3D Imaging Techniques for Li-ion Battery Research
- NASA Aerospace Battery Workshop

Zhao Liu, Materials & Structural Analysis Division
Nov 2019
Multiple Analytical Solutions for Battery Research and Production
Motivation: Understanding Microstructure-Performance Correlation of Li-ion Battery

- Understanding battery microstructure is critical for:
  - Analyzing microstructure-performance correlation
  - Identifying degradation mechanisms
  - Improving battery performance
    ✓ Energy density
    ✓ Power density
    ✓ Cycle life
    ✓ Safety
- Imaging technique is one of the key approaches for microstructural characterization
2D Imaging vs. 3D Imaging on Batteries

2D Imaging

2D imaging mainly provides qualitative understanding on battery microstructure

3D Imaging

3D imaging provides in-depth quantitative understanding on battery microstructure
Overview

• 3D Imaging Solution via microCT
  • 3D imaging on 18650 full cell via HeliScan microCT

• 3D Imaging Solution via DualBeam (FIB-SEM)
  • 3D imaging analysis of battery electrode
Introduction: HeliScan microCT

- **Stitch-free** scanning of larger volumes and regions of interest with *Helical* scanning trajectory
- **Fast acquisition** and high resolution (below 400nm) with new *high cone-angle x-ray source*
- **Highest image fidelity** with iterative reconstruction algorithm and *patented* correction software
- Built for **versatility**
HeliScan microCT 3D Imaging Workflow

1. Acquisition

2. Reconstruction

   - Iterative reconstruction on GPU-Cluster

3. Visualization & analysis

   - Avizo for Materials Science software
3D Imaging at The Cell Level: microCT

X (Y) – Z Plane

X – Y Plane
Comparing Cu Collector Shape on Each Horizontal Slice

- Extracting spirals following the Cu collector, starting and ending where the cathode starts & ends
- Segmentation is automated and performed on every x,y slice

Sample courtesy: University of College London
Geometry Extraction from the microCT Dataset

In each horizontal slice; the foil locations are tracked and co-registered to allow comparison between the new state and the cycled state.
Comparing Cu Collector Shape

Collector shape change is quantified by digitally correlating the position of the spiral in the fresh and aged battery and plotting the distance from the center versus the rotation angle $\alpha$.
Comparing Cu Collector Shape on Slice Z=2mm

At the center, the cycled battery collector is closer to the middle; this situation reverses towards the edge of the battery. It suggests an electrode volumetric expansion.
Comparing Central Rod Area

- Area around the central rod, ‘inside’ the anode

Average area:
New = 8.76 mm², Cycled = 6.78 mm² (77%)

HeliScan microCT + Avizo allows to quantify structural changes in a cycled battery
Challenges of the microCT for Battery Imaging

- Not enough resolution to see fine-structure within the electrode particles (e.g. cracks)
- Lack of chemical compositional/elemental information
- DualBeam (FIB-SEM) technique provides 3D imaging solution at electrode and particle level
What is DualBeam (FIB-SEM)?

A DualBeam is defined by two primary components...

**Scanning Electron Microscope (for imaging)**

+ **Focused Ion Beam (milling)**

The sample and stage are maneuvered beneath the beams to optimize imaging and milling.
The DualBeam in Action…

FIB

SEM
DualBeam Technologies

Increasing 3D Volume for Analysis

Helios (FIB)
- 40x40x40 μm³/hr (Si) @65nA
- Ga⁺ LMIS

Helios Hydra DualBeam
- 150x150x150 μm³/hr (Si) @2.5μA
- (~40x faster than FIB)
- Multi chem plasma source (Xe, Ar, N, O)

Helios PFIB UXe Laser
- 1200x1200x1200 μm³/hr
- (>15.000x faster than FIB, or >1000μA)
- Ultra short pulse laser (515nm./1030 nm)
DualBeam 3D Imaging Workflow

Data Acquisition

Image Processing
- Alignment
- Curtaining remove
- De-noising
- Shading correction

3D Exploration
- Volume rendering
- Slicing
- Color mapping

Segmentation
- Watershed segmentation
- Multi-phase segmentation
- Masking

Quantification, Simulation & Analysis

3D Model Reconstruction

3D SE/BSE Images

3D EDS

3D TOF-SIMS

DualBeam can perform accurate multi-scale, multi-modal 3D characterization
3D Characterization through BSE/SE Image

Volume
Connectivity
Tortuosity
3D EDS on Li\((\text{Ni}_x\text{Mn}_y\text{Co}_{1-x-y})\text{O}_2\) Cathode
3D EDS on SiO/C Anode: Enables 4-phase Identification

- High-res 3D EDS on SiO/C anode at low kV to enable 4-phase identification in 3D volume
  - Not achievable via SE/BSE imaging due to low contrast among phases

- Identification of each phase provides critical information to understand structure-performance correlation in the battery
  - Phase distribution
  - CMC coating on SiO
  - Carbon/SiO ratio optimization
3D EDS on SiO/C Anode: Enables 4-phase Identification
TOF-SIMS: $^7$Li$^+$ Distribution in 3D

- TOF-SIMS on DualBeam enables the measurement of 3D distribution of the lithium ions within the electrode
  - Effective in mechanism study, e.g. SEI layer, fresh vs. cycled cell structure
Summary

- 3D imaging technique provides a quantitative approach to understand battery structure-performance correlations
  - Heliscan microCT allows for quantitative study of the battery structure evolution at the cell levels
  - DualBeam technique allows for 3D characterization at electrode level for both morphology and chemical information