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Lithium Quantification in Lithium-Ion Batteries Using Operando Neutron Depth Profiling

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Commercial Lithium-Ion Battery (LIB) cells are mostly based on graphite as anode material. During the first inter¬calation of Li into graphite, the electrolyte gets reduced at the anode, forming a nm-thick surface layer, the so-called solid electrolyte interphase (SEI). The SEI stops further electrolyte reduction but consumes Li during its formation. Neutron depth profiling (NDP) is a non-destructive technique and a suitable tool to measure Li concentrations as a function of depth. When irradiating the sample with a cold neutron beam, 6Li nuclides emit charged particles after neutron capture. The residual energy and the signal rate of the emitted 3H particles are correlated to depth and amount of Li. Thus, SEI growth and Li (de)intercalation in graphite anodes can be studied up to a depth of ca. 30 µm. Here, we present operando NDP data for the first charge/discharge cycle of a graphite anode vs. a LiFePO4 cathode, using a custom-designed coin cell casing with 0.5 mm diameter holes which are sealed with a 7.5 µm Kapton window. We will demonstrate that the cycling performance of the operando cell is comparable to a standard laboratory cell, show that it was possible to quantitatively track the Li concentration across the graphite electrode during cycling, and thus to correlate the amount of Li in the SEI layer with the first cycle irreversible capacity.1

1 Linsenmann, Trunk, Rapp, Werner, Gernhäuser, Gilles, Märkisch, Revay, Gasteiger, J. Elec. Soc. 167 (2020) 100554.

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