

Imaging dynamic Li electrode processes via an *in-situ* TEM liquid cell

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The electrode/electrolyte interface in electrochemical devices is largely studied by indirect methods, but advances in microfabrication allow the design of a miniature electrochemical cell to be used in a transmission electron microscope (TEM). We have developed a TEM liquid cell specifically designed for imaging nanoscale electrochemistry that incorporates quantitative electrochemical control, multiple electrodes spaced for easy nanoparticle assembly, and sealable fluid fill ports for operation with air-sensitive materials. In the study of battery materials, we have focused on the electrode/electrolyte interactions on anode materials in lithium-ion batteries such as the formation, growth, and dynamics of the solid-electrolyte interphase (SEI). Filling the liquid cell with aprotic battery electrolyte containing LiPF₆ salt, lithium was clearly and controllably electroplated on nanoscale titanium electrodes under galvanostatic control, and a formation of natural and/or beam-induced SEI was visible on the lithium deposits under scanning mode (STEM) with minimal beam dose. Stripping the lithium showed direct evidence of stranded Li and SEI upon cycling such as might occur in secondary lithium batteries (e.g., Li-S or Li-air). The TEM can provide new, direct information about the structure and dynamics of battery materials and interfaces in the *in-situ* liquid environment.

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