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A Multiscale, Correlative, Air Free Workflow for the Analysis of Li Distribution in Batteries via ToF-SIMS

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The ability to link the macro and nano world has long been a challenge due to correlation and sample preparation difficulties. X-Ray microscopy is widely accepted as the standard for non-destructive interior tomography imaging of large objects with high resolution. The ability to access these interior objects for further analysis has previously been impossible due to sample damage and lack of precision in correlation when manually cutting the sample. Moreover, this technique requires careful chemical handling for systems of interest such as intact batteries which may have undergone cycling. The adaptation of a FIB-SEM with a femtosecond laser in a dedicated separate chamber has made this workflow feasible.

In this work, we present a correlative workflow whereby an intact battery is imaged with an X-Ray microscope and a specific location is targeted for ToF-SIMS analysis through interpretation of the AI reconstructed X-Ray data. To do this, we introduce a major update of the ZEISS Crossbeam Laser, the world's first commercial femtosecond laser FIB-SEM system. Significant enhancements of the laser workflow are described, including the ability to remotely operate the system via robotic sample transfer and automated vacuum handling between the laser chamber and FIB chamber. We furthermore present the ability to automatically load the laser chamber via a robotic arm. Automation of calibration procedures are also enhanced allowing for a 2 micron precision for laser milling.

A new capability of the femtosecond laser system, burst mode, further increases ablation rates whilst constraining sidewall slopes to allow much more precise cuts and therefore object creation. Laser cut finish is also improved, reducing the need for polishing with an ion beam to remove Laser Induced Periodic Surface Structures (LIPSS). This further demonstrates the requirement of a separate ablation chamber for high ablation rate processing of samples and debris handling with a gas exhaust system.

In the context of this workflow, we cut the battery open with the femtosecond laser to precisely access the region identified in the X-Ray microscope tomogram. The cut surface is then transferred automatically via robot from the laser chamber to the main FIB chamber under vacuum where ToF-SIMS analysis is performed on the pristine surface. Li concentrations of the surface are then measured [2].

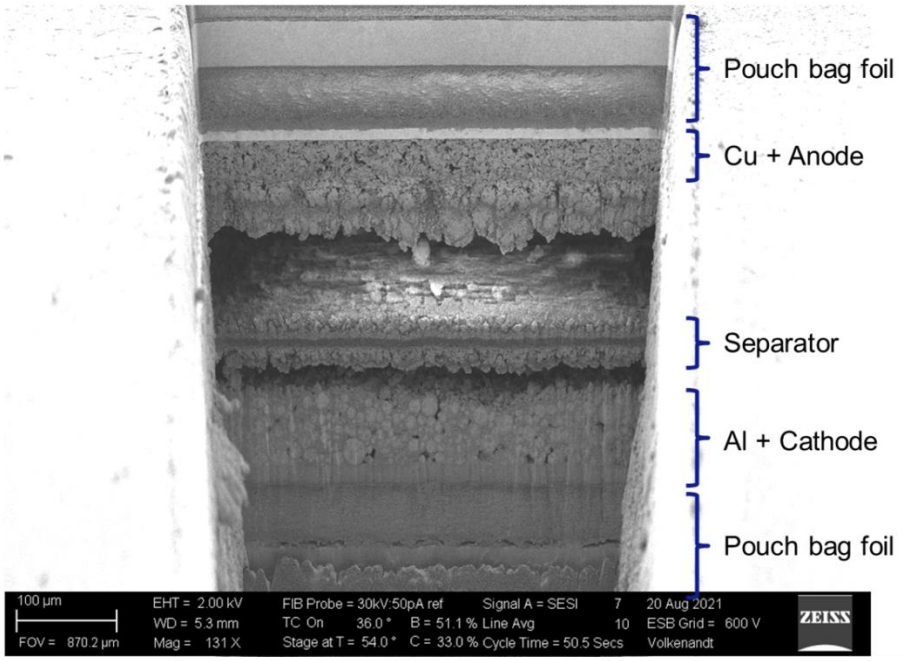

Figure 1: Cross-sectional SEM view of intact pouch cell battery opened using fs-laser mill prior to air-free transfer to the FIB-SEM instrument analysis chamber.

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[1] B Tordoff et al., Appl. Microsc. **50**(24) (2020). https://doi.org/10.1186/s42649-020-00044-5
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