# Study of Lithiation Dynamics in Cathode Materials by in situ TEM Electrochemical Liquid Technics.

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Lithium-ion (LIB) batteries' technology has evolved rapidly to be on par to the increasing demands of the market. The EV (Electric Vehicle) market requires batteries presenting high safety, higher energy density and duration. For instances, one of the cathode materials suitable for EV application is NMC 811, however, its accelerated degradation leads to a short battery life. For the further development of EV suited batteries, the understanding of NMC 811 degradation dynamics is crucial.

For a proper study of the dynamics of (de)lithiation in NMC 811 cathode material, an in-situ analysis at the primary particle scale is proposed. The electrochemical liquid TEM analysis configuration [1] allows the performance of battery cycling inside the TEM for imaging at different SoC (state of charge), inducing less of perturbations during data acquisition and imaging analysis. The information obtained could reveal the behavior of lithium inside the crystallographic, as well as other phenomena that lead to battery degradation.

One of the main causes of degradation in NMC 811 is due to the formation of a passivation layer during cycling, the CEI (cathode electrolyte interface) [2]. The CEI formation take place during the first cycles of the batterie, at the interface between the active material and the electrolyte, organic and inorganic compounds (LiF, LiOH, Li2Co3, etc.) are formed, consuming active Li which leads to the reduction of the batter capacity and degradation of the electrolyte. The CEI layer is not stable, dissolution and reformation of it occurs during each cycle, reducing the batterie life. STEM-EDX and 4D-STEM microscopy technics can be used to follow the formation of the CEI.
4D-STEM technique allows us to obtain structural information based on electron diffraction pattern collections (50k patterns) with a spatial resolution of 1-2 nm. [3-5]. Using this method, it is possible to reconstruct it via pattern matching, a phase mapping of the present inorganic components in a CEI as has already being done for ex situ analysis of NMC 811 (figure 1) [2]. Coupling 4D-STEM and STEM-EDX analysis for different SoC (State of Charge) will allow us to follow the behavior of the CEI layer and determine the dynamics behind it.
The obtained information could lead to the comprehension of the strains present at the primary particle level that could eventually lead to the fracture

of secondary particles and the degradation of the material as reported several times in literature.

**Graphic:**

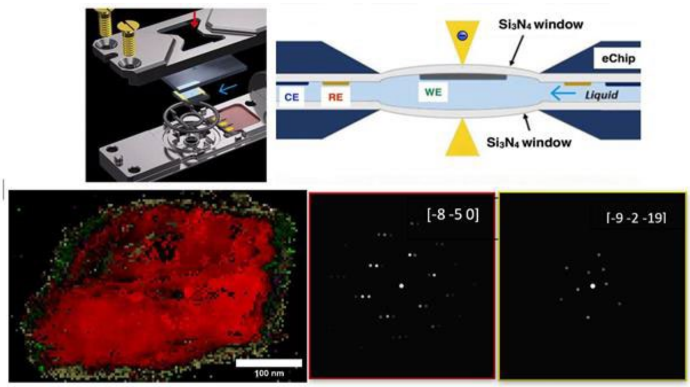


Figure 1 : a) Poseidon sample holder and schematics of liquid electrochemistry cell, b) 4D-STEM ex situ analysis for a NMC811 particle cycled in coin cell, Diffraction pattern corresponding to c) NMC 811 and d) LiF, after pattern matching determination.

**Keywords:**

In-situ, 4D-STEM, NMC, CEI

**Reference:**

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