

Experiment Brief

K3 IS Camera and Model 626 Cryo-Transfer Holder

Title

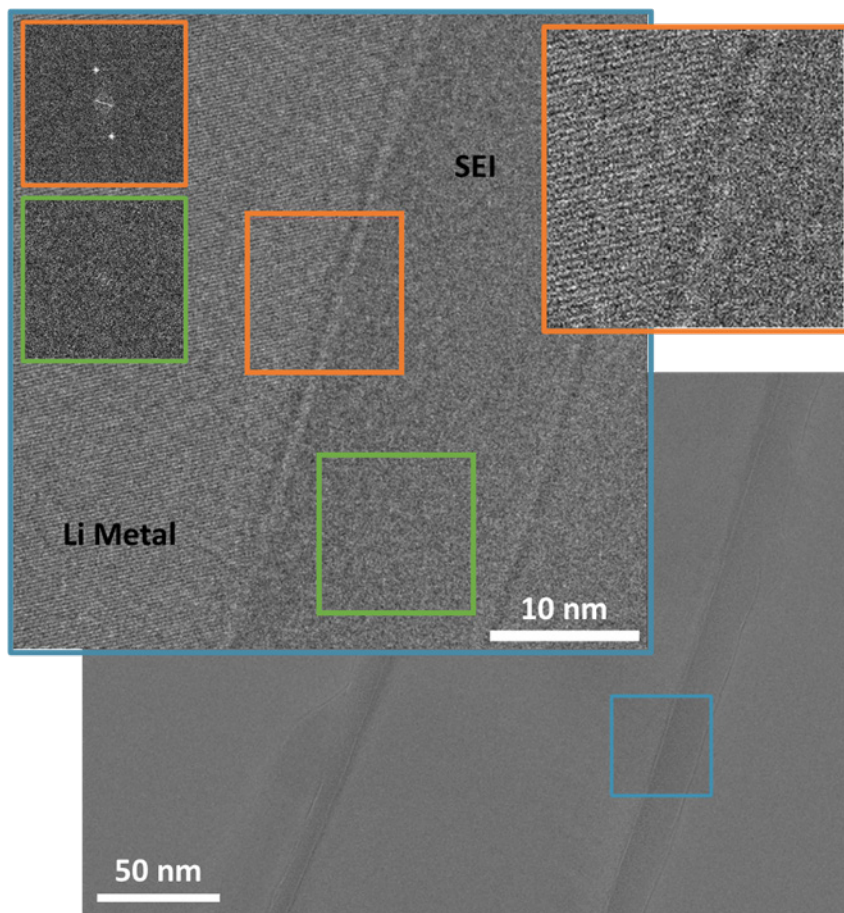
Imaging a lithium metal battery solid electrolyte interphase

Gatan Instrument Used

The K3[®] IS camera delivers simultaneous **low-dose imaging via real-time electron counting**, fast continuous data capture, and a large field of view. The model 626 cryo-transfer holder enables **frost-free low-temperature specimen transfer** and **subsequent imaging of radiation-sensitive frozen samples at high resolution** with precise temperature measurement.

Background

Developing better batteries is critical for advancing a number of technologies for energy and the environment, and lithium (Li) metal is an attractive anode material based on its fundamental properties. One of the issues limiting the use of Li metal is the formation and instability of a solid electrolyte interphase (SEI). Therefore, understanding the exact structure and structural dynamics of the SEI in new battery designs is valuable. Imaging the SEI is challenging. It is a thin and possibly heterogeneous layer, thus requiring high-resolution imaging, but it is also quite sensitive to the electron beam which makes standard HR TEM difficult. Here, we observe the SEI in a new electrolyte as described in [1].



Materials and Methods

The lithium metal in the novel electrolyte was prepared by plunge-freezing on a standard Cu transmission electron microscope (TEM) grid. The temperature was maintained below 100 K, using a model 626 cryo-transfer holder, as the sample was inserted into an image-corrected Titan ETEM. This image was collected with a K3 IS camera in counted mode and a dose rate of 40 e⁻/Å²/s. The total dose was 70 e⁻/Å². Even with a pixel size of only 0.54 Å, a large sample area could be imaged, as seen in Figure 1. Data is from the same dataset shown in Figure 4 of [1].

Summary

The TEM image in Figure 1 shows that the Li filament is a single crystal over the observed region. The SEI on the Li surface is amorphous and could be successfully imaged despite its sensitivity to the electron beam due to the low electron dose and dose rate applied, and the low temperature maintained using the model 626 cryo-transfer holder.

Figure 1. Cryo-TEM image of Li metal filament with SEI. The full field of view is shown along with a smaller region (blue) showing the amorphous SEI and its interfaces with the crystalline Li and solvent more clearly. The orange region shows the visibility of the lattice fringes that exist only within the Li filament. Green and orange insets show FFTs from the corresponding boxed regions.

Credit(s)

A special thanks to Stanford University, including Hansen Wang, Zhiao Yu, William Huang, Zewen Zhang, Zhenan Bao, and Yi Cui [1] Wang, H. et al. Dual-Solvent Li-Ion Solvation Enables High-Performance Li-Metal Batteries. *Adv. Mater.* 33, 2008619 (2021).

Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.