Process Development and Scale-Up of Advanced Active Battery Materials

Ozge Kahvecioglu (PI)

Argonne National Laboratory
Project ID: BAT167

June 23, 2021

Overview

Timeline

- Project start date: Oct. 2020
- Project end date: Sept. 2021
- Percent complete: 80%

Budget

- Total project funding:
  - $912.5K in FY20
  - $800K in FY21

Barriers

- Cost: Reduce manufacturing costs with scalable continuous processes
- Availability: Advanced cathode materials needed for research are not commercially available with the desired composition or morphology
- Performance & Life: High energy density advanced cathode materials have major performance and life issues

Partners

- Collaborators in Deep-Dive into Next-Generation Cathode Materials (BAT251, BAT252, BAT253), ANL, ORNL, NREL, BNL, PNNL
- CAMP Facility (BAT030), ANL
- Jianlin Li (BAT164), ORNL
- Venkat Srinivasan (BAT402), ANL
- Feng Wang (BAT183), BNL
- Prof. Mark Hersam's Group, Northwestern University
- Ted Seo, Volexion
- Zheng Chen, UC San Diego, ReCell (BAT377)
- Christopher Johnson, ANL

OBJECTIVES AND RELEVANCE

- The objective of this program is to provide a systematic research approach to:
  - Produce and provide **sufficient quantities of high quality** battery materials for large scale evaluation and to support further research
  - Evaluate **emerging synthesis technologies** for the production of experimental cathode materials
  - Develop **cost-effective, scalable** processes for manufacturing of advance materials that are not commercially available
- The relevance of this program to the DOE Vehicle Technologies Program is:
  - The program is a key missing link between invention of new advanced cathode materials, market evaluation of these materials and high-volume manufacturing
  - Enabling full-scale evaluation by multiple R&D groups
  - Reducing the risk associated with the commercialization of new battery materials
- This program provides large quantities of materials with consistent quality
  - For prototyping in large format cells
  - To allow battery community access to new materials and advance further research

MILESTONES

Month/Year	Description of Milestones	Status
July 2020 – May 2021	**Synthesis of Ni-based hydroxide precursors- Deep-Dive into Next-Generation Cathode Materials (BAT030,BAT251, BAT252, BAT253, BAT476)** Reproduction & scaling up baseline materials at 10L TVR: $Ni_{0.6}Co_{0.2}Mn_{0.2}(OH)_2$ and $Ni(OH)_2$ Optimization & reproduction of $Ni_{0.90}Co_{0.05}Mn_{0.05}(OH)_2$ at 10L TVR: diagnostic studies; pouch cell evaluation **Introduction of higher nickel content chemistries**: Optimization of $Ni_{0.95}Co_{0.25}Mn_{0.25}(OH)_2$ at 1L TVR & Scaling up at 10L TVR Optimization of $Ni_{0.95}Mn_{0.05}(OH)_2$ at 1L TVR Comparative studies: TVR (continuous) vs CSTR(batch) methods for $Ni_{0.95}Mn_{0.05}(OH)_2$ **Calcination optimization for LNO-based oxides:** Calcination method (box furnace vs tube furnace), heating/cooling profile, calcination atmosphere and temperature Issues were identified; calcination scale-up still remains as a challenge **Preliminary synthesis of Li/Mn-rich cathode material at 1L TVR for next year activities** **Milestone1:** Materials delivery to CAMP Facility (BAT030) **Milestone2:** Several journal publications are on-going in collaboration with the whole team	**On track** Complete Complete Complete Complete On-going On-going **On-going** On track On track **Complete** **Complete** **On-going**
September 2020 February 2021	**Synthesis of NCM622 and NCM811 – Chris Johnson (ANL)** New lot of $Ni_{0.6}Co_{0.2}Mn_{0.2}(OH)_2$ precursor synthesis at 10L Taylor Vortex Reactor – 500 g delivery New lot of $Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)_2$ precursor synthesis at 10L Taylor Vortex Reactor – 650 g delivery	**Complete** Complete Complete
December 2020	Determine the structure and morphology evolution during the sintering process in synthesis of $LiNiO_2$ and NMC811 **(BAT183, BAT402)** $Ni(OH)_2$ and new lot of $Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)_2$ precursor material was synthesized at 10L TVR and delivered $Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)_2$ precursor material with different porosities	**On-going** Complete On-going
January 2021	**ReCell (BAT377) & University of California, San Diego** $Ni(OH)_2$ precursor material delivery @ 200 g for "Hydrothermal Recycling/Upcycling of Spent Lithium-ion Battery Cathodes"	**Complete** Complete
August 2020 April 2021	**Northwestern University & Volexion** New batch of LNO cathode material (~10 µm): applicability of graphene coating method on larger particles **Milestone1:** Journal publication is in submission	**On track** Complete On track

APPROACH AND STRATEGY

- To address US battery community material needs as quickly as possible;
  - Providing high quality cathode precursors & cathodes that are not commercially available
  - Scaling up promising chemistries up to kg quantities for large scale evaluation
- Collaborating with basic R&D teams, companies, start-ups and academia across the nation to enable their research
  - Tailoring particle properties & compositions upon request for the project initiation
- By implementing emerging synthesis technologies;
  - Reducing the process optimization time to get high quality materials with reproducibility
  - Reducing the waste, time and cost using continuous process
- Our strategy relies on the feedback from the collaborators and recipients of our materials;
  - Taking the formula and the synthesis method from the bench scale studies; develop scalable processes based on target specs

TECHNICAL ACCOMPLISHMENTS

Precursor Scale-up Enables systematic LNO-based oxide studies

- Extensive collaboration between the subgroups within the low-cobalt deep-dive program (see **BAT030, BAT251, BAT252, BAT253, BAT476**); 8 different compositions were synthesized using Taylor Vortex Reactors (TVRs).
- Majority of compositions are optimized and scaled up: 1L TVR (FY19-20) and 10L TVR (FY20-21) within a 1.5 year time-frame

MERF Precursor via TVR → **RNGC Calcination** → **CAMP Facility Electrode Fabrication**

Composition	RNGC Reference (Ni-Mn-Co %)
$Ni_{0.6}Mn_{0.2}Co_{0.2}$	60-20-20
LNO	100-0-0
$Ni_{0.95}Co_{0.05}$	95-0-5
$Ni_{0.9}Co_{0.1}$	90-0-10
$Ni_{0.95}Mn_{0.05}$	95-5-0
$Ni_{0.9}Mn_{0.1}$	90-10-0
$Ni_{0.95}Mn_{0.025}Co_{0.025}$	95-2.5-2.5
$Ni_{0.90}Mn_{0.05}Co_{0.05}$	90-5-5

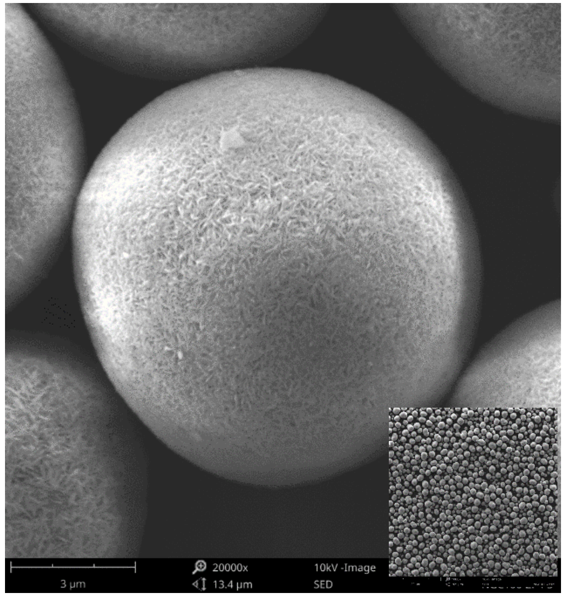
Precursor scale-up resulted in numerous calcined samples (30~200 grams), then provided to the CAMP Facility for electrode fabrication

Electrodes designed for proper capacity matching against a baseline anode A-A015(A)

TECHNICAL ACCOMPLISHMENTS

Effect of Calcination Temperature & Atmosphere

- **Physicochemical baseline material, LNO,** was synthesized at different conditions to exploit the best performances: $Ni(OH)_2$ precursor, is mixed with stoichiometric amount of $LiOH.H_2O$ using acoustic mixer and calcined in a tube furnace;
  - **$T_{calc.}$= 665°C & *Po_2* = 1.0 atm → optimal conditions with highest capacity retention, lowest Li/Ni mixing**



$Ni(OH)_2$

Reproduced -10L TVR

Production rate ~170 g/h

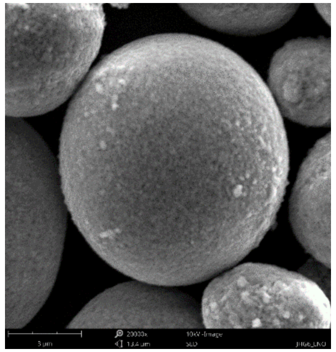
Tap density = 2.01 g/cc

PSA = 4.9 / 9.4 / 16.5 µm

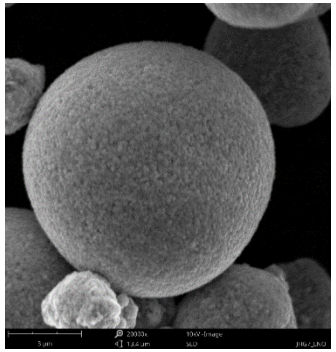
BET = ~10 m^2/g

Effect of Temperature @1.0 O_2 atm: Primary particle sizes increases with increasing calcination temperature

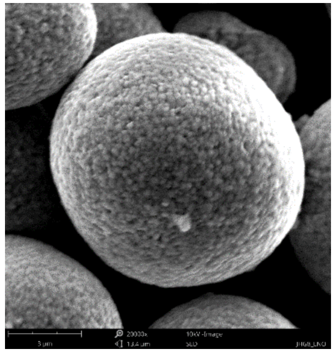
650°C



665°C

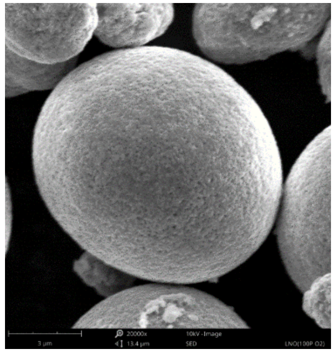


680°C

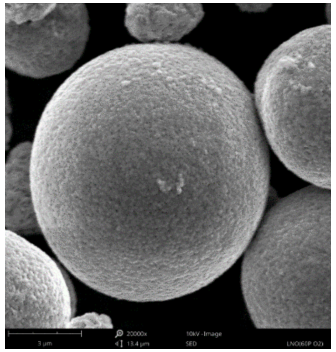


Effect of Atmosphere @ 665°C: Primary particle sizes increases with increasing O_2 partial pressure, (c/a ↑)

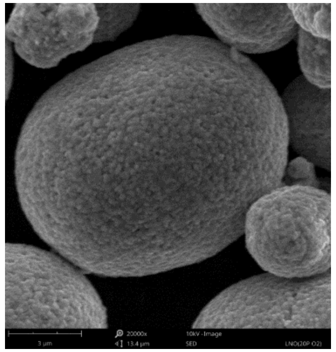
100% O_2



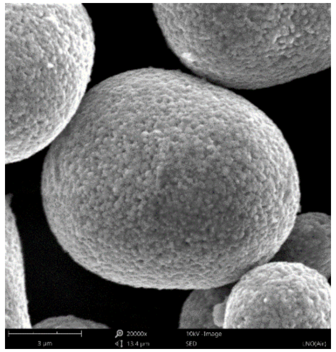
60% O_2



20% O_2



20.9% O_2



✓ **Capacity Retention** @50th vs 4th cycle; **90% (100%O_2)** → 88% (60%O_2) → **84% (21%O_2)**

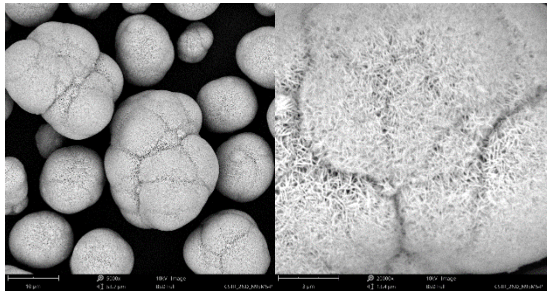
See also BAT251

TECHNICAL ACCOMPLISHMENT

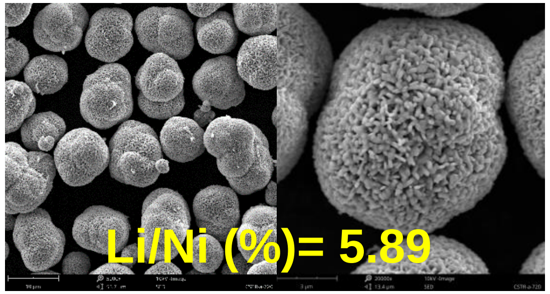
Comparison of Co-precipitation and Calcination Methods

- ➢ Nodular secondary particles in CSTR synthesis vs more spherical particles in TVR synthesis
- ➢ Narrow particle size distribution of CSTR product compared to TVR product

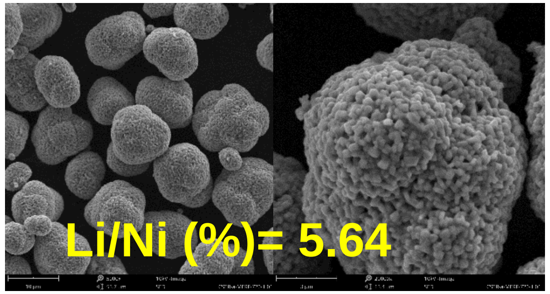
CSTR batch, ~13μm



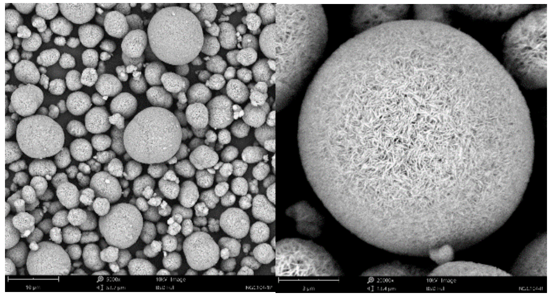
Tube furnace

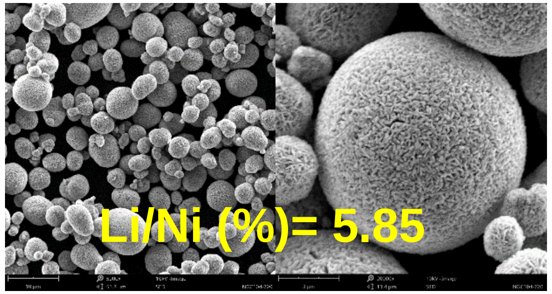


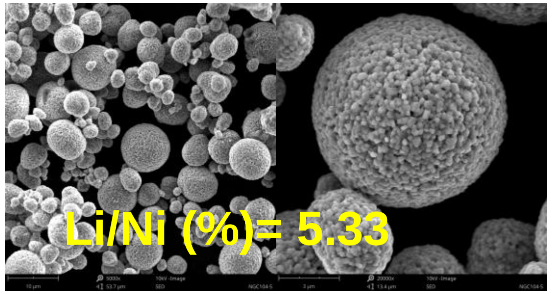
Box furnace



TVR continuous, ~8μm







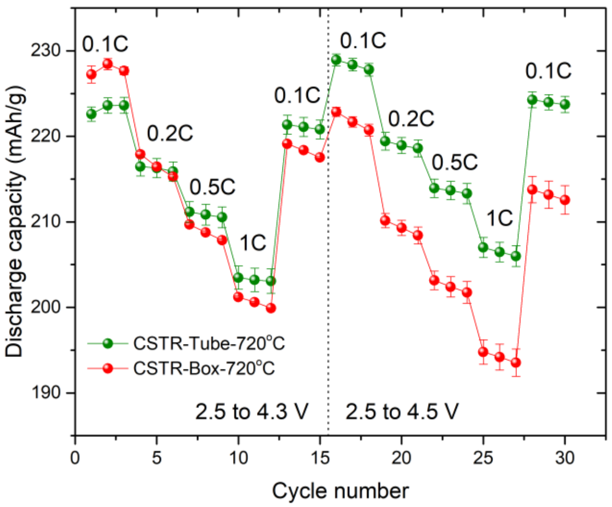
720°C; 12 h; heating/cooling under pure O_2 flow

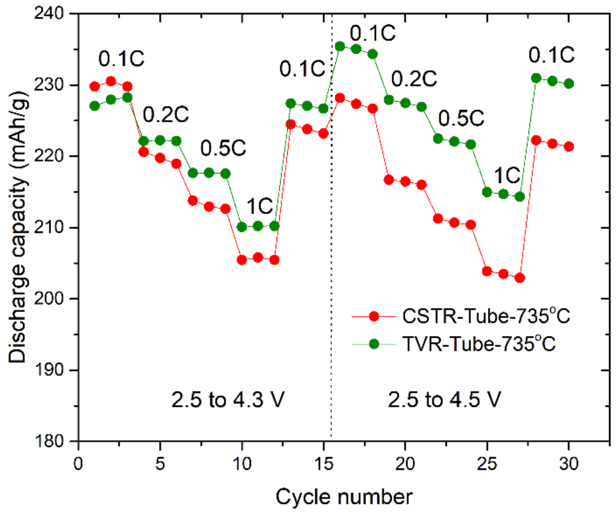
- ➢ **Tube furnace:** inhomogeneous primary particle morphologies
- ➢ **Box furnace:** homogeneous primary particle morphologies

See also BAT030, BAT251

Process/T (tube furnace)	Cap. Retention @100th cycle	Li/Ni Mixing, %
CSTR-720° C	**76%**	**5.89**
TVR-720 ° C	78%	5.85
CSTR-735 ° C	**68%**	**5.59**
TVR-735 ° C	**81%**	**4.65**
CSTR-750 ° C	70%	5.64
TVR-750 ° C	79%	4.45

Deep-Dive RNGC Protocols (see BAT476)



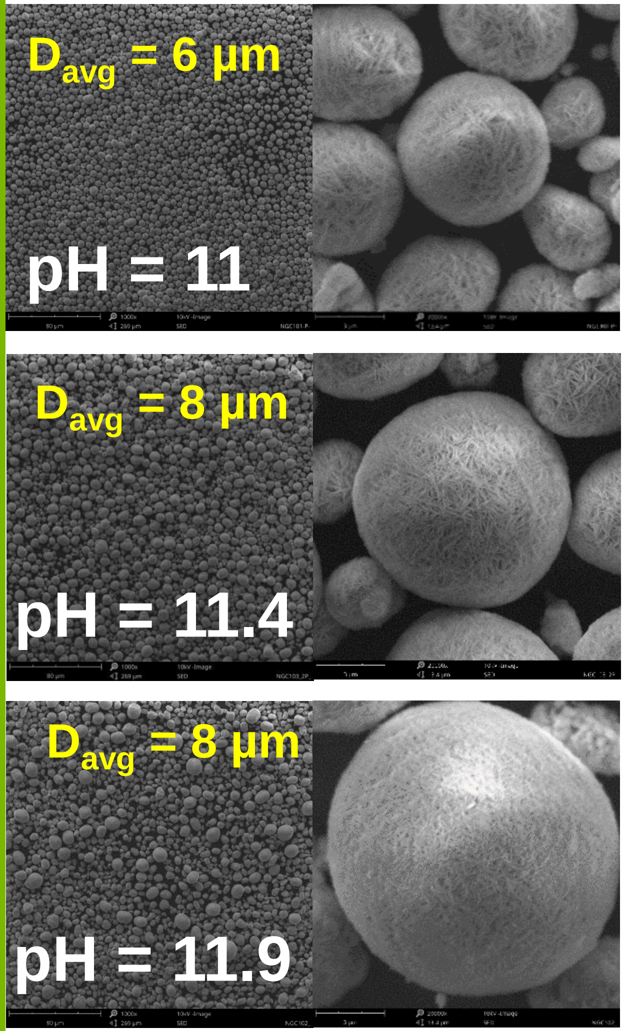


$LiNi_{0.95}Mn_{0.05}O_2$ → under optimization

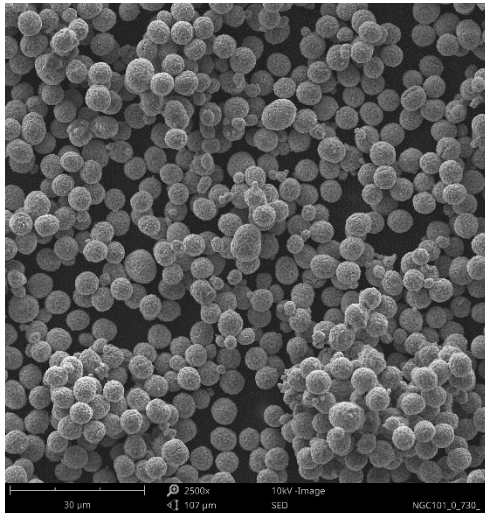
Argonne NATIONAL LABORATORY

TECHNICAL ACCOMPLISHMENT

Exemplary Optimization Studies @ TVR for $Ni_{0.95}Co_{0.025}Mn_{0.025}(OH)_2$: Effect of pH



- Nickel based hydroxide precursors require high pH conditions to obtain high density particles
  - Tap density increases with increasing reaction pH: 1.7 g/cc → ~ 2 g/cc
- Increasing the pH of the reaction results in bi-modal particle size distribution: large particles are dense and spherical, and small particles are quasi-spherical
- Surface area of the secondary particles reduces with increasing reaction pH: ~15m²/g→~9m²/g

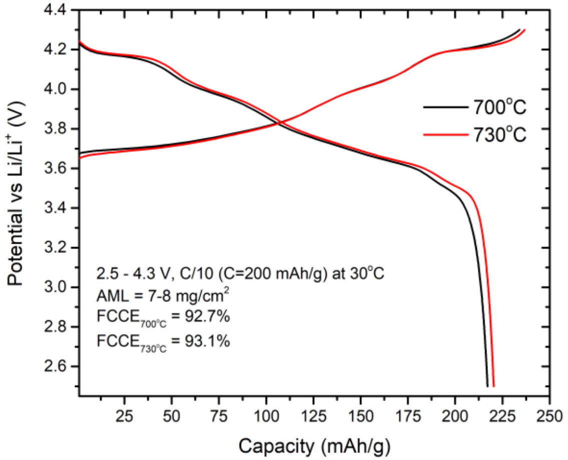


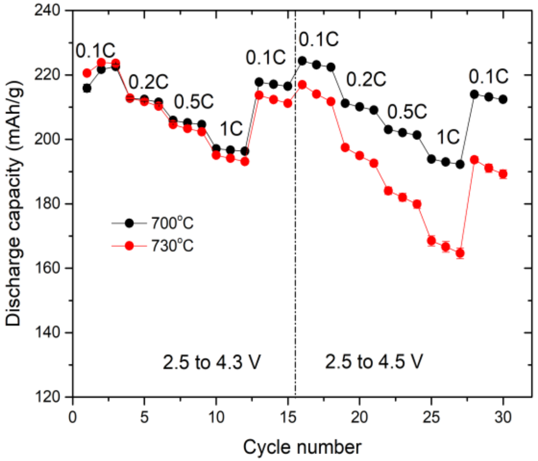
$LiNi_{0.95}Co_{0.025}Mn_{0.025}O_2$

Box furnace calcination

Li/Ni (%) = 2.6-3.6% (with ↓T)

Low/No-Co Deep Dive Protocols (Half-Cell vs Li)





Two cells average

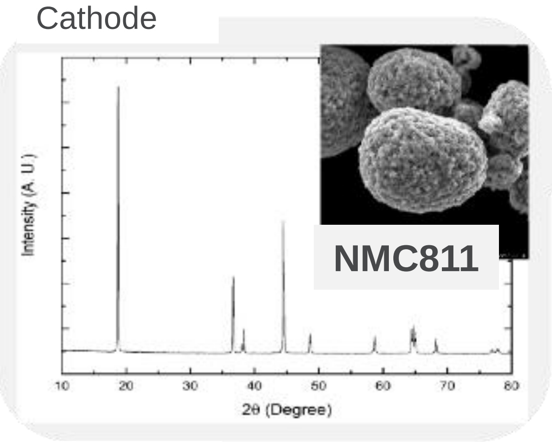
- Ni-based compositions with ≥95% requires lower calcination temperatures <730°C for better e-chem performance

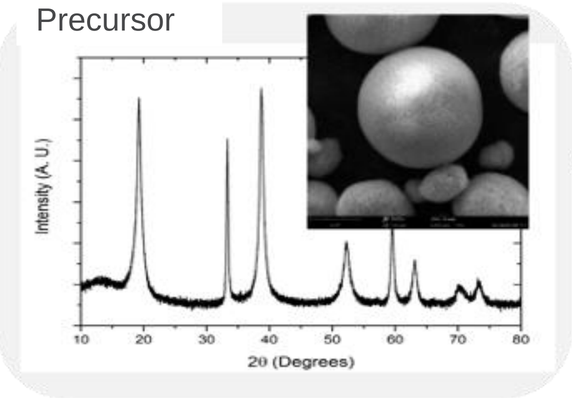
See also BAT251,BAT252, BAT476

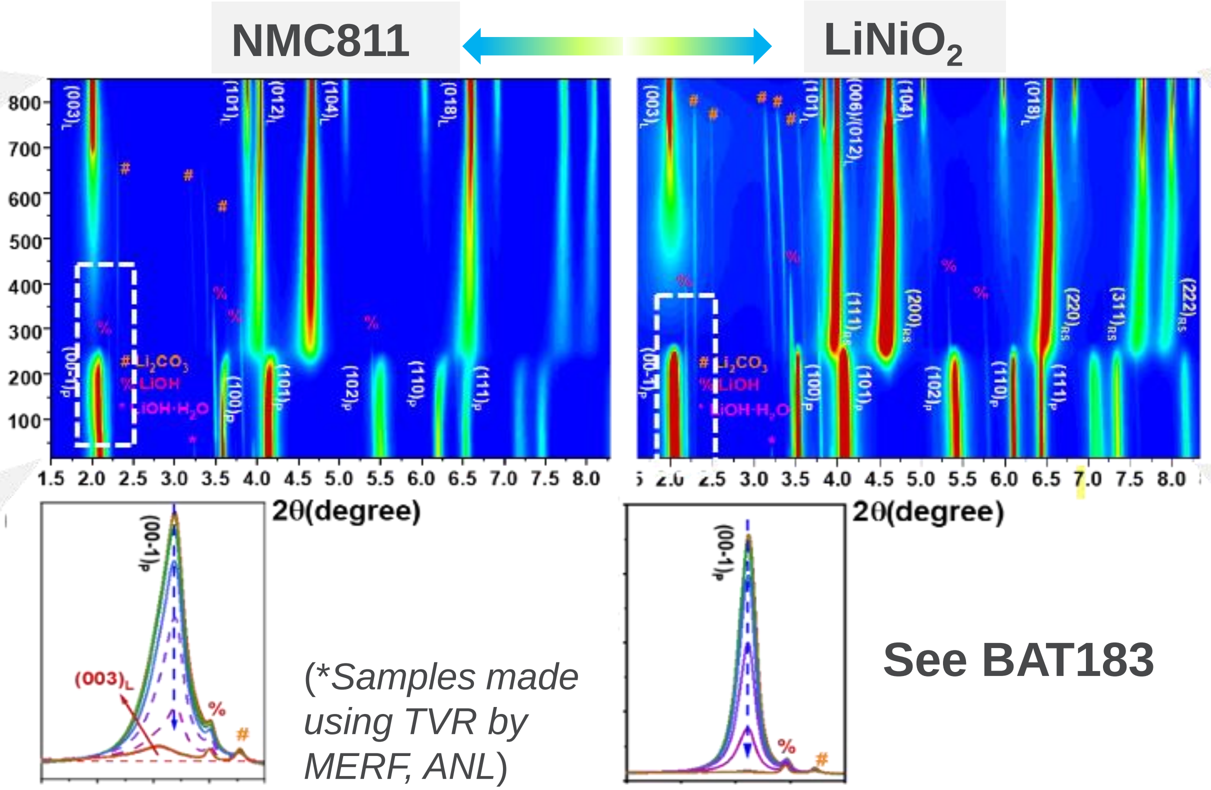
Argonne NATIONAL LABORATORY

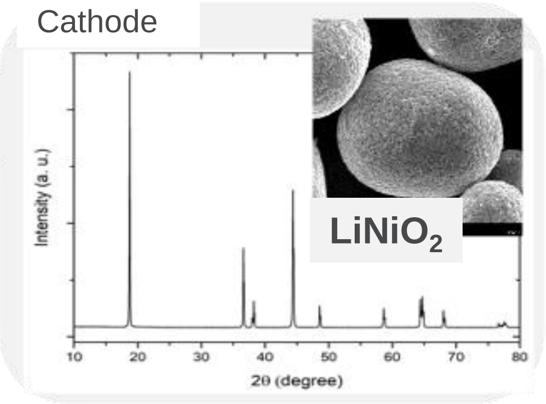
TECHNICAL ACCOMPLISHMENTS

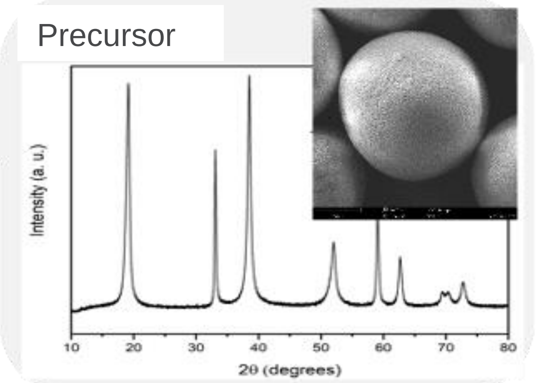
Structure and Morphology Evolution During the Sintering Process in Synthesis of NMC811 and $LiNiO_2$

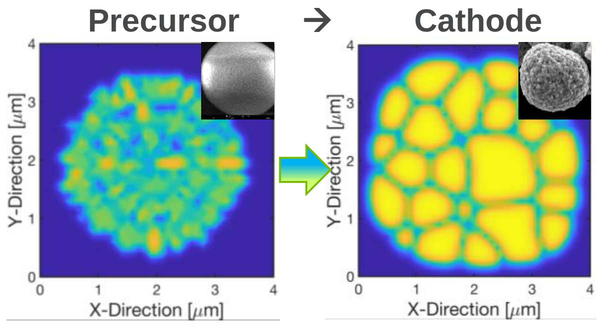












Multi-scale simulation (BAT402)

- **Reaction pathways**: overall similar in the two systems, exhibiting significant structure and morphology change
  - **NMC811**: early formation of the layered phase from hydroxide (*as low as ~ 240 ºC*) and Li-containing rocksalt;
  - **$LiNiO_2$**: formation of Li-containing rocksalt and delayed transformation into the layered phase (at ~360 ºC).

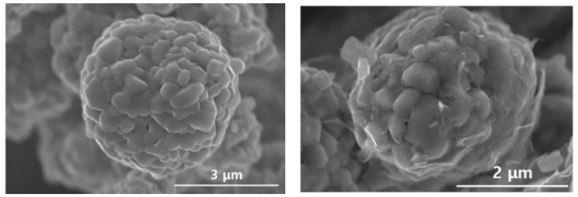
➢ **Role of Mn/Co**: facilitating Li/O incorporation during structural ordering and crystal growth.

Future work: Identify the impact of size/morphology of hydroxide precursors on the sintering process in synthesis of NMC811 → Well-controlled particle morphology and porosity using TVR

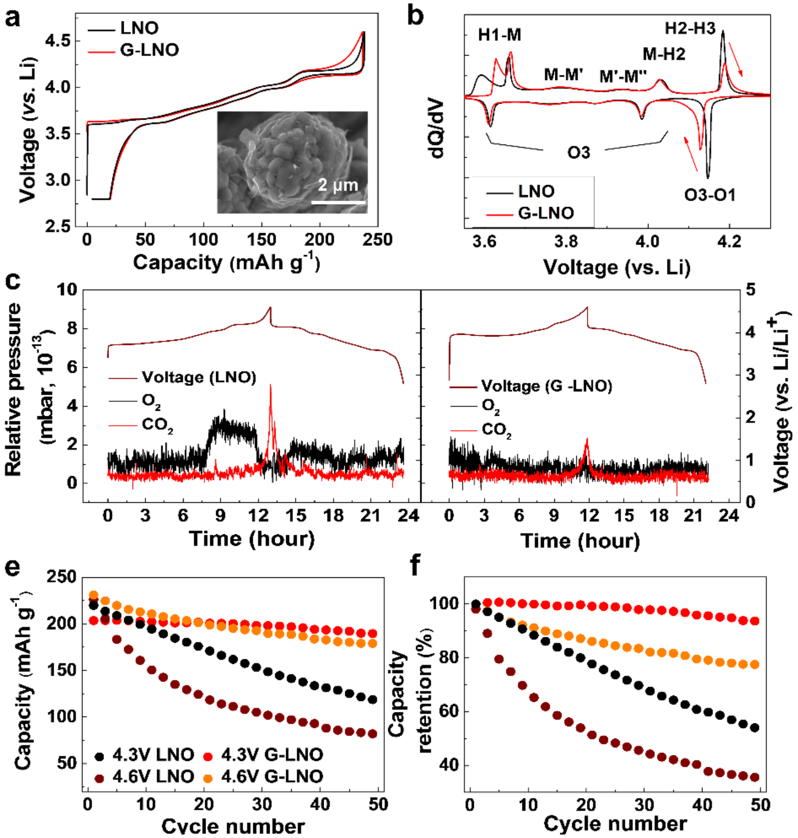
TECHNICAL ACCOMPLISHMENTS

Material Support: Northwestern University

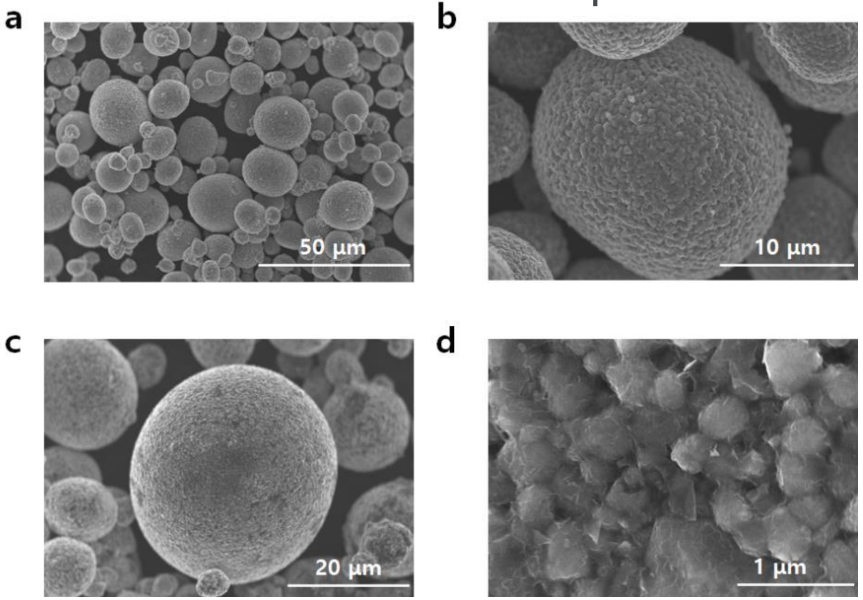
MERF-LNO ~3 µm



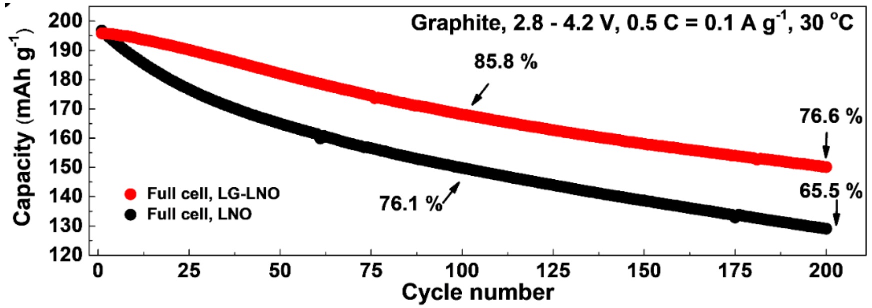
SEM images of as-synthesized ~3 µm $LiNiO_2$ (left) and graphene coated $LiNiO_2$ (right).



MERF-LNO ~10 µm

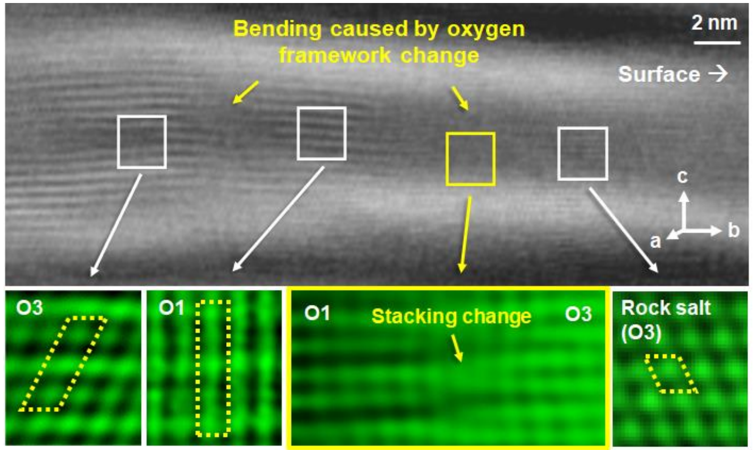


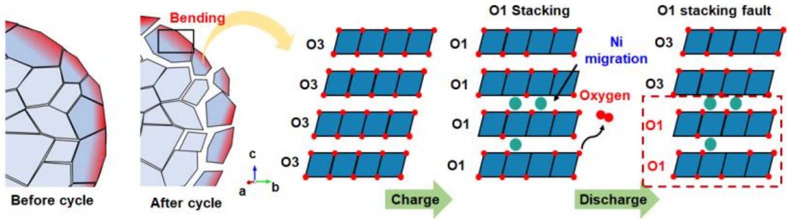
SEM images of as-synthesized ~10 µm $LiNiO_2$ (a,b) and graphene coated $LiNiO_2$ (c, d).



Long-term cycle characteristic of surface stabilized LNO

- Key solution for realizing high voltage operation is to suppress the oxygen evolution from the surface, thus mitigating the destructive oxygen stacking transition





Institutions:

Dept. of Materials Sci. & Eng., NU

NUANCE Center, NU

MERF, ANL

KIST, Korea

Dept. of Materials Sci. & Eng., Korea University

Volexion, Inc.,

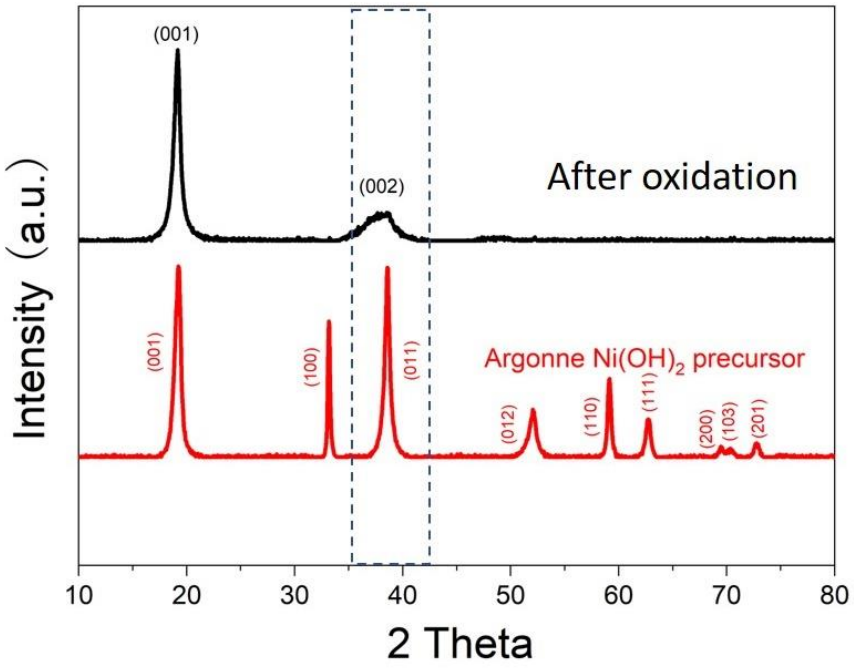
Dept. of Chemistry, NU

Dept. of Electrical and Computer Eng., NU

TECHNICAL ACCOMPLISHMENTS

Providing Materials for Other DOE-VTO Funded Projects

- **ReCell Project (BAT377)**
  - Hydrothermal recycling/upcycling of spent lithium-ion battery cathodes
  - Objective: Ni precursor is used to convert low-Ni cathode into high Ni-cathodes

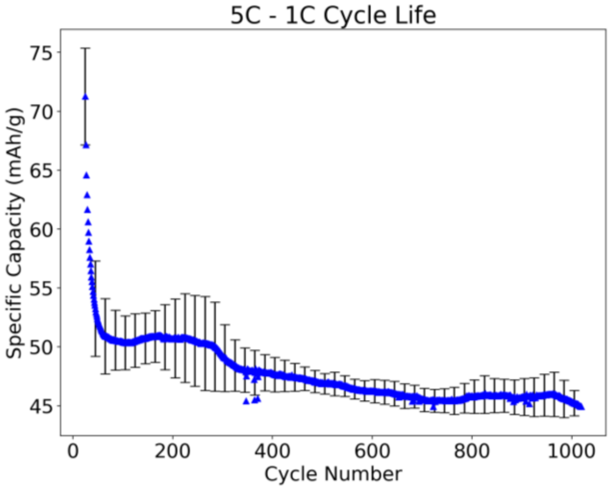


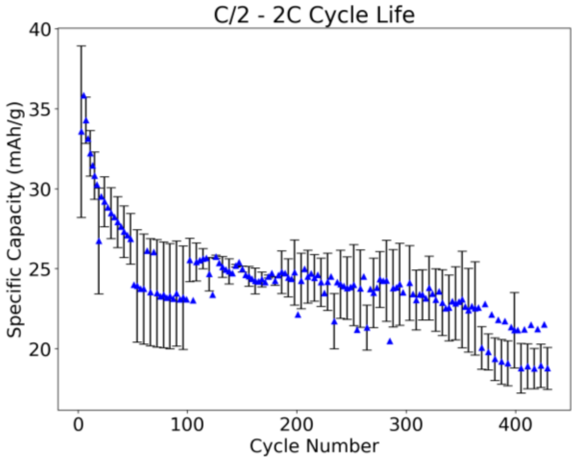
TVR-made pure β-$Ni(OH)_2$ → pure β-NiOOH

Prof. Zheng Chen, Dr. Xiaolu Yu
University of California, San Diego

- **ORNL, BAT164**
  - Graded bilayer cathodes (NCM811) pouch cells for XFC Program (see **BAT164**)

XFC Program Protocols



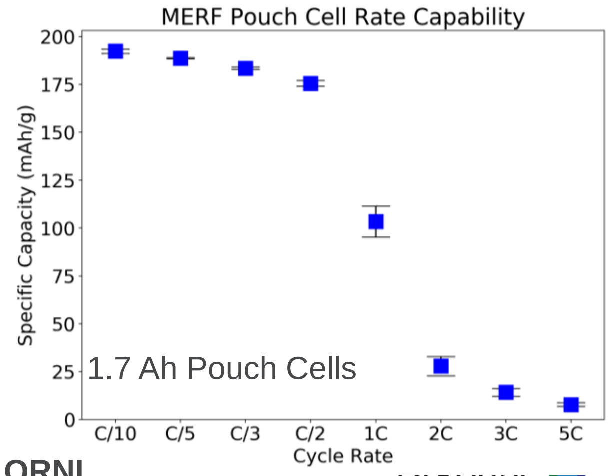


6 ~1.7 Ah cells were constructed

- 90/5/5 NMC 811/Carbon Black/PVDF composition
- 3 for rate capability and XFC protocol & 3 for cycle life protocol

~6 mAh/cm² (~35 mg/cm²) double pass NMC 811

- Bottom layer was 12 μm Targray, **top layer was 6 μm MERF material**



Dr. Jianlin Li and Dr. Alexander Kukay **ORNL**

RESPONSES TO PREVIOUS YEAR REVIEWERS' COMMENTS

- This project was not reviewed last year

COLLABORATIONS

- Cathode Materials for Next Generation LiBs: Design, Synthesis, and Characterization of Low-Cobalt Cathodes (BAT030, BAT251, BAT252, BAT253, BAT476)
  - ANL, ORNL, NREL, BNL, PNNL
- Improving Battery Performance through Structure-Morphology Optimization (BAT402)
  - Venkat Srinivasan, Tim Fister, Xiaoping Wang, Pallab Barai (ANL) → In-situ characterization and modelling
- In Situ Spectroscopy of Solvothermal Synthesis of Next-Generation Cathode Materials (BAT183)
  - Feng Wang, Jiangming Bai, Sizhan Liu (BNL) → In-situ calcination of LNO, NCM811
- Volexion and Northwestern University, Prof. Mark Hersam group
  - Ted Seo, Kyu-Young Park → various particle sizes of LNO and NCM811 for enabling the development of graphene coating to suppress H2-H3 transition research
- Thick, Low-Cost, High-Power Lithium-Ion Electrodes via Aqueous Processing (BAT164)
  - Jianlin Li and Alexander Kukay (ORNL) → Electrode cracking studies using scaled up ~7µm NCM811 cathode particles (synthesized at 10L TVR) and XFC rate capability research
- ReCell–Overview and Update; UC San Diego (BAT377);
  - Prof. Zheng Chen, Dr. Xiaolu Yu
- Chris Johnson, ANL
  - NCM622 and NCM811 hydroxide precursors

REMAINING CHALLENGES AND BARRIERS

Material Storage & Sensitivity & Scalable Methods

- Precursors of (Mn-containing) NMCs requires protective storage conditions;
  - Producing large amounts of hydroxide precursors and sampling several times introduces air flow to the bulk and causes material degradation (Mn oxy-hydroxide formation) over time;
    - Continuously synthesizing/reproducing immediately fresh materials upon requests
- Doping of some elements at the co-precipitation step to provide dopants at the intra-particle level, requires reactor feedline modification and fundamentally different chemical reaction conditions to be tested;
  - Different chelating agents to complex elements that don't chelate with ammonia (e.g.; Al)
- LNO-based NMCs with low/no cobalt cathode oxide materials are highly sensitive to:
  - The calcination conditions; Li-ratio, calcination temperature and atmosphere, furnace type, calcination recipe (heating/cooling rates)
    - Scalable calcination furnaces needs modified oxygen flow inside to maximize the oxygen penetration: furnace internal fixture designs
  - The storage conditions; Humid and CO_2 → creating surface impurities affecting the electrode manufacturing conditions and ultimately the performance of the material
  - Scalable surface protection methodologies are still under investigation: time/cost

PROPOSED FUTURE RESEARCH

- Optimization of the calcination processes by internal fixture designs in scalable box furnaces to create efficient oxygen flow to the bath of loading: to create higher quality cathode materials with less surface impurities
  - Super Alloys (with or without surface treatments-resistant against high temperature oxidative conditions) for efficient oxygen delivery to the cathode mixture
  - Applying oxygen flow in two-folds: purging gas and sheath gas
- Continue supporting battery research community (National Laboratories, Universities, Companies) by providing and making available advanced cathode materials
  - Commercially unavailable hydroxide precursor materials (e.g.; NMC hydroxides)
  - Commercially unavailable cathode compositions (e.g.; LNO-based cathodes, Li/Mn-rich cathodes)
  - Commercially unavailable cathode particle sizes (e.g.; good performing small particles (D_{50}= 3-8 µm))
  - Continue on synthesis of high-Ni NMC by design (e.g.; in-situ calcination → modelling)
    - Identify the impact of size/morphology (porosity) of hydroxide precursors on the sintering process in synthesis of NMC811
      - 3 batches of precursors of different porosity and general characterization (BAT167, ANL)
      - In situ, ex situ characterization (BAT183, BNL)
      - Modelling of morphology evolution (BAT402, ANL)
- Scale up and process optimization of promising new cathode materials
  - Li/Mn-rich cathode chemistries with dopants (BAT251, BAT252, BAT253, BAT030)
  - Dopants using modified co-precipitation techniques
  - Suggestions are welcome for scaling up newly invented, promising battery materials

Any proposed future work is subject to change based on funding levels.

SUMMARY

Several Different Cathode Materials: Synthesis, Scale up and Delivery

- Synthesized and scaled up commercially unavailable cathode precursor materials
  - Materials synthesized using 1L Taylor Vortex Reactor (TVR) in **FY20**, were scaled up using 10L TVR in **FY21**:
    - $Ni_{0.95}Co_{0.05}(OH)_2$ (scaled up), $Ni_{0.95}Mn_{0.05}(OH)_2$ (preliminary scale up; under optimization)
    - Reproduced materials; $Ni(OH)_2$, $Ni_{0.90}Co_{0.05}Mn_{0.05}(OH)_2$, $Ni_{0.80}Co_{0.10}Mn_{0.10}(OH)_2$, $Ni_{0.60}Co_{0.20}Mn_{0.20}(OH)_2$
    - Calcination studies focusing on optimal Li ratio and optimal temperature
    - Materials were delivered to collaborators and are being reported in FY21 (BAT030, BAT402, BAT251, BAT252, BAT253, BAT183, BAT164, BAT377)
  - New materials were introduced to the Low-Co Deep Dive Program (BAT251, BAT252, BAT253) in FY21:
    - $Ni_{0.95}Co_{0.025}Mn_{0.025}(OH)_2$ (optimized & scaled up)
    - $Ni_{0.95}Mn_{0.05}(OH)_2$ (under optimization)
    - $Ni_{0.60}Co_{0.04}Mn_{0.35}Al_{0.01}(OH)_2$ (preliminary synthesis at 1L TVR)
    - Comparative studies on both co-precipitation and calcination methods: CSTR vs TVR and box furnace vs tube furnace ($LiNi_{0.95}Mn_{0.05}O_2$)
    - Pretreatment of hydroxide precursors: $Ni_{0.9}Mn_{0.1}(OH)_2$; $Ni_{0.9}Co_{0.1}(OH)_2$; $Ni(OH)_2$; $Ni_{0.95}Co_{0.05}(OH)_2$
  - Calcination temperature; recipe; atmosphere ($LiNiO_2$ material)
    - Temperature; O_2 gas flow; varying ratios of O_2+N_2 gas flow
  - NCM622, NCM811 and $LiNiO_2$ cathode materials at different particle sizes (3-5-10 µm) (NU, Volexion)
  - NCM622 (~10µm) & NCM811 (~8µm) hydroxide precursors (Chris Johnson, ANL)
  - NCM523 (~5µm) & NCM622 (~7µm) & NCM811 (~6µm) hydroxide precursors and co-precipitation liquid waste (NUMiX Materials)

ACKNOWLEDGEMENTS AND CONTRIBUTORS

- Continuous support from Peter Faguy and David Howell of the U.S. Department of Energy’s Office of Vehicle Technologies is greatly appreciated.
- Contributors
  - Kris Pupek
  - Shankar Aryal
  - Nick Sovronec
  - Gerald Jeka
  - Andrew Turczynski
  - Jason Croy
  - Adam Tornheim
  - Jihyeon Gim
  - Eungje Lee
  - Jinhyup Han
  - Venkat Srinivasan
  - Pallab Barai
  - Andrew Jansen
  - Steve Trask
  - Bryant Polzin
  - Alison Dunlop
  - Fulya Dogan Key
  - Daniel Abraham
  - Arturo Gutierrez
  - Marco Rodrigues
  - Seoung-Bum Son
  - Jianlin Li (ORNL)
  - Alexander Kukay (ORNL)
  - Feng Wang (BNL)
  - Jianming Bai (BNL)
  - Zheng Chen (UC SD, ReCell)
  - Kyu-Young Park; Prof. Mark Hersam’s group (Northwestern University)
  - Ted Seo (Volexion)

Samples request and further information:

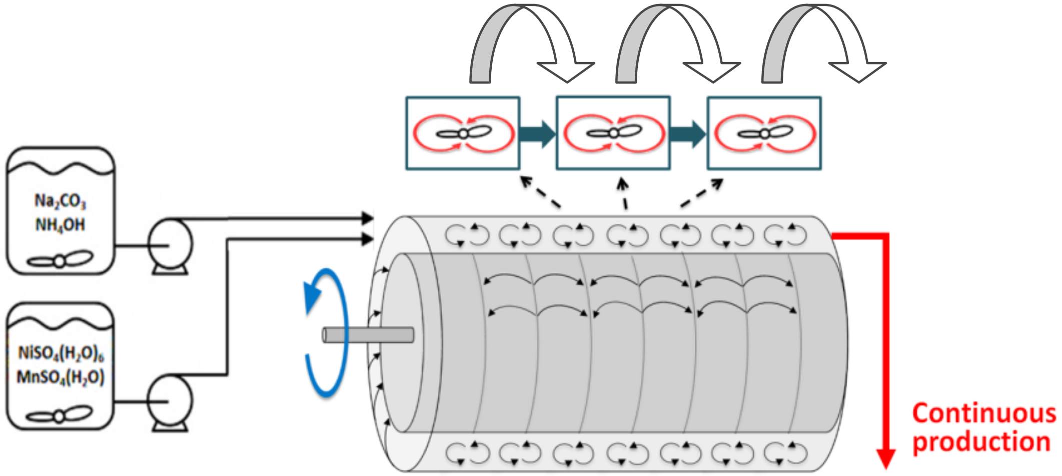
okahvecioglu@anl.gov

TECHNICAL BACK-UP

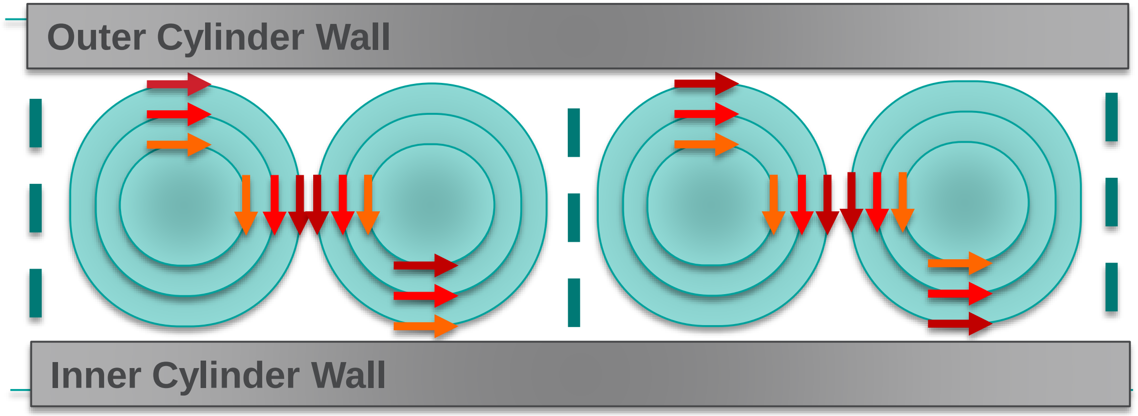
EMERGING MANUFACTURING PROCESS: TAYLOR VORTEX REACTOR

TVR

- Simplified operation
- Product uniformity
- Shorter residence time
- Plug-in flow + Tank



Each unitary vortex cell : enabling micro-mixing



<u>Homogeneous intense micro-mixing zone: faster reaction kinetics</u>

- ➢ High mass and heat transfer: high degree of uniform supersaturation
- ➢ Self particle size control: high fluid shear→ breakage and re-dispersion
- ➢ No dead-zone : improvement of purity, morphology, particle size & distribution, degree of crystallinity

➢ Key variables affecting fluid motion are hydrodynamic intensity and dimensions of Taylor vortex

Rotation speed (hydrodynamic intensity) — Gap size (dimensions)

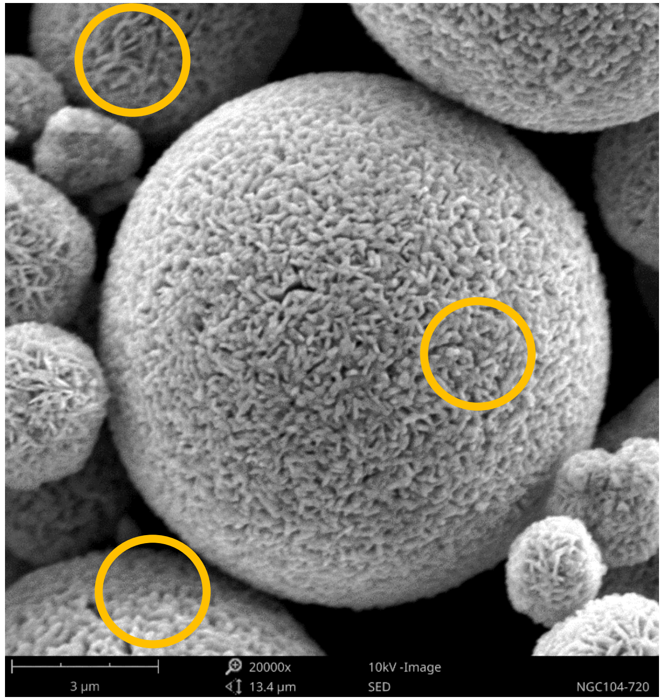
Argonne NATIONAL LABORATORY

20

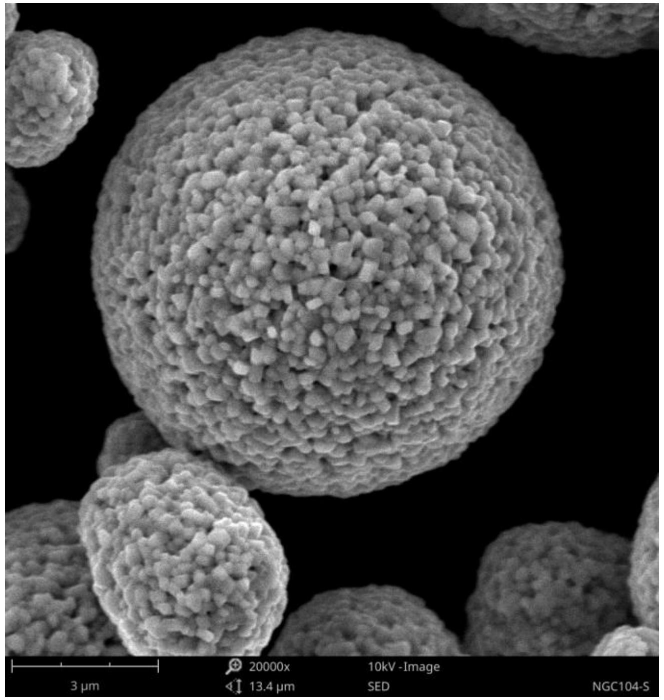
BACKUP

Effect of Calcination Method: Tube Furnace vs. Box Furnace

- An approach to understand the heat profile and lithiation homogeneity in both calcination furnaces;
  - **TVR-made** $Ni_{0.95}Mn_{0.05}(OH)_2$ material was mixed with $LiOH.H_2O$ using an acoustic mixer and calcined at T_{calc} = 720°C under pure O_2 flow for 12 hours



Tube Furnace



Box Furnace

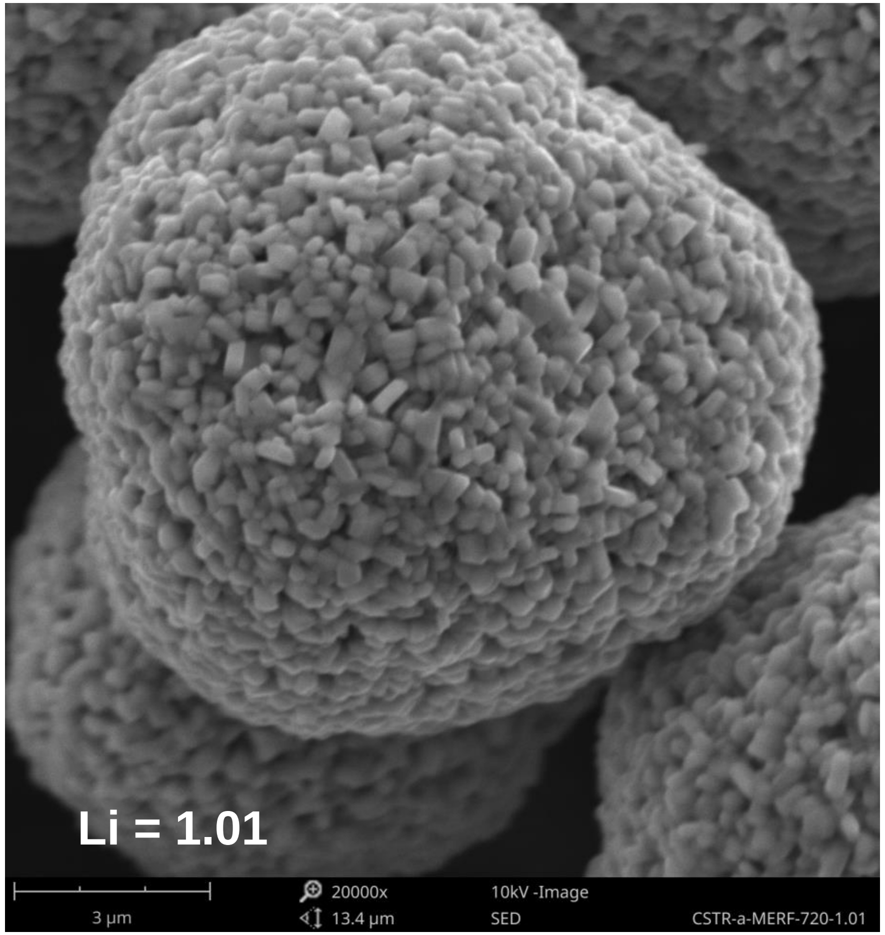
➢ (In)Homogeneous Li-salt + TM-hydroxide physical-mixing prior to calcination: leading to inhomogeneous lithium diffusion

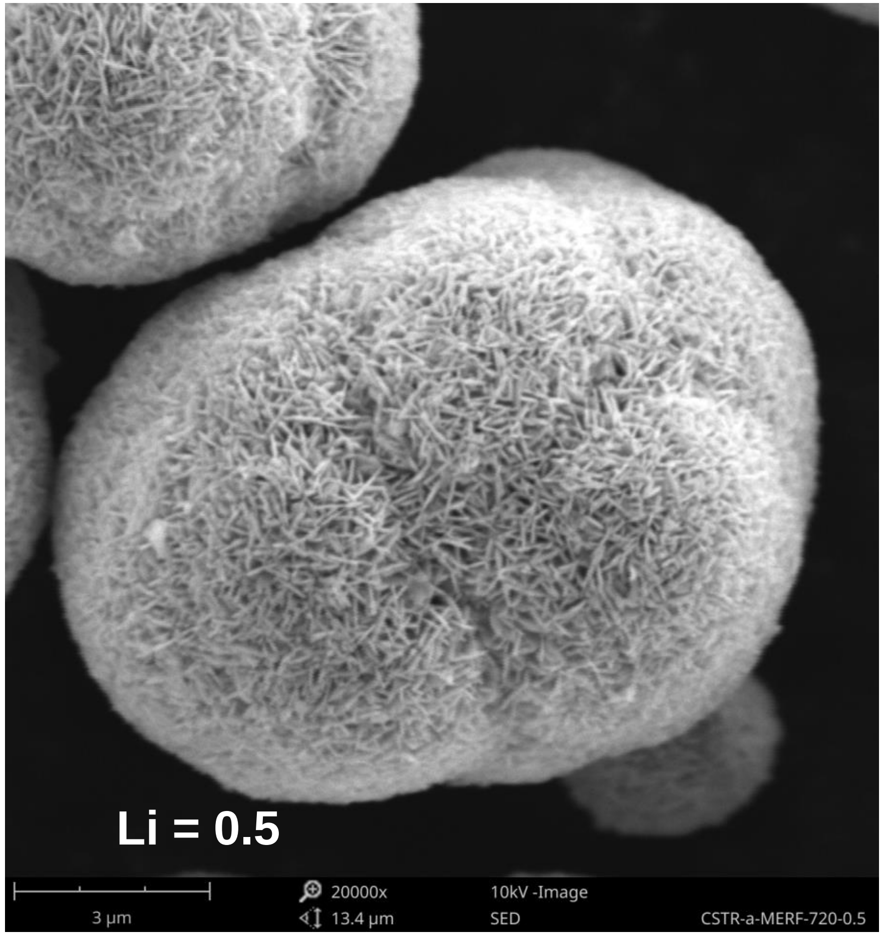
- Calcination in tube furnace shows inhomogeneous primary particle morphologies throughout the batch; grain-like primaries along with preserved-plate like morphologies
- Temperature profile in box furnace was tested using ceramic doughnuts (Range: 500-900°C) → heat distribution was homogeneous
- The lithiation throughout the batch for box furnace calcination is homogeneous and primary particle morphologies shows grain-like structures
- This material is still under optimization

BACKUP

Effect of Li ratio on Primary Particle Morphology

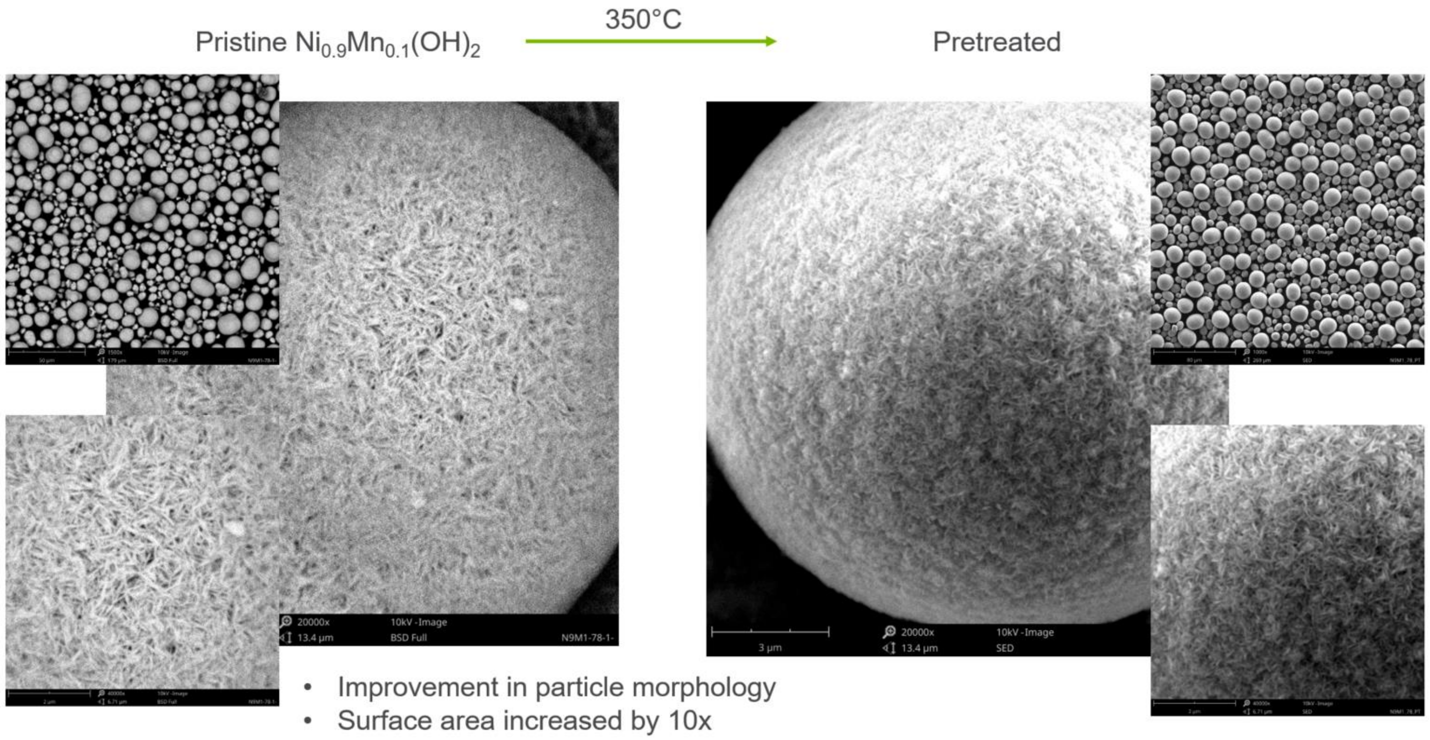
- In order to understand the inhomogeneous morphologies of primaries and lithiation in cathodes of $Ni_{0.95}Mn_{0.05}$ chemistry; two samples were prepared by mixing $LiOH.H_2O$ and **CSTR-made** $Ni_{0.95}Mn_{0.05}(OH)_2$ precursor at 1.01 & 0.5 ratios. The mixtures were calcined in a box furnace at 720^o C under oxygen flow for 12 h.

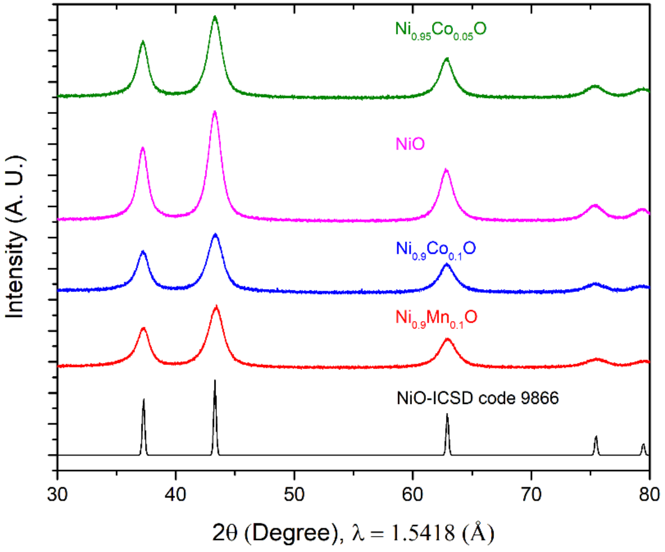




- Degree of physical homogenous mixing; Li ratio and calcination temperature and atmosphere are effective parameters on primary particle morphology; porosity; and ultimately the e-chem performance
- Under lithiated material (Li = 0.5) preserved its plate-like primary morphology as similar to the its hydroxide precursor

Pretreatment (PT) of hydroxide precursors



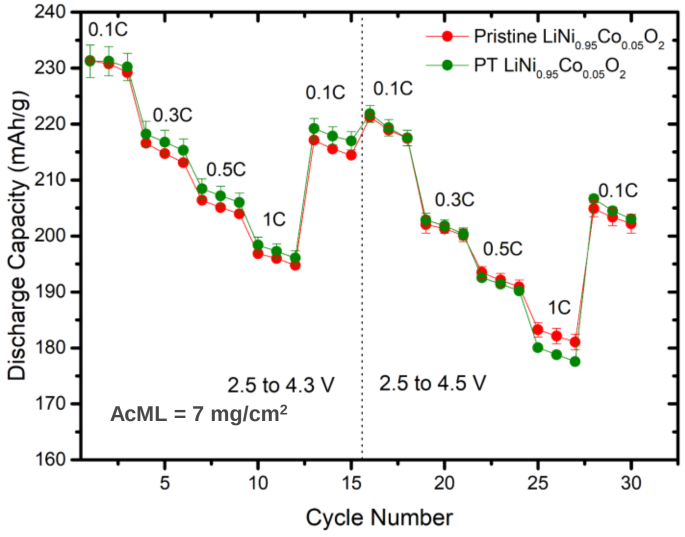


All PT oxides matched with NaCl rock salt structure (NiO)

Pretreatment of precursors @350°C for 5 h under O_2 flow (heating/cooling under O_2)

Precursors studied include: $Ni_{0.9}Mn_{0.1}(OH)_2$; $Ni_{0.9}Co_{0.1}(OH)_2$; $Ni(OH)_2$; $Ni_{0.95}Co_{0.05}(OH)_2$

- While the morphology improvement (sphericity) observed for pretreated $Ni_{0.9}Mn_{0.1}O$ precursor; no apparent changes were observed for others in terms of morphology
- All showed increased surface area by at least ~10x → by the conversion from hydroxide to oxide; losing the water content



No apparent improvement is observed for the rate capability of $LiNi_{0.95}Co_{0.05}O_2$ after pretreatment