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APPLICATION NOTE

Multiscale Image-Based Control and Characterization of Lithium-Ion Batteries

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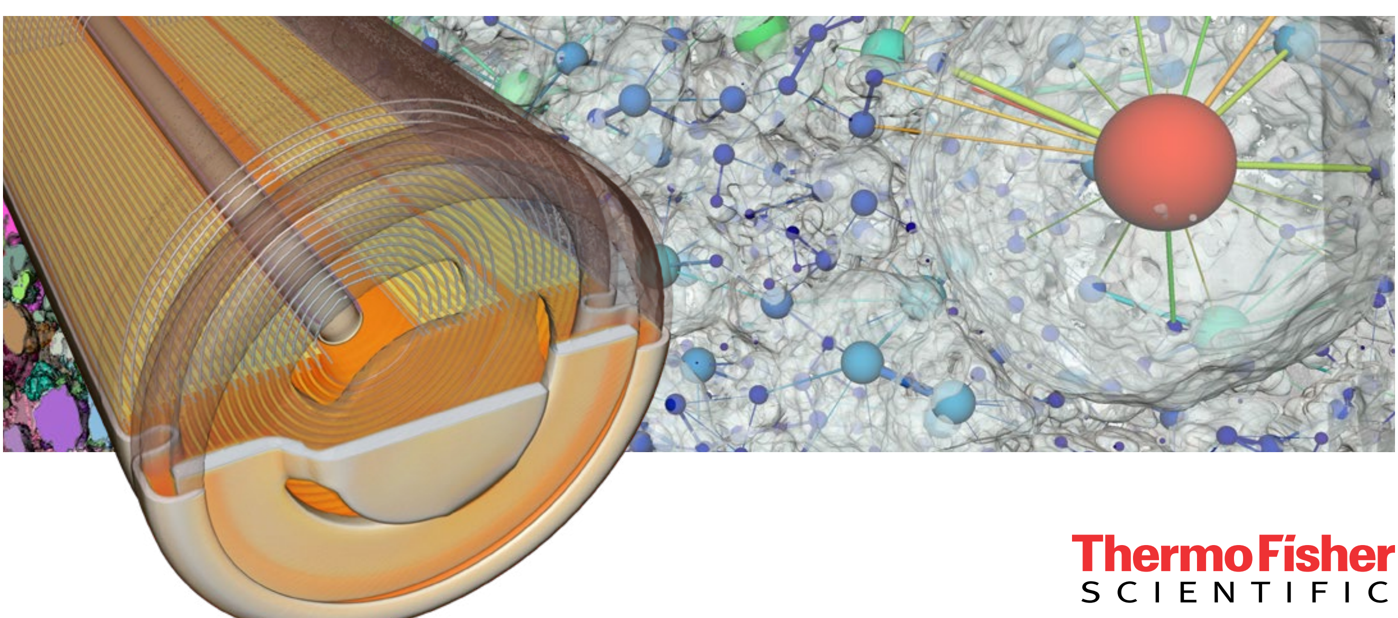
Lithium-ion batteries (LIB) are the predominant energy storage device for a number of everyday applications, ranging from portable electronics to electric vehicles. This is largely because LIBs have a high energy density, low self-discharge and low memory effect, allowing for a large number of charging cycles without diminishing storage capacity.

Performance, cost and safety are now the main factors driving ongoing battery research, with variations in battery chemistry enhancing performance at a lower cost with increased safety. Direct observation, down to the micro-scale, can be a significant benefit in the research and development of batteries and fuel cells. By combining techniques such as X-ray tomography, transmission electron microscopy (TEM) as well as focused ion beam scanning transmission microscopy (FIB-SEM) or plasma FIB (PFIB), images of the whole assembly can be obtained and observed at a variety of scales.

With advanced image processing and segmentation techniques, Thermo Scientific™ Avizo™ Software is ideally suited for this multi-scale approach. At the macro level, Avizo Software can be used to assess leakage, porosity or delamination. It can also examine aging processes by monitoring the quality of the foil, cathode and anode. At the microscopic level, Avizo Software allows for the estimation of the tortuosity and permeability of the porous electrode and separator. Effective transport parameters and the cell's performance can be further analyzed with electrochemical performance simulations as well as quantification of the triple phase boundary (TPB), phase distribution and connectivity.



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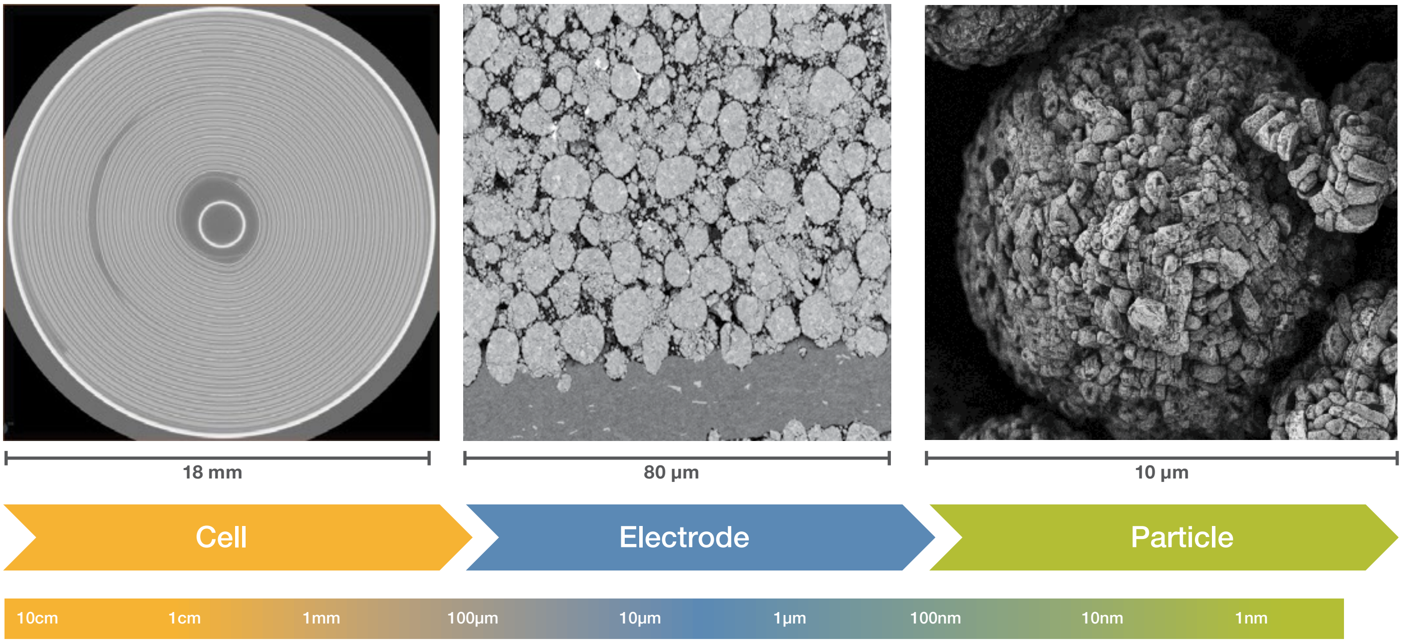
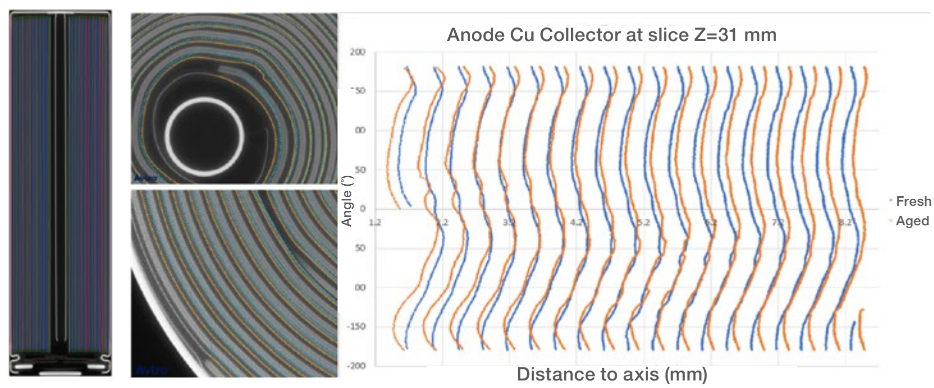


Structural analysis using microCT

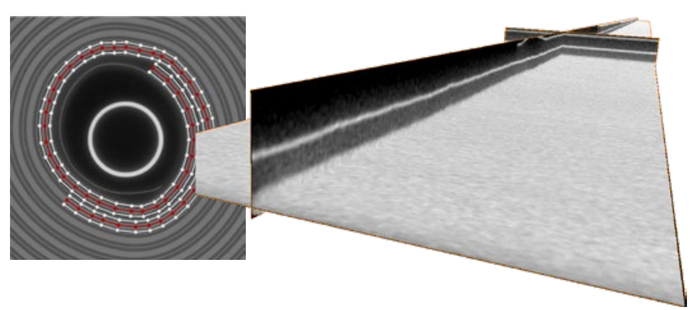
Micro-computed tomography (microCT) instruments non-destructively generate 3D reconstructions of samples by placing them on a rotating stage and illuminating them with a micro-spot X-ray source. The transmitted X-rays, influenced by the material, are captured by a detector, creating a 2D projection (the tomogram). As the sample rotates a series of these projections is collected, which are subsequently recombined into a digital 3D model. MicroCT is useful in a variety of applications including;



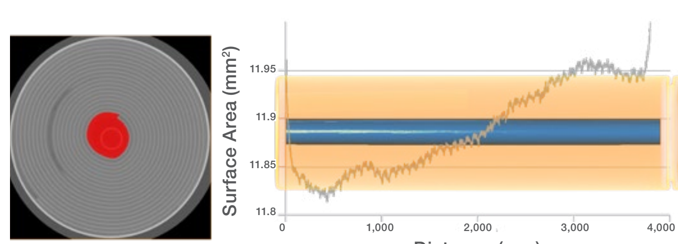
Electrode length: the structure of a lithium ion battery, changed by numerous cycles of charging and discharging. It is possible to control this degradation by monitoring the length of the electrodes. As the electrode curves the radial distance between its ends changes.

Quality control: Non-destructive analysis of a battery can identify possible internal defects that may have occurred during manufacturing (e.g. soldering, leakage, delamination, porosity, etc.). This information can lead to more efficient and safer batteries.

Aging and degradation: Incremental changes occur to a battery during multiple charging cycles, leading to degradation. MicroCT enables non-destructive investigation of the mechanisms behind this process. The number of cycles is one of most important factors for a rechargeable battery and is impacted by changes to foil, anode and cathode morphology (length, radial distance, etc.) as well as core leakage.



Battery unfolding: for a lithium ion cylindrical cell, the cathode can be unrolled along with the anode and separator. In order to ease the inspection of the full cathode, an unfolding algorithm specially designed to reduce distortions has been developed.[1]



Central rod area: in order to control battery cell design, specific regions of the surface area can be segmented and measured in 3D or in 2D slices. Here the central rod of the cell is analyzed, and the results are plotted in a variation plot.

Nano-structure analysis using electron microscopy

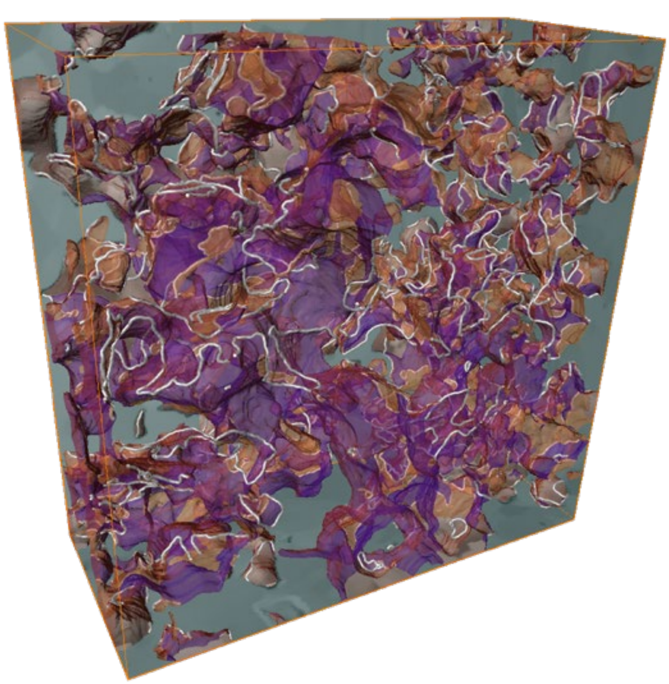
Structural information is critical for interpretation of battery performance, influencing parameters such as the speed of charging, the total capacity as well as the rate of capacity degradation. Electron microscopes (EM) can observe structural details at the nano-scale, revealing the intricate details of particle connectivity and the porous network through which ions are transported.

Cell properties such as tortuosity, porosity, as well as pore and particle size distributions influence how effectively ions diffuse within the electrodes and the electrolyte. The electrical conductivity of an electrode is also dependent on its microstructure (the contact area between the different solids at the TPBs). Heterogeneity within the electrodes is known to contribute to cell degradation.

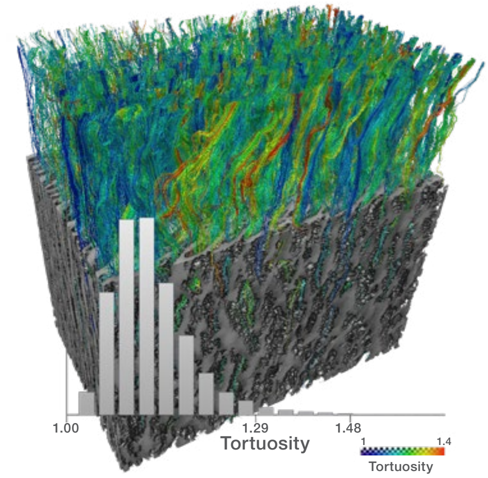
Our EM 3D imaging systems are essential for the analysis of transport properties (calculated from representative volumes) to understand and predict percolation in battery and fuel cell systems.

Connectivity reveals how each phase within the cell is connected and how effectively the materials are utilized. While pore connectivity informs the transport path of lithium ions, carbon phase connectivity is critical for electrode materials that do not have good electrical conductivity (e.g. $LiFePO_4$, sulfur). Phase connectivity can only be determined with 3D datasets.

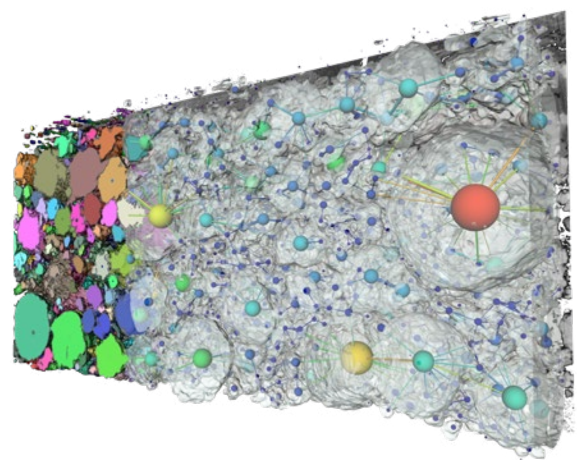
Tortuosity describes the influence of morphology on electrode transport. Specifically, pore tortuosity describes the Li-ion transport capability within the electrode whereas carbon tortuosity is a critical parameter for materials that are not good electronic conductors. Tortuosity distribution analysis is useful for evaluating the homogeneity of electrode transport and can only be determined with 3D datasets.



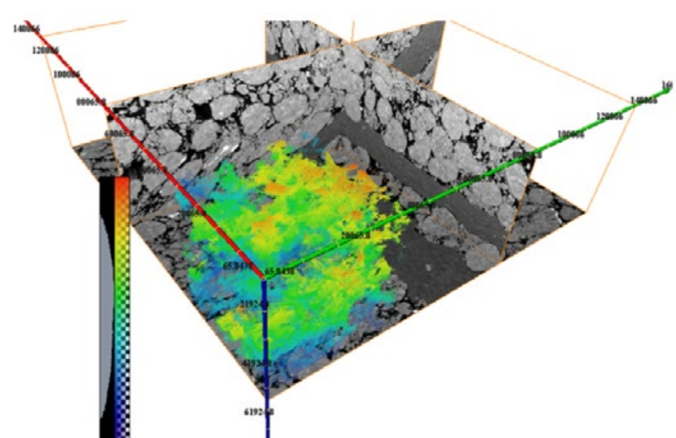
Triple phase boundary length and density is directly correlated to the rate of the electrochemical reactions and, therefore, have a direct impact on fuel cell performance.[2] With FIB-SEM these interfaces are immediately accessible after segmentation. Data courtesy of Sabanci University, Turkey.



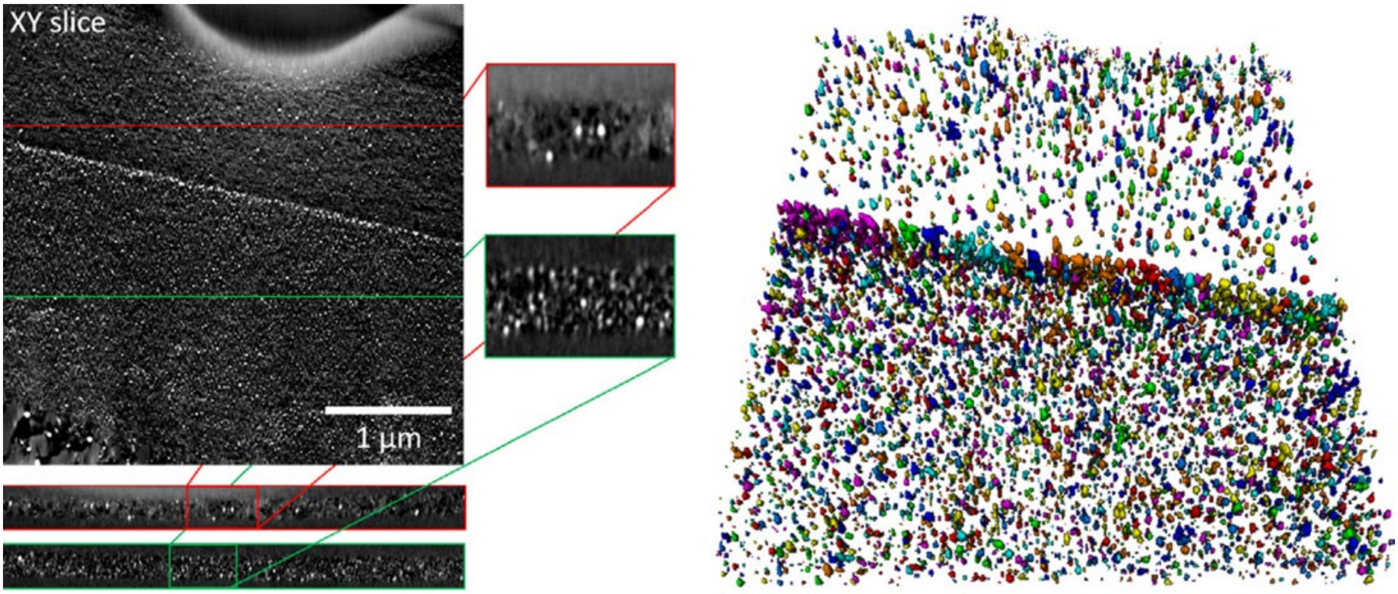
Tortuosity of the battery separator relates to electrical insulation and ion transport properties. This measurement can either be derived purely geometrically or based on simulations. Using a FIB-SEM data reconstruction, averaged 3D tortuosity and a distribution map can be obtained for a lithium ion battery separator.[3]



Pores and/or particles networks extraction: Connectivity of the active material with the conducting network has a critical role in the performance of a battery.[4] After identifying the activate material isolated from the binder, it is possible to generate a model of the particle network for the active material, as is shown here for a lithium-ion battery cathode. Information like particle size and contact surface area are calculated. Data acquired on a Thermo Scientific Helios PFIB DualBeam.



Permeability simulation and estimated pressure field generated from data acquired on the Helios PFIB DualBeam.



Workflow connectivity: A thin lamella was extracted from a pore-filled membrane using a Thermo Scientific Helios 660 DualBeam; subsequent STEM/EDX acquisition was performed on a Thermo Scientific Talos F200A STEM. EDX data was processed using Thermo Scientific Velox Software and 3D tomography was performed using Thermo Scientific XPlore3D Software. STEM and Velox datasets were then loaded into Avizo Software, where volume fraction and distribution of Au and Pb were computed.

Conclusions

Tomography and electron microscopy provide a vast amount of information on the material properties of battery and fuel cell samples; however, simply having this data is not enough without corresponding contextual information. Thanks to advanced image processing and segmentation techniques, Avizo Software makes it possible to take microCT and EM data and extract key quantitative parameters for materials at the micro- and macro- scales.

Avizo Software provides a reliable, fully automatable solution for material analysis in academia and industry, allowing for faster innovation, reduced time to market, and more reliable batteries/fuel cells with enhanced performance.

References

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3. Duda, A., Koza, Z., & Matyka, M. “Hydraulic tortuosity in arbitrary porous media flow.” Physical Review E, 84(3) (2011), 036319.
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