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Phase transition of nanoparticulate LiFePO₄ during high rate cycling

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A fundamental understanding of an electrode material requires the elucidation of its phase transformation mechanism during charge and discharge. Ex situ methods, which are carried out under equilibrium condition, have been widely used in characterizing the thermodynamic phases at different states of charge, from which a thermodynamic phase transformation pathway can be constructed. However, ex situ measurements do not always reflect the process occurred in an operating battery as the non-equilibrium operating condition might result in deviations from the thermodynamic process, especially for high-rate materials, such as LiFePO₄, which is predicted to exhibit a fundamentally different phase transformation process at high rates [1,2]. To probe the process at high rate, an in situ method with reasonable temporal resolution must be employed. In this work, the high rate galvanostatic cycling process of LiFePO₄ nanoparticle electrode in a customised AMPIX cell [3] was investigated in situ by time-resolved synchrotron X-ray powder diffraction. Formation of continuous non-equilibrium solid solution phases between LiFePO₄ and FePO₄ was observed at 10 C rate. The in situ diffraction patterns were analysed by a refinement strategy that accounts for the asymmetrical diffraction peak profiles due to Li composition variations.

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