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Cryogenic Electron Microscopy for Understanding Energy Storage

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Energy storage systems are complex, reliant on multiple simultaneous processes to occur with compatible materials to achieve high performance and longevity. For lithium ion batteries, even when these systems are constructed identically within a batch, the performance of each system can vary greatly, causing some cells to fail 100's of cycles prior to the average. [1] The understanding for these variations is only speculation, as failure analysis of these batteries is not straight forward. The cells are generally deconstructed and analyzed in pieces, instead of viewing the composite structure together. Holistic characterization of a battery in structure and chemistry has been limited, as methods for imaging within an unperturbed cell does not offer nanoscale spatial resolution or chemical mapping in three dimensions. [2]

Cryogenic freezing of materials resists damage when imaging with high energy particles and reduces localized heating which can distort light materials, including lithium. [3] In the past several years, researchers have demonstrated the use of cryogenic freezing for lithium metal characterization within an electron microscope. [4,5] One study has enabled a specimen to be sectioned from an electrode surface to characterize the liquid-solid interface between lithium and the electrolyte in both structure and chemistry. [6] This technique presents a very powerful tool for understanding the dominant mechanisms in batteries which lead to capacity loss and failure. Free access to these tools are available within a user facility at the Center for Integrated Nanotechnologies in New Mexico.

This work will review these cryogenic sectioning and imaging techniques that can be used for battery electrodes and nanoscale interfaces. Early results will demonstrate their use for carbon and lithium metal anodes. Nanoscale imaging and energy dispersive x-ray spectroscopy of cycled electrodes and their interfaces will be presented. We have observed that contrast variations from the surfaces of lithium electrodes was caused by density variations in the electrodeposited lithium. Future advancements of these tools will enable failure analysis of intact batteries, whereby the interfaces between electrodes, electrolyte, and separator will be imaged over large distances of the structure. [7]

References:

[1] SJ Harris, DJ Harris, and C Li, Journal of Power Sources **342** (2017), p. 589.
[2] S Waluś, et al., Chemistry Communications **49** (2013), p. 7899.
[3] RF Egerton, Micron **119** (2019), p. 72.
[4] X Wang, et al., Nano Letters **17** (2017), p. 7606.
[5] Y. Li, et al., Science **27** (2017), p. 506.
[6] MJ Zachman, Z Tu, S Choudhury, LA Archer, and LF Kourkoutis, Nature **560** (2018), p. 345.
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