Materials Scale-up and Cell Performance Analysis

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DOE AMR

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Project ID #
ES029

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Overview

Timeline

- Start: June 2009
- End: September 2012
- Percent complete: 30%

Barriers

- Barriers addressed
  - High Cost (<150 $/kWh)
  - Low Energy Density (>230 Wh/l)

Budget

- Total project funding: $580 k
- Funding received in FY09: $290 k
- Funding for FY10: $290 k

Supports 1⅛ Research Associates

Partners

- BATT PIs

Dillon (NREL)	Kumta (Pitt. U.)
Doeff (LBNL)	Lucht (U.R.I.)
Dudney (ORNL)	Thackeray (ANL)
Ceder (MIT)	Zaghib (HQ)

Objective

Evaluate New Materials Being Developed in the BATT Program Against DOE Goals and Baseline Performance Markers

- For FY ’10 we expected to evaluate at least four new materials.
- Such evaluation provides guidance to the researchers as to what degree they have surpassed the baseline performance and how much farther we need to go toward meeting the DOE/USABC Performance Targets.
- Tracking progress is critically important to making progress.

Approach

1. Make inquiry to BATT PIs for new materials they deem ready for the next step
   - Scale-up to 10 g batch
   - Evaluation in full cell
2. Test in half cell against Li using electrode fabrication techniques developed in BATT program.
   1. If capacity density and first cycle irreversible capacity improve on baseline then go to next step.
   2. Measure rate capability at different C-rates at a reasonable loading. If improvements over baseline, then go to next step.
   3. Work with BATT cell modelers to design electrodes for full cells.
3. Evaluate cycle life in full cells.
   - Attempt to identify performance attributes and limitations.
4. Report performance results
   1. To PI
   2. At semi-annual ABRT meetings.
   3. If performance is favorable, at DOE AMR meeting.

Received 8 responses from BATT PIs expressing interest in scale-up and evaluation

- Completed evaluation of 4 materials.
- In the process of testing 3 materials
- With work with the last PI in the coming months.

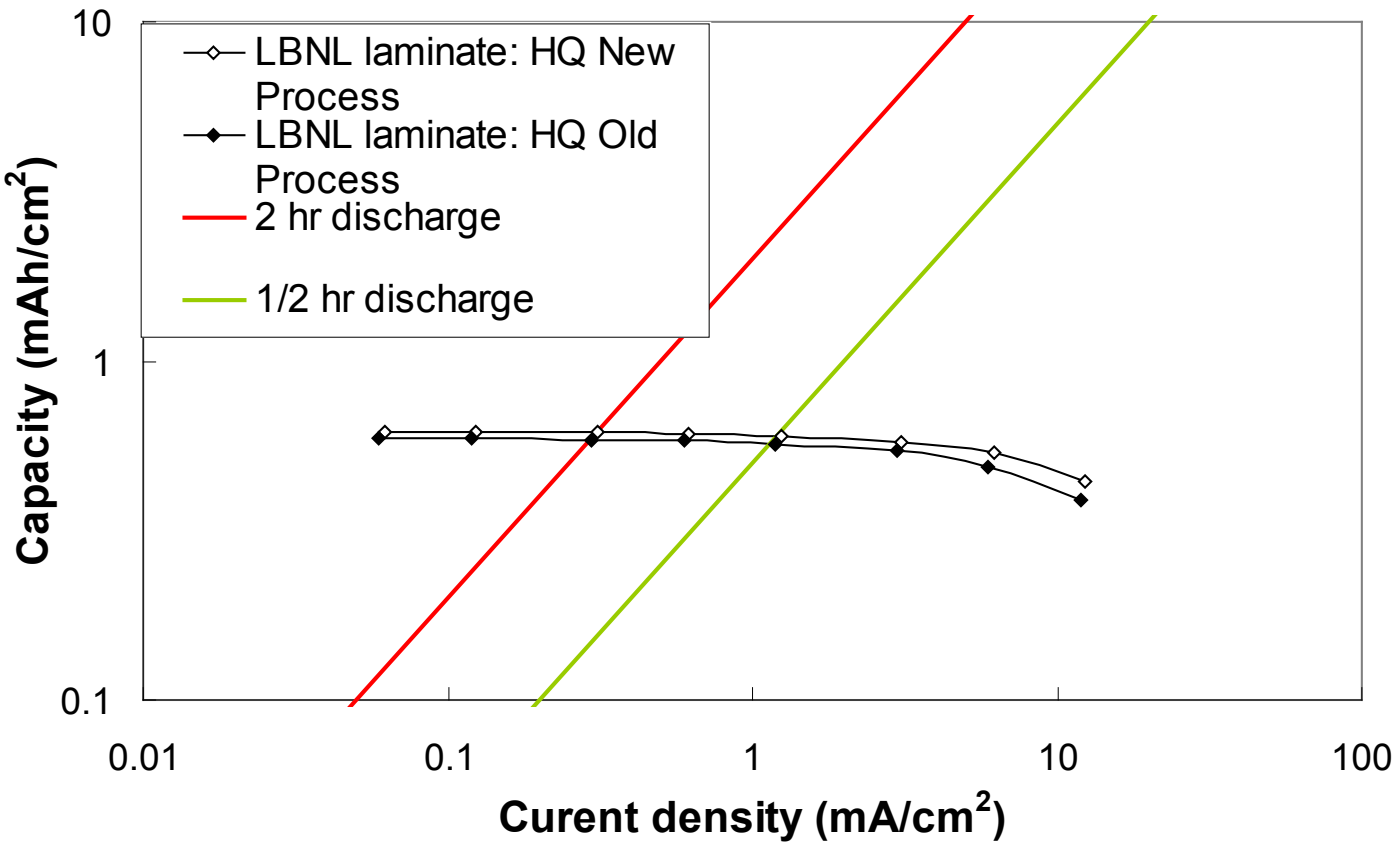
Technical Accomplishments

LBNL

Investigator	Institution	Material	Barrier	Feedback	Status
Respondents in FY09					
G. Ceder	Massachu-settes Inst. of Technology	High-rate $LiFePO_4$	High system cost	We made the material w/ their guidance	**Cycle-life testing**
M. Thackeray	Argonne National Laboratory	High-capacity NCM material	Low energy density	Sent us materials and electrode formulations	**Cycle-life testing**
N. Dudney	Oakridge National Laboratory	$LiFePO_4^-$ in carbon mat – no Cu cur. col.	High system cost	Sent us anodes	**Tests complete (low cap. dens.)**
M. Doeff	Lawrence Berkeley Nat. Laboratory	Al-doped NCM material	High material cost	We will make material w/ their guidance	**To be initiated**
P. Kumta	University of Pittsburgh	Si-C nanocomposite	Low energy density	Sent 1st gen anodes.	**Tests complete (high 1st cycle ICL)**
K. Zaghib	Hydro-Québec	Lower cost $LiFePO_4$	High cost	Sent 50 g of powder and laminates	**Cycle-life testing**
A. Dillon	National Renewalble Energy Lab.	High capacity MoO_3 anode	Higher energy density	Sent us anodes	**Tests complete (high 1st cycle ICL)**
B. Lucht	University of Rhode Island	$LiPF_4C_2O_4$ thermally stable salt	Poor high temperature performance	Sent us 10 g of salt	**Tests complete (high 1st cycle ICL)**

Technical Accomplishments

A comparison of two HQ materials: one received a year ago and one received 6 months ago. The new material was made *via* a less costly process.



When making comparisons, one needs to fabricate electrodes of identical loadings.

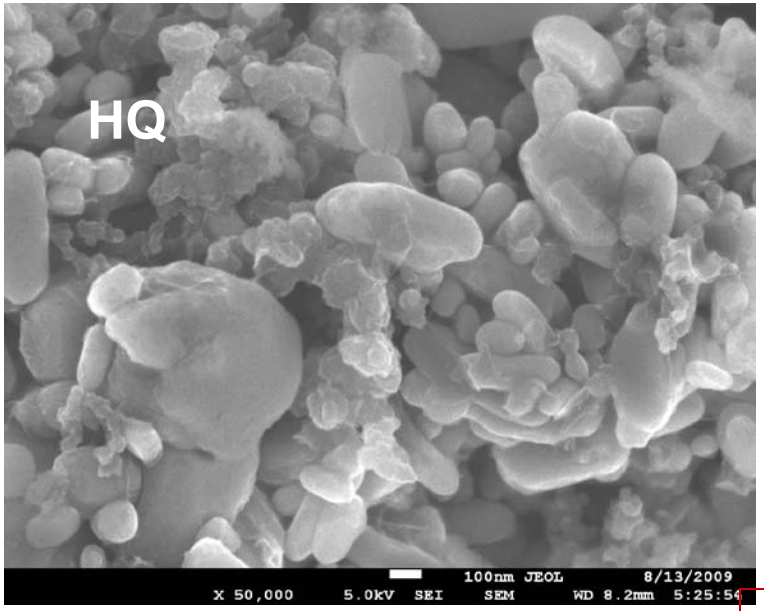
MIT Nano-$LiFePO_4$ Material

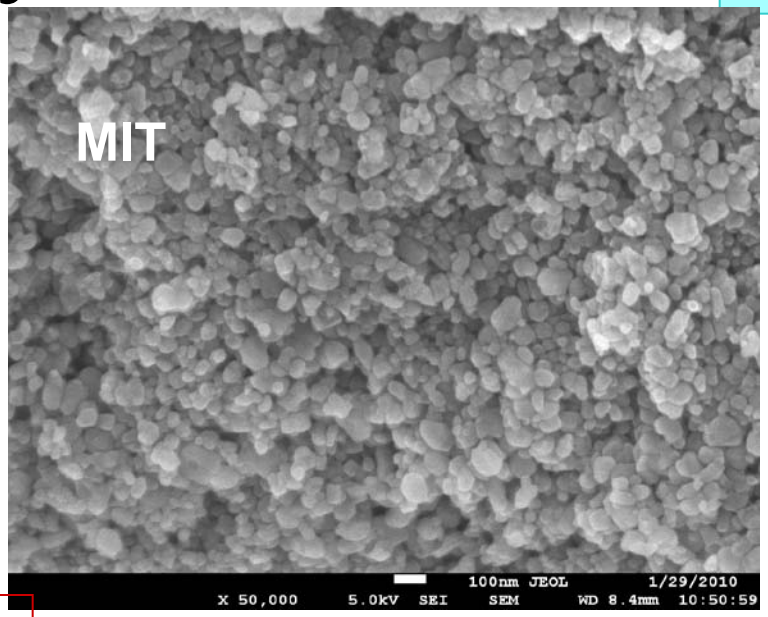
- We sent two scientists and Gao Liu to MIT for one week at the end of November during the MRS meeting in Boston
  - We made a batch of material there.
  - We were given *ca*. 1 gram of powder.
  - We were given an electrode laminate and Swagelok hardware for cell testing.
- Since then we have:
  - Made several 7 g batches of the material.
  - Characterized the materials with SEM, BET, and PSA.
  - Made laminates following their recipe using PTFE
  - Made laminates following our recipe using PVdF.
  - Exchanged testing results.
  - Prof. Ceder visited our lab in March.
  - Sent samples out for XPS analysis.
  - Been working to make higher rate electrodes.
  - Been working with the Molecular Foundry to do further surface analysis.

This has been a very open collaboration between all scientists involved!

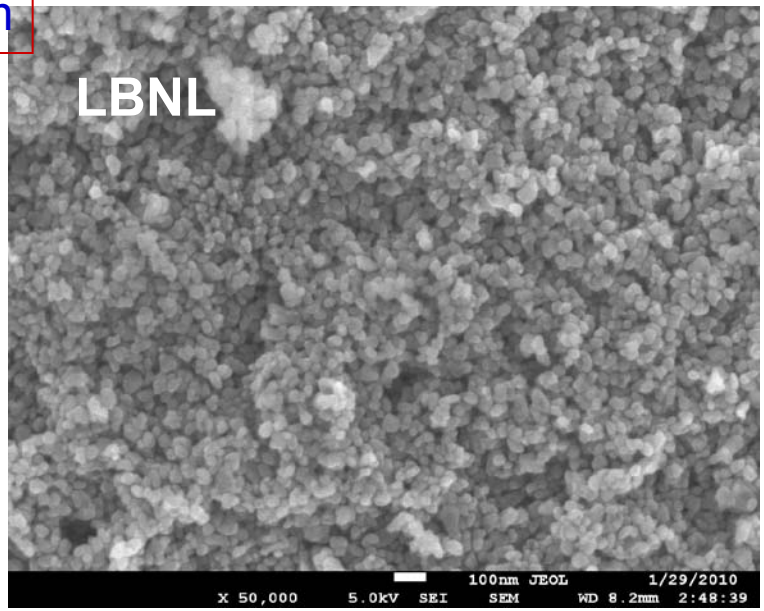
SEM - Primary Particles

X. Song





– 100 nm

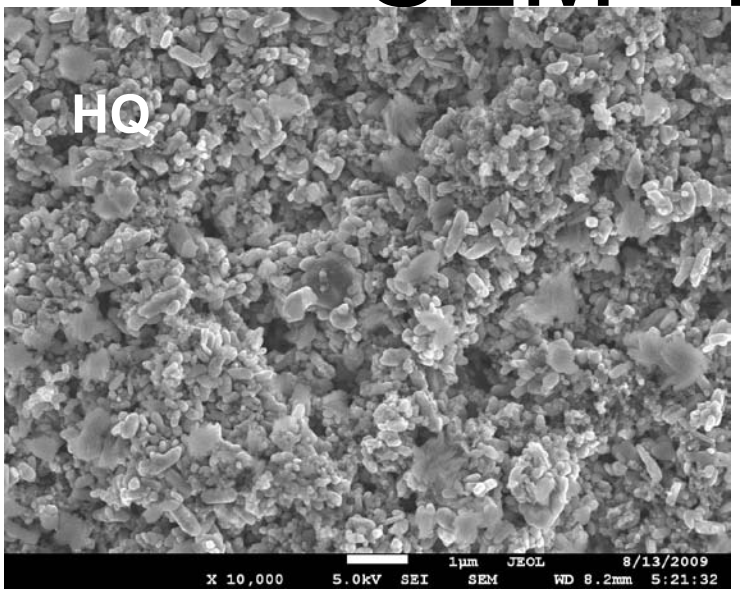


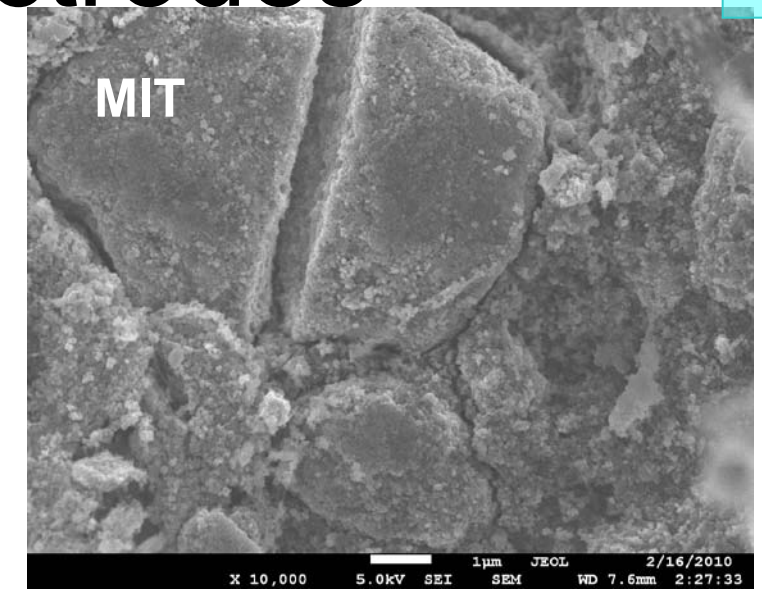
SEMs at 100 nm

- **For the HQ material, primary particles range from 50 to 500 nm**
- **For the MIT and LBNL, material primary particles are around *ca*. 25 nm**

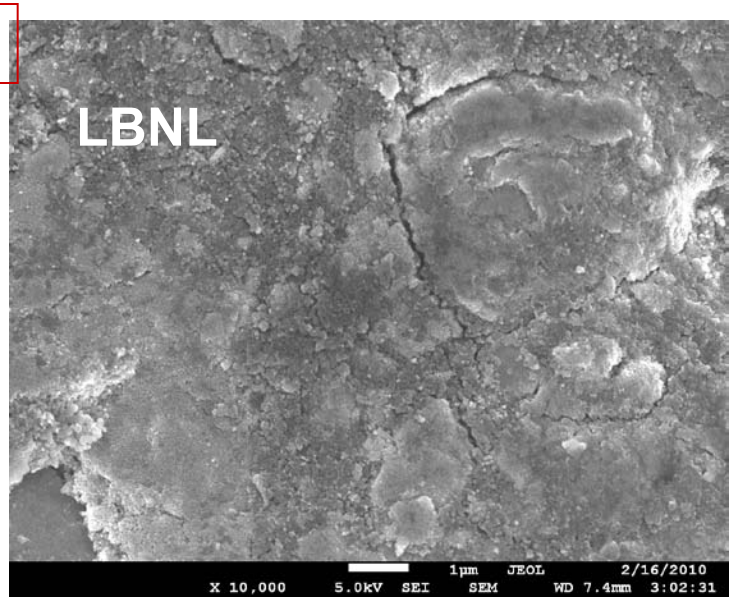
SEM - Electrodes

X. Song





— 1 µm



Electrodes

- **HQ shows uniform mixing of additives and primary particles.**
- **For the MIT and LBNL, a fraction of the powder was still clumped together.**

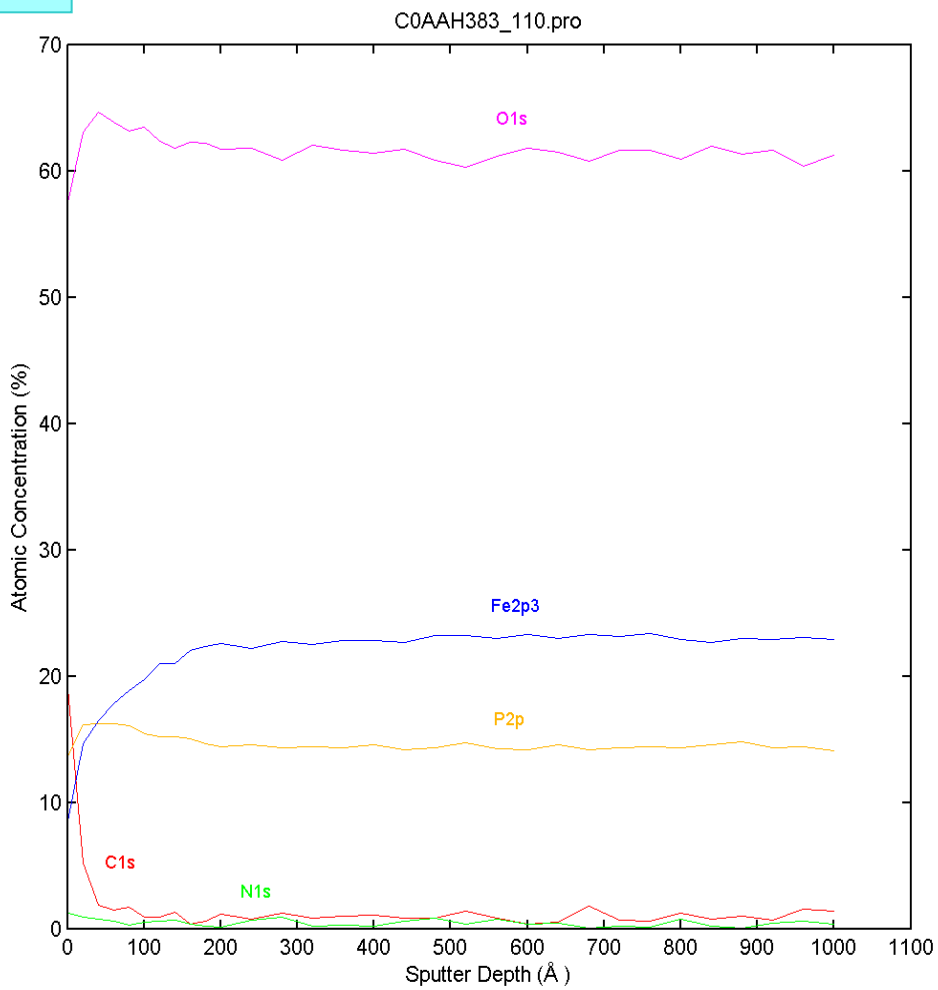
Technical Accomplishments

The MIT nano-$LiFePO_4$ is jet black.

- EDX indicated that there is some carbon on the surface.
- We sent this material out for XPS analysis, along with a sample of HQ's material.

XPS & Ion Sputtering of MIT $LiFePO_4$

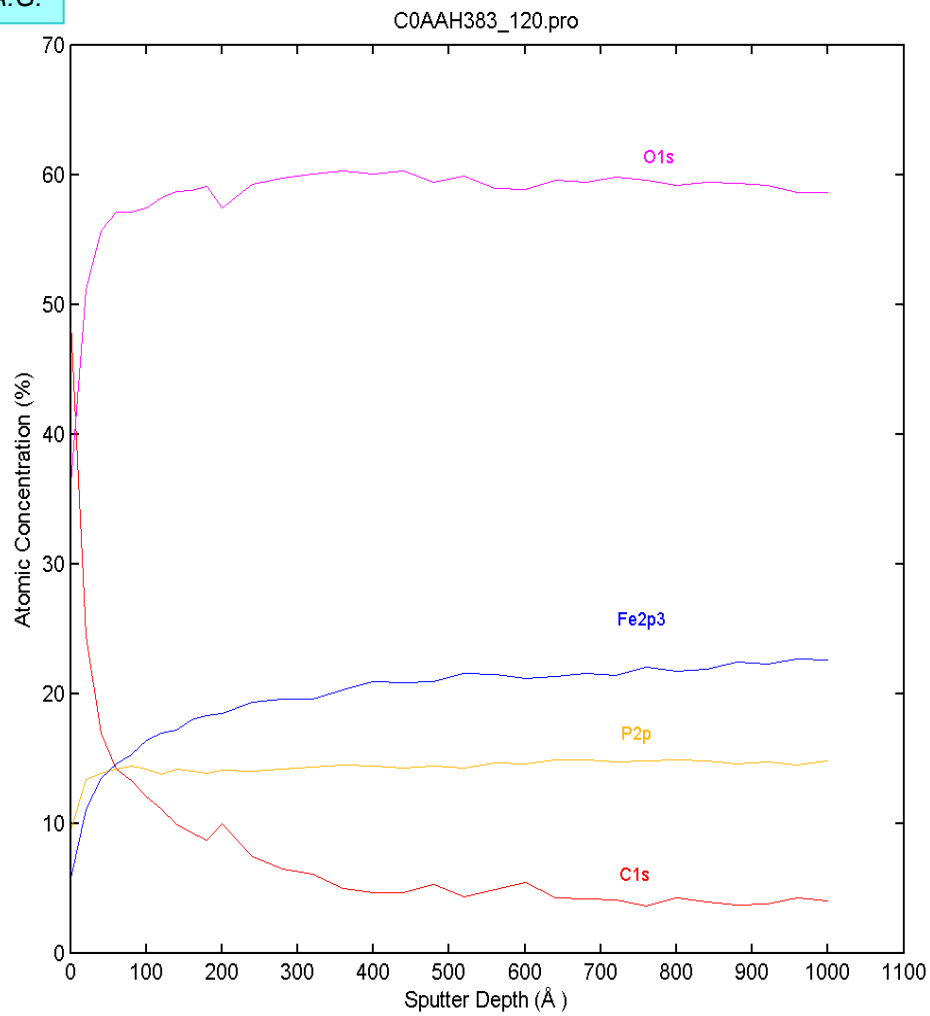
E.A.G.



- Points of interest
  - Slight carbon coating of 1 to 2 nm, believed to be residue of $FeC_2O_4 \cdot 2H_2O$ precursor.
  - Oxygen and phosphorous are uniform and of the same ratio from surface to interior P:O = 1:4.
  - Accumulation of data suggests this sample is coated with 2 to 5 nm of Li_3PO_4.

XPS & Ion Sputtering of HQ $LiFePO_4$

E.A.G.



- Points of interest
  - Carbon coating 5 nm thick, believed to be an elemental carbon.
  - Oxygen and phosphorous are uniform and of the same ratio from surface to interior P:O = 1:4.
  - There may be a thin coating of Li_3PO_4 on the surface of this material as well, underneath the carbon.

Collaborations

Investigator	Institution	Interaction	Investigator	Institution	Interaction
G. Ceder	MIT	We make material w/ their guidance	P. Kumta	U.Pitt.	Will send 30 to 100 g of 1st gen anodes
M. Thackeray	ANL	Will send us materials and electrode formulations	K. Zaghib	H.Q.	Will send 30 to 100 g of powder and laminates.
N. Dudney	ORNL	Will send electrodes	A. Dillon	NREL	Will send laminates
M. Doeff	LBNL	We make material w/ their guidance	B. Lucht	U.R.I	Will send 10 g of salt

We are also working with V. Srinivasan of the Modeling Group of the BATT program to design cells.

P. Ross helped us with acquiring and interpreting the XPS data.

Acknowledgment

The majority of this work was carried out by Jin Chong and Honghe Zheng.

Proposed Future Work

- Rest of this year
  - MIT material looks promising as a high rate material
    - Finish electrode processing and testing.
    - Work with BATT Modeling Group and MIT to determine best automotive application.
    - Design electrodes and test cells to that application.
    - We have additional experiments planned to understand why this sample is black.
  - ANL's materials are in early phases of evaluation
    - Finish initial tests.
    - Decision point - If improvement over baseline materials, we will:
      - Work with BATT Modeling Group and ANL to determine best automotive application.
      - Design electrodes and test cells to that application.
  - H.Q. laminates are in the early stages of evaluation
    - Finish initial tests.
    - Decision point - If improvement over baseline materials, we will:
      - Work with BATT Modeling Group and ANL to determine best automotive application.
      - Design electrodes and test cells to that application.
  - Low-Co NCM is important, scale-up LBNL's Al-substituted NCM.

- All milestones completed

Summary

- Eight BATT PIs answered the call for materials evaluation.
  - 4 sent laminates
  - 1 sent powders
  - 1 sent both
  - 2 asked us for assistance in scale-up
- Benefits and limitations of materials have been confirmed and conveyed back to PIs.
- New, low-cost HQ material performs as well as previous material.
- MIT material needs further processing to make good electrodes – this work has begun.
- Both MIT and HQ materials have coatings and perform well.
  - HQ's has 100 nm primary particles with a 5 nm carbon coating that improves the electronic conductivity and leads to good electrode performance.
  - MIT's has 30 nm primary particles with a 5 nm phosphate coating that mitigates secondary particle formation and minimizes solid-state diffusion limitations that leads to good electrode performance.