# Developing Next-Generation Multimodal Chemical Imaging Capability by Combining STEM/APT/STXM/HIM

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Pacific Northwest NATIONAL LABORATORY
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Project 1.9; October 2012 – September 2015

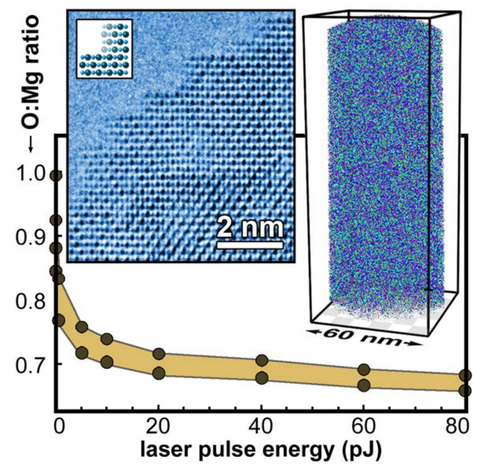
## Purpose

- Understand structure, composition, and charge state of Li and Na ion battery cathode materials at sub-nanometer spatial and chemical resolution and ppm-level mass sensitivity
- Develop a common analysis platform for integrating aberration-corrected transmission electron microscopy (TEM)/scanning TEM (STEM) tomography with atom probe tomography (APT), scanning transmission X-ray microscopy (STXM), and helium ion microscopy to understand complex materials for energy storage

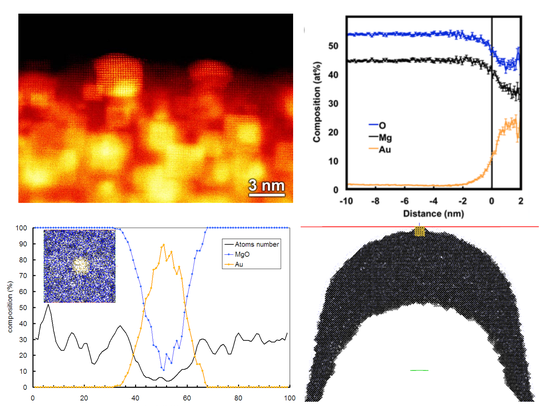
## Key Successes

- Two invited talks: M&M 2013 and AVS 2013, 1 contributed talk MRS 2013
- First publication on direct coupling of aberration-corrected TEM and APT in *Journal of Physical Chemistry Letters*. 1 more manuscript under review and 2 more under preparation
- Submitted full research proposals for DOE Vehicle Technology Office and International Energy Technology's Network of Experts in Energy Technologies (NEET)

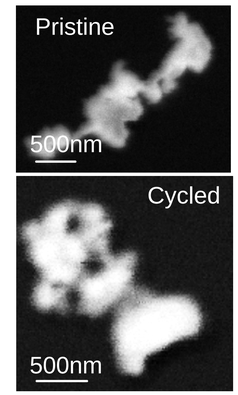
## Research Accomplishments




Understanding laser-oxide interaction during APT analysis by direct coupling of aberration-corrected TEM and APT analysis
Devaraj A et al. 2013. *Journal of Physical Chemistry Letters* 4(6):993-998.

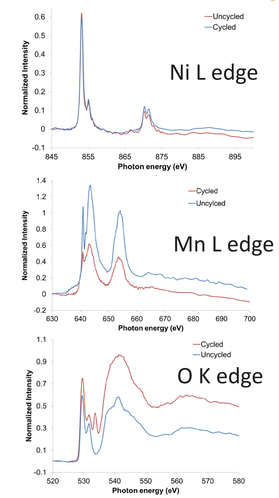



Direct coupling of aberration-corrected STEM with APT analysis and computational modeling to understand local magnification artifacts during APT analysis of complex heterogeneous metal-dielectric composites (Manuscript in preparation).

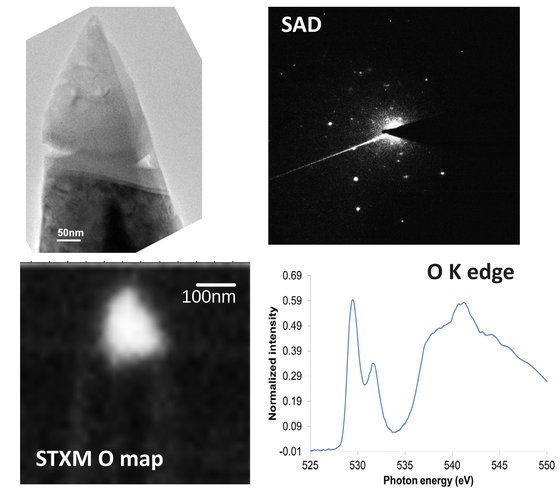



STXM Ni map of Li-ion battery cathode material: $Li_{1.2}Ni_{0.2}Mn_{0.6}O_2$ nanoparticles

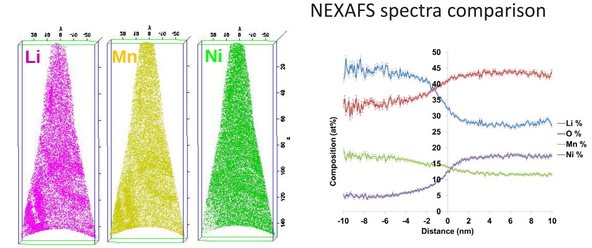
### STXM-TEM-APT analysis of Li-ion battery cathode materials



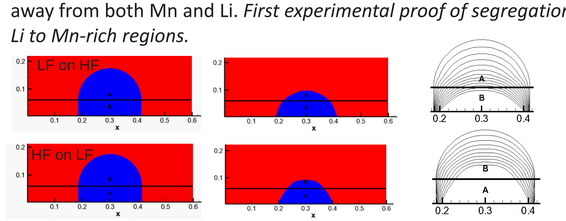

NEXAFS spectra comparison




Results of direct coupling of TEM-STXM: $Li_{1.2}Ni_{0.2}Mn_{0.6}O_2$ nanoparticles were analyzed by the multimodal capability for performing TEM imaging, diffraction, and STXM. APT analysis of the same sample is currently in progress.




APT analysis results of pristine $Li_{1.2}Ni_{0.2}Mn_{0.6}O_2$ nanoparticles showing segregation of Li toward regions with Mn segregation. Ni segregating away from both Mn and Li. *First experimental proof of segregation of Li to Mn-rich regions.*




Level set simulations for understanding tip shape evolutions during APT analysis of heterogeneous materials. Important for high-fidelity reconstruction of APT data and studies about evaporation laws.

## Next Steps

- Extend and refine the use of STXM-TEM-APT multimodal capability for analyzing phase transformations in Na-ion battery cathode materials to ultimately understand mechanism for capacity loss on cycling
- Conduct experiments for combining TEM tomography with STXM, APT, and computational modeling
- Develop discrete and compressed sensing tomography algorithms

PNNL-SA-96746