PROCESS DEVELOPMENT AND SCALE UP OF ADVANCED ACTIVE BATTERY MATERIALS

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Project ID: ES167

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Overview

Timeline
- Project start date: Oct. 2010
- Project end date: Sept. 2017
- Percent complete: on going

Budget
- Total project funding:
  - $1.2M in FY15
  - $1.2M in FY16
  - $500K for Flame Spray Pyrolysis
  - $300K for 10 L Taylor Vortex Reactor

Barriers
- Cost: Reduce manufacturing costs with advanced processing methods
- Performance: Synthesis route selection and process optimization for maximum performance

Partners
- Active material process R&D:
  - Argonne’s Applied R&D Group
    - Material synthesis and scale-up
  - University of Illinois at Chicago
    - 3D elemental mapping
  - Technische Universität Braunschweig
    - Particle stress study
  - A123
    - Cathode precursor micronization
  - PPG Industries
    - Cathode material customization
  - SiNode Systems
    - Si-graphene composite synthesis
  - Cabot
    - Flame spray pyrolysis
  - Swiss Federal Institute of Technology
    - Flame spray pyrolysis
Objectives - Relevance

The objective of this program is to provide a systematic engineering research approach to:

- Develop *cost-effective* processes for the scale-up of advanced battery materials.
- Provide *sufficient quantities* of these materials produced under rigorous quality control specifications for industrial evaluation of further research.
- Evaluate *material purity profiles* and their influence on battery performance.
- Evaluate *emerging manufacturing technologies* for the production of these materials.

The relevance of this program to the DOE Vehicle Technologies Program is:

- The program is a key missing link between discovery of advanced battery materials, market evaluation of these materials and high-volume manufacturing.
  - Reducing the risk associated with the commercialization of new battery materials.
- This program provides large quantities of materials with consistent quality.
  - For industrial validation in large format prototype cells.
  - For further research on advanced materials.
## Milestones

<table>
<thead>
<tr>
<th>Year</th>
<th>Category</th>
<th>Description</th>
<th>Status</th>
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</thead>
<tbody>
<tr>
<td><strong>FY15</strong></td>
<td>R&amp;D</td>
<td>Layered-layered material – Kilogram production</td>
<td>Completed 15-Jun</td>
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<tr>
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<td>R&amp;D</td>
<td>Layered-layered-spinel material – Evaluate the effect of spinel content</td>
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<td>R&amp;D</td>
<td>Gradient material – Identify target composition</td>
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<td>– Complete preliminary assessment</td>
<td>Completed 15-Sep</td>
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<td>– Complete precursor optimization (NCM811 as Core composition)</td>
<td>Completed 16-Feb</td>
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<tr>
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<td>– Preliminary synthesis of Core-Gradient material</td>
<td>Completed 16-Mar</td>
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<td>– Kilogram production of Core-Gradient material</td>
<td>Ongoing 16-Q2</td>
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<tr>
<td></td>
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<td>– Optimize the synthesis of Surface composition</td>
<td>Target 16-Q3</td>
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<tr>
<td></td>
<td></td>
<td>– Optimize the thickness of Gradient layer</td>
<td>Target 16-Q4</td>
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<tr>
<td></td>
<td></td>
<td>– Kilogram production of optimized Core-Gradient material</td>
<td>Target 17-Q1</td>
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<tr>
<td></td>
<td></td>
<td>– Core-Shell material synthesis for comparison</td>
<td>Target 17-Q1</td>
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<td><strong>FY16</strong></td>
<td>Ind.</td>
<td>Spray drying – Micronization of nano-size LFP material</td>
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<td>– Reactive spray drying for Si-graphene composite</td>
<td>Ongoing 16-Q2</td>
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<td></td>
<td>R&amp;D</td>
<td>FSP* set-up – Process basic design and installation</td>
<td>Ongoing 16-Q2</td>
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<td>– Identify target composition and produce preliminary material</td>
<td>Target 16-Q4</td>
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<td>Both</td>
<td>TVR** scale-up – 1 L TVR NCM material synthesis for electrodeposition R&amp;D</td>
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<td>– 10 &amp; 40 L TVR installation</td>
<td>Ongoing 16-Q3</td>
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<td>– Begin scale-up research using 1, 10 and 40 L TVRs</td>
<td>Target 16-Q4</td>
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</table>

* Flame Spray Pyrolysis  ** Taylor Vortex Reactor
Approach - Strategy

**Material Synthesis with Process R&D**

- Define target active material
  - Evaluate bench-scale samples
- Select synthesis process and synthesis route
  - Batch, CSTR, TVR, FSP
  - Carbonate and hydroxide
- Produce intermediate material
  - 10 gram scale
  - Preliminary synthesis
  - Material evaluation
- Synthesis condition optimization by DoE
- Production and distribution
  - 1 ~ 10 kilogram scale
  - Assist other DOE programs

**Layered-layered Layered-layered spinel**
**Gradient materials**
**NCM523/622/811**

- **Solid state**
  - High E. Ball Mill
    - 200 g/batch
- **Co-precipitation**
  - Batch
    - 2 kg/batch
  - CSTR
    - 200 g/hr
  - TVR
    - 10 g/hr
  - FSP
    - 10 g/hr
- **Next generation**
  - Future
    - Hydro-Thermal
      - 10 g/batch

**Evaluation of Emerging Manufacturing Technologies**

- Taylor Vortex Reactor (evaluate process scalability)
- Flame spray pyrolysis (establishing capability)
- Hydrothermal synthesis (future)
Technical Accomplishments

Gradient Material

Gradient material will have the best of Core and Surface compositions:

- Ni-rich material: high capacity, low stability
- Gradient layer: prevents the crack and segregation between Core and Shell
- Mn-rich material: low capacity, high stability

Core composition

Gradient layer

Surface composition

Core-Shell
- Low particle strength

Core-Gradient (FY16)
- Low capacity due to smaller Ni portion

Full Concentration-Gradient
- Low capacity due to smaller Ni portion

1. To increase Ni portion for higher capacity
2. To optimize Core composition without internal porosity
3. To prepare small Core particle with better particle strength

Synthesis optimization of Core NCM811 by DoE
Technical Accomplishments

Gradient Material

- Precursor optimization for Core NCM811
  - 5μm Core NCM811: Not commercially available
  - Dense particle
  - Spherical morphology
  - Narrow particle size distribution
  - DoE: Multilevel Factorial Design
  - 12-time 20 hr co-precipitations using 20 L Batch reactor

- 3D mesh plot for 12 precursors

  - pH 11.5 shows 5 μm dense spherical particles.
  - pH = 11.5 & NH4/TM = 2 condition was selected to prepare Core NCM811 at MERF.
Technical Accomplishments

Gradient Material

- Comparison between optimized MERF NCM811 and commercial NCM811

- Quality of 5 μm Core NCM811 was verified.
- Both NCM811s show ~210 mAh/g.
- 50g MERF NCM811 was sent to Technische Universität Braunschweig / CAMP for particle stress study.

Synthesis of Core-Gradient material was started.
Technical Accomplishments

Gradient Material

- Synthesis of Core-Gradient materials
  - First, Core TM solution feeding to batch reactor
  - Then, Surface TM solution feeding to Core TM solution tank
  - Core TM solution changes to Surface TM solution gradually

- 2 Core-Gradient materials were prepared to evaluate the effect of Gradient layer thickness

- Thickness control of Gradient layer from normal NCM811 to FCG

Elemental mappings

Core-Gradient 1 (thin layer) Core-Gradient 2 (thick layer)

Ni Co Mn
## Technical Accomplishments

### Gradient Material

#### Comparison of prepared materials

<table>
<thead>
<tr>
<th>Material</th>
<th>NCM622</th>
<th>NCM811 (no layer)</th>
<th>Core-Gradient 1 (thin layer)</th>
<th>Core-Gradient 2 (thick layer)</th>
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</thead>
<tbody>
<tr>
<td>Scale / status</td>
<td>Commercial product</td>
<td>MERF pre-pilot Optimized</td>
<td>MERF pre-pilot Preliminary</td>
<td>MERF pre-pilot Preliminary</td>
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<td>SEM 3,000x</td>
<td><img src="image1.png" alt="SEM image" /></td>
<td><img src="image2.png" alt="SEM image" /></td>
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<td><img src="image3.png" alt="SEM image" /></td>
<td><img src="image4.png" alt="SEM image" /></td>
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<tr>
<td>Composition</td>
<td>NCM622</td>
<td>NCM811</td>
<td>~ NCM622</td>
<td>~ NCM523</td>
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<td>ICP-MS analysis</td>
<td>Li$<em>{1.04}$Ni$</em>{0.60}$Co$<em>{0.20}$Mn$</em>{0.20}$O$_y$</td>
<td>Li$<em>{1.06}$Ni$</em>{0.77}$Co$<em>{0.12}$Mn$</em>{0.12}$O$_y$</td>
<td>Li$<em>{1.07}$Ni$</em>{0.57}$Co$<em>{0.17}$Mn$</em>{0.26}$O$_y$</td>
<td>Li$<em>{1.1}$Ni$</em>{0.46}$Co$<em>{0.19}$Mn$</em>{0.35}$O$_y$</td>
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<td>Particle size D$_{50}$ [μm]</td>
<td><strong>11.3</strong></td>
<td><strong>4.7</strong></td>
<td><strong>5.1</strong></td>
<td><strong>7.0</strong></td>
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<tr>
<td>Tap density [g/cc]</td>
<td>2.3</td>
<td>1.7</td>
<td>1.8</td>
<td>2.5</td>
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<tr>
<td>BET [m$^2$/g]</td>
<td>0.34</td>
<td>0.77</td>
<td>1.56</td>
<td>Ongoing</td>
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<tr>
<td>* FCE [%]</td>
<td>90.5</td>
<td>92.1</td>
<td>93.1</td>
<td>93.2</td>
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<tr>
<td>* Initial discharge capacity [mAh/g]</td>
<td><strong>188</strong></td>
<td><strong>207</strong></td>
<td><strong>185</strong></td>
<td><strong>178</strong></td>
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</table>

- Core-Gradient materials have smaller primary particles and higher surface area than commercial NCM622.
- Core-Gradient 1 shows similar overall composition and discharge capacity compared to commercial NCM622.
Technical Accomplishments

Gradient Material

- Comparison of electrochemical performance for 3 materials

- CG1 shows lower capacity than commercial NCM622 at 1C. Gradient layer need to be optimized for better conductivity.
- CG1 shows superior capacity retention at high C-rate.
- CG2 (thick layer) needs further improvement.
- Core-Gradient structure has the best of Core (high capacity) and Surface (high stability) compositions.

For further improvement:

1. Optimize the synthesis of Surface composition
2. Optimize the thickness of Gradient layer to determine trade-off between Core-Gradient and Full Concentration-Gradient
Technical Accomplishments

LL 1kg Scale-up and LLS Synthesis

- Layered-layered material, 1 kg
  - Total 61 g provided to CAMP and R&D group.
  - 960 g available for the HE-HV program.

- Collaboration with M. Thackeray’s group
  - Stabilizing spinel component incorporated into ‘layered-layered’ structure.
  - Layered-layered-spinel’ system shows improved:
    - Capacity
    - Rate performance
    - First-cycle efficiency

- Synthesized LLS materials at MERF to optimize spinel content

<table>
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<tr>
<th>Spinel Content</th>
<th>Chemical Formula</th>
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<td>2% spinel</td>
<td>( \text{Li}<em>{1.236}\text{Ni}</em>{0.273}\text{Mn}<em>{0.536}\text{Co}</em>{0.191}\text{O}_y )</td>
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<td>5% spinel</td>
<td>( \text{Li}<em>{1.210}\text{Ni}</em>{0.273}\text{Mn}<em>{0.536}\text{Co}</em>{0.191}\text{O}_y )</td>
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<td>10% spinel</td>
<td>( \text{Li}<em>{1.176}\text{Ni}</em>{0.274}\text{Mn}<em>{0.536}\text{Co}</em>{0.190}\text{O}_y )</td>
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<td>15% spinel</td>
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Technical Accomplishments

Electrochemical Performance of LLS Materials

- 5~10% spinel content shows higher discharge capacity.
- Spinel content more than 10% lowers the capacity.
- Spinel content more than 5% shows improved stability.
- Higher spinel content shows better rate capability.

Effect of spinel content was clearly shown to collaborators for their further basic research.

ES049: Tailoring Spinel Electrodes for High Capacity, High Voltage Cells
Technical Accomplishments

**TVR: NCM Synthesis and Process Scale-up**

- **Taylor Vortex Reactor**
  - TVR provides a homogeneous intense micro-mixing zone and produces spherical precursors with narrow size distribution.
  - Simplified operation
  - Product uniformity
  - Shorter residence time

- **NCM materials from 1 L TVR**
  - Tap density = 2.1 g/cc
  - D50 = 5 µm
  - BET = 0.56 m²/g

  - Tap density = 2.0 g/cc
  - D50 = 8 µm
  - BET = 0.59 m²/g

  - Tap density = 2.4 g/cc
  - D50 = 14 µm
  - BET = 0.66 m²/g

- **Collaboration with equipment manufacturer to evaluate process scalability**

  - 1 L TVR in place
  - 10 L TVR ongoing
  - 40 L TVR ongoing
FSP: Nano-material synthesis

- Combustion of precursor aerosol solution w/o organic content
  - A system to produce nano-size active battery materials using a combustion flame spray unit
  - In collaboration with Miki Oljaca at Cabot Corp. and Sotiris Pratsinis at Swiss Federal Institute of Technology
  - Production rate target: 100 g/day

Technical Accomplishments

Spray Drying Application with Industry

- Spray drying of nano-size LFP slurry for micronization (A123)
  - Water evaporation: 3 kg/hr
  - Inlet air temperature: 250°C
  - Production rate: ~ 1 kg/hr
  - Particle size was increased from 500 nm to 5.6 μm.
  - 4.2 kg product was delivered to A123.

- Reactive spray drying for Si-graphene composite (SiNode)
  - Scale Si-graphene composite to Kg quantity.
  - Control particle size and distribution.

**ES240:** High Energy Anode Material Development for Li-Ion Batteries
Technical Accomplishments

**Active Material Synthesis with Tailored Properties**

- **MERF CRADA activity**
  - Active materials proof of concept for compatibility with PPG’s e-coat system
    - Prepare size-controlled NCM523
    - Lithium dissolution
    - Transition metal dissolution
    - Monitor pH for particle suspension stability

- Custom cathode synthesis for DOE funded Electrodeposition for Low Cost Water Based Electrode Manufacturing project

  **Electrophoretic deposition:**
  - Charged monodispersed particles migrate to oppositely charged Al-foil.
  - Requires small particle size (5µm and below) to obtain stable suspension in water-based baths.

**ES263:** Electrodeposition for Low-Cost, Water-Based Electrode Manufacturing
Responses to Previous Year Reviewers’ Comments

THIS PROJECT WAS NOT REVIEWED LAST YEAR
Collaborations

- **Active materials process R&D:**
  - Argonne National Lab (HE/HV program)
    - Material Synthesis
  - Argonne National Lab (Michael Thackeray)
    - Material synthesis
  - PPG Industries - CRADA (Stuart Hellring)
    - Custom cathode materials
  - Cabot Corporation – (Miki Oljaca)
    - Flame spray pyrolysis
  - Technische Universitétat Braunschweig (Wolfgang Haselrieder)
    - Particle stress evaluation
  - Laminar Co., Ltd – CRADA
    - Process scalability evaluation
  - Oak Ridge National Lab (Claus Daniel)
    - Custom material for R2R collaboration
  - Swiss Federal Institute of Technology (Sotiris Pratsinis)
    - Flame spray pyrolysis

- **Materials provided:**
  - University of Illinois at Chicago (Prof. Jordi Cabana)
  - NanoResearch Inc. (David Noye)
  - A123 Systems, Johnson Matthey, PPG
  - Argonne National Lab (various researchers)
  - Technische Universitétat Braunschweig

- **Electrochemical evaluation of scaled materials:**
  - Argonne’s Materials Screening Group (Wenquan Lu)
  - Argonne’s CAMP facility (Andrew Jansen, Bryant Polzin, Steve Trask)

Open to working with any group developing advanced active materials that will be beneficial for the ABR program.
Remaining Challenges and Barriers

- New battery materials are continually being discovered and developed.
- There is a strong demand from the research community for high quality experimental materials in quantities exceeding bench scale synthesis.
- Production of high performance active materials is extremely complex. A detailed understanding of how process variables effect performance is critical to fully understand material cost and capability.
- Emerging manufacturing technologies need to be evaluated to further reduce production costs and increase performance of battery materials.
- Development and scale-up of material engineering technology like surface coating is challenging but has great promise to improve the performance of battery materials.
Proposed Future Work

- **Continue work on Gradient material (Core NCM811 + Surface NCM424)**
  - Kilogram scale-up of preliminary Core-Gradient material
  - Optimize the synthesis of Surface composition
  - Optimize the thickness of Gradient layer from normal NCM811 to FCG
  - Kilogram scale-up of optimized Core-Gradient material
  - Core-Shell and Full Concentration-Gradient material synthesis for comparison
  - Pouch cell evaluation of prepared materials

- **Active material engineering**
  - Complete reactive spray drying synthesis of Si-graphene composite (SiNode)
  - Synthesize custom material for electrophoretic deposition (PPG)
  - Synthesize custom material for AMO R2R program

- **Evaluate emerging manufacturing technologies**
  - Investigate process scalability with Taylor Vortex Reactors
  - Design and construction of FSP system and material synthesis
Summary

- **Layered-layered material**
  - Material synthesis at kilogram quantity and delivery have been completed

- **Layered-layered spinel material**
  - 5~10% spinel content shows improved capacity, rate performance and stability

- **Gradient material (Core NCM811 + Surface NCM424)**
  - Core 5 μm NCM811 was optimized by DoE.
  - 2 Core-Gradient materials were prepared and analyzed by elemental mapping
  - Core-Gradient material shows valid capacity and improved stability

- **Material Engineering with Industry**
  - 4.2 kg production of 5.6 μm powder from nano-size LFP slurry by spray dryer
  - Synthesis of sized-controlled NCM523 for electrophoretic deposition by 1 L TVR

- Installation of 10 L & 40 L TVRs is ongoing
- Design and construction of FSP system is ongoing
Acknowledgements and Contributors

- Support from David Howell and Peter Faguy of the U.S. Department of Energy’s Office of Vehicle Technologies is gratefully acknowledged.

- Argonne National Laboratory
  - Michael Thackeray
  - Daniel Abraham
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  - Eva Allen
  - Jason Croy
  - Joseph Libera
  - Chris Claxton

- SiNode systems
  - Samir Mayekar

- UIC
  - Jordi Cabana

- Cabot
  - Miodrag Oljaca

- PPG Industries INC.
  - Stuart Hellring

- Swiss Federal Institute of Technology
  - Sotiris Pratsinis
Technical Backup Slides
## Material Delivery to R&D Group and Industry

<table>
<thead>
<tr>
<th>FY</th>
<th>Date</th>
<th>Material</th>
<th>Method</th>
<th>To</th>
<th>Purpose</th>
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<td>Mechanical testing</td>
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