In situ 3DED in liquid to study the structural transformation of functional materials during electrochemical reactions; challenges and progress

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3D electron diffraction (3D ED) is used to study the crystal structure of functional nanomaterials that are too small to study with X-ray or neutron single crystal diffraction. Recently, 3D ED has been employed in situ to show the structural transformation of nanomaterials during phase transformations upon reactions such as heating and oxidizing and reducing reactions [1-2]. However, 3D ED still faces several challenges to be implemented in electrochemical reactions. In the presence of the liquid, the quality of the data will be decreased because of scattering by the liquid, large inelastic scattering and reactions between the beam and the liquid. In literature, the crystal structure transformation of LiFePO4 upon charging in situ in liquid was studied by decreasing the liquid thickness using the beam shower method [1]. However, this method cannot be employed to study the structure transformations across several cycles as it changes the properties of the electrolyte dramatically.

Here, we used a low dose method to acquire data on LiNi0.6Mn0.2Co0.2O2.5 in the liquid environment with 3D ED without changing the electrolyte properties. LiNi0.6Mn0.2Co0.2O2.5 is a cathode material for lithium-ion batteries. We will discuss the quality of the data collected in the liquid environment compared with the normal 3D ED of the crystal, the adaptations of the data acquisition strategy to optimally acquire the data from the sample and the remaining challenges to perform electrochemical measurements along with 3D ED.
