 https://doi.org/10.1051/bioconf/202412925050

Innovative Nanoanalytical Approaches for Lithium Metal Interface Analysis

Clementine Warres[1], Philipp Karl Albrecht[1], Dr. Tarek Lutz[1], Dr. Wilfried Nisch[1]
[1]NMI Natural and Medical Sciences Institute at the University of Tuebingen, Reutlingen, Germany

Background
Lithium metal anodes are important for next generation high-energy batteries, enhancing energy density in terms of both volume and mass while remaining cost effective. They are already implemented in lithium-sulfur batteries nowadays. However Interfacial instability reactions during operation still represent a challenge for the commercialization of solid-state batteries and require further research.

Challenges in Characterization
The analytical characterization of lithium metal interfaces with high lateral resolution is essential for understanding chemical reactions. These interfaces are highly reactive in ambient air and in particular sensitive to electron and ion beam interactions at room temperature. Employing an argon filled glove box prevents Lithium from reacting with moisture and oxygen. Nevertheless, the reaction with the residual nitrogen content in argon-flooded glove boxes is often overlooked. Additionally, conventional electron microscope air locks are typically vented with nitrogen and are therefore another nitrogen contamination source when samples were transferred between microscopes.

Methods
We deposited lithium layers and other thin test layers by thermal evaporation in a vacuum glove box and flooded afterwards with pure argon gas. With this approach we achieved an oxygen-, water- and nitrogen-free environment in the glove box. Our workflow also involves nitrogen free transfer between the scanning electron microscopes (SEM) and transmission electron microscopes (TEM) under argon atmosphere as well as cryogenic electron microscopy. Chemical characterization of the interfaces with high spatial resolution is achieved by focused ion beam secondary ion mass spectroscopy (FIB-SIMS), energy dispersive x-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS).

Results and Conclusion
We demonstrate the preservation of metallic lithium via the analysis chain. We also show the possibility of depositing additional thin test layers within the glove box. The methodology and workflows shown in this work give rise to an understanding of Li metal / coating interfaces and can be adapted to study other thin coatings or reaction products sensitive to ambient conditions.

Graphic:

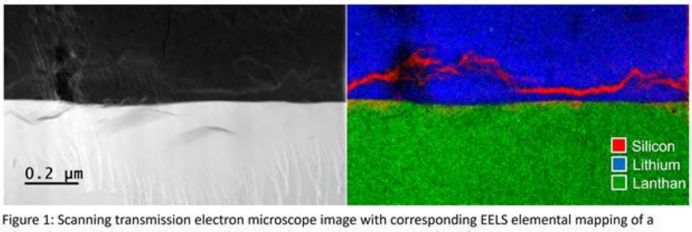

Figure 1: Scanning transmission electron microscope image with corresponding EELS elemental mapping of a lithium layer with 20 nm silicon on Lithium Lanthanum Zirconium Oxide (LLZO)

Keywords:

Li-battery, cryo-/electron-microscopy, inert-transfer,nitrogen-free, Lithium-deposition