3D Imaging Characterizations of Li-ion Battery via X-ray Microtomography and Electron Microscope

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Market Development Manager
Thermo Fisher Scientific

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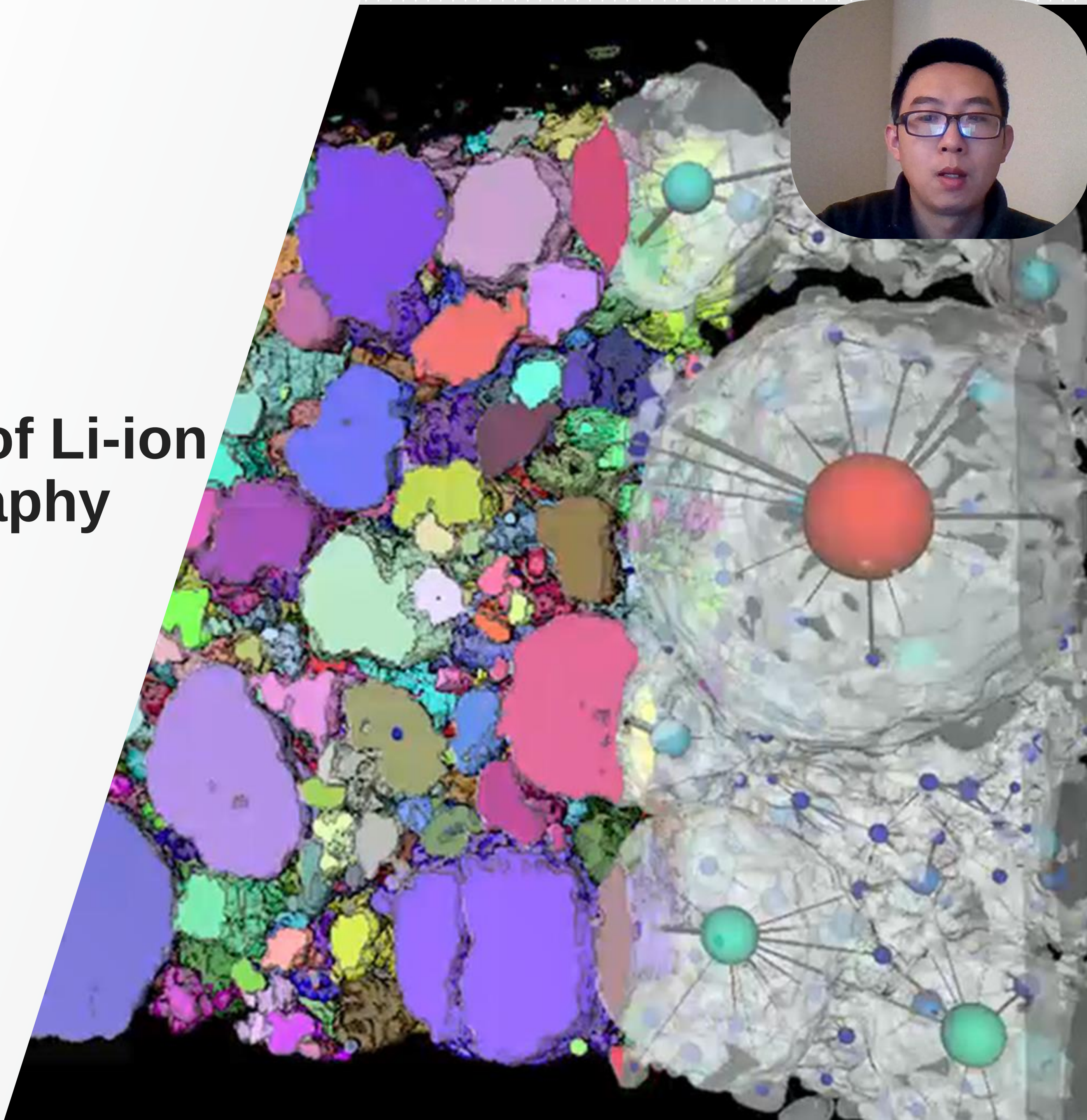
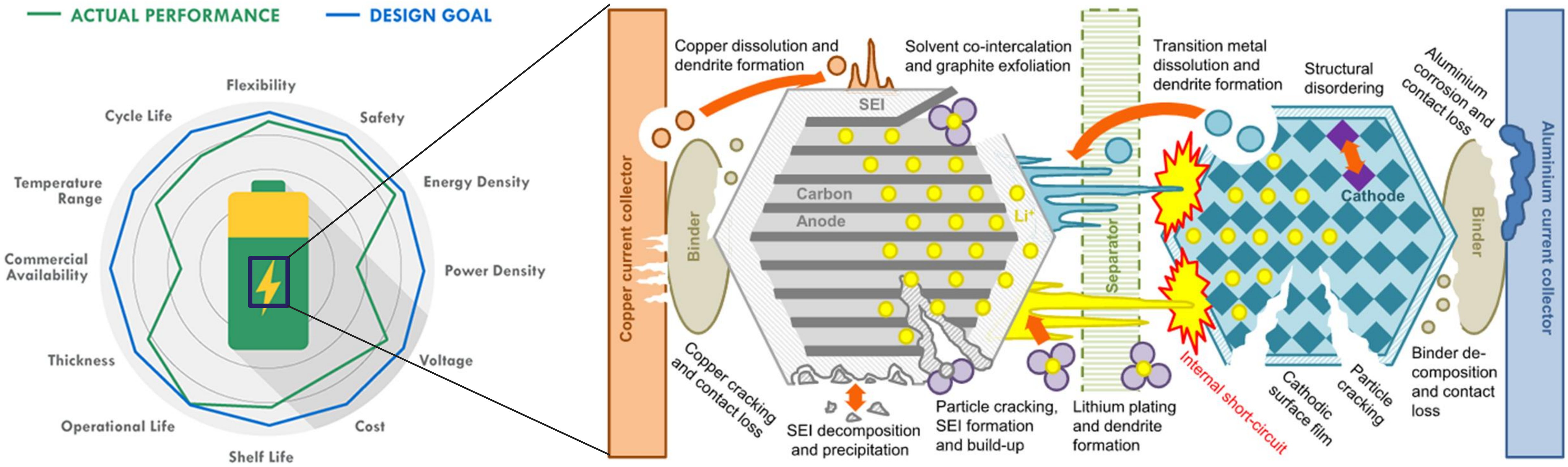


Design vs. Actual Performance

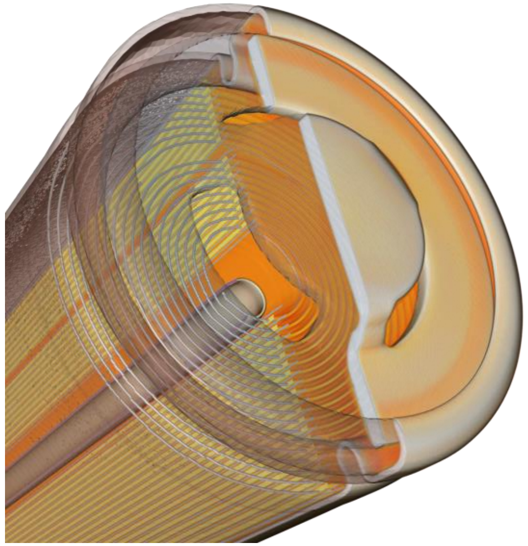


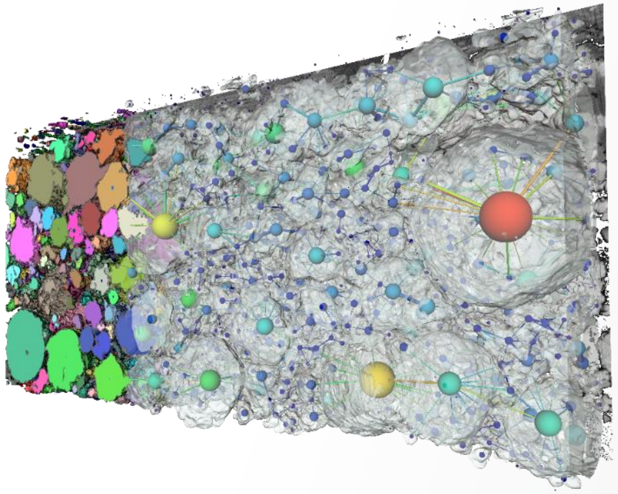
- Many interactions, at different length scales, all impacting the battery performance
- Understand the structure and performance correlation is critical

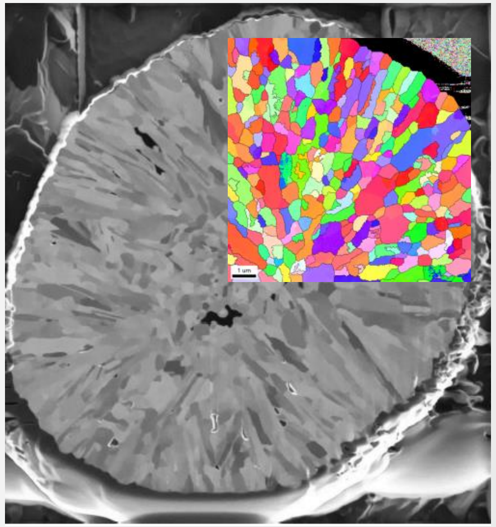
Birkl et al., J. Power Sources, 2017

Multiscale Imaging Analysis Approach

ThermoFisher SCIENTIFIC





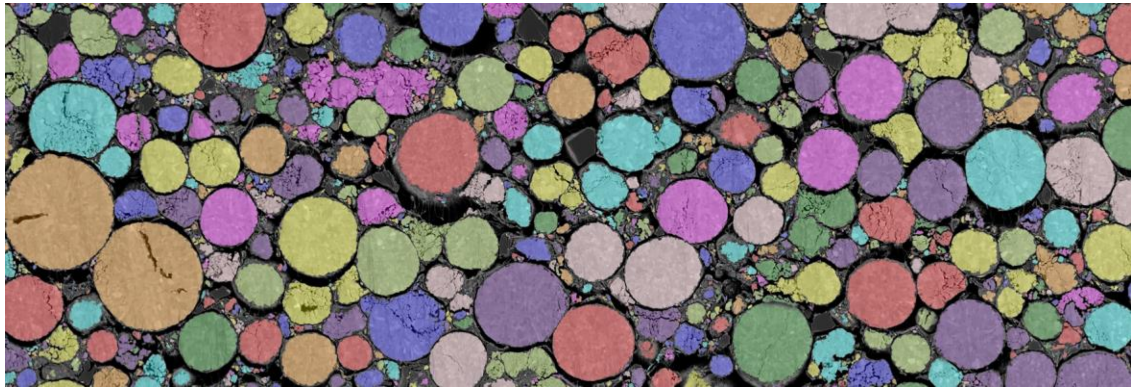


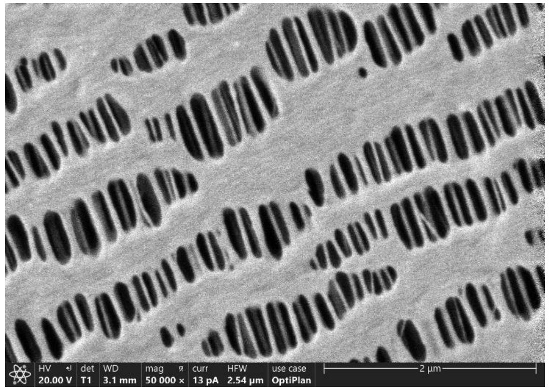
Cell → Electrode → Particle

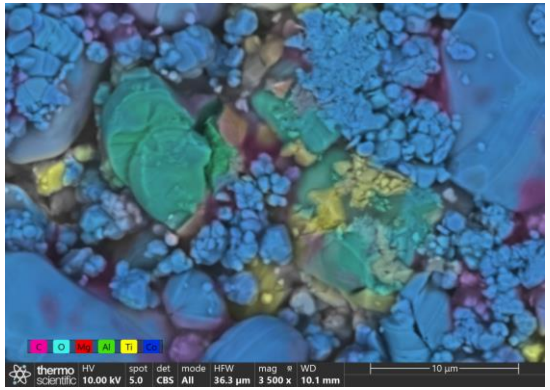
10 cm | 1cm | 1mm | 100µm | 10µm | 1µm | 100nm

Outline

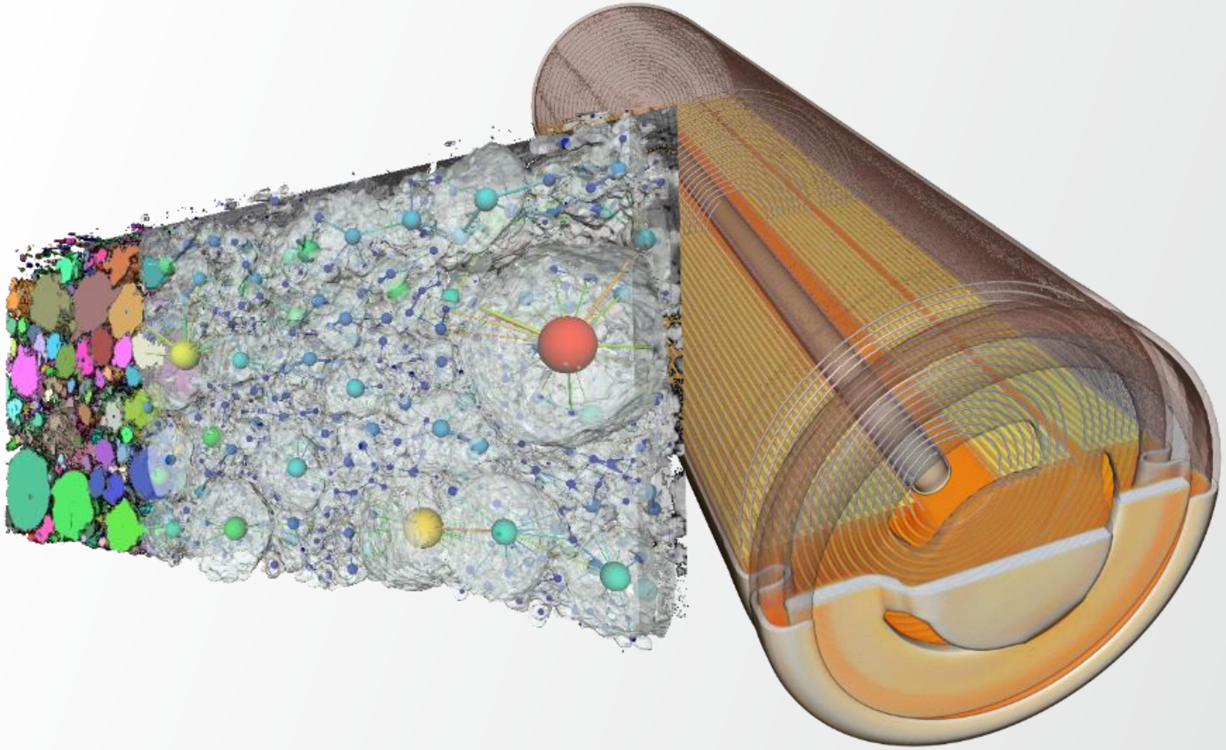
2D Imaging







3D Imaging



- Characterization methods for 3D imaging analysis
- How 3D imaging analysis help battery research?

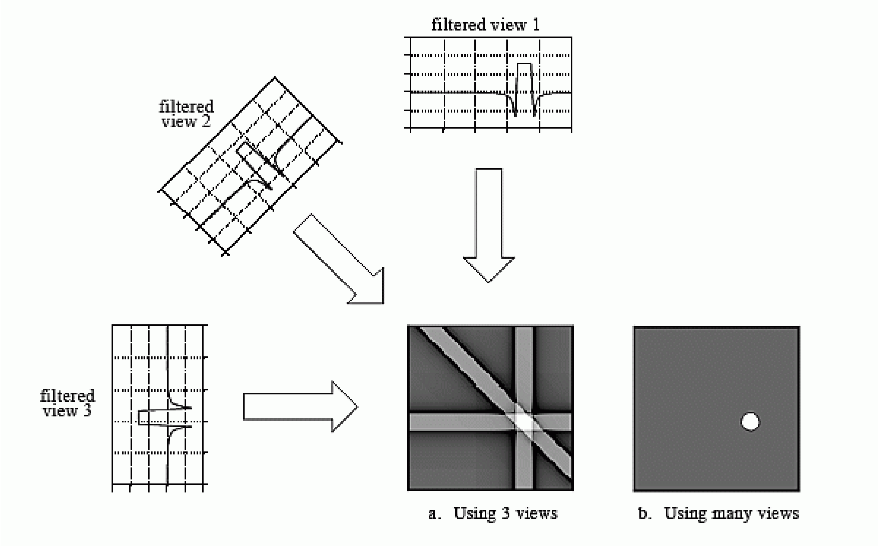
Data courtesy Devin Wu, Alice Scarpellini, Harold Phelippeau , VSG Team , Thermo Fisher Scientific

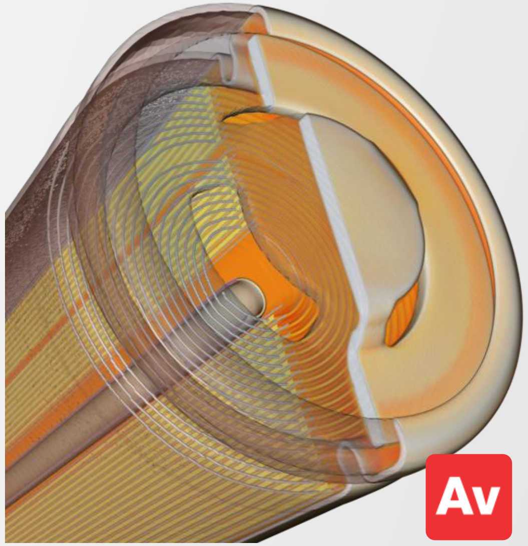
Heliscan microCT Analysis on Battery Cell

Data Acquisition

Reconstruction

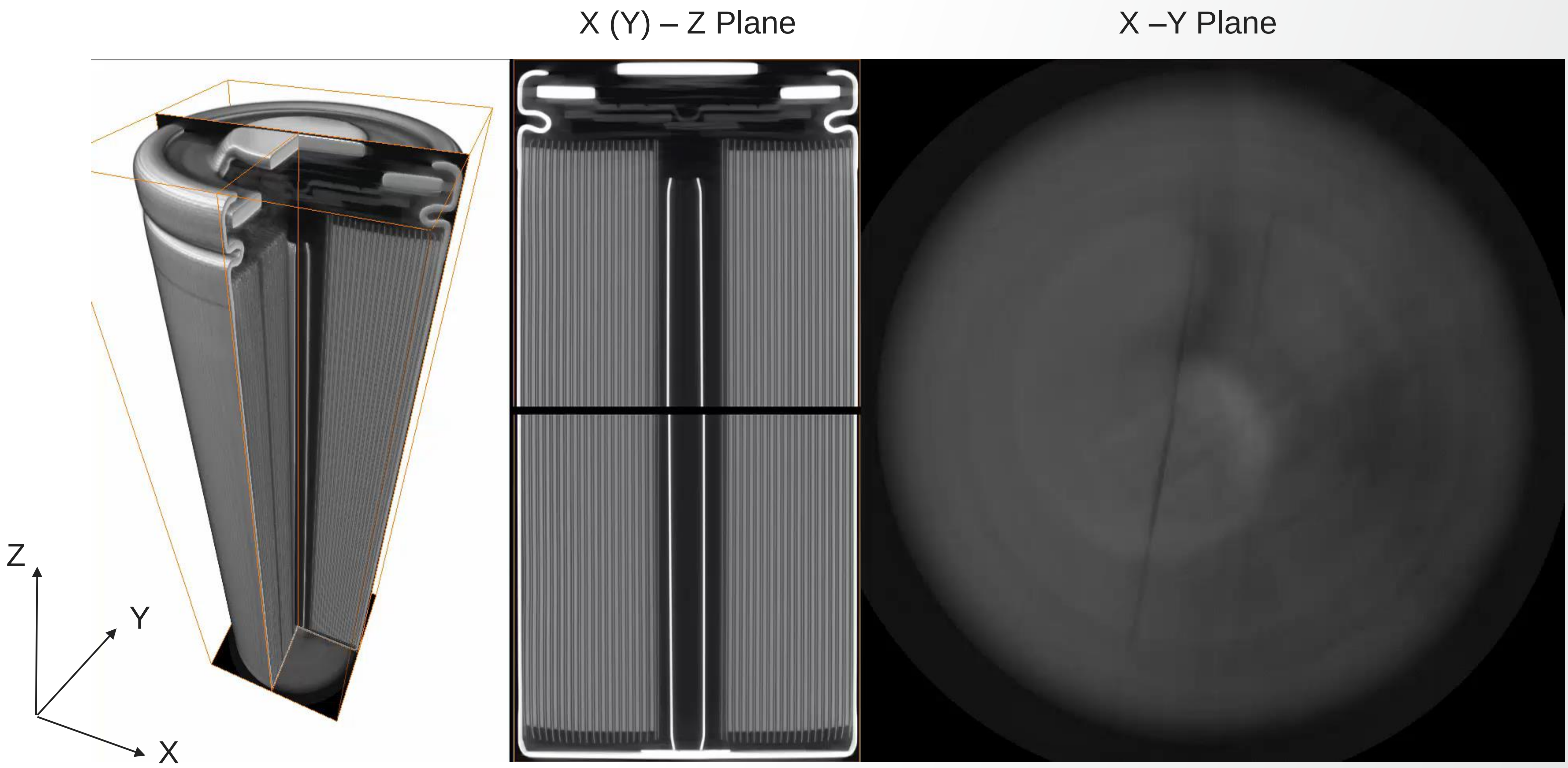
Visualization & analysis





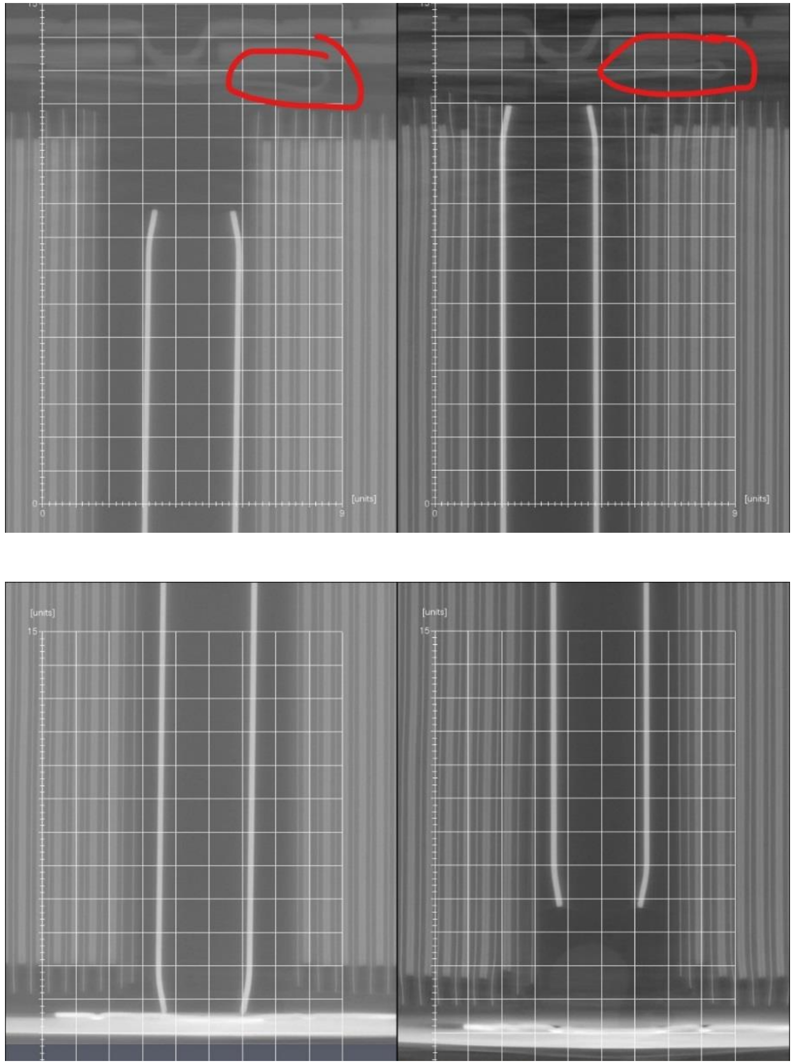
Non-destructive 3D imaging method

3D Reconstruction of a 18650 Cell



Sample courtesy: Electrochemical Innovation Lab, UCL

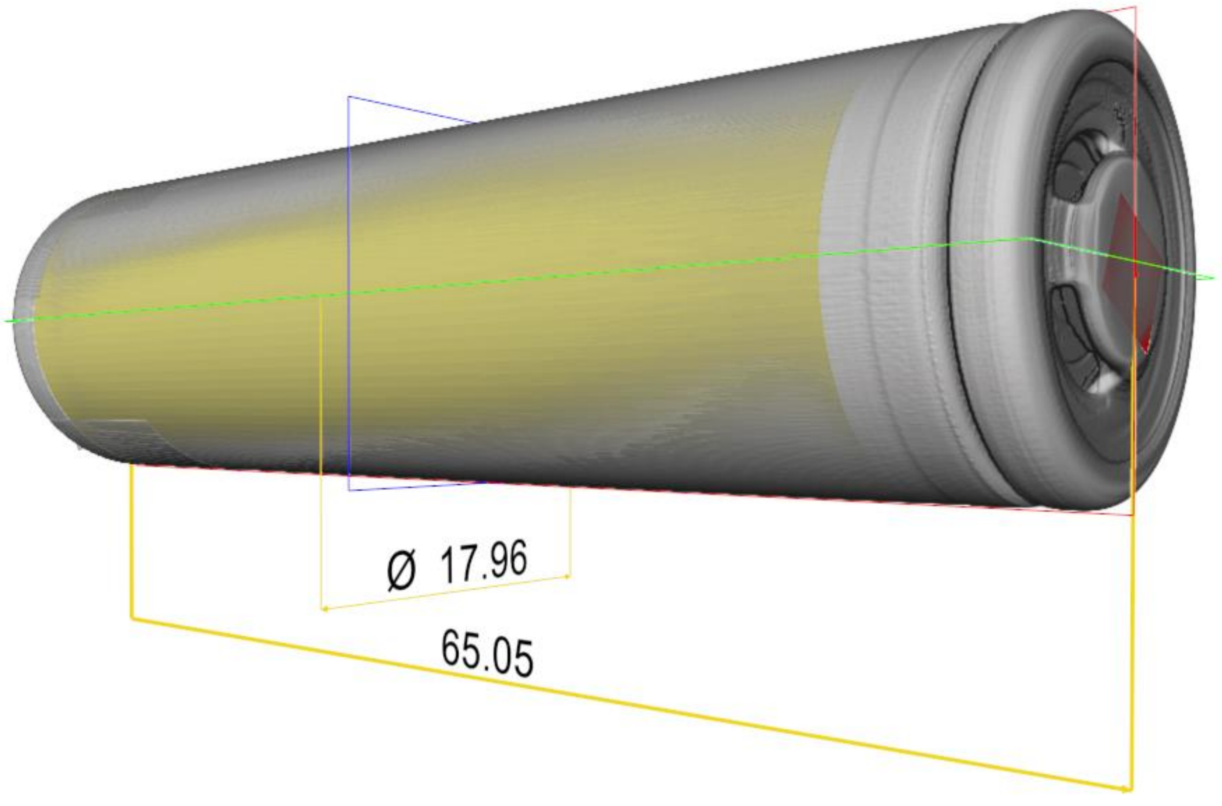
Cycling Effects Case Study on 18650 Cell



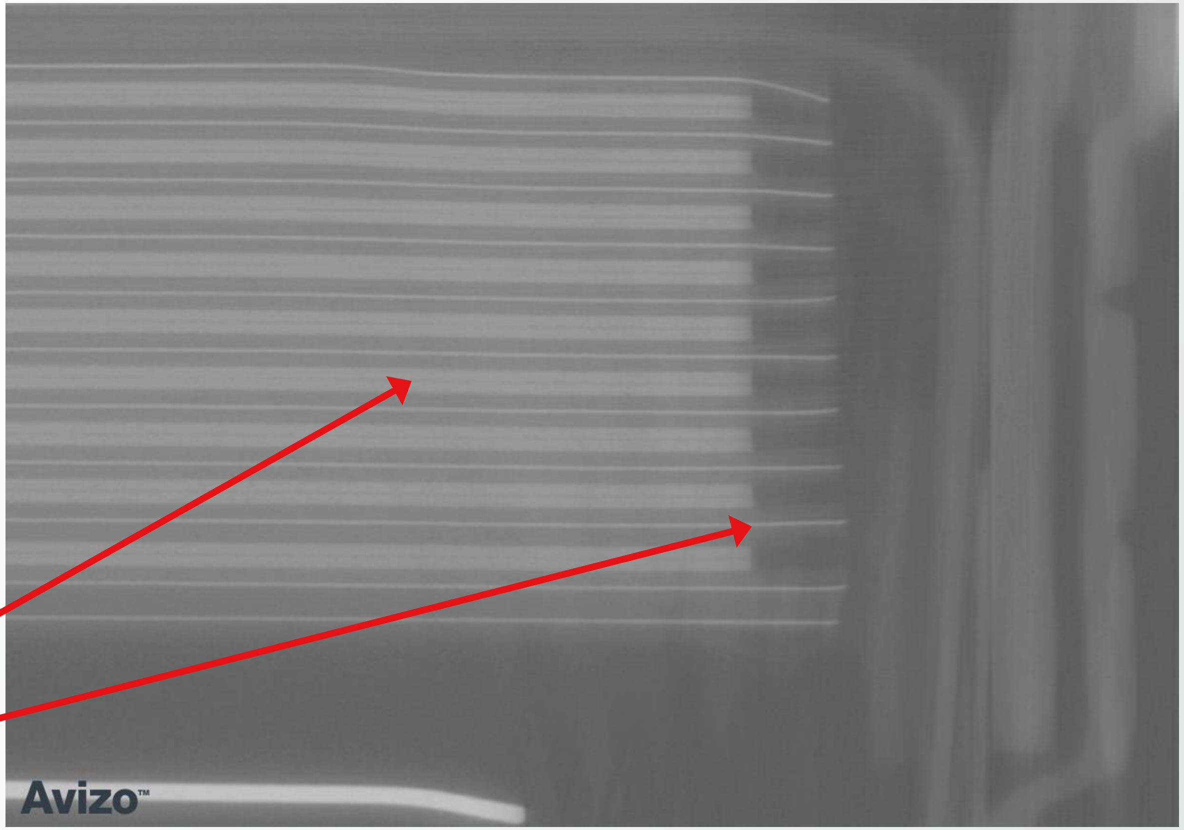
Fresh Cycled

Sample courtesy: Electrochemical Innovation Lab, UCL

Cycling Effects Case Study on the 18650 Cell



- Voxel size: 7.7µm

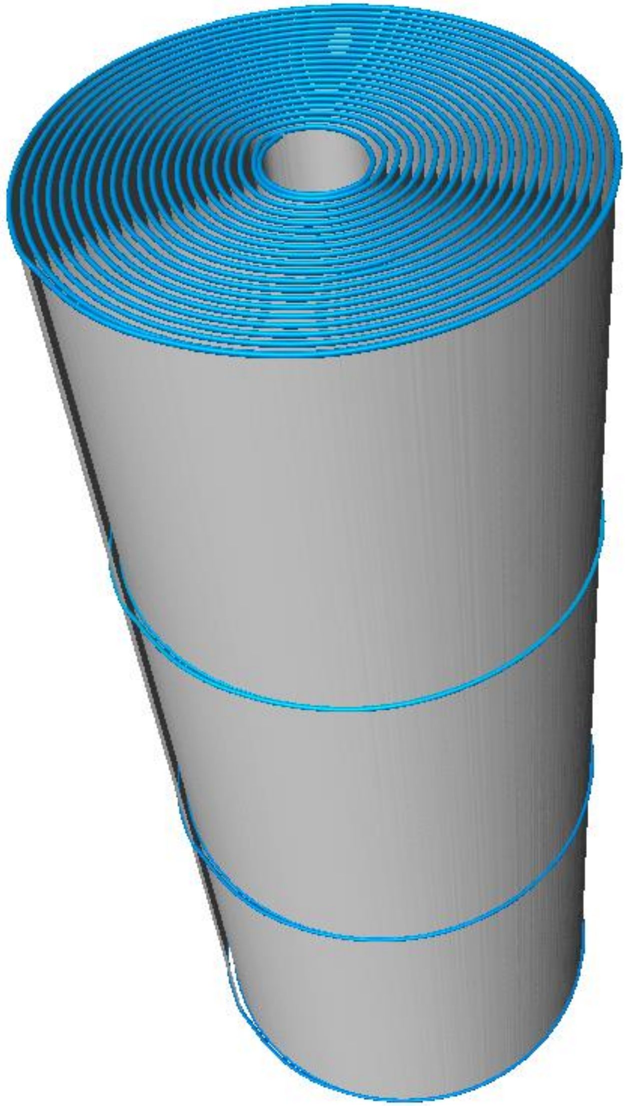


Cathode: bright + faintly visible Al collector

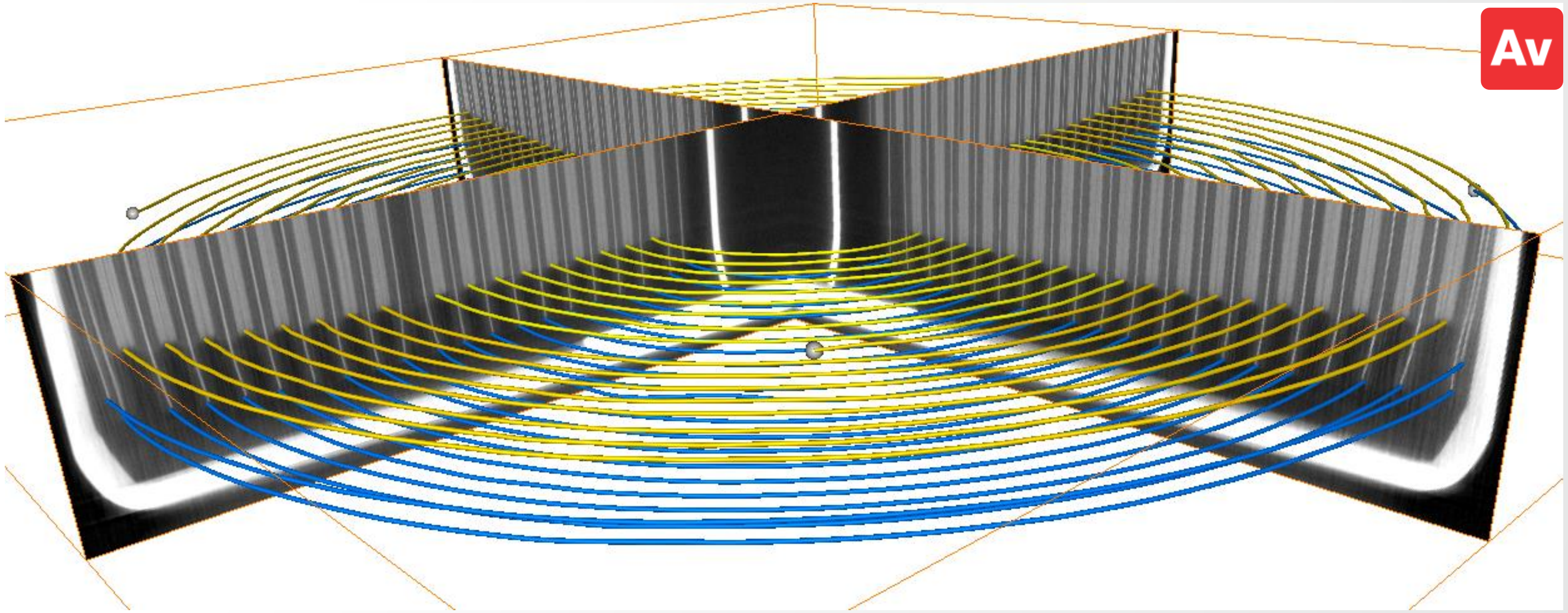
Anode: dark + bright Cu collector

Sample courtesy: Electrochemical Innovation Lab, UCL

Cycling Effects Case Study on the 18650 Cell



Tracing the current collector for quantification

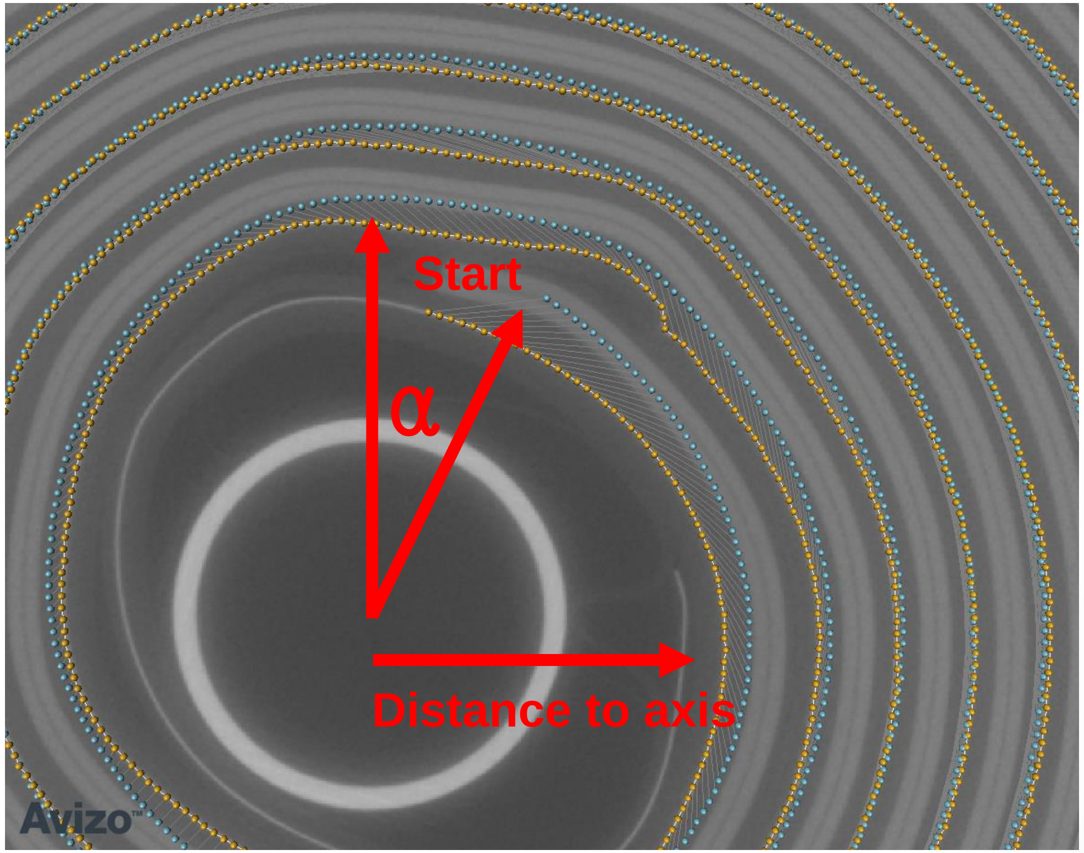


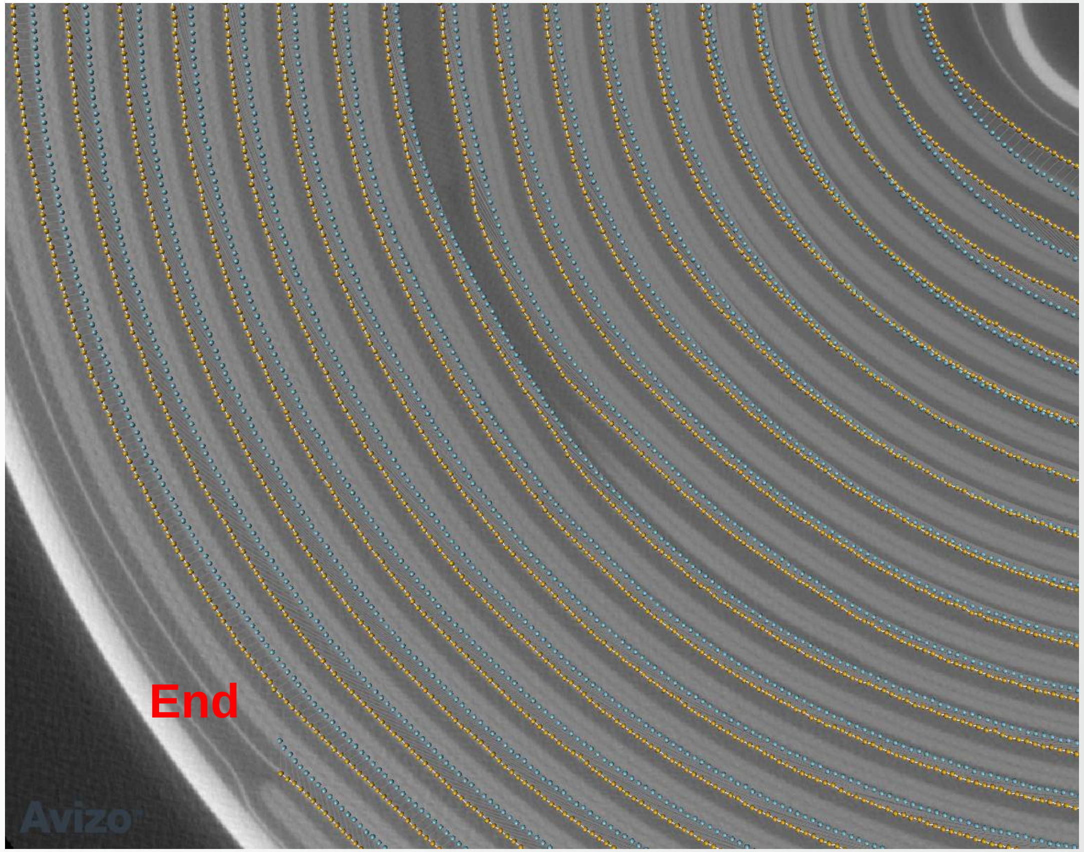
Fresh

Cycled

Sample courtesy: Electrochemical Innovation Lab, UCL

Cycling Effects Case Study on the 18650 Cell



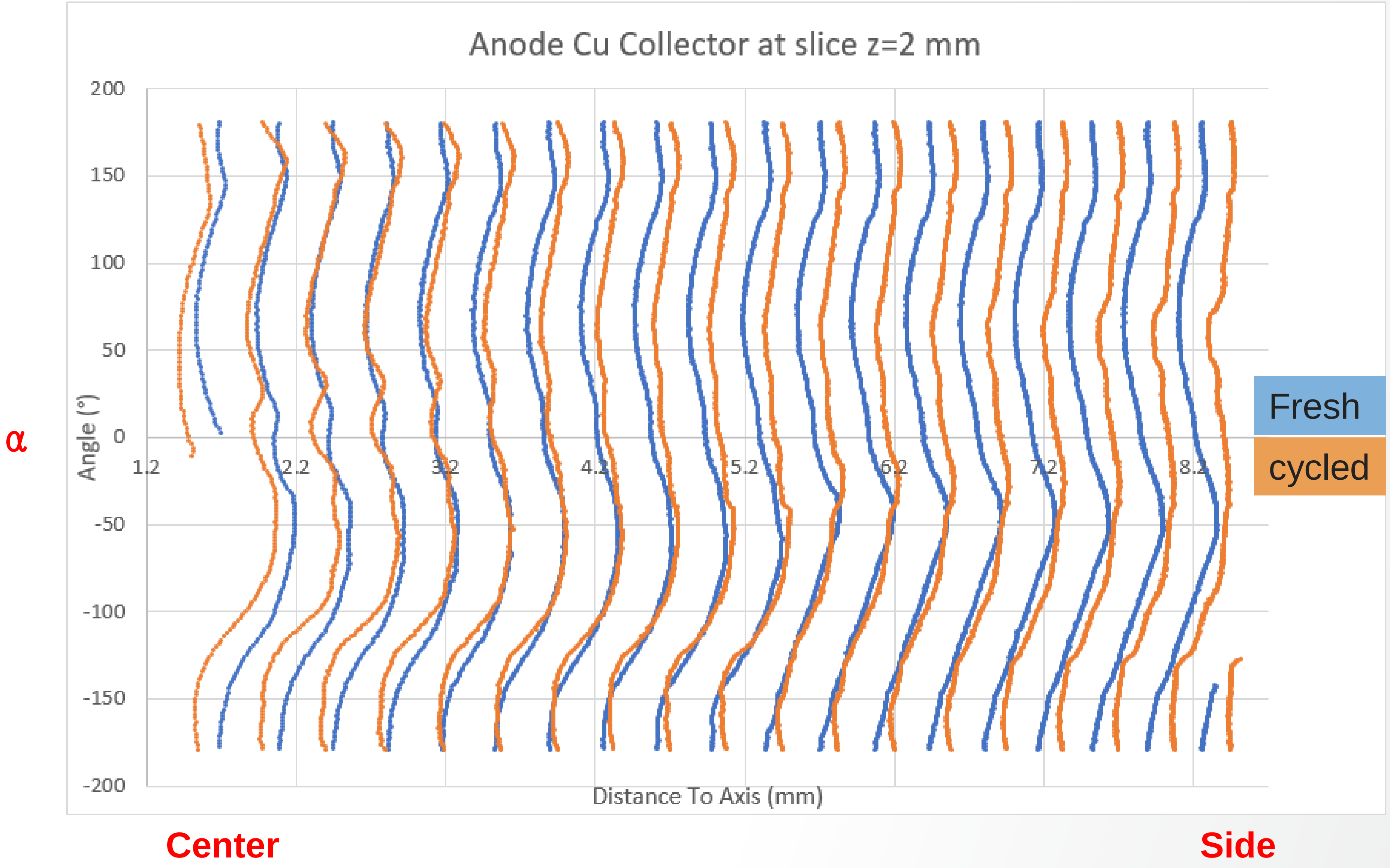


Fresh Cycled

Collector shape change is quantified by digitally correlating the position of the spiral in the fresh and cycled battery and plotting the distance from the center versus the rotation angle α

Sample courtesy: Electrochemical Innovation Lab, UCL

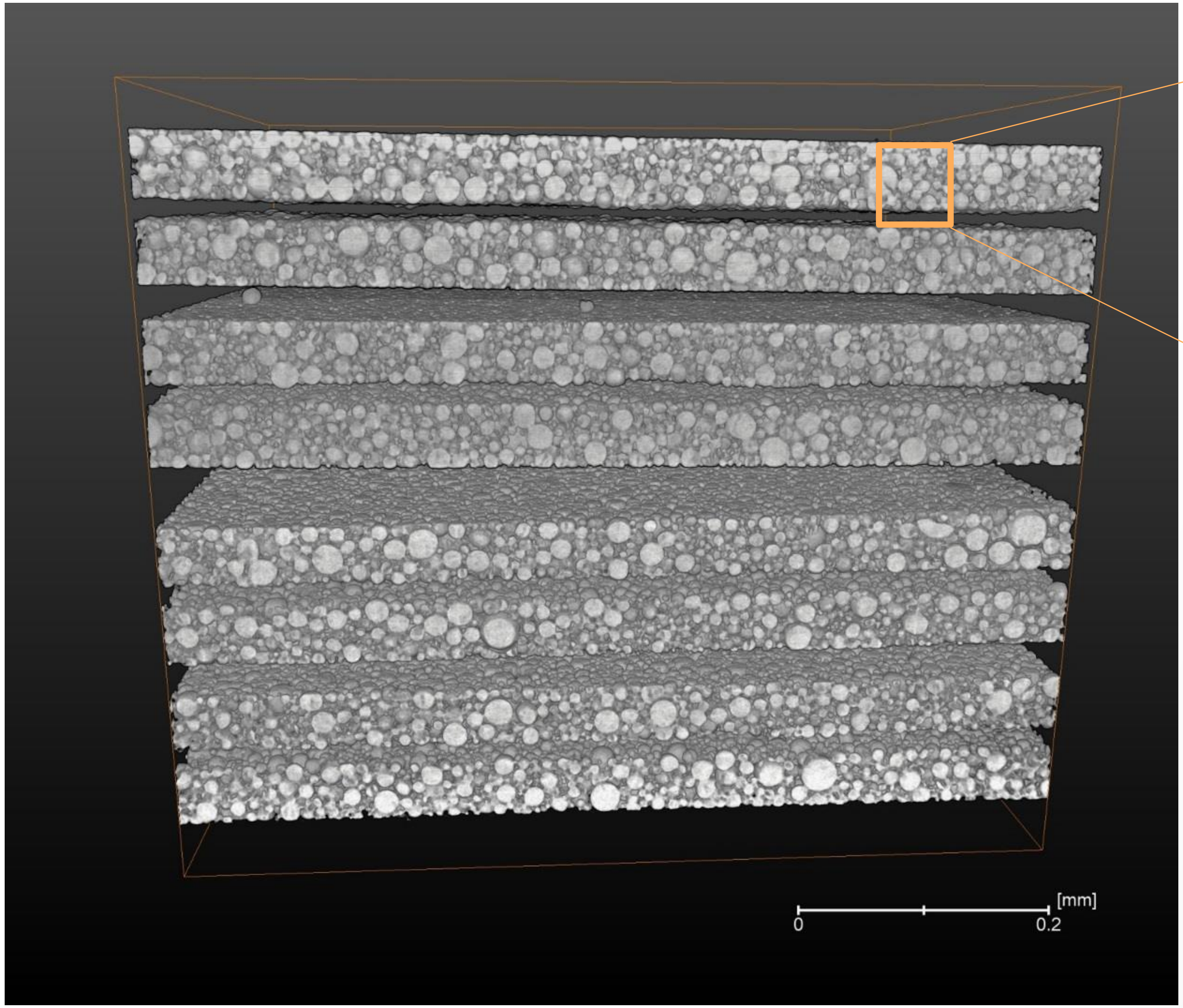
Cycling Effects Case Study on the 18650 Cell

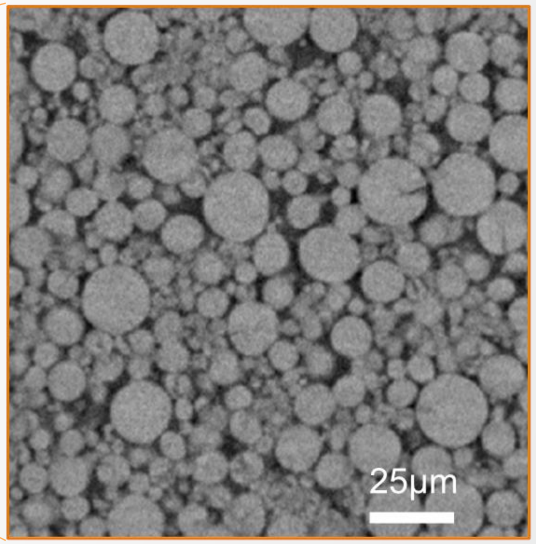


Detailed position at each point in the cell can be further explored and modeling for failure analysis and structural optimization

Sample courtesy: Electrochemical Innovation Lab, UCL

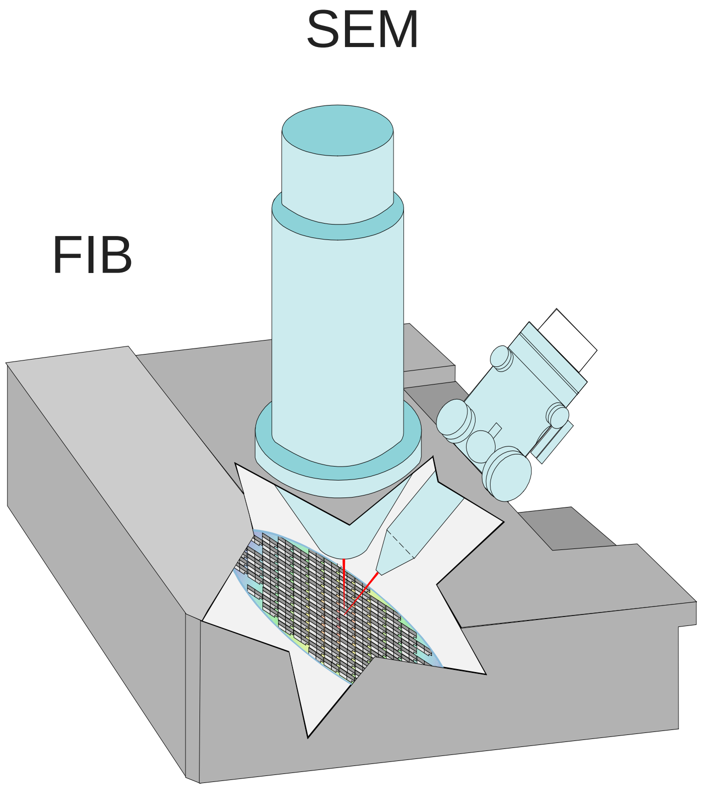
Challenges of the microCT for Battery Imaging

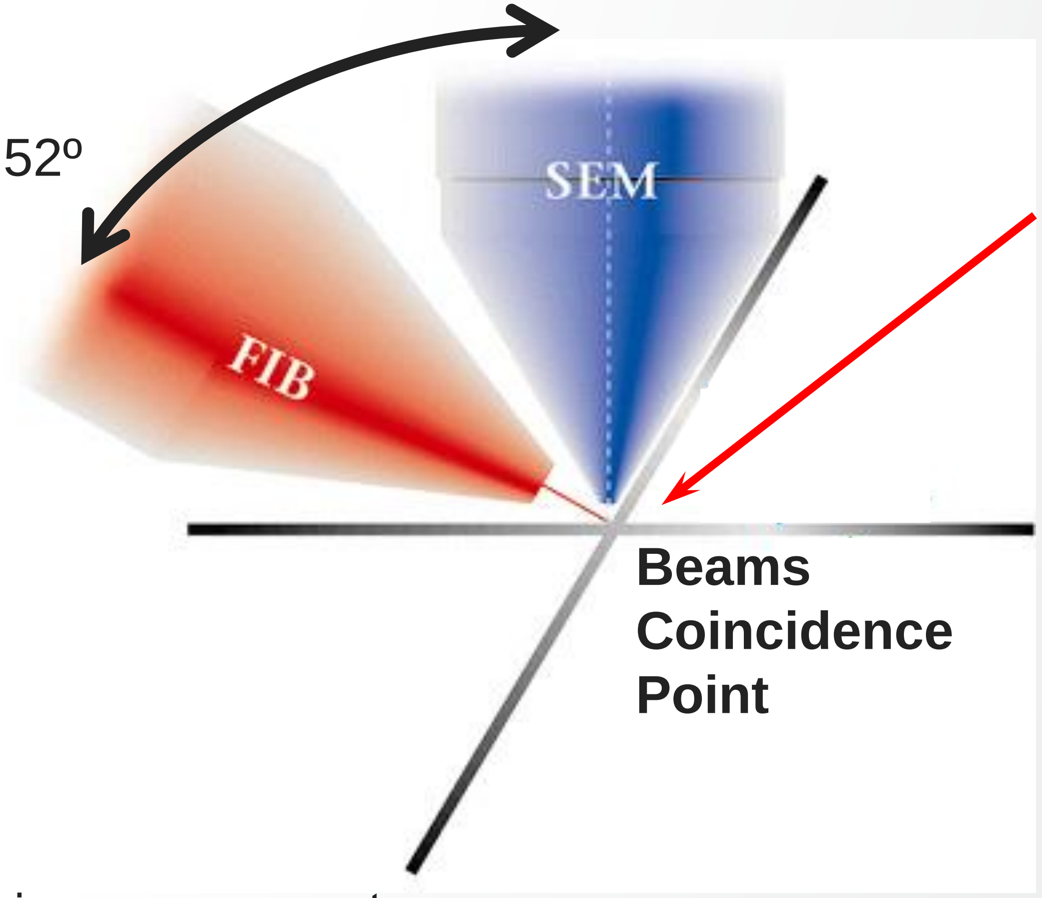


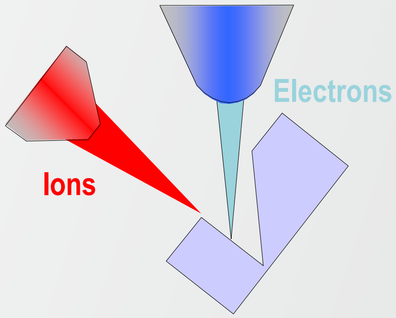


- Not enough resolution to see fine-structure at electrode and particle level (e.g. cracks)
- Lack of chemical and crystallographic information

DualBeam Application on Battery







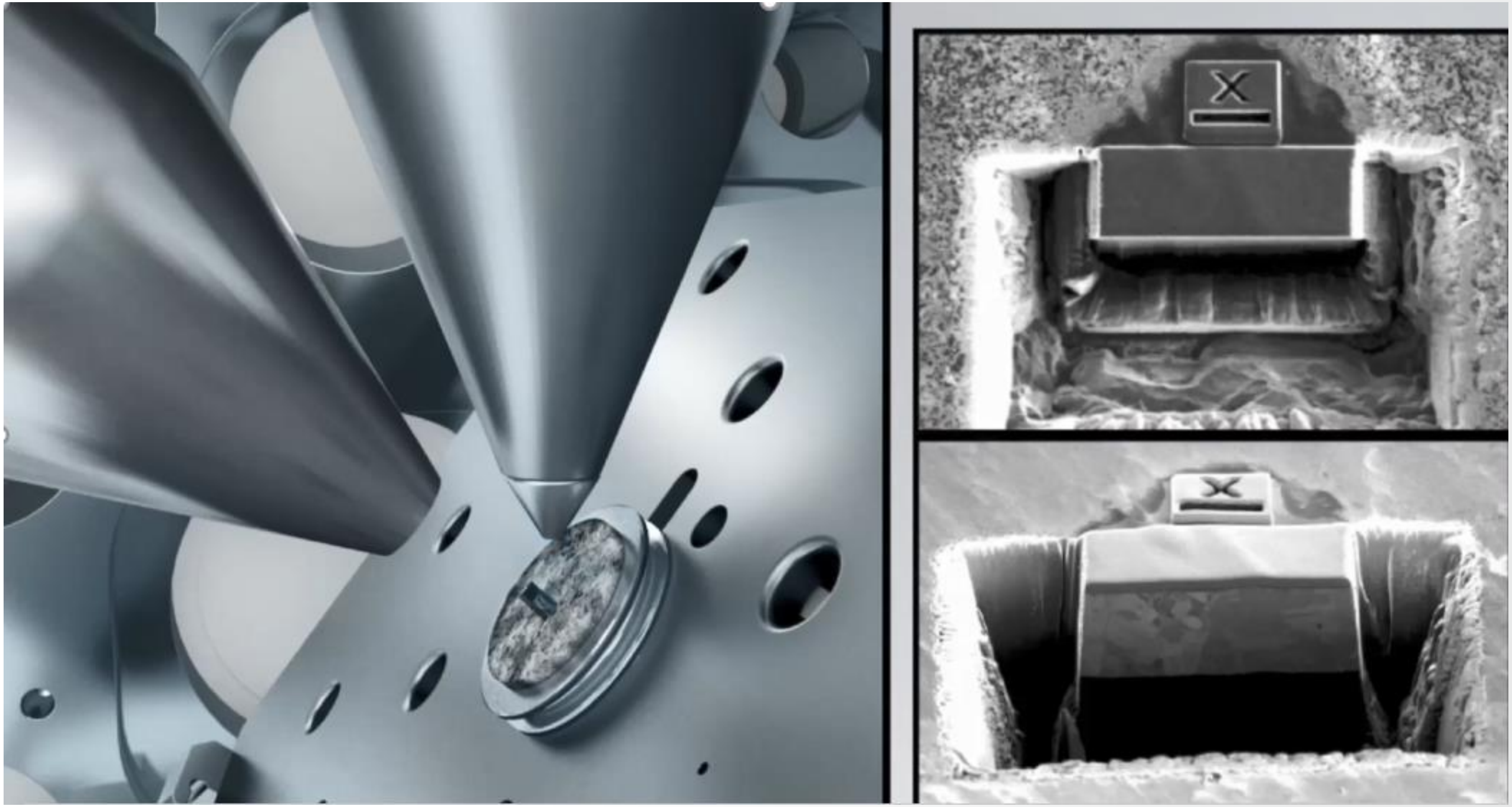
A DualBeam (FIB-SEM) is defined by of two primary components…

Scanning Electron Microscope for imaging

Focused Ion Beam for sample modification (milling)

The sample and stage are maneuvered beneath the beams to optimize imaging and milling

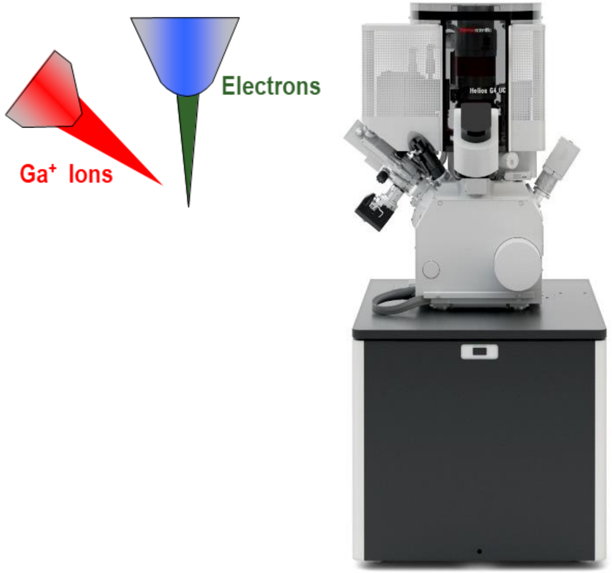
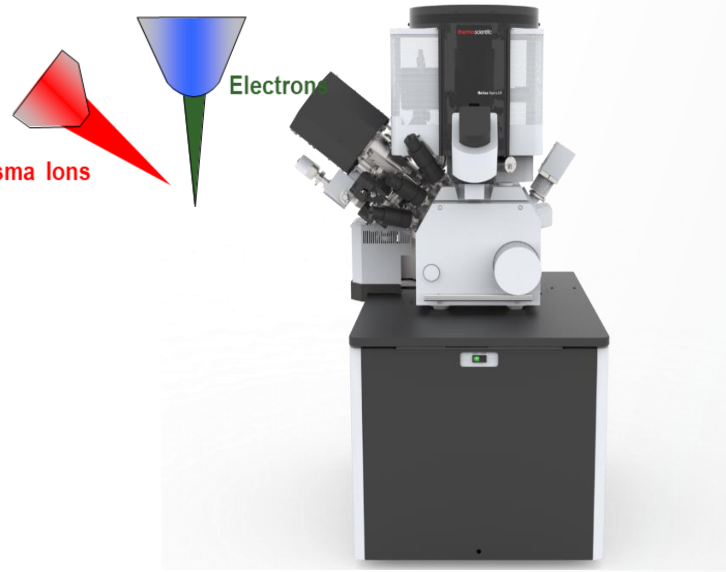
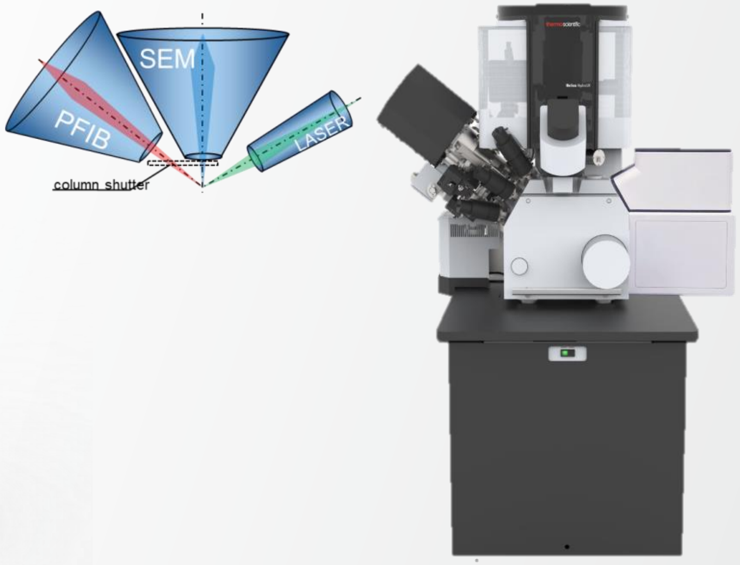
The DualBeam in Action



FIB

SEM

DualBeam Technologies

Ga-FIB	Plasma-FIB	Laser-PFIB
		
Ga^{+} ion source	Plasma ion source (Xe, Ar, N, O)	Femtosecond Laser + Plasma ion source

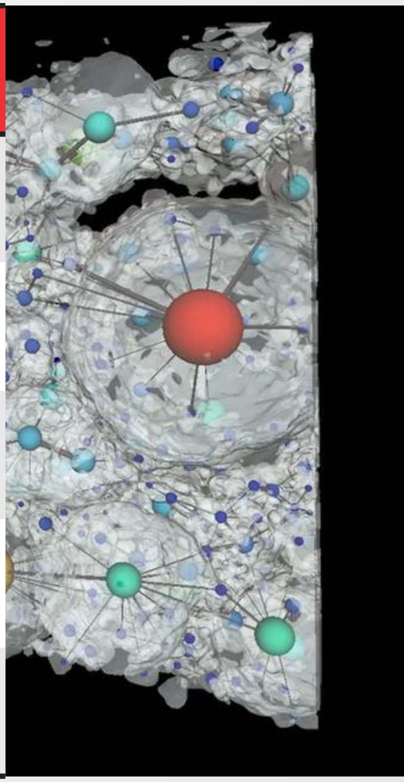
- Different ion sources (laser) to cover wide number of applications in different materials system
- Increasing volume for analysis: from Ga^{+}-FIB, P-FIB to Laser PFIB

DualBeam Analysis on Electrode via 2D and 3D

2D SEM Image on battery electrode

3D reconstruction of battery electrode

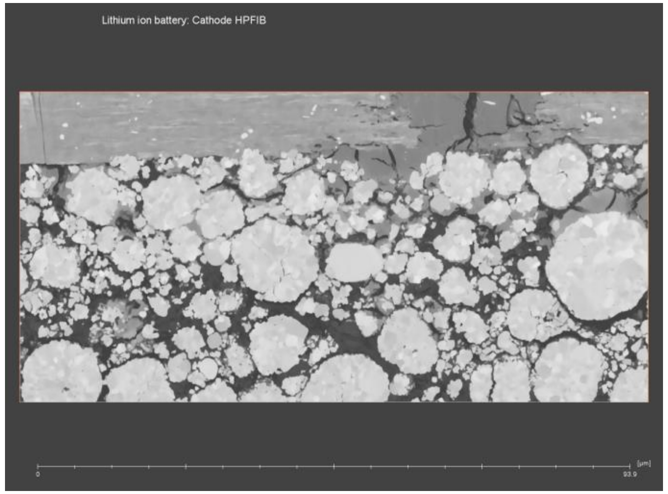
Microstructural Analysis	2D Imaging	3D Imaging
Volume Fraction	Yes	Yes
Surface Area	Limited	Yes
Particle Size Distribution	Limited	Yes
Connectivity	No	Yes
Tortuosity	No	Yes



3D imaging provides more information than 2D imaging for structural quantification, which is crucial for understanding the structure-performance correlation

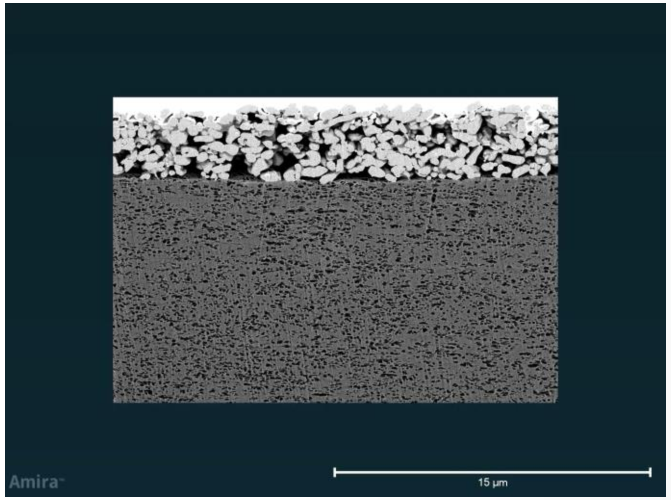
3D Imaging Analysis on Different Battery Components

PFIB: NMC cathode



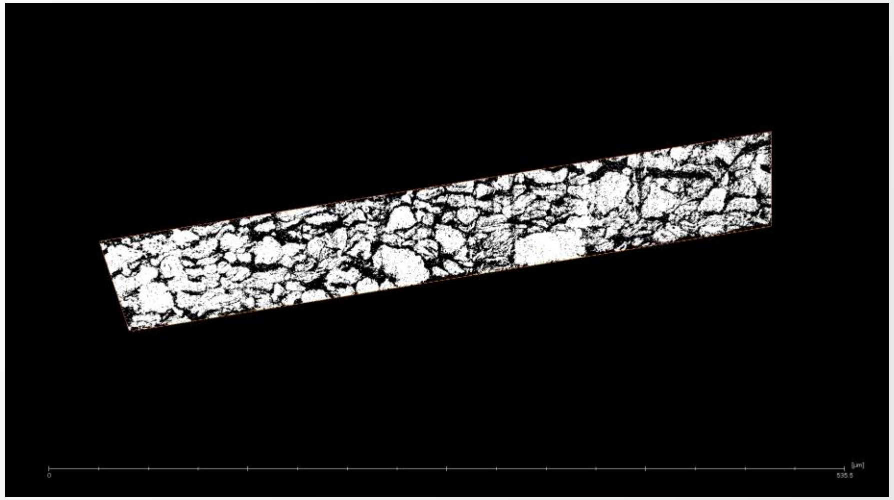
100 µm

Ga-FIB: separator

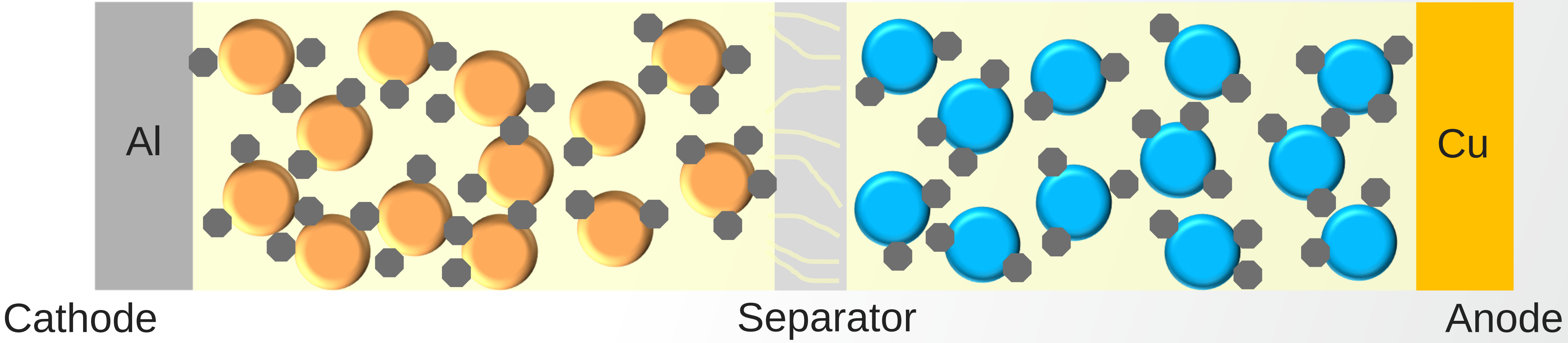


30 µm

Laser-PFIB: graphite anode

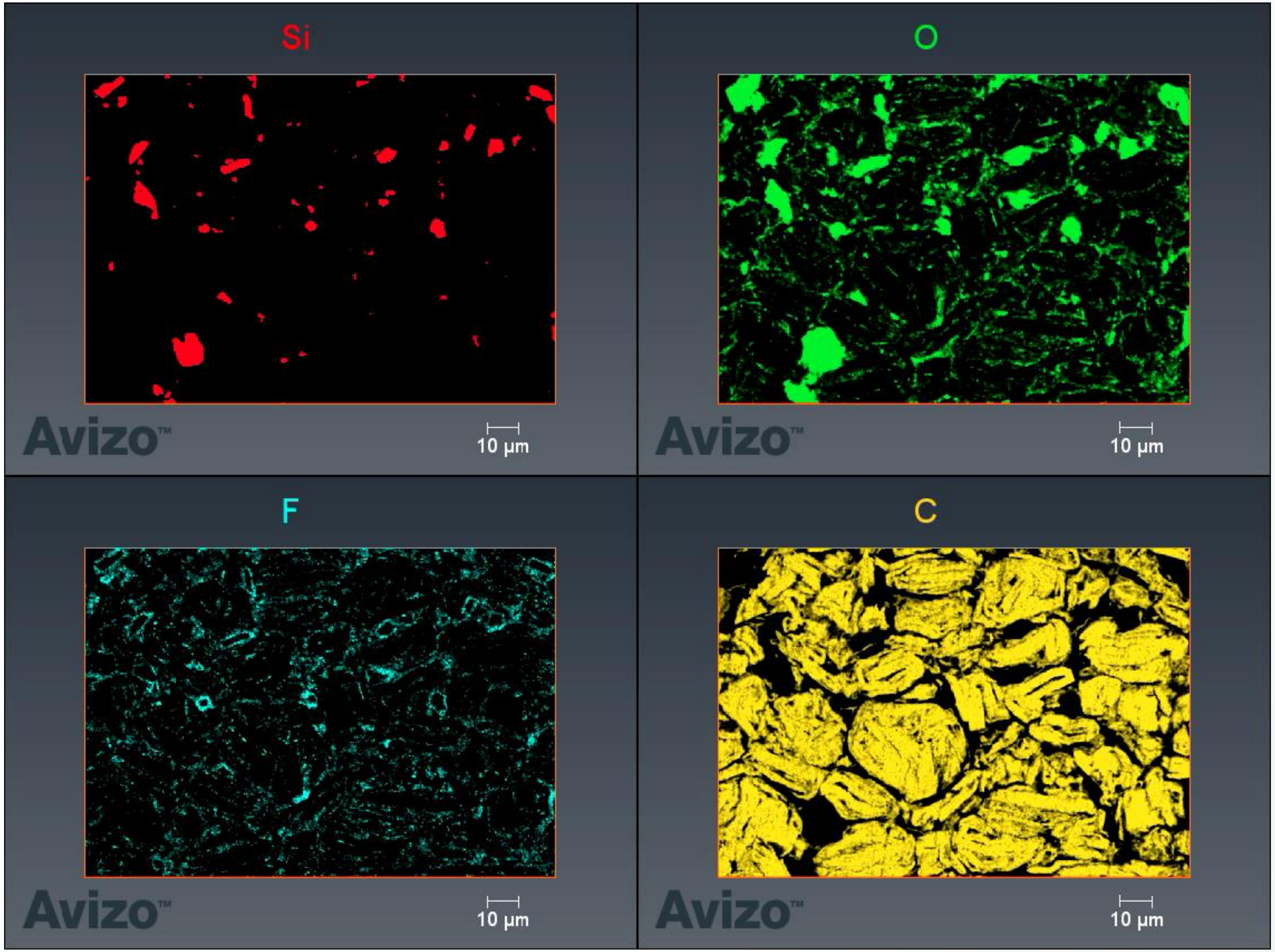


500 µm



Courtesy: Devin Wu, Remco Geurts, Stephen Randolph ,Bartlomiej Winiarski, Ken Wu, Chenge Jiao Thermo Fisher Scientific

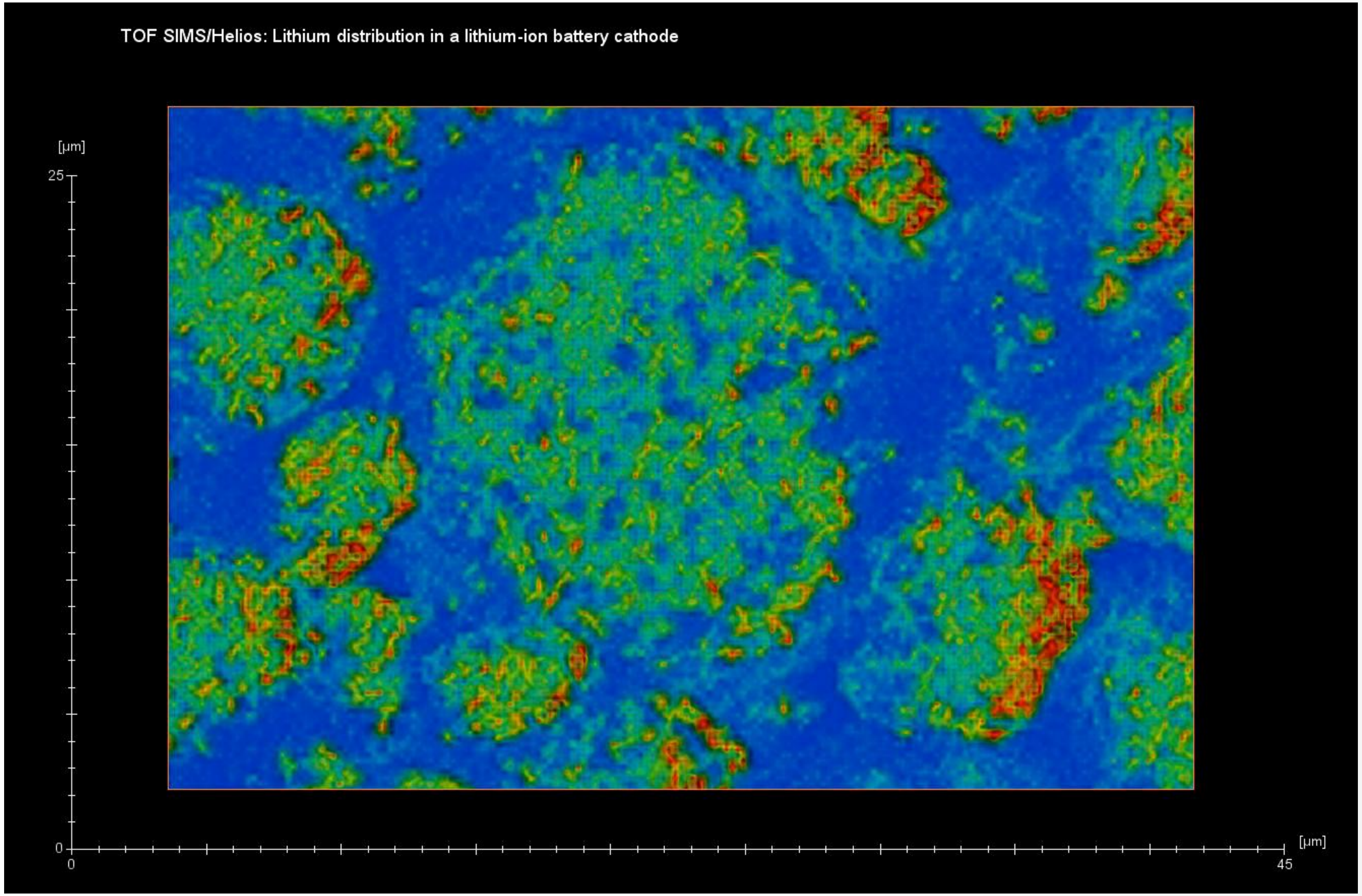
3D EDS Analysis of a Si-C Anode

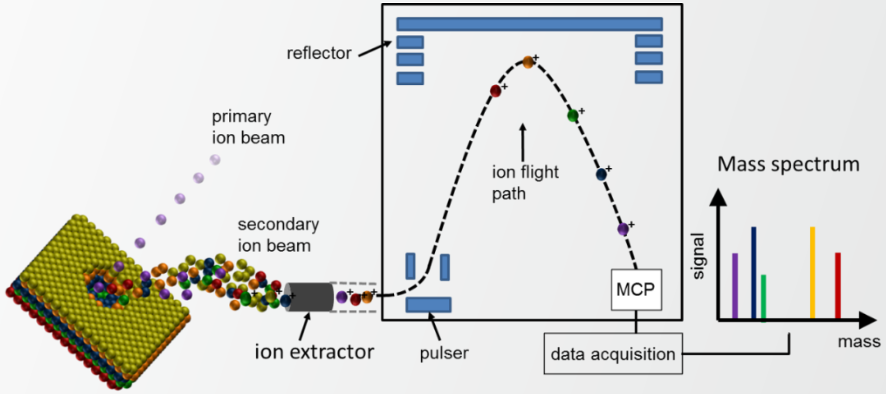


- **High-res 3D EDS on SiO/C anode at low kV to enable 4-phase identification in 3D volume**
  - Not achievable via SE/BSE imaging due to low contrast among phases
- **Identification of each phase provides critical information to understand structure-performance correlation in the battery**
  - Phase distribution
  - CMC coating on SiO
  - Carbon/SiO ratio optimization

Courtesy: Devin Wu, Thermo Fisher Scientific

TOF-SIMS Analysis of $^{7}Li^{+}$ Distribution in 3D

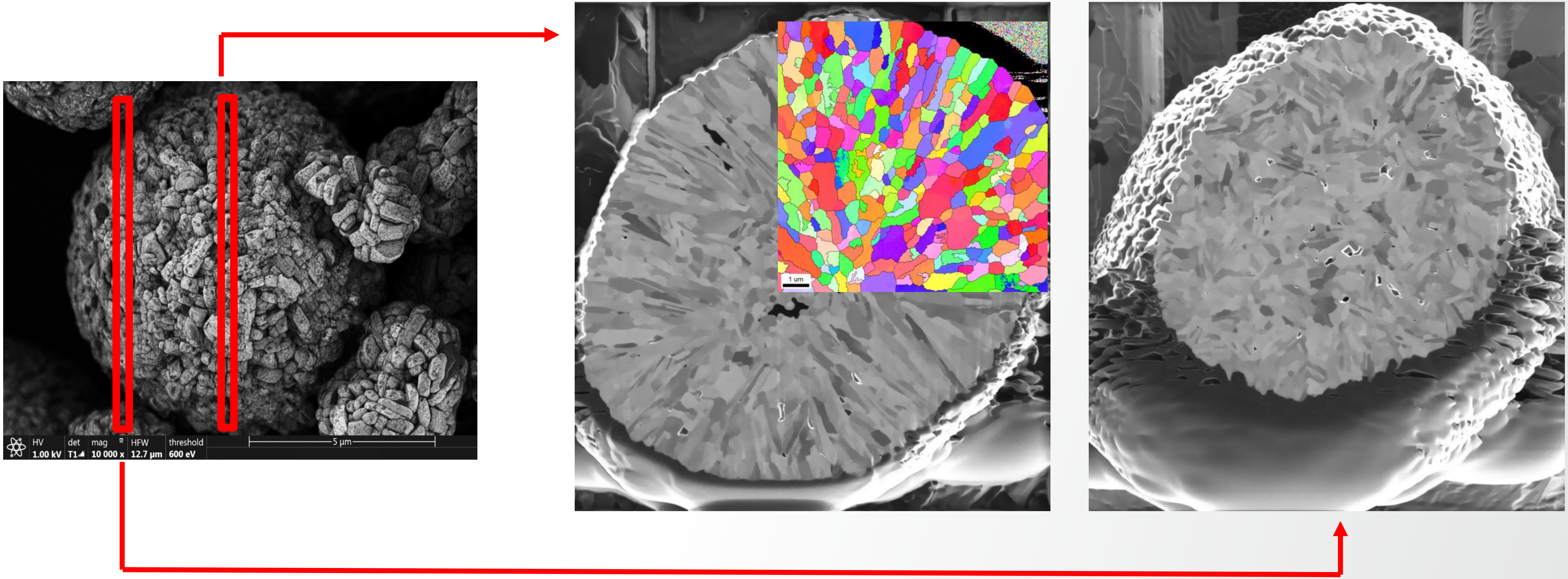




- **TOF-SIMS in DualBeam enables the measurement of 3D distribution of the lithium ions within the electrode**
  - Effective in mechanism study, e.g. Li distribution change at different cycling stage

Courtesy: Chengge Jiao, Thermo Fisher Scientific

3D Analysis Approach of a Single Particle

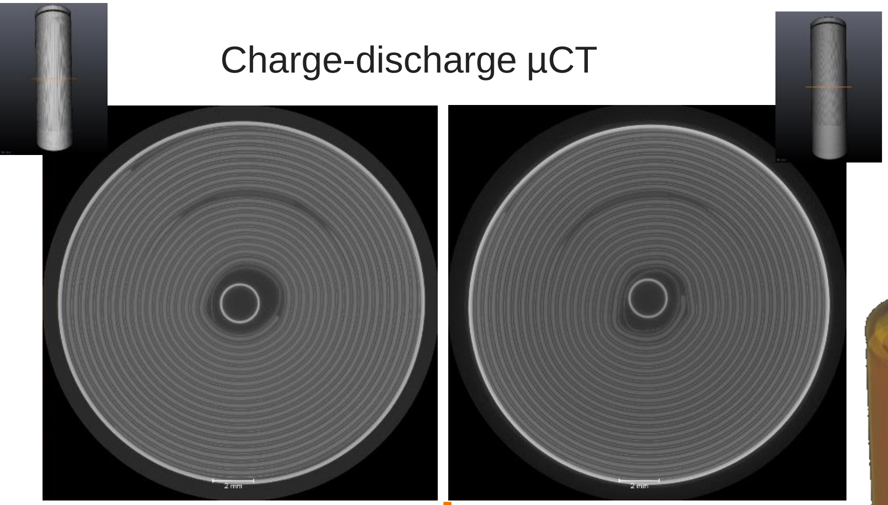


- Imaging was done at different “depths” in the particle to see how the structure changes from outside to inside of the particle
- EBSD analysis for primary particle grain orientation

Courtesy: Devin Wu, Thermo Fisher Scientific

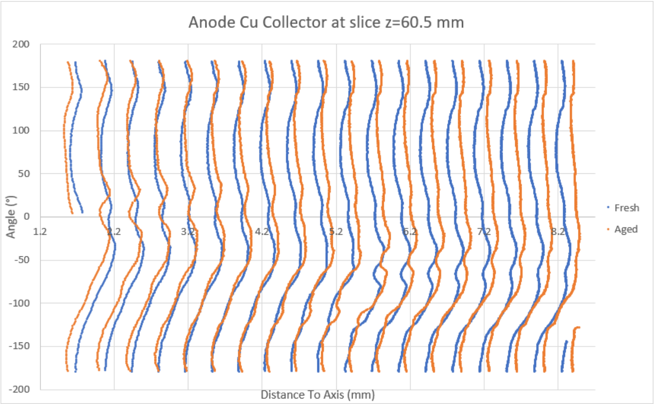
Correlative Multiscale Imaging across Tools

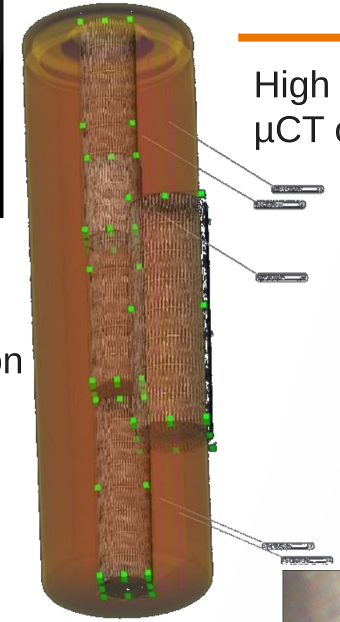
Charge-discharge µCT



Digital volume correlation & structural quantification

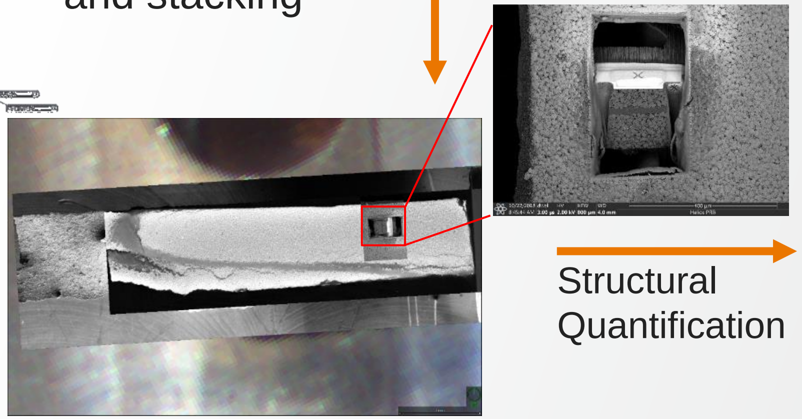
Damage-based region of interest selection





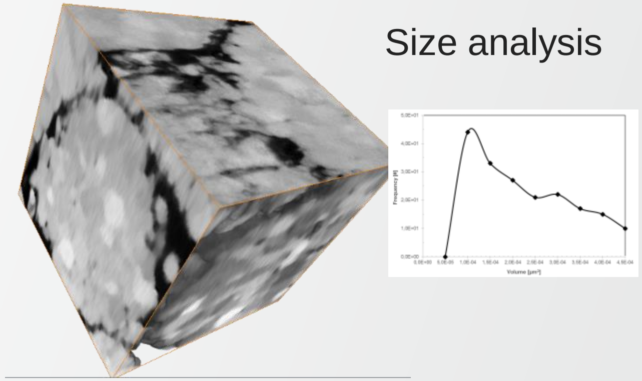
High resolution µCT on ROIs

FIB/SEM digging and stacking

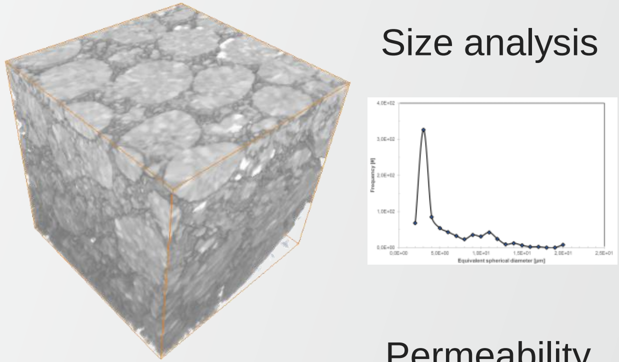


Structural Quantification

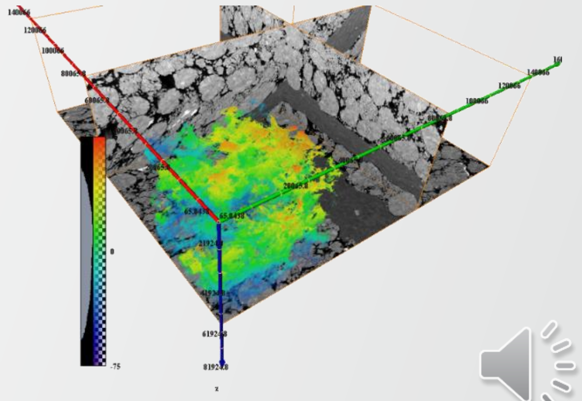
Size analysis



Size analysis



Permeability



Courtesy: Ali Chirazi, Bartlomiej Winiarski ,Thermo Fisher Scientific

Summary

- 3D imaging techniques provides an approach to in-depth understand battery structure-performance correlations
  - Heliscan microCT allows for quantitatively study of the battery structure evolution at the cell levels
  - DualBeam techniques enables 3D characterization of representative electrode volume and particle analysis for both morphology quantification, chemical and crystallographic analysis
  - Correlative imaging workflow enable analysis across multiple length scale in 3D

Thank You

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Thermofisher.com/battery-research