Poster Presentation

MS79.P01

A New Electrochemical Cell for Rietveld Refinements of In-Situ Powder ND Data

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In-situ techniques proved to be exceptionally useful tools to understand electrode materials for Li-ion batteries (1). However in-situ neutron diffraction (ND) knew a slow development, due to the intrinsic difficulties it held (2). The two main limiting factors are: the high amount of electrode active material required for ND, which makes electrochemistry challenging; and the strong scattering coming from the different parts of the battery (current collectors, protective case, organic solvent-based electrolytes…). To overcome these problems, we have designed a new electrochemical cell, manufactured with a completely neutron-transparent (Ti,Zr) alloy. Used with deuterated electrolytes, the cell was able to combine good electrochemical properties and the ability to collect ND patterns operando, with good statistics and no other Bragg peaks than those of the electrode material of interest. This allows detailed structural determinations by Rietveld refinement during operation. The cell was validated using well-known battery materials such as LiFePO4 and Li1.1Mn1.9O4 (3). Li batteries using our in-situ cell were assembled and measured on the D20 high flux neutron powder diffractometer (ILL Grenoble). Rietveld refinements were used to analyze the data and the results showed good agreement with the expected values (obtained from the same materials measured as powders on the D2B high-resolution powder diffractometer). A real operando experiment was also conducted (Figure), where the active material LiFePO4 was charged (Li+ extracted) to reach FePO4 while collecting ND patterns (1 hour each for 24h of charge). Details can be found in (3). The cell is now ready to be used to study new challenging materials. As an example, we measured the charge operando of a series of spinel materials Li1+xMn2-xO4 (x=0, 0.05, 0.1). From the data, we observed how the material evolution upon charge strongly depends on x. Results from the refinements of these data will be reported and discussed.

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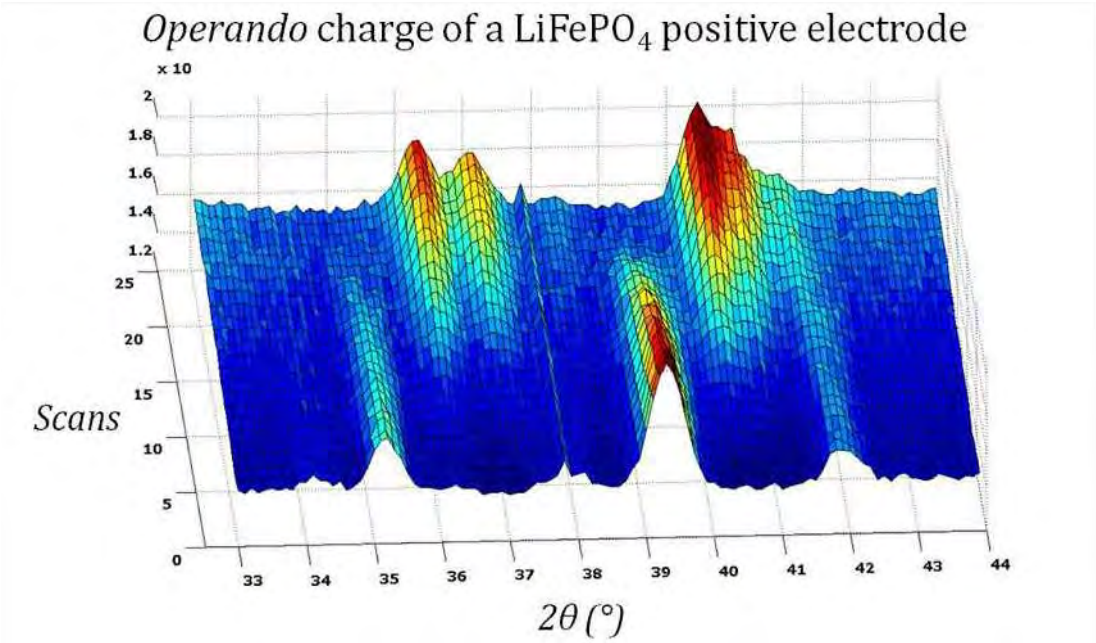

Keywords: Neutron diffraction, In-situ, Lithium batteries