

Supporting Information

Nanoscale Detection of Intermediate Solid Solutions in Equilibrated Li_xFePO_4 Microcrystals

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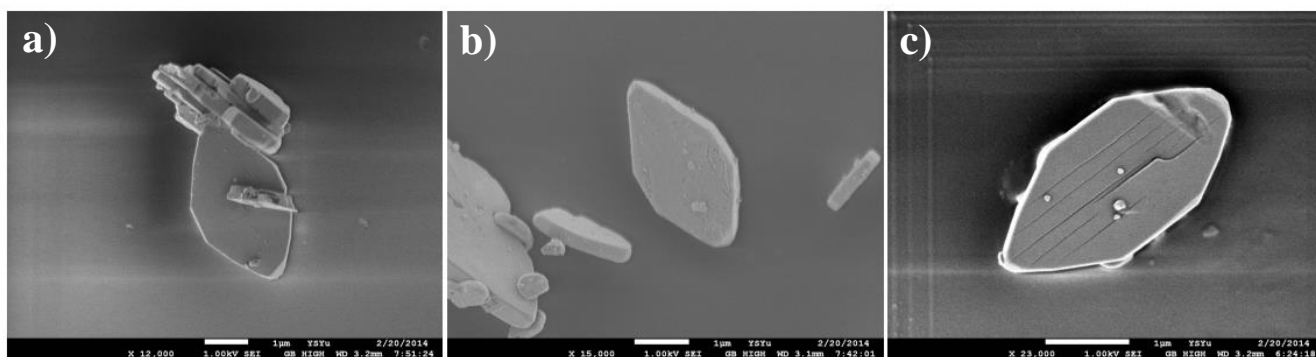


Figure S1. Scanning electron microscope images of the Li1(a), Li0.5 (b), and Li0 (c) particles used in this study.

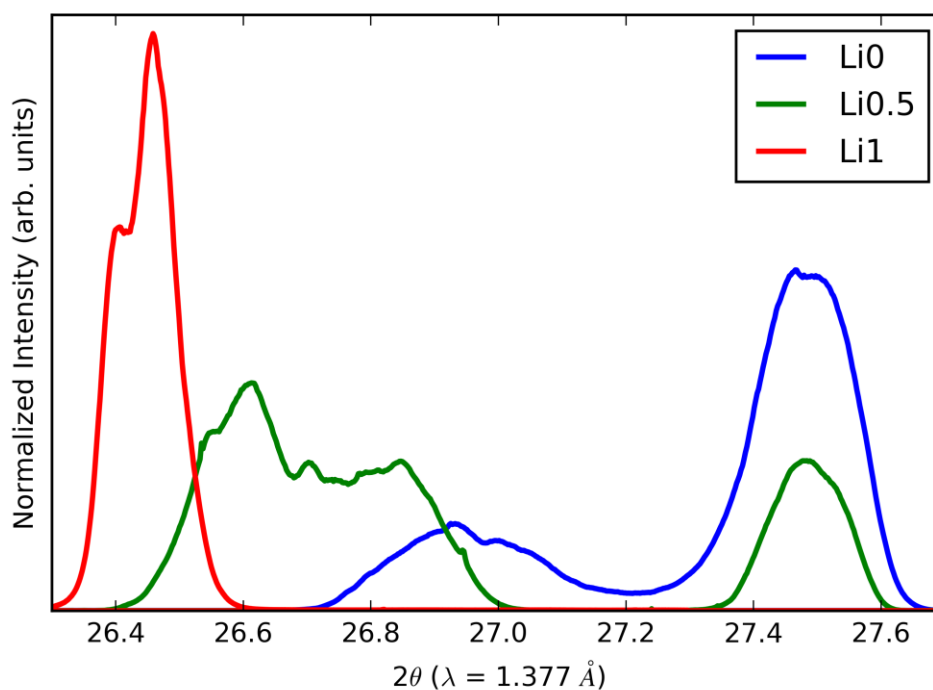


Figure S2. Intensity vs. 2θ plots for the summed diffraction data for each particle analyzed by SXDM. The Li1 (red) is found to contain one phase. The Li0.5 (green) displays a broad range of intensity between lithiated and an intermediate value of $\sim\text{Li}_{0.6}\text{FePO}_4$ (low angle), with a miscibility gap between the intermediate and delithiated phases. The Li0 (blue) sample also contains intermediate phases and the delithiated phase.

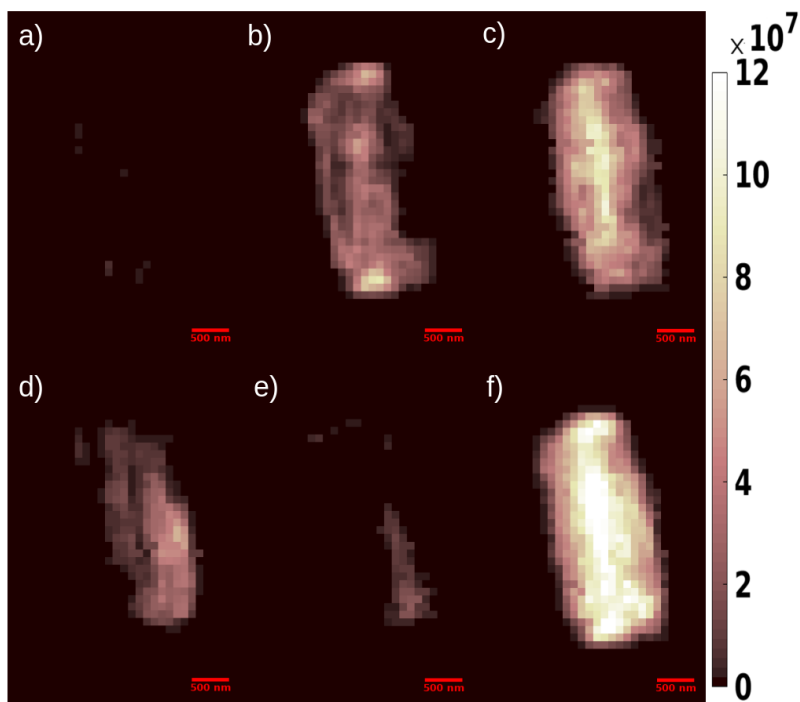


Figure S3. Individual scans of the Li1 particle at different angles (a-e). Also shown is the composite image (f) from the individual scans.

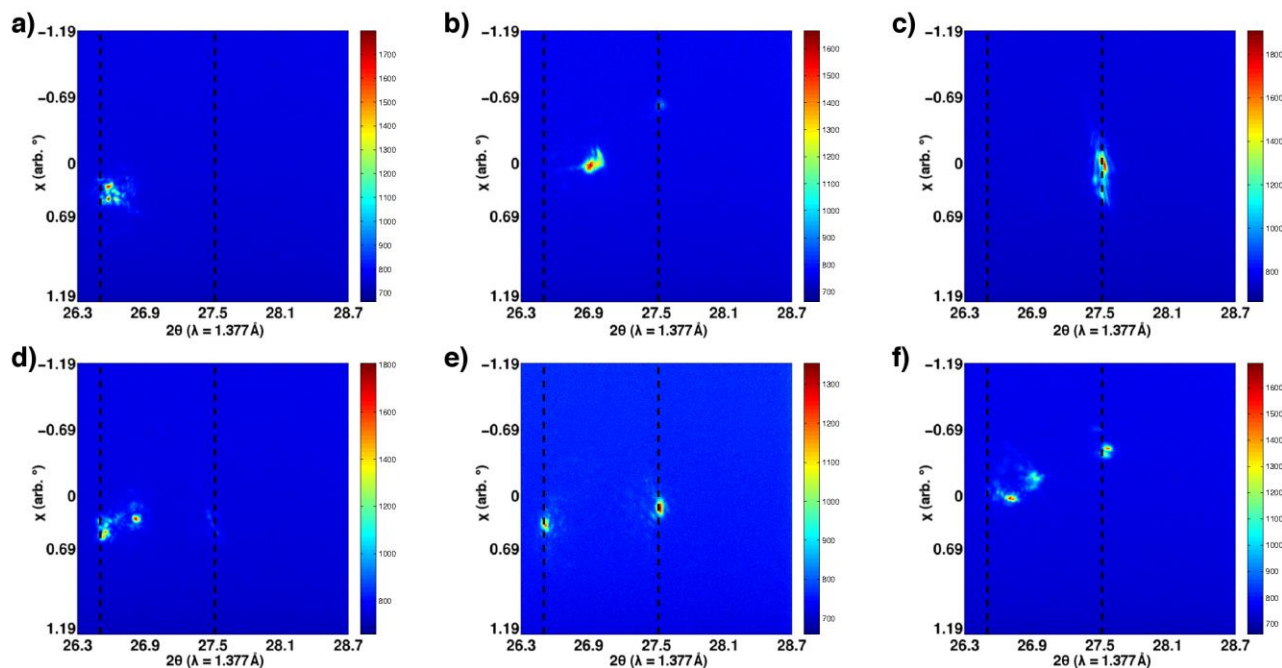


Figure S4. Individual diffraction patterns representative of the phases present in the Li0.5 sample. Patterns pertaining to lithiated (close to LiFePO_4) (a), intermediate (b), and delithiated (essentially, FePO_4) (c) phases are shown, as well as ones that include a mixture between lithiated and intermediate (d), lithiated and delithiated (e) phases, and three different phases (f). The patterns highlight the variability in 2θ values between 26.4° and 26.9° , reflecting the broad peaks in the summed patterns of Figure S2. Dashed lines denote the literature values for LiFePO_4 (left) and FePO_4 (right) in each image.

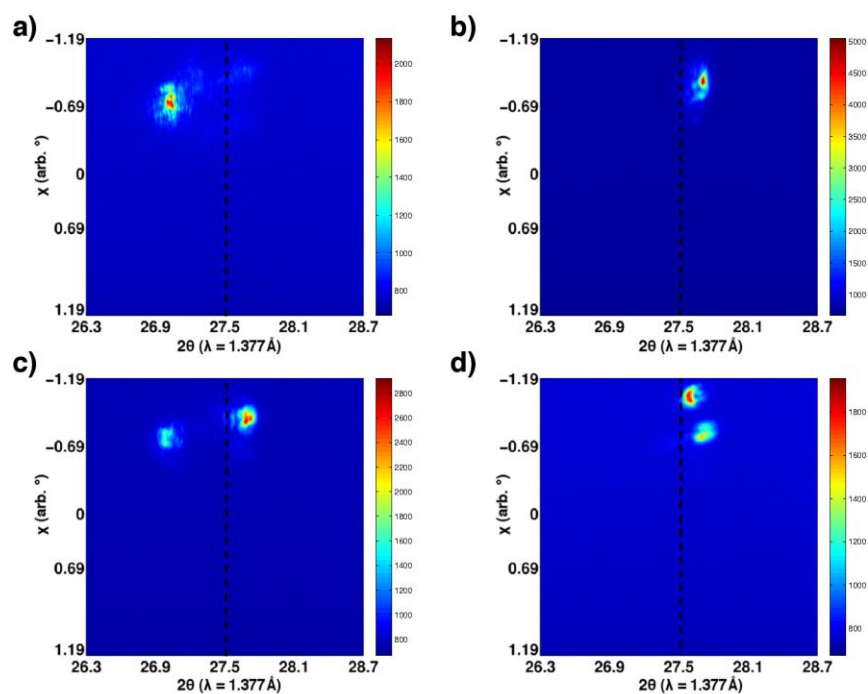


Figure S5. Individual diffraction patterns representative of the phases present in the Li0 sample. The intermediate (a) and delithiated (b) phases are shown, as well as a location containing both phases (c), and one containing multiple orientations of the delithiated phase (d). Dashed lines denote the literature value for FePO_4 .