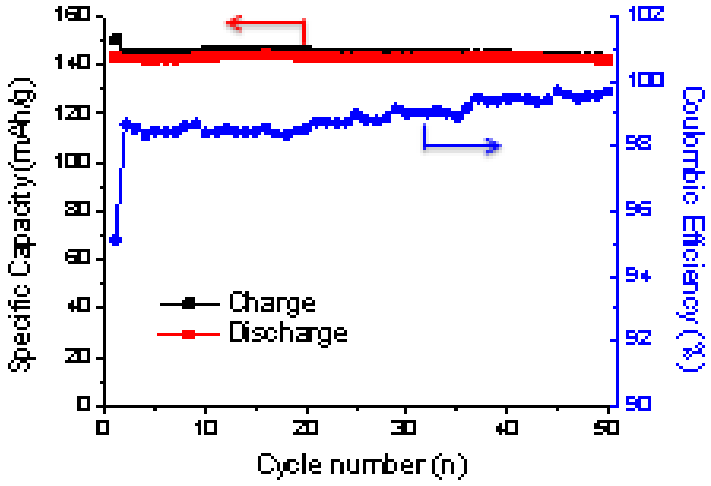
Half-cell cycling conditions

-Anode: Li metal
-Electrolyte: Gen 2, 1.2 M LiPF6 in EC:EMC (3:7 by wt.)
-Separator: Celgard 2325
-Potential window: 3.0-4.3 V

1st formation cycle @C/10



Long-term cycling @C/3



Summary

- Foundations in place:
  - ✓ Personnel, equipment, materials, and new facility
- Since October 2011, Miltec UV has successfully qualified candidate UV Curable constituents including: Oligomers, Monomers, Photoinitiators, and Dispersants
- NMC based cathode tests confirmed:
  - ✓ Layered coating
  - ✓ Success at lower porosity
  - ✓ Confident of making NMC cathode with performance at least equal, most likely better than baseline
- $LiFePO_4$ based cathode work initiated
  - ✓ Latest results very encouraging
- Anode work initiated
  - ✓ Evaluating MCMB and silicon anode materials
- Initial Polysiloxane work encouraging
- Baseline cells delivered, on schedule for Interim cells

Miltec UV Dual Coater Prototype