

**Structural evolution of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode material by low temperature (S)TEM**

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$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  (LNMO) is a promising cathode material for the new generation of high voltage lithium-ion batteries. However, various factors such as oxidation state, atomic coordination of Mn and Ni cations and the morphology of the particles influence the performance of LNMO as cathode material [1], [2].

Previous results have shown the presence of different crystallographic phases (spinel,  $\text{Mn}_3\text{O}_4$  – like, rocksalt) at different state of charge using high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) imaging techniques [3] which is mainly sensitive to Mn and Ni in LMNO. However, the phase transition from spinel to rocksalt phase was found to be due to the electron beam irradiation in  $\text{LiMn}_2\text{O}_4$  (LMO) having the same spinel structure as LNMO. In this work, our purpose is to investigate structural and chemical changes in LNMO at different states of charge that are due to electrochemical cycling and not due to the electron beam exposure. To do so, high-resolution STEM and electron energy loss spectroscopy (EELS) investigations at cryo-T are implemented and prove to delay electron beam degradation. Careful STEM characterizations at low temperature allowed to follow the structural and chemical changes due to the cycling and separate them from the electron beam induced transformations. Our results indicate room temperature TEM characterizations lead to misinterpretation of the degradation mechanism.

- [1] H. Duncan *et al.*, *Chem. Mater.*, vol. 26, no. 18, pp. 5374-5382, Sep. 2014.
- [2] R. Qiao, *et al.*, *J. Phys. Chem. C*, vol. 119, no. 49, pp. 27228-27233, Dec. 2015.
- [3] M. Lin *et al.*, *Chem. Mater.*, vol. 27, no. 1, pp. 292-303, Jan. 2015.