Scale-up of Low-Cost Encapsulation Technologies for High Capacity and High Voltage Electrode Powders

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www.pneumaticoat.com

Project ID: ES239

Overview

Timeline (Phase I & II)

- June 10, 2013
- July 26, 2016
- Phase II is 35% Complete

Budget

- Total SBIR Project Funding
  - 100% by DOE
- FY13: $104K
- FY14: $218K
- FY15-YTD: $192K
- Remaining: $642K

Barriers

- Poor stability of high energy density materials
- Perceived cost of stabilizing coatings
- Overbuilding requirements to meet cycle life targets

Collaborators

- Nader Hagh, NEI Corp.
- Sung-Jin Cho, NC A&T Univ.
- Fabio Albano, XALT Energy LLC
- Corporate Partnerships

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Main Objectives

PHASE I

- ✓ Compare performance of stabilizing approaches:
  ALD vs. co-precipitation
- ✓ Demonstrate low-cost ALD using an innovative semi-continuous manufacturing approach
- ✓ Validate economic viability of semi-continuous ALD
- ✓ Develop new ALD coating chemistries for high capacity (HC) and high voltage (HV) materials

PHASE II

- ✓ Construct and validate a 100 kg/shift Particle ALD reactor
- Demonstrate and down-select new ALD coating chemistries for HV cathodes
- Implement QA/QC strategy for ALD manufacturing
- Produce over 400 kg of material for strategic partners
- Demonstrate < 5% capacity fade over 200 cycles in 2+ Ah pouch and 18650 cells

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Phase II Milestones

Milestone # and Description	Milestone Verification Process	Month
M1: Pilot Reactor Installation	Successful system construction and ability to control critical process variables such as pressure, temperature and valve firing in automatic operation	5 **Complete**
M2: 2Ah Baseline Cells	Successful fabrication of viable 2Ah cells and completion of 200 testing cycles at 1C rates	6 **Complete**
M3: Pilot Reactor Commissioning	Completed recipe builds for Al_2O_3 and TiO_2 ALD processes yielding > 80% material collection and < 5% variation in coating content per cycle	11
M4: Year 1 Report	Successful completion of Tasks 1-1 to 1-8	12
M5: Final Down-Selection	Successful identification of the coating processes for LNMO and graphite powders providing the greatest value proposition.	18
M6: Year 2 Report, Phase III	Successful completion of Tasks 2-1 to 2-6	24

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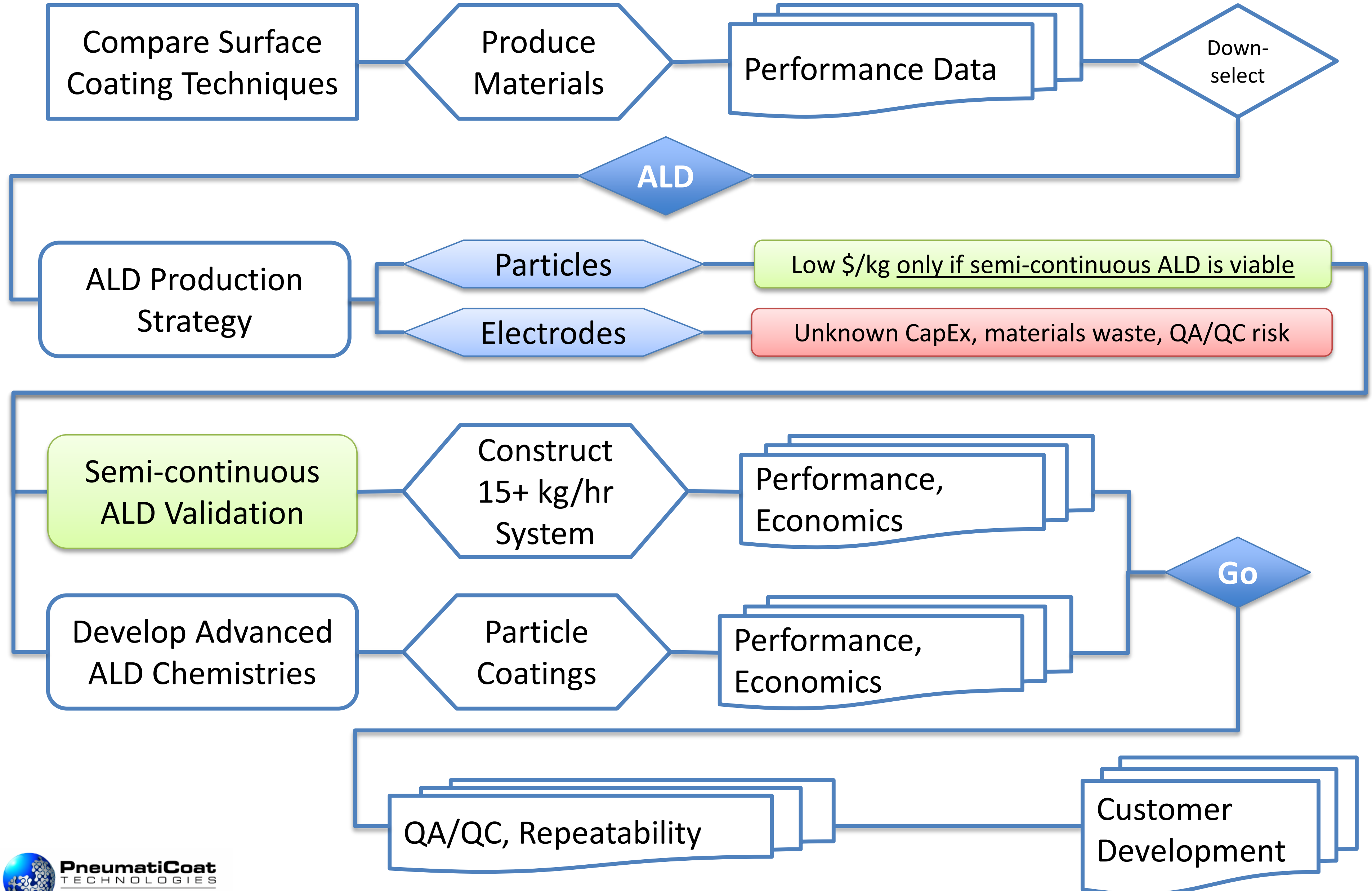
Technical and Strategic Approach



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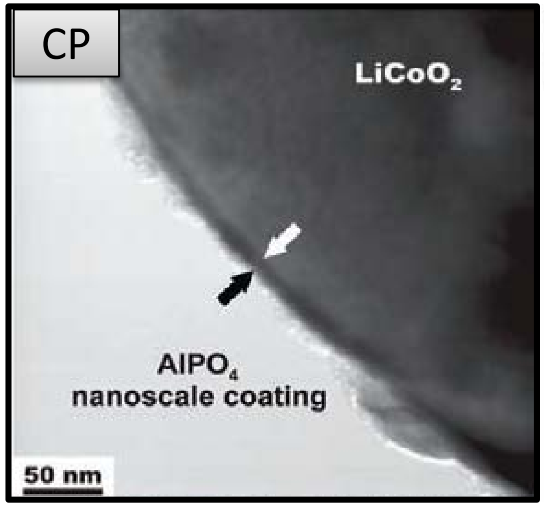
Technical and Strategic Approach

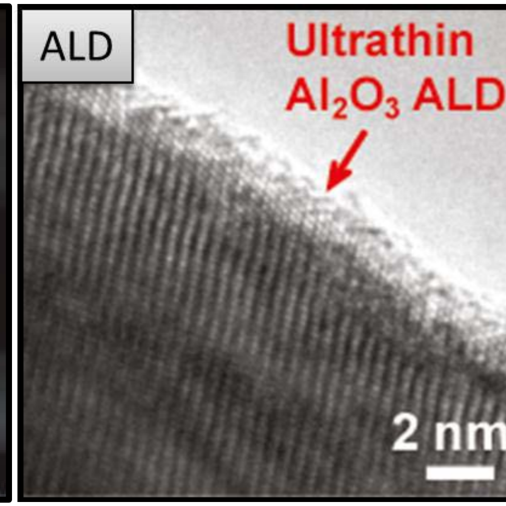
Balanced value proposition assessment between gas-phase and liquid-phase surface coating techniques:
Co-Precipitation (CP) versus ALD

Select Particles:
HV LNMO,
HC NMC (layered)

Select Coatings:
Al_2O_3, TiO_2



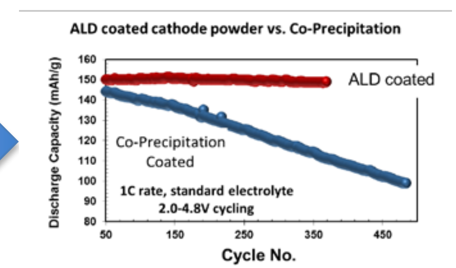
K.T. Lee, et al.



I.D. Scott, et al

CP → NEI Corporation

ALD → PneumatiCoat TECHNOLOGIES Commercializing "nano"



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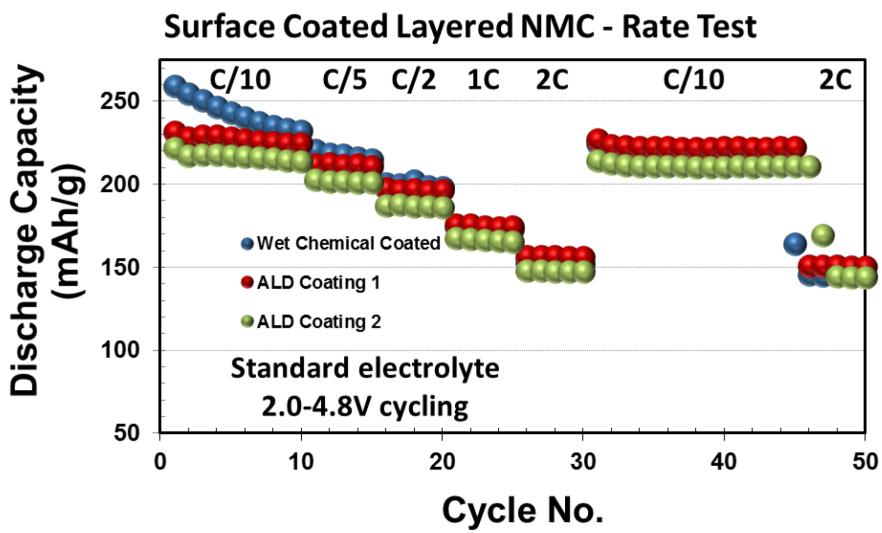
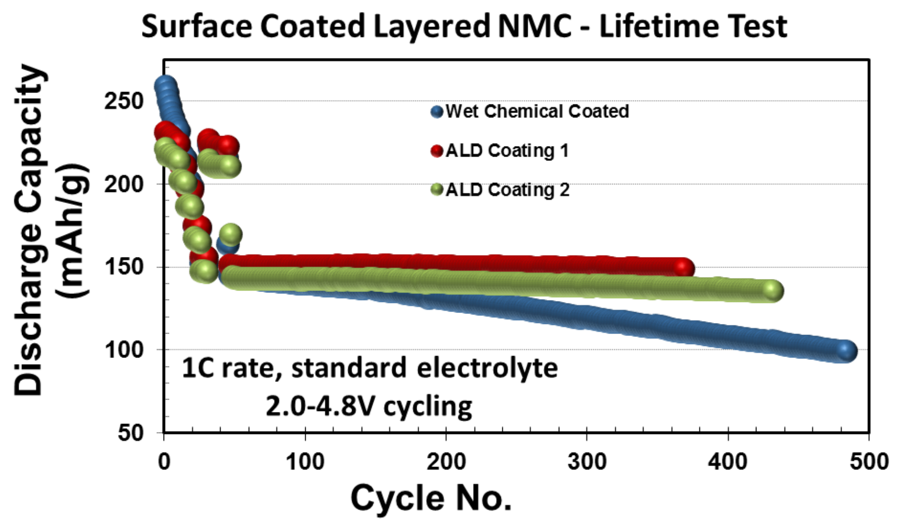
K.T. Lee, et al., "Roles of Surface Chemistry on Safety and Electrochemistry in Lithium Ion Batteries" Accounts Chem. Res., DOI:10.1021/ar200224h
I.D. Scott, et al., "Ultrathin Coatings on Nano-$LiCoO_2$ for Li-Ion Vehicular Applications" *Nano Letters,* 2011. 11(2): p. 414-418.

ALD vs. CP for HV and HC Cathode Powders



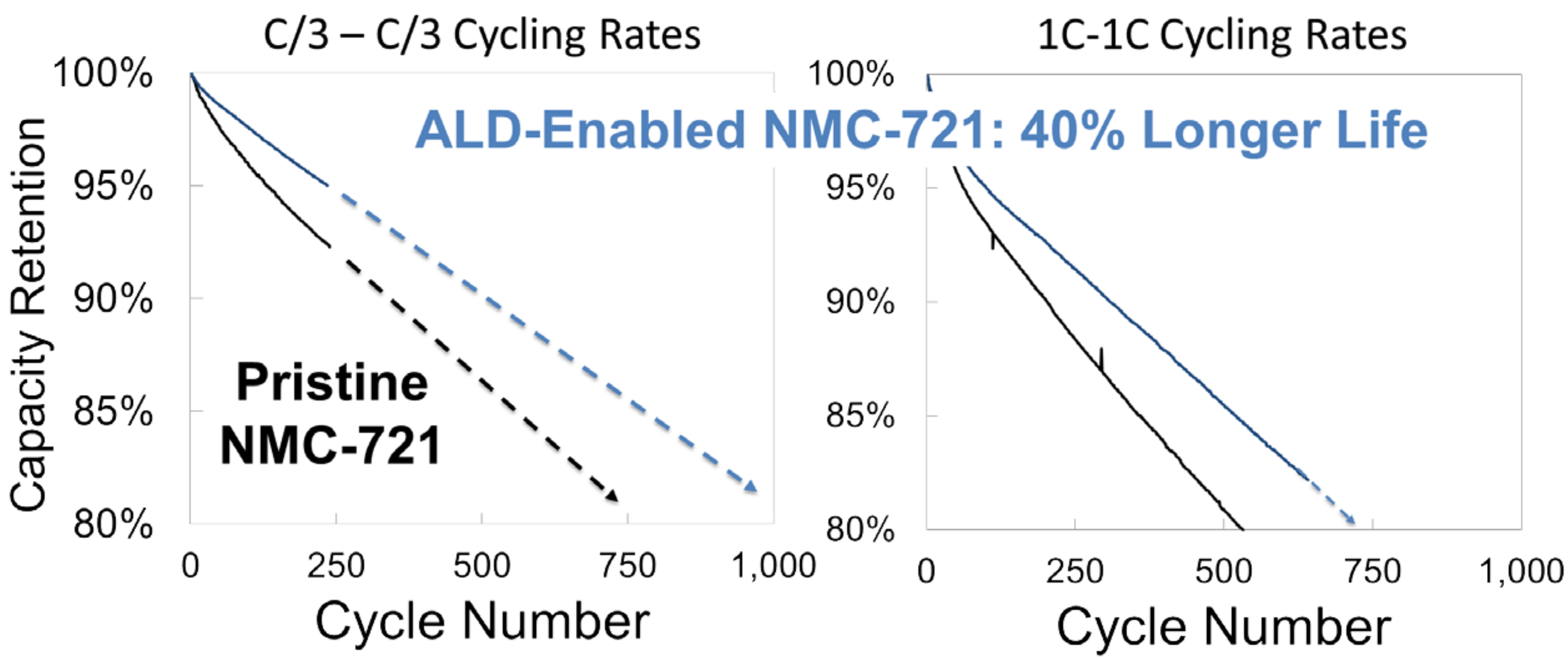


- Initially, CP coatings (2-4 wt%) maintained high performance, but were not robust and failed during long-term testing
- ALD coatings (0.1-0.5 wt%) demonstrated robust performance over long-duration cycling for both 5 V LNMO and layered LNMC

> - Decision Point: GO for ALD coatings based on more robust performance and lower materials consumption
> - Optimal ALD thickness and materials down-selected for each

Pouch Cell ALD Performance Validation

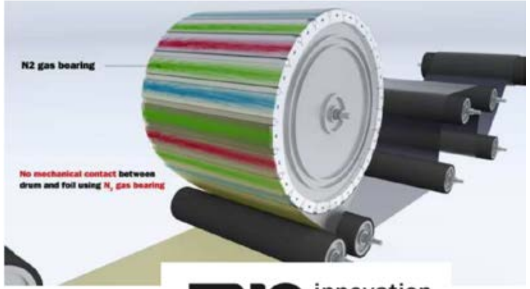
2 Ah pouch cells fabricated by XALT Energy using PCT's ALD-enabled NMC-721 and Pristine Graphite



Demonstrated performance: 190 Wh/kg, 430 Wh/L, 1000 cycles at C/3 rate

ALD Production Strategy: Particles vs. Electrodes

- ALD on electrodes requires Roll-to-Roll processing
  - Highest likelihood of success: TNO / Holst Centre efforts
  - *However precursor waste is 50-70% (Holst)*
  - Strong Benefits: functions as additional separator, maintains interparticle contacts
  - ALD/MLD-coated electrodes benefit from diffusion time, particularly for thick electrodes: “slow” R2R is anticipated, increasing $/m^2
  - Process risk: failure would scrap embedded cost of finished electrode
  - Still many unanswered questions for large global investment to date



- ALD on particles requires high-throughput processing
  - Batch Fluidized Bed Reactor ALD: expensive and not scalable
  - Semi-continuous ALD: low-cost, focus of this program
  - Continuous ALD: infeasible due to costs, entrainment losses

If Successful, Semi-continuous Particle ALD Coating Provides Lowest $/kWh

Empirical Fluidized Bed Reactor Scalability

Figure 1: Fluidization Profiles of 5 µm NCA Powder in 3” and 6” FBRs

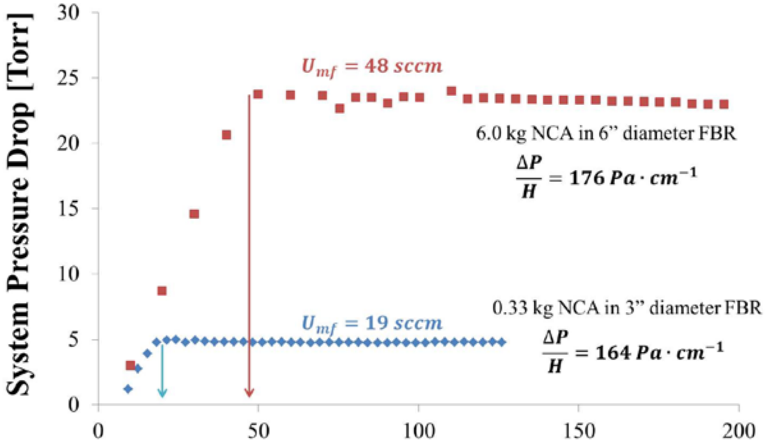


Figure 2: Vacuum Fluidization Process Window for Practical Bed Height to Diameter Ratios

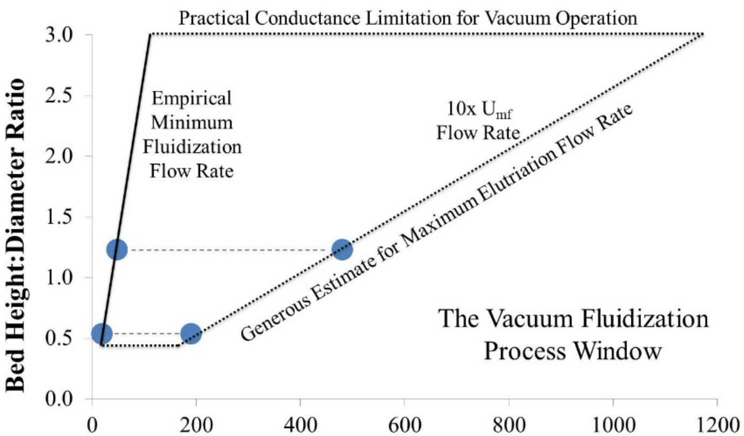


Figure 3: Total Bed Surface Area and Process Times for Practical FBR Diameters with various H/d Ratios

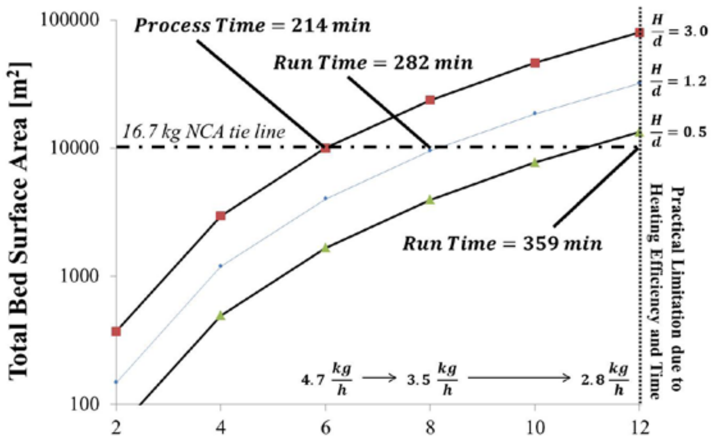
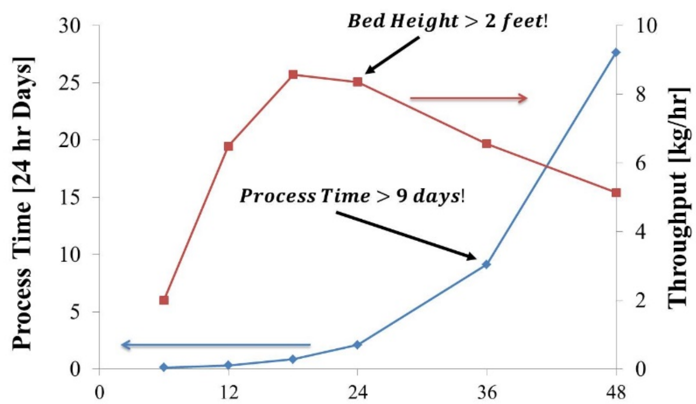


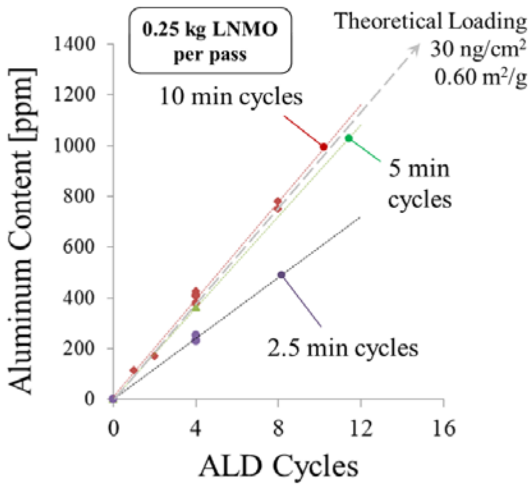
Figure 4: Calculated ALD Process Time, Volume and Throughput Limitations for Li-ion Battery Materials MANUFACTURED IN FLUIDIZED BED REACTORS

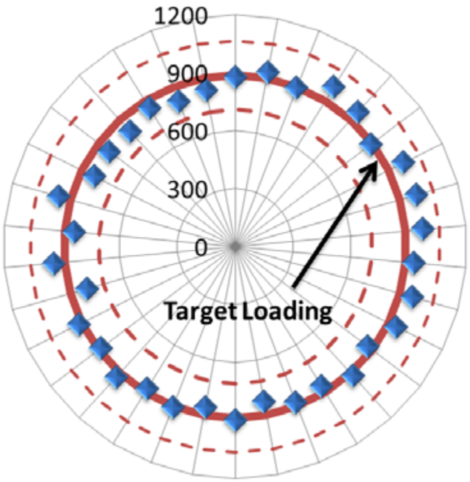


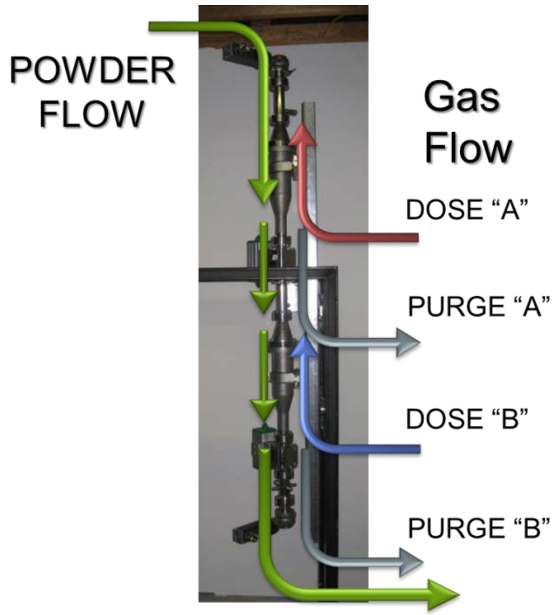
Fluidized Bed ALD Reactors are not Scalable/Suitable for Manufacturing

Semi-Continuous Particle ALD Validation

Phase I Prototype Scale (3 kg/hr)



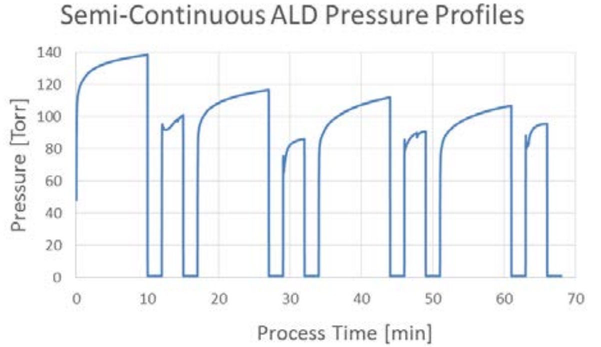


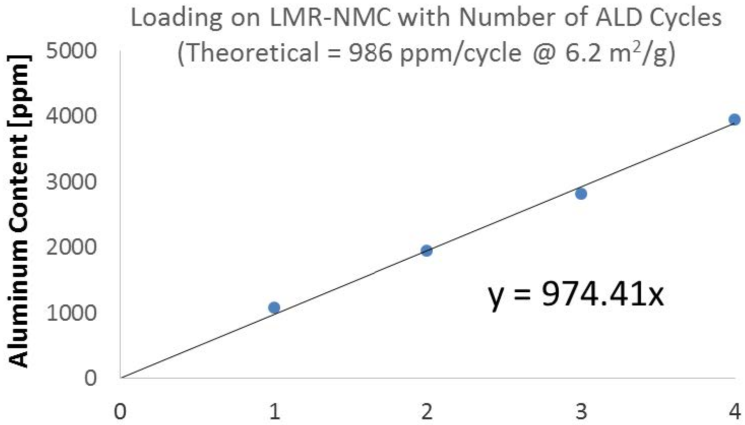


- Linear ALD growth with cycles achieved
- Adds residence time as variable
- Minimizes water exposure for sensitive electrode powders
- Low CapEx and utilities costs; 200x throughput over batch systems
- Fully automated using conventional powder handling equipment
- <u>Makes powder flow, not precursor flow, rate determining step</u>

Phase II: Validation of Pilot-Scale Manufacturing

- 200 kg/shift 4-ALD cycle system
- Partners providing 400 kg of powder
- Linear growth with cycles
- Fully-automated controls
- Similar CapEx to 1 kg/shift batch Fluidized Bed Reactor





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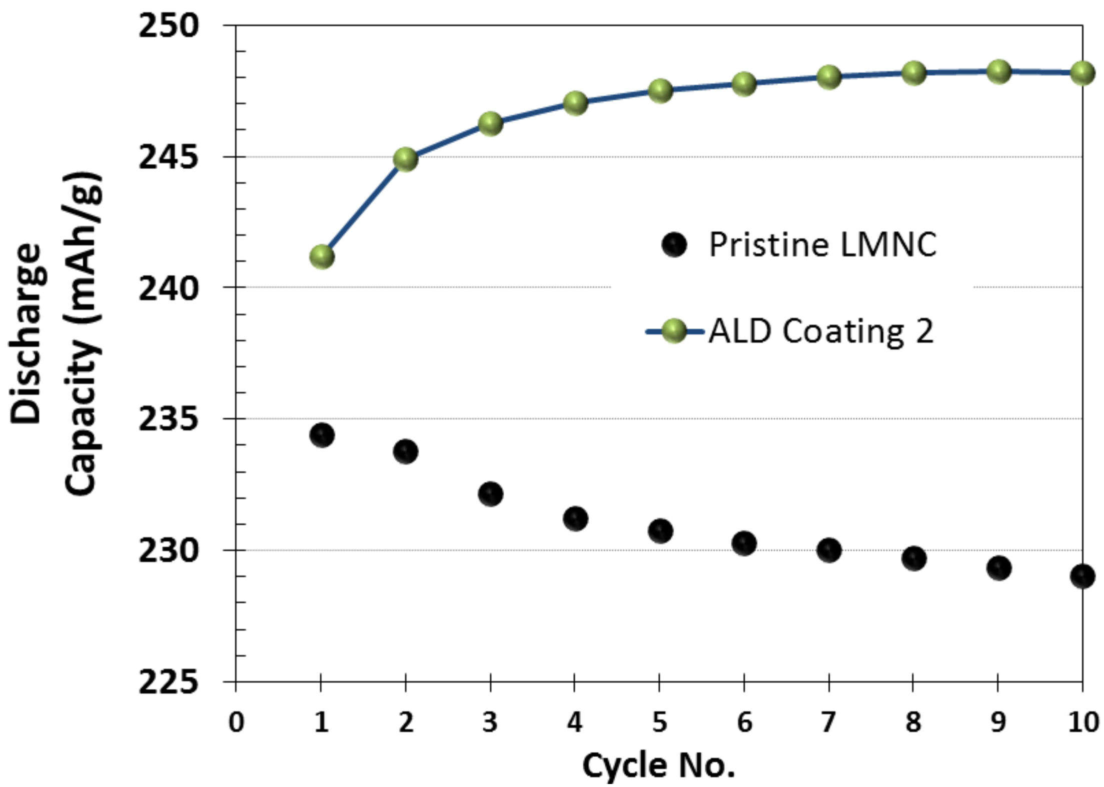
Phase II: Validation of Pilot-Scale Manufacturing

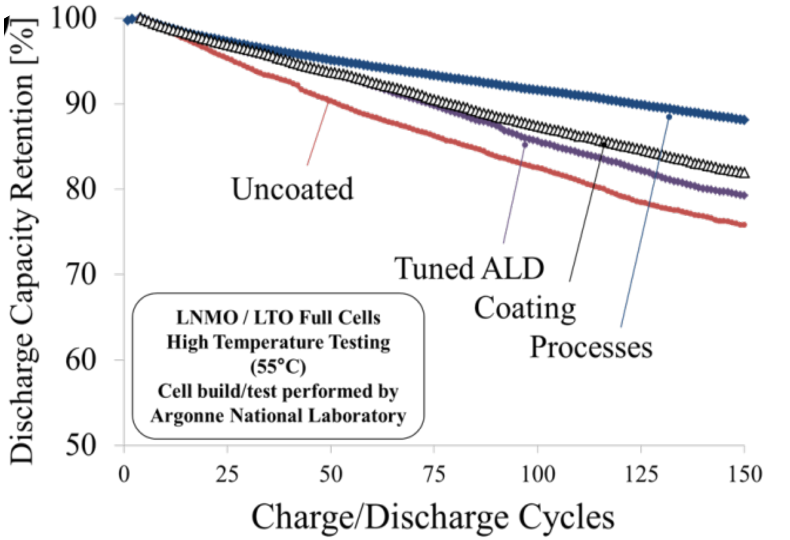
- Semi-continuous production of ALD-enabled LMR-NMC: First performance data (April 2015)

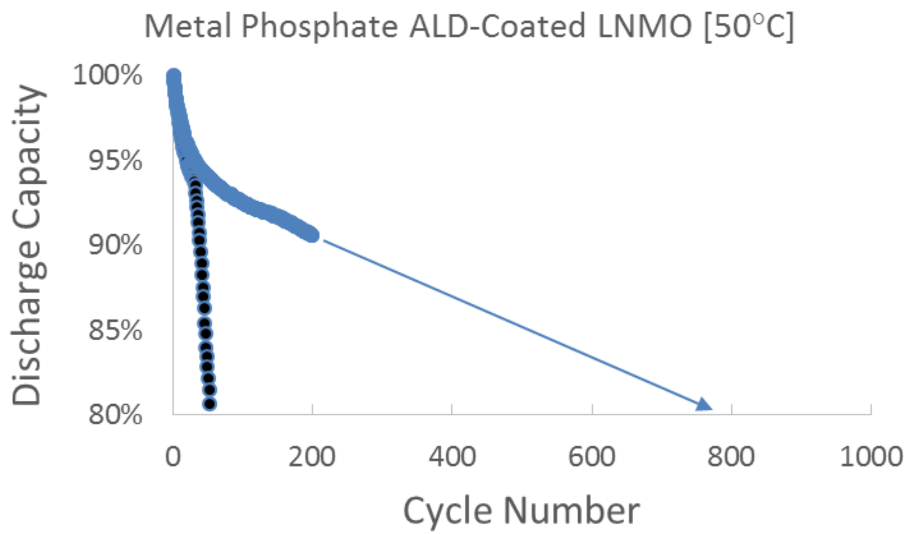


Performance of Semi-Continuous ALD comparable to or better than Batch ALD

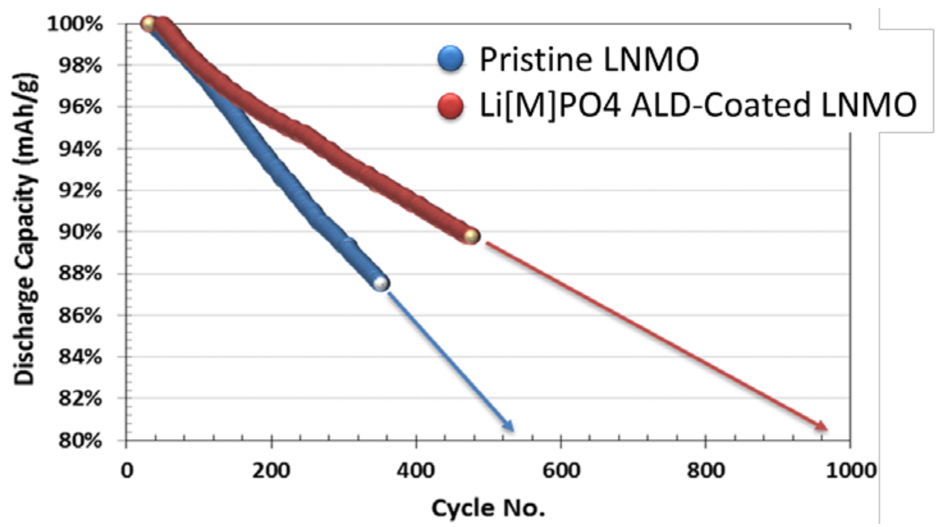
Alternate ALD Chemistry Development: Li[M]PO_4

ALD-Oxide LNMO/LTO EOL performance: 300 cycles at 55°C





Transitioning to [M]PO_4 and Li[M]PO_4 ALD Processes significantly enhances ALD-enabled LNMO



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PneumatiCoat SBIR Program Summary

- ✓ Confirmed ALD delivers highest value proposition of all surface coating techniques
- ✓ Validated scalability of Particle ALD using semi-continuous manufacturing approach
  - ✓ Pilot-scale system capable of 4 ALD cycles at 200 kg/day
  - ✓ Fewer unanswered questions vs. ALD electrode coating
- ✓ Developed ALD processes for fluorides, phosphates, Li-containing coatings, and others
  - ✓ No-Go for fluorides: hygroscopic, waned commercial interest
  - ✓ Li-containing coatings provide 20% reduction in 1st cycle ICL
- ✓ Issued Patent: US 8,993,051 for ALD-enabled battery materials

Future Work

- System reliability and electrochemical reproducibility studies using 200 kg of cathode powders
- Screening designs for ALD-enabled graphite anodes
- Benchmark ALD-enabled LMR-NMC performance
- Down-selection and demonstration of alternate ALD coatings for HC and HV cathodes in 2 Ah pouch cells
- Performance validation in 18650 cells
- Expand collaborations into BATT, USABC, ABR/EERE programs by providing sample evaluation materials
- Goal: ALD-enabled battery materials supply contracts

Backup Slides

PCT’s R&D Systems Brochure

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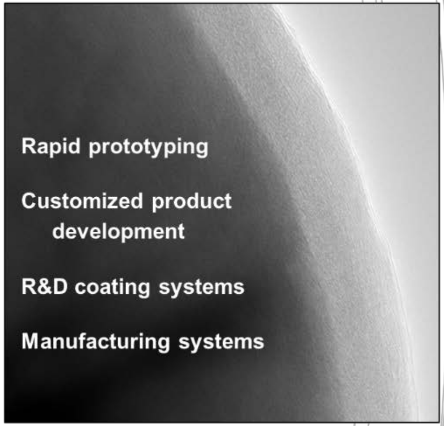

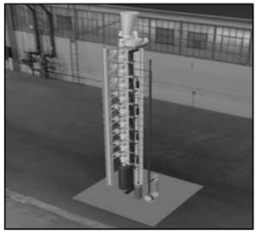

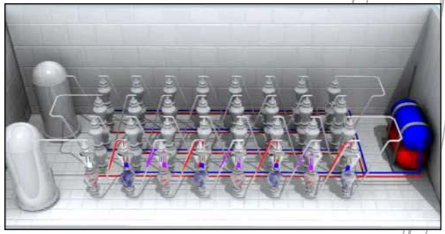

Particle Coating Reactor Systems

R&D Tools for Atomic Layer Deposition

PneumatiCoat Technologies presents the world’s most robust, flexible and economical Atomic Layer Deposition (ALD) R&D tool designed with the researcher in mind. The PCR Series reactor is well-suited to apply nanoscale encapsulating coatings on 10’s to 100’s of grams of powders, facilitating groundbreaking R&D, new product development and enabling intellectual property generation.

Technology at work for you

The PCR Series combines the most advanced features for control and in situ analysis to execute carefully designed coating recipes to perfection. In line mass spectrometry provides real-time insight for new chemistry development, studying fundamental interactions between gas-phase precursors and sensitive substrates, and allows for lean operation to minimize the total cost of ownership of the system. The dimensions of each reactor body have been optimized such that any vessel can be passed through a glovebox antechamber, and isolation valves provide for a complete solution to air-free handling of sensitive powders. Cohesive and nano-sized powders can also be processed with integrated high-shear tools to minimize secondary aggregation caused by charge build-up or liquid bridging during the fluidization and coating process. Multiple precursor zones with independent heating controls allow the PCR Series to utilize solid and low vapor-pressure precursors with ease.

Functional. Flexible. Economical.

PneumatiCoat Technologies routinely customizes capabilities, configurations and designs to meet the technical and budgetary objectives of both Corporate R&D groups and Academic partners alike. Decades of nano-coating process and system design experience makes PneumatiCoat Technologies an ideal partner for small-scale customized product development, IP protection, through to low-cost lean manufacturing in high-throughput systems.

Experience the PneumatiCoat™ advantage.

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Technical Specifications

PCT Fluidized Bed ALD Reactor	Lab-scale fluidized bed ALD reactor with inert handling and up to 500°C capability
Base operating diameter	2” vertical chamber with 3” expansion zone for effective fluidization; Larger systems and object coating modules available and can be added at an additional cost
Design options	Inert handling, Custom reactor chamber sizes, Fluidization aids, water-free chemistries and many more options
Construction	Stainless Steel Conflat flanged reactor body, VCR-based piping, 304SS standard
Gas Flow	Three MFCs depending on required fluidization conditions. N_2 MFCs standard unless specialty gases are requested; additional MFCs and ozone/plasma generators can be incorporated at an additional cost
Precursor Channels	Capabilities for processing with three precursor types (standard) with separate temperature set point capabilities up to 150°C for low vapor pressure liquids and solids; additional dosing capabilities can be added at an additional cost
Maximum reactor temperature	500°C (vertical clamshell furnace)
Heated process lines	Automated temperature control of all dosing lines independently
Pressure gauges	2 heated vacuum pressure gauges
Optional Mass spectrometer	Mass spectrometer with direct integration into PCT software, including alarms for filament protection for maximizing up-time
Pumping and Abatement Module • **Vacuum Pump** • **Foreline Traps** • **External Oil Filtration Unit**	Rotary vane mechanical vacuum pump prepared for use with Fomblin oil; additional abatement strategies included to maximize up-time, implemented based on targeted ALD coatings of interest
User interface	Computer Control (laptop or desktop)
Control system ***LabVIEW development software license NOT included**	Easy to use HMI for control and data logs Custom temperature and flow controls Highly customizable recipe building Proprietary software in English language National Instruments LabVIEW hardware Fail-safe operation Inherently safe alarm scheme Emergency stop logic