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Contrast Matched SANS for Observing SEI and Pore Clogging in Silicon-Graphite Anodes

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Silicon-graphite (SiG) electrodes are attractive candidates as anodes for Li-ion batteries due to their high theoretical specific capacity. However, repeated lithiation/delithiation during charge/discharge cycling causes significant morphological changes of the silicon particles. This results in the formation of highly porous silicon structures and severe side reactions at the silicon/electrolyte interface. To quantify these morphological changes, small-angle neutron scattering (SANS) was applied with selective contrast matching of Si nanoparticles (200 nm diameter) and the surrounding electrolyte decomposition products. Using electrolytes consisting of 1.5 M LiPF6 dissolved in either deuterated or protonated ethylene carbonate (EC) resulted in solid-electrolyte-interphase (SEI) compounds with scattering length densities either matching or mismatching that of the Si nanoparticles. SiG anodes with 35 wt% silicon nanoparticles were aged for 10 and 20 charge/discharge cycles against capacitively oversized LiFePO4 cathodes. Afterwards, the morphological changes and size distribution of the SEI compounds were evaluated by means of ex-situ SANS measurements of the SiG electrodes in their fully discharged state. Transmission electron microscopy (TEM) images of the pristine and cycled silicon nanoparticles complement the interpretation of the SANS analysis.

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