

Unlocking insight in lithium-ion batteries

Analytical solutions for
advancing battery technology

Lithium-ion batteries have dominated the energy market over the last few years, helping drive the transition to a low-carbon society. While battery breakthroughs are happening rapidly, the market continues to demand lower costs, enhanced energy density, and greater safety.

Improving batteries starts with deeply understanding the relationship between structure and performance. Comprehensive analytical characterization with a suite of electron microscopy (EM) techniques complemented by X-ray photoelectron spectroscopy (XPS) makes it possible to ensure that batteries meet required standards and specifications.

Currently, EM enables a suite of characterization workflows for fundamental battery research, as well as quality control (QC) and failure analysis (FA), which can help battery engineers and researchers analyze different parts of battery components and raw materials from the microscale to nanoscale. XPS complements EM's microstructural analysis with detailed surface analysis, helping to identify and quantify the chemical state and associated stability of electrochemical interfaces within a battery system. Together, they enable analytical workflows that can help you overcome the challenges frequently faced when characterizing battery materials and components.

Introduction to lithium-ion battery technology

From a basic electrochemical perspective, lithium-ion batteries are metaphorically referred to as “rocking chair batteries.”

Their operating principle, as illustrated in Figure 1, involves the reversible electrochemical reaction of lithium ions between the crystal lattices of positive and negative

electrode materials to achieve mutual conversion of chemical and electrical energy. However, in practical terms, lithium-ion batteries constitute a highly complex system.

Lithium-ion batteries comprise intricate components such as positive electrodes, negative electrodes, separators, electrolytes, and packaging components. The positive and negative electrodes, as well as the electrolyte, consist

of complex mixtures containing various organic and inorganic components. Dozens of composite ingredients must be combined and processed to manufacture a commercial battery, which involves highly complex procedures and auxiliary materials. Due to this complexity, even identically designed batteries may exhibit different characteristics under different testing conditions and environments, impacting performance across the entire lifecycle of the battery.

The primary scope and core of research and development work on lithium-ion batteries revolve around addressing the scientific prediction and diagnosis of battery performance from the perspectives of materials and processes. However, the complex multi-interface processes influenced by chemical and physical factors during the operation and degradation of lithium-ion batteries is a big challenge. To gain a thorough understanding of the operational mechanisms of lithium-ion batteries and to qualify the manufacturing process, it is essential to establish systematic and reliable research and measurement platforms across multiple dimensions and length scales.

Within the battery industry, it's best to apply these platforms to research and development, quality control, and failure analysis at the same time. Effectively integrating these processes is essential for continuous improvement and innovation in lithium-ion battery technology, addressing challenges, and ensuring the reliability and safety of these energy storage systems.

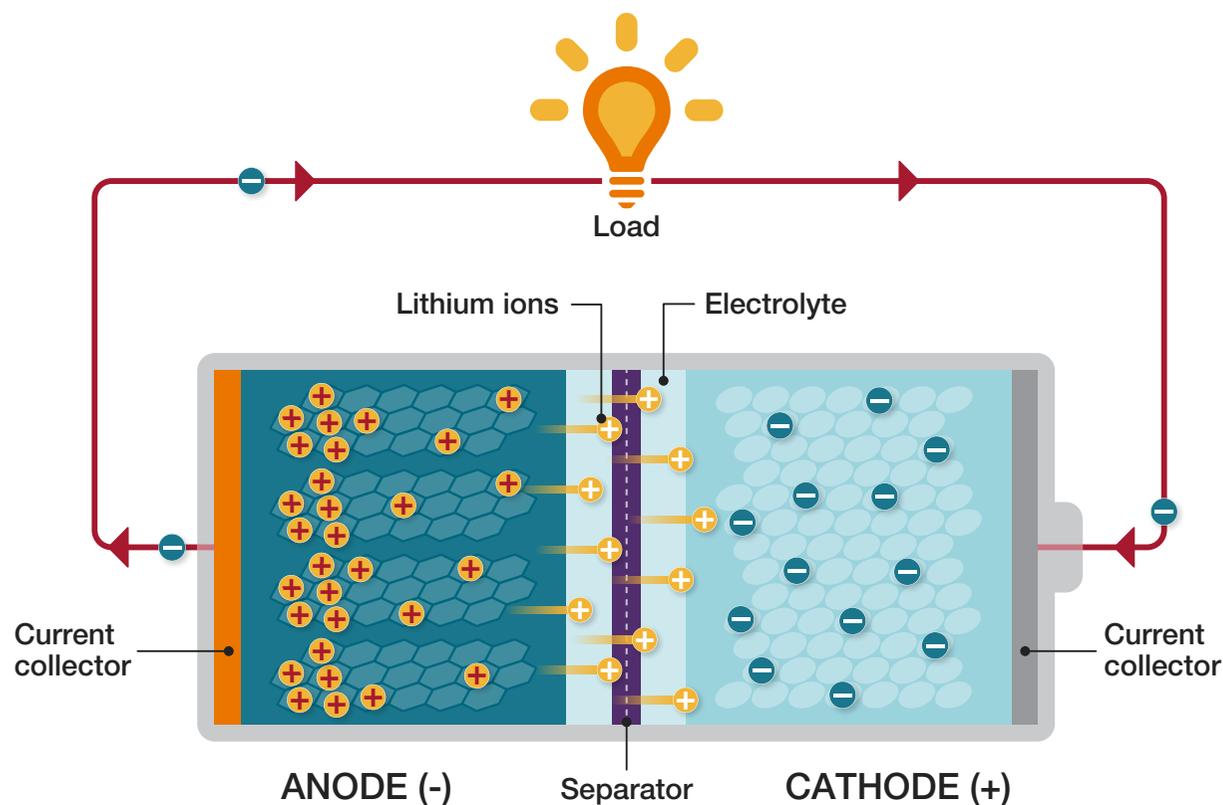


Figure 1: Working principle of a lithium-ion battery.

Research and development

In the research and development (R&D) phase, scientists and engineers work on advancing the fundamental aspects of lithium-ion battery technology.

This includes exploring new materials for electrodes and electrolytes, improving cell design, and enhancing overall performance characteristics such as energy density, cycle life, and safety. The integration of novel electrode materials, particularly with regard to cycling stability, is frequently influenced by the interplay between these materials and their corresponding operational environment. This interaction is evidenced by the formation of an interfacial layer between the active material and the electrolyte, commonly referred to as the solid electrolyte interphase (SEI). Characterizing the SEI in lithium-ion batteries is crucial for understanding battery performance, degradation mechanisms, and safety.

SEI characterization

Due to the air sensitivity and reactivity of the electrolyte and electrode with the electron beam used in an electron microscope, it can be difficult to characterize the SEI layer with EM. However, cryo-electron microscopy (cryo-EM) techniques, in which cryogenic temperature can help stabilize sensitive battery materials and interfaces during sample preparation and characterization, make it possible to maintain sample integrity during EM imaging.

Figure 2 shows the evolution of SEI on silicon nanowires while lithiating and de-lithiating the sample. The sample was held at cryogenic temperature during preparation and imaging to minimize the sample damage from ambient conditions and the imaging beam with the help of a cryo-transfer holder. Figure 2c, a cryo-TEM image created using a Thermo Scientific™ Talos™ F200X TEM, shows that a core-shell structure is formed, wherein crystalline silicon is the core and the amorphous lithium and silicon is the shell (shown in Fig. 2b). Meanwhile, the SEI layer also forms during the first lithiation as an additional shell on the silicon nanowire. Figure 2d, cryo-STEM high-angle annular dark field (cryo-STEM-HAADF) images, shows that the silicon nanowires become porous after de-lithiating, revealing a rough surface and deterioration of the SEI layer. In Figure 2e, 3D cryo-STEM-EDS demonstrates the evolution of SEI and silicon nanowire with increasing

cycles. Cross-sectional segmentation analysis of the 3D chemical composition of the nanowire reveals a gradual growth of the SEI toward the silicon interior with cycling.

Integrating advanced EDS tomography, cryo-STEM, and Thermo Scientific™ Avizo™ Software can elucidate the interconnected structural and chemical transformations occurring within the SEI and silicon constituents of lithium-ion battery electrodes in three dimensions. Moreover, cryo-EM has the potential to uncover previously unknown structures, resolve unsolved issues, and elucidate unclear mechanisms related to cathodes, electrolytes, anodes, and their interfaces in electrochemical devices. For example, it can reveal the nanostructure changes in cathode materials during charging and discharging, provide insights into the structure of solid electrolyte material and its interface with the electrode for solid-state batteries, and track the evolution of nanoscale features within anode materials along with their interfaces when discharging. These findings support the development of more efficient and sustainable energy storage technologies.

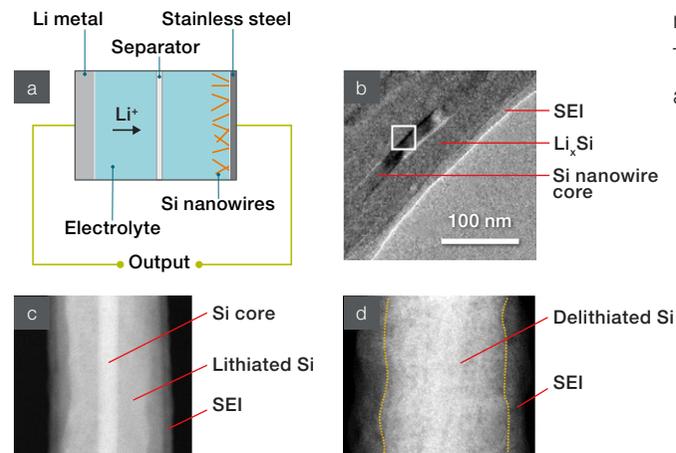
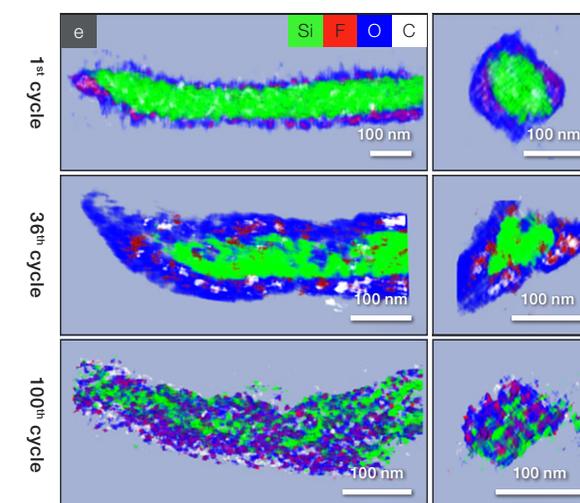


Figure 2: SEI evolution on silicon nanowires while lithiating and de-lithiating captured on a Talos F200X TEM with a Super-X Detector. a.) Schematic of the coin-cell setup. b.) Cryo-TEM images of a silicon nanowire during the first lithiation. c. and d.) Cryo-STEM-HAADF images of the first lithiation and de-lithiation. e.) View of 3D cryo-STEM-EDS chemical composition from two directions segmented in Avizo Software, illustrating the spatially correlated evolution of the silicon an SEI layer with battery cycles. He, Y., Jiang, L. et al. *Nat. Nanotechnol.*, 2021.



Maintaining sample integrity

Many materials used in battery manufacturing, such as lithium metal, sulfide-based solid electrolytes, and charged electrodes, are highly air sensitive, reacting to both moisture and oxygen in the air. When dealing with air-sensitive materials, it's essential to handle them in an environment free of oxygen and moisture to prevent degradation or alteration of their properties. The Thermo Scientific™ Inert Gas Sample Transfer (IGST) Workflow uses inert gases such as argon to provide a controlled environment free of oxygen and moisture.

Lithium metal is well known for being a promising anode material for next-generation rechargeable batteries due to its high theoretical capacity. However, the practical

application of lithium metal batteries is significantly hindered by unstable interfaces, uncontrolled dendrite formation, and rapid capacity degradation. In addition, lithium is difficult to detect in a TEM due to its mobility, beam sensitivity, and light weight.

The IGST Workflow, shown in Figure 3, is specifically designed to reliably and repeatably characterize air-, moisture-, and beam-sensitive battery materials. The IGST Workflow protects both the bulk sample and the prepared lamella against air and moisture contamination by maintaining an inert argon atmosphere with the Thermo Scientific™ CleanConnect™ Sample Transfer System, which moves the sample between the glovebox and the instruments in an inert gas sample transfer

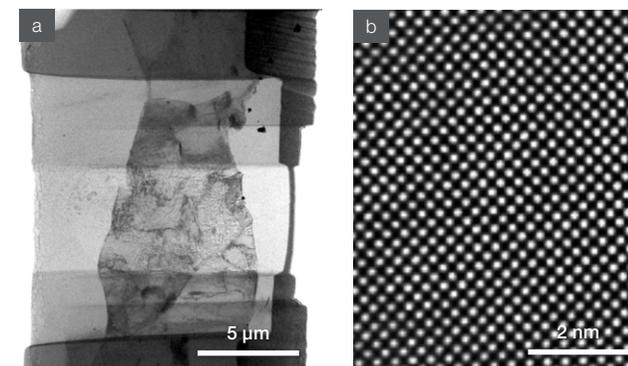


Figure 4: a.) Li-metal TEM lamella prepared with the IGST Workflow. b.) Atomic-resolution TEM image of Li-metal atom columns. The IGST Workflow allow the Li-metal lamella to stay crystalline with minimal signs of surface oxidation.

holder. If the sample is vulnerable to beam damage, characterization at cryogenic temperature via the SEM, FIB-SEM, or (S)TEM can be incorporated into the workflow.

Figure 4a shows the high-angle annular dark field (HAADF) (S)TEM image of lithium metal lamella. In this study, a bulk Li-metal piece was successfully transferred from a glovebox to a Thermo Scientific™ Helios™ 5 Hydra™ DualBeam™ FIB-SEM without signs of surface oxidation, followed by TEM lamella preparation at cryogenic temperature (–178 °C). Figure 4b shows a high-resolution TEM image of the lithium lamella that proves the sample was well protected during the entire workflow.

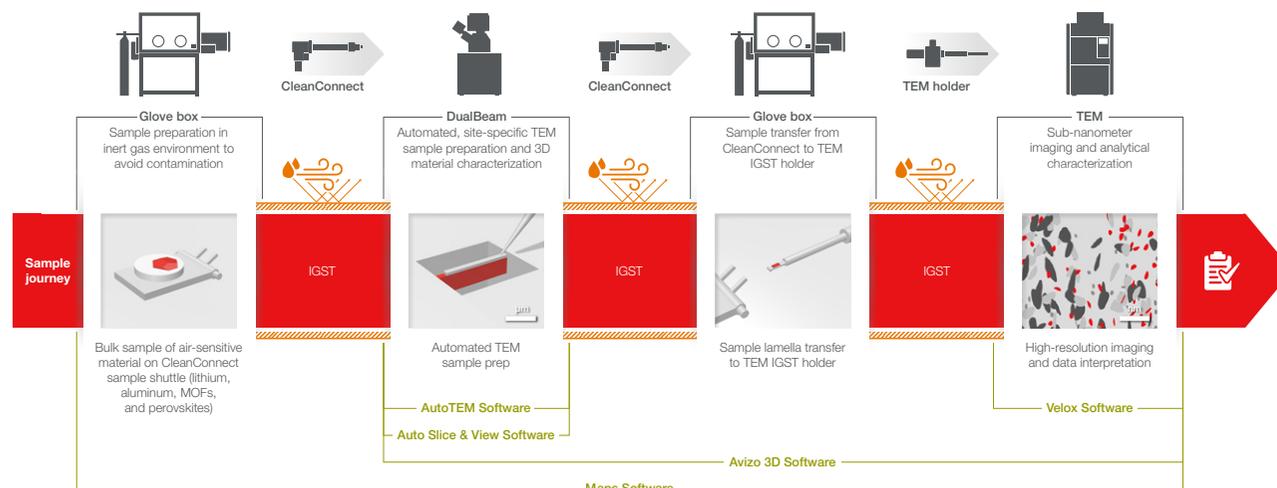


Figure 3: IGST Workflow for nanoscale analysis using Thermo Scientific™ DualBeam™ FIB-SEM and TEM instruments.

Surface residual evaluation

LiNiO₂-based cathode materials, such as Li[Ni_xCo_yMn_{1-x-y}]O₂ (NCM) and Li[Ni_xCo_yAl_{1-x-y}]O₂ (NCA), have been extensively studied and used to develop lithium-ion batteries. However, it is important to note that high nickel LiNiO₂-based cathode materials can be sensitive to moisture and CO₂, leading to the formation of residual lithium compounds on the surface, including LiOH and Li₂CO₃. The presence of these residual lithium compounds can pose challenges in the practical production and application of high-nickel cathode materials. Firstly, they can negatively impact the capacity retention capability of the cathode, leading to a decrease in the overall performance of the battery. Additionally, these compounds can increase the pH of the electrode coating slurry, as well as induce gelation during the electrode preparation process. This can generate a large amount of gas during cycling, further deteriorating the electrochemical performance of lithium-ion batteries.

Consequently, identifying and quantifying surface reaction byproducts from nickel-rich NMC cathodes plays an important role in evaluating the air and water stability of these materials, both before and after modification. Given XPS's efficacy in detecting the chemical composition of battery material surfaces, it can function as a diagnostic tool for assessing the effects of surface modifications in the development of high-nickel cathodes.

Figure 5 shows the XPS spectrum of pristine LiNiO₂ as the air exposure time increased to 28 hours. The Thermo Scientific™ Nexsa™ XPS System was used to analyze the surface of undesired residues of LiNiO₂ particles. Due to the air-sensitive nature of these materials, the Nexsa Vacuum Transfer Module (VTM) was used to safely transport the pristine samples from a glove box to the

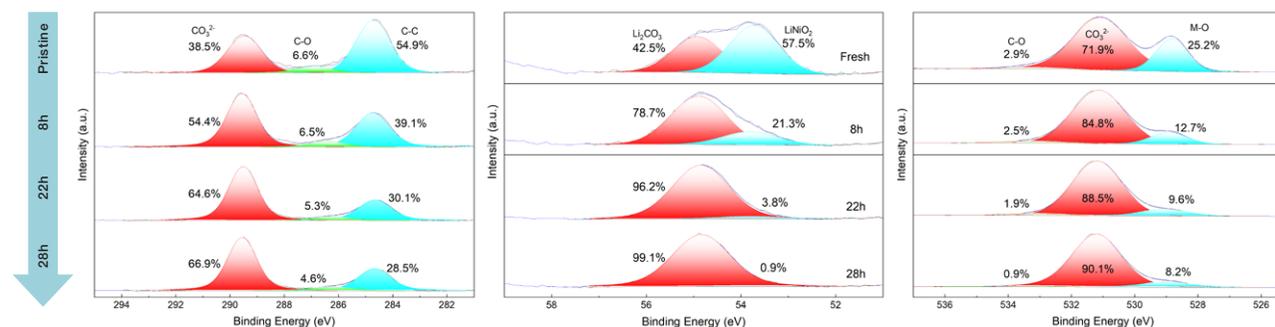


Figure 5: XPS spectra of LiNiO₂ samples under various conditions, including pristine and after being exposed to air for 8, 22, and 28 hours.

instrument without exposure to ambient atmosphere. This ensured that the surface represented the LiNiO₂ material as it was received.

The XPS reveals three spectra (C 1s, Li 1s, and O 1s) related to lithium residuals. To simplify the comparison, the C 1s peak was deconvoluted into two main groups: organic carbon groups and inorganic carbonates (–CO₃) located below 288 eV and above 289 eV, respectively. The organic carbon groups (C–O and C=C) likely originate from adventitious carbonaceous species, while the peak centered at 289 eV can be associated to Li₂CO₃.

The Li 1s spectrum shows a low-binding energy component from lattice Li in LiNiO₂, along with undesired surface residual species of LiOH and Li₂CO₃. The proportion of Li 1s peak intensity indicates an increasing presence of lithium carbonate species and a decreasing amount of LiNiO₂ with increasing exposure time.

A similar trend can be observed in the O 1s spectrum, which demonstrates that the O lattice signal decreases due to the formation of residual lithium species covering the LiNiO₂ particles.

To address the challenges mentioned earlier, various approaches have been employed to modify LiNiO₂-based cathode materials. Among these strategies, doping has emerged as a crucial and highly effective method for enhancing the air and water stability of these materials. Introducing small amounts of cation or anion atoms into the lattice of Ni-rich cathode materials can significantly improve their air and water stability. Ongoing research aims to optimize doping techniques further, with the goal of enhancing the stability and overall performance of Ni-rich cathode materials.

XPS analysis can be used to quantify the amount and distribution of residual lithium compounds on the surface of the cathode material. By combining the insights gained from XPS analysis and optimizing doping techniques, researchers can work toward enhancing the air and water stability of Ni-rich cathode materials, ultimately helping to develop more efficient and reliable lithium-ion batteries.

Quality control

Quality control (QC) is essential in ensuring that the manufacturing processes and the produced lithium-ion batteries meet specific standards and requirements.

This involves monitoring and controlling various parameters throughout the production process, including material quality, cell assembly, and final product testing. The goal is to identify and rectify any deviations from the desired specifications to maintain consistency and reliability.

The quality of raw materials in lithium-ion batteries directly determines the processing performance and operational capabilities of the finished battery. That's why testing incoming materials is indispensable when researching, developing, and manufacturing lithium-ion batteries.

Impurity analysis

Impurities are always introduced when gathering raw material and producing batteries. Impurities can compromise the safety and durability of batteries by causing an internal short circuit, which can occur when large impurity particles mechanically pierce the separator or when metal impurity particles in the high-potential cathode undergo a dissolution-precipitation process. Among these impurities, copper (Cu), characterized by its low dissolution potential and commonly generated in raw material manufacturing, poses a high risk by dissolving on the cathode side, diffusing to anode side,

depositing along the separator's pores connecting the anode and cathode, and causing a micro short circuit. Thus, the battery industry needs an effective methodology to detect and characterize impurity particles in QC.

To address this issue, Thermo Scientific™ Perception Software for batteries automates the analysis process and reduces the time per particle to less than one second, providing quick results and generating detailed reports with conclusions.

The example report in Table 1 includes impurities identified in NMC powders with both composition and size information. It shows whether particles within different size ranges meet the specified standards. Rows represent particle categories and columns show the size distributions of individual particle categories.

ISO16232 / VDA-19 Results					
Size class		B	C	D	E
Size Range (µm)	Total	5≤X<15	15≤X<25	25≤X<50	50≤X<100
Cu-rich	1104	888	163	52	1
Iron-rich	1192	83	678	427	4
Al-rich	7	6	1		
Zn-rich	3	3			
Al-silicates	0				
Si-rich	400	394	5		1
Total counts	2706	1374	847	479	6
Clean level		11	10	9*	3
Component clean code (CCC): V(B11/C10/D9/E3/F00/G00/H00/I00/J00/K00)					
Specifications: V(B12/C10/D8/E6/F0/G0/H0/I0/J0/K0) *Does not pass specification					

Class	Color	Sub total
High conductivity		2296
Mid conductivity		10
Low conductivity		400

Table 1: Custom report and classification of identified particle contamination in NMC powders generated by Perception Software.

It is critical for battery industries to trace and pinpoint the source of impurities. That's why impurity particle details, including morphology and chemical composition, are crucial for efficiently identifying the source of impurity particles. Perception Software can help you precisely reposition the particles of interest and conduct in-depth analysis.

Table 2 shows the detailed information for impurity particles detected in the sample. This information can be extremely helpful for establishing a database of impurities.

Morphology control

Cathode precursor morphology plays a critical role in the final structure of cathode materials that impact the materials' performance.

The morphology of precursor can determine the particle size, surface area and porosity, and pore structure of the final cathode material. A controlled precursor morphology can result in desired particle size distribution and higher surface area, which enhances the electrochemical performance of the cathode material through improved lithium-ion diffusion, electrode compacted density, and electrolyte wettability. Moreover, the morphology of precursor affects the distribution of nickel, manganese, and cobalt atoms within the cathode material, as well as the crystal structure and phase purity of the cathode material, which impact the electrochemical stability, capacity retention, and overall behavior of the cathode material. Therefore, analyzing and monitoring the nanometer-scale features (e.g., primary particles) on the cathode precursor surface is critical in production QC.

Using the characterization of $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})_2$ particles as an example, as shown in Figure 6, imaging at a higher acceleration voltage of 5 KeV does not well capture the fine structure information on the particle surface due to the large interaction volume. Low-kV SEM imaging at 800 eV provides high-quality data, with details of fine structures clearly captured. This imaging approach is an effective method for characterizing the finely detailed surface structure on battery materials. If the material is beam sensitive, using low-energy imaging will minimize or eliminate any damage the SEM might induce.

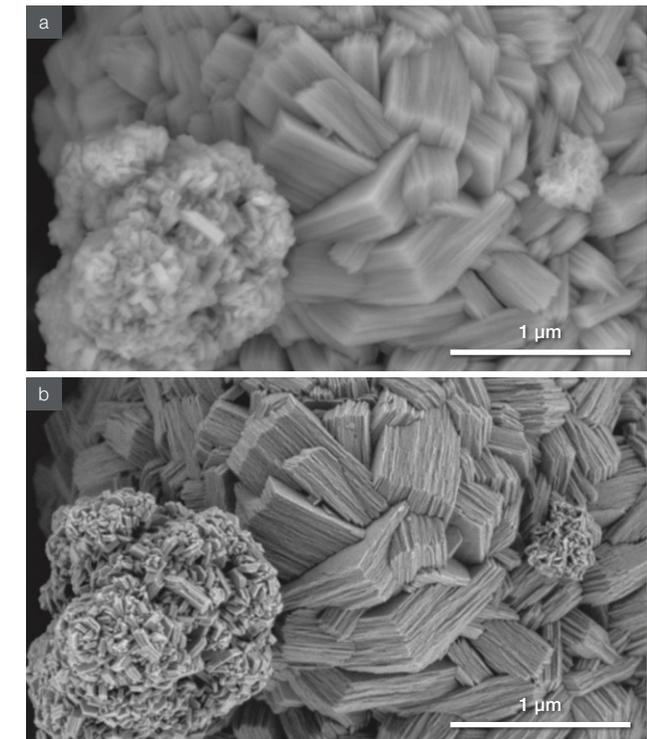


Figure 6: Images of $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})_2$ at 5 keV (a) versus 800 eV (b) using the Apreo ChemiSEM System.

Rank	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge roughness	ECD
1	246	1	53.702	30.757	Iron-rich	-7.477	0.742	1.751	1294.059	0.571	0.556	1.306	40.591

Rank	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge roughness	ECD
2	612	1	50.219	44.017	Cu-rich	-6.457	-5.879	1.177	1230.267	0.621	0.348	1.131	39.578

Rank	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge roughness	ECD
3	522	1	48.919	25.796	Iron-rich	7.447	-3.004	2.199	860.571	0.458	0.502	0.745	33.102

Table 2: Particle information table showing contaminant particles detected in NMC powder.

Binder imaging

When manufacturing lithium-ion battery electrodes, various materials are dispersed in a solvent to form a slurry. The slurry is coated onto a current collector, then the solvent is removed through a drying process to produce the electrode. However, conditions during the drying process, such as temperature, can impact the distribution of binders within the electrode. This may lead to uneven binder distribution, reduced bonding strength, or accumulation of binders on the surface of the electrode during solvent evaporation, causing localized electron resistance and hindering lithium-ion transport. These issues can decrease the battery's electrical performance. Therefore, characterizing the distribution of binders in the electrode is crucial for optimizing the research and manufacturing processes of high-performance batteries.

The binder employed in the commercial graphite anode electrode consists of sodium carboxymethyl cellulose (CMC) and styrene-butadiene rubber (SBR), primarily composed of light carbon, hydrogen, and oxygen elements. This makes it extremely challenging to characterize binder distribution with EM due to its beam sensitivity and lack of contrast to graphite background with traditional SEM imaging.

The images in Figure 7 depict secondary electron images of two different formulations of graphite electrode surfaces, captured using low-voltage imaging technology with the Thermo Scientific™ Apreo ChemiSEM™ System. In the images, the darker regions attached to the graphite surface represent the distribution areas of SBR, which are circled by yellow dashed lines.

In Figures 7a and 7b, the contact area between SBR and graphite is larger, with SBR spread evenly across the graphite surface. This configuration typically results in

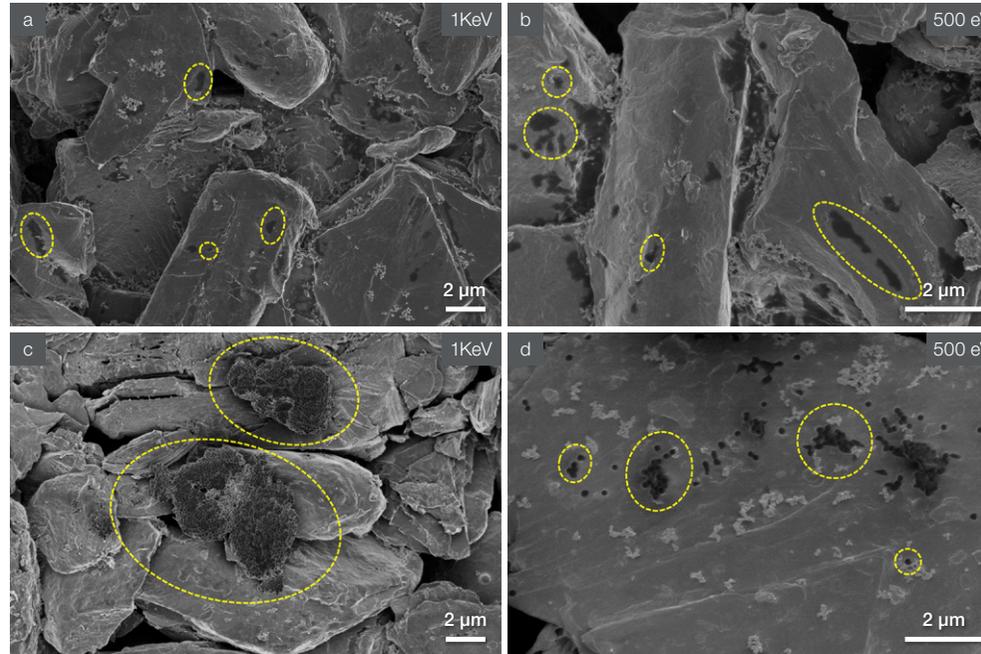


Figure 7: Various graphite and electrode surfaces imaged at low voltage using the Apreo ChemiSEM System.

higher binding strength and cohesion in the electrode. In contrast, Figures 7c and 7d show smaller contact areas between SBR and graphite, with the binder maintaining a spherical shape adhering to the graphite electrode surface and localized agglomeration. This configuration may lead to lower binding strength and cohesion in the electrode.

However, in Figures 7a and 7b, the larger contact area between the binder and graphite electrode reduces the electrolyte's contact area with the graphite surface. This reduction impedes the diffusion of lithium ions on the graphite surface, affecting the electrochemical reactions at the graphite surface and resulting in poorer rate performance. On the other hand, in Figures 7c and 7d, the smaller contact area between the binder and

graphite allows the electrolyte to adequately infiltrate the graphite surface. This, in turn, reduces the impedance of lithium-ion transport on the graphite surface, resulting in improved rate performance.

Thanks to the Apreo ChemiSEM's unique Thermo Scientific Trinity™ In-Column Detection System, combining low-energy imaging with T2 in-lens second electron detector, you can image battery materials with high resolution and make it possible to visualize the binder in the anode and displays the connectivity between the binder and active particles. This enables qualitative elucidation of the binder's role and distribution within the electrode structure, which can be helpful when monitoring binder distribution and thus optimizing the mixing and coating process.

However, visualizing and qualitatively analyzing the binder is insufficient to meet the demands of research and production. Therefore, conducting quantitative analysis to understand the distribution of binders and establishing correlations between binders and cell performance has become a point of interest in both the academic and industrial sectors.

In some graphite and SiO_x anodes, constituents such as PVDF and CMC contain carbon and are composed of low atomic number elements like fluorine and oxygen. Although it is challenging to separate these features using scanning electron microscopy (SEM) imaging alone,

3D energy-dispersive X-ray spectroscopy (EDS) can effectively address this issue, as it can be combined with SEM images to study the spatial distribution of low atomic number elements in electrode components. Meanwhile, Thermo Scientific™ Auto Slice & View™ Software with analysis capabilities can collect EDS maps from focused ion beam-polished cross-sections and output elemental distribution maps for each layer.

As shown in Figure 8, after serial sectioning and subsequent 3D characterization with the Thermo Scientific™ Scios™ 2 DualBeam™ FIB-SEM, EDS maps of each cross-section clearly display the distribution of

silicon (SiO_x particles), oxygen (CMC), fluorine (PVDF), and carbon (graphite). EDS mapping continuously analyzes elemental information for each layer throughout the entire FIB polishing process, forming a 3D volume containing chemical information. After processing with Avizo Software, the spatial distribution of various components in the 3D electrode volume can be obtained, including the binder, within the electrode volume.

When combined with EDS, SEM and FIB-SEM serve as potent tools for characterizing binder materials in lithium-ion batteries. These techniques make it possible to create detailed 2D and 3D representations of binder dispersion throughout the electrode. Incorporating Avizo Software into the analytical process further enhances visualization of binder spatial distribution within the electrode volume.

The information from these characterization methods could provide critical insights into further optimizing the binder composition and monitoring the electrode fabrication process to enhance battery performance and longevity.

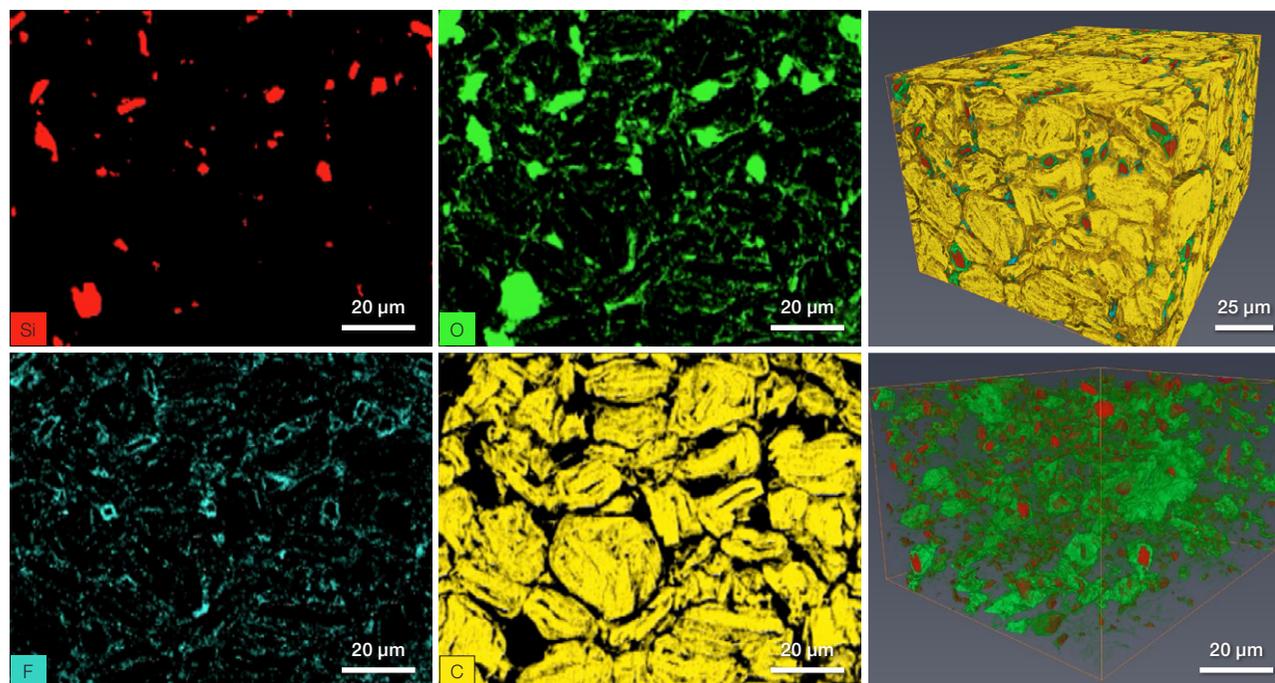


Figure 8: 3D-EDS results showing the spatial distribution of CMC additives, PVDF binder, and SiO and graphite particles in a graphite anode.

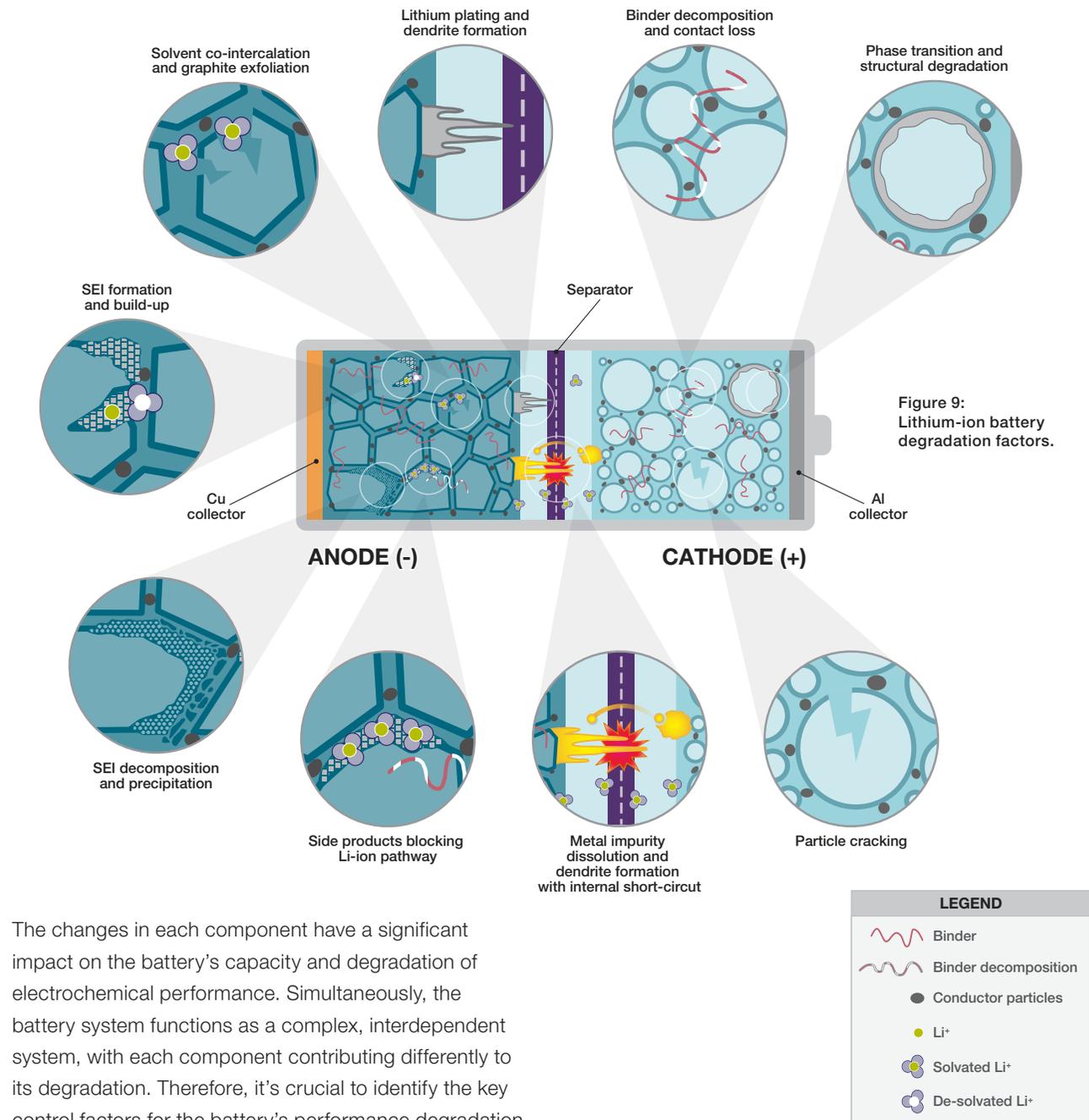
Failure analysis

Failure analysis (FA) comes into play when issues or failures occur during the R&D or QC phases or when batteries in the field experience problems.

The FA process involves investigating and diagnosing the root causes of failures, which may include examining manufacturing defects, material issues, design flaws, or external factors impacting battery performance. The insights gained from failure analysis help refine R&D efforts and improve QC procedures.

Commercial lithium-ion batteries often exhibit various failure phenomena during cycling or storage, including capacity degradation, increased internal resistance, decreased rate capability, gas generation, leakage, short circuits, deformation, thermal runaway, and lithium plating. These failures significantly degrade the performance, consistency, reliability, and safety of lithium-ion batteries, and they arise from a complex interplay of chemical and physical mechanisms within the battery.

Battery degradation is the gradual decline of a battery's ability to store and deliver energy, which leads to reduced capacity and overall efficiency. As summarized in Figure 9, a series of chemical and electrochemical reactions occur inside the battery during the charging and discharging cycles, leading to changes in various raw materials. This can include changes in the cathode structure, fracturing of particles in the cathode and anode's active materials, damage to the conductor and binder network, oxidative decomposition of the electrolyte, and the formation, growth, and repair of the SEI on the surface.



The changes in each component have a significant impact on the battery's capacity and degradation of electrochemical performance. Simultaneously, the battery system functions as a complex, interdependent system, with each component contributing differently to its degradation. Therefore, it's crucial to identify the key control factors for the battery's performance degradation.

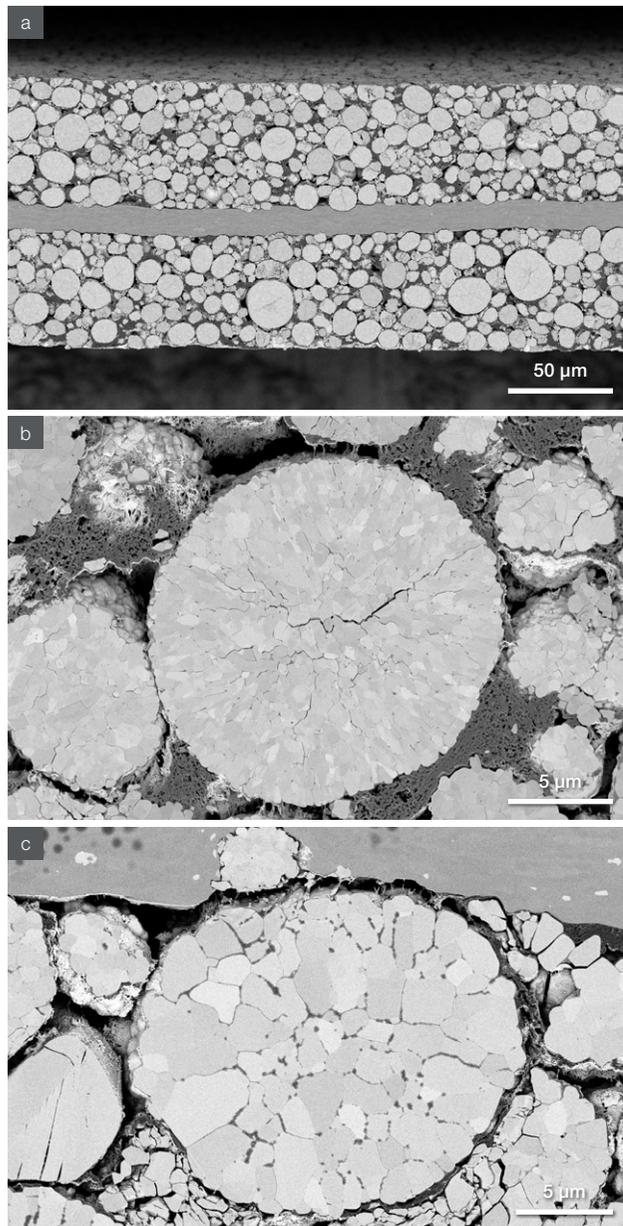


Figure 10: a. and b.) BSE images acquired at 2 keV of a Li-NMC oxide sample that was transferred to the Apreo 2 SEM with the CleanConnect System. c.) BSE image of a Li-NMC oxide sample acquired in the Apreo 2 SEM without air protection.

2D characterization

Post-mortem analysis of disassembled cells is commonly used in the battery industry to conduct failure analysis. To characterize the as-disassembled electrode samples in their native states, it is critical to protect the sample from air and moisture to avoid side reactions. Figure 10 shows a cross section of a cycled Li-NMC cathode sample analyzed to understand the impact of cracks on battery degradation.

The sample was loaded into the CleanConnect System in an argon-filled glovebox and transferred into the Thermo Scientific™ CleanMill™ Broad Ion Beam System (BIB) to create a wide cross section through the whole electrode that was rapidly polished into a high-quality surface. From there, it was transferred to an Apreo 2 SEM, once again inside the CleanConnect System, for imaging, as shown in Figure 10a and 10b. High-resolution BSE images showed clear primary particle contrast and particle cracks. Figure 10c shows the cross-section of an air-polluted sample that was moved without the CleanConnect System.

In this sample, contamination filled the cracks and there is poor contrast between the primary particles. The contamination on the surface and cracks will impact the root cause analysis of the cell failure.

3D characterization

Traditional 2D characterization techniques, such as SEM with BIB for sample preparation, can provide plenty of insights from the cross-sectional structure to battery failure. But when evaluating the complex morphologies and interconnected nature of different components in electrodes, such as pores, active material, carbon additive, and binder, a full 3D image characterization and analysis workflow is necessary, as shown in Figure 11. This approach delivers quantifiable evidence for the observed features, which is helpful in precisely determining the relationship between structure and performance. This allows for a comprehensive understanding of the spatial distribution, connectivity, and interactions between these components, as well as the relationship of structure and performance.

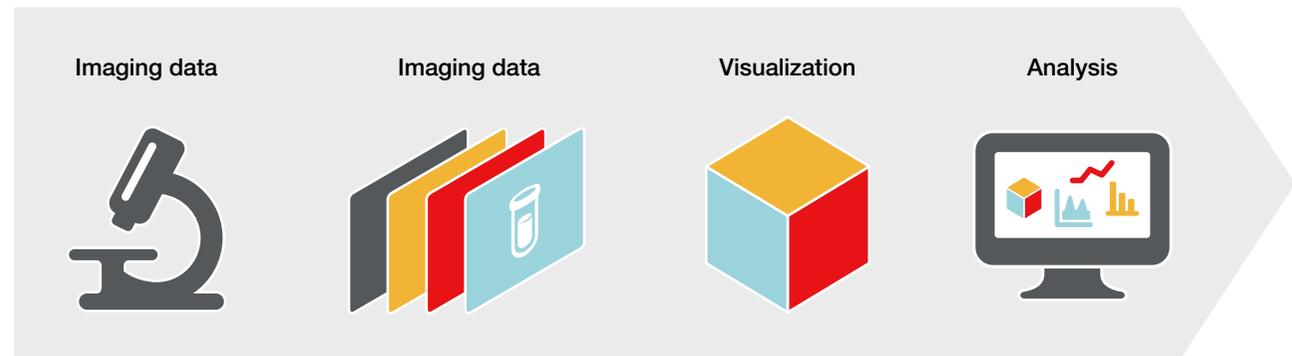


Figure 11: Avizo Software imaging analysis workflow.

Auto Slice & View Software is an automated application for DualBeam FIB-SEMs that acquires data by milling serial sections (slices) and then imaging each slice of a user-defined region of interest within the sample. Figure 12 shows the analysis of a cycled NMC cathode from serial raw 2D data achieved by Auto Slice & View Software and the final reconstruction using Avizo Software. The raw data (Figure 12a) shows microstructural features in the cathode sample, including cracks inside the NMC particle, the carbon and binder domain (CBD), and pores between the particles.

Avizo Software segments and quantifies various features, as illustrated in Figure 12b. Different levels of image contrast distinguish and segment distinct components, such as NMC particles (red), cracks (green), carbon binder (dark blue), and pores (light blue). Following the segmentation process, Avizo Software facilitates quantification of these identified features.

It is important to note that Figure 12a and 12b specifically present data from a single slice. However, the comprehensive Auto Slice & View Software dataset comprises multiple images. These images are cross

sectioned sequentially, maintaining a consistent 100 nm distance between slices. Avizo Software can analyze both individual 2D images and the complete 3D reconstruction, allowing for quantification of segmented features in a three-dimensional context, as depicted in Figure 12c.

Accurately quantifying these segmented features can help you identify signs of failure and pinpoint the root causes of various failure modes. This, in turn, can guide strategies to improve battery design and material selection, leading to more durable and reliable lithium-ion batteries.

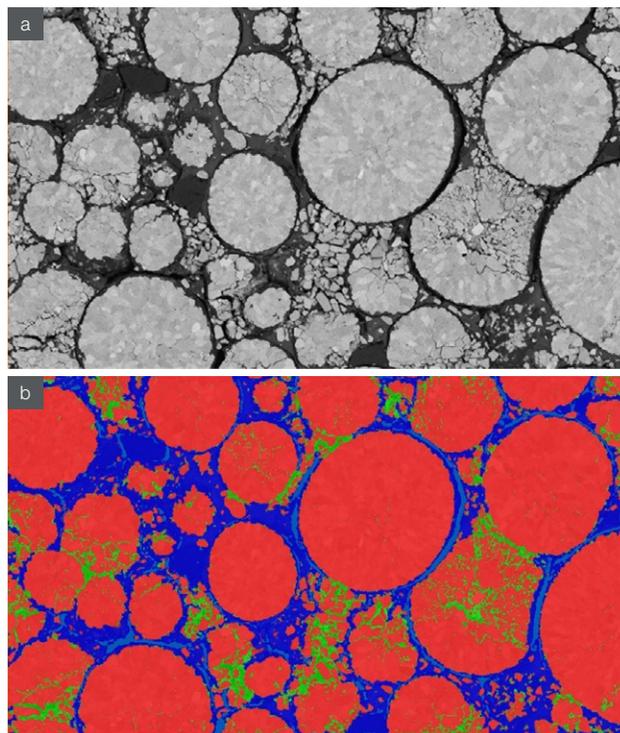
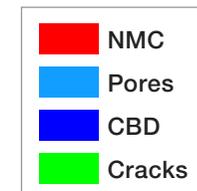
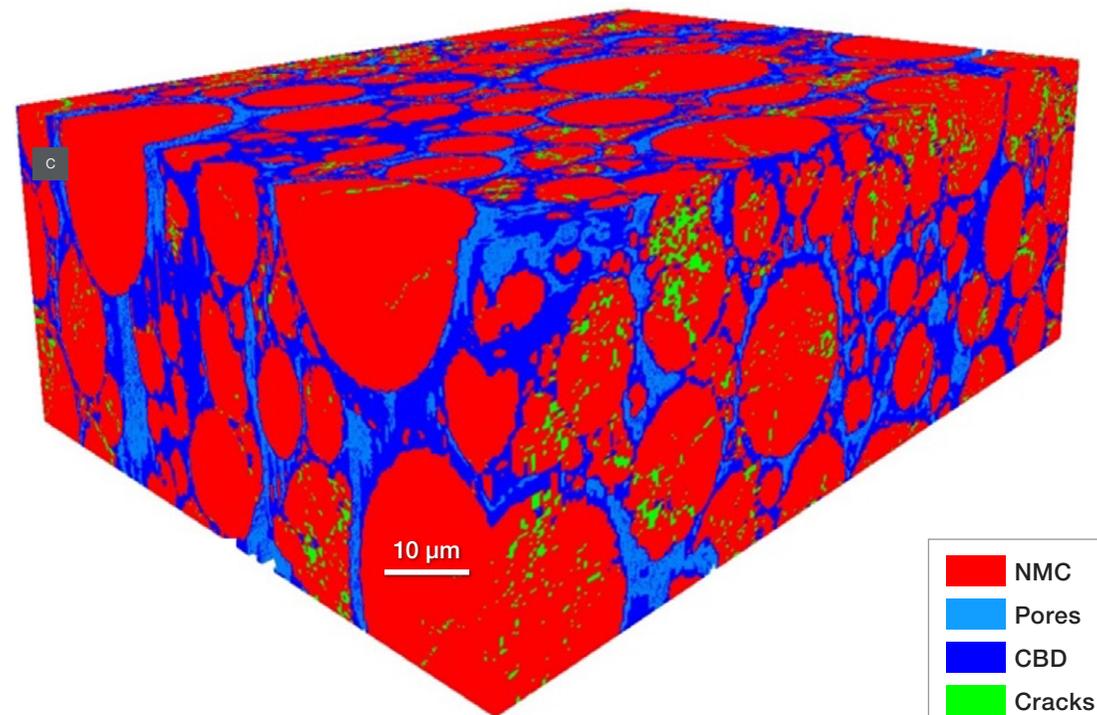


Figure 12: Advanced image processing with Avizo Software allows quantification of different microstructural features of an NMC cathode from Auto Slice & View Software 3D data acquired with a Helios 5 PFIB. Zhang, Minghao, et al. *Joule* 7.1 (2023): 201-220.



Summary

Thermo Fisher Scientific's EM and XPS solutions are essential for comprehensive battery development, providing analytical capabilities from atomic to macroscopic scales. TEMs offer nanoscale examination of critical structures such as dendrites and SEI layers, which is crucial for understanding battery performance. DualBeam™ technology facilitates precise, site-specific, cross-section preparation and 3D characterization, aiding in the identification of defects and detailed analysis of battery electrode structures. SEMs contribute to the identification of surface and cross-sectional defects, while XPS systems provide vital surface chemistry analysis, especially for the SEI layer.

In addition, the Thermo Scientific™ IGST Workflow enables the study of air-sensitive and beam-sensitive materials in their native states, which is vital for the comprehension of battery chemistry and material impacts on performance and safety.

These advanced analytical instruments from Thermo Fisher Scientific provide researchers with the detailed information necessary to innovate and refine battery technologies, contributing to the development of more efficient, safer, and longer-lasting energy solutions.

Thermo Scientific instruments

From research and development to quality control and failure analysis, there's a Thermo Scientific instrument to support every step of your battery workflow.



CleanMill™ Broad Ion Beam System

High-quality cross-section and surface sample preparation with protection for air-sensitive samples.

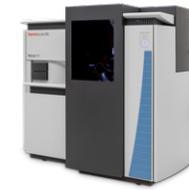
[Learn more >](#)



Phenom™ ParticleX™ Desktop SEM

Desktop scanning electron microscope for advanced battery materials analysis.

[Learn more >](#)



Nexsa™ G2 Surface Analysis System

X-ray photoelectron spectrometer with automated surface analysis and multi-technique capabilities.

[Learn more >](#)



Apreo ChemiSEM™ System

Scanning electron microscope for high-performance imaging with integrated chemical analysis and structural characterization.

[Learn more >](#)



Scios™ 2 DualBeam™ FIB-SEM

Focused ion beam scanning electron microscope for ultra-high resolution, high-quality sample preparation and 3D characterization.

[Learn more >](#)



Helios Hydra™ DualBeam™ PFIB-SEM

PFIB SEM with multiple ion species for 3D EM and TEM sample preparation.

[Learn more >](#)



Talos™ F200X TEM

TEM microscope for high-resolution TEM and STEM with accurate chemical quantification.

[Learn more >](#)



Iliad™ (S)TEM

Advanced scanning transmission electron microscope for high-resolution imaging and analysis for a wide range of applications.

[Learn more >](#)

About Thermo Fisher Scientific

We are the world leader in serving science. Our Mission is to enable our customers to make the world healthier, cleaner and safer.



Learn about the mission of Thermo Fisher Scientific. Duration 1:23.

Our innovative solutions for electron microscopy, surface analysis, and microanalysis help materials science researchers advance their sample characterization to gain deeper insight into the physical and chemical properties of materials from the macroscale to the nanoscale. Our multiscale, multimodal solutions cover a broad range of applications across dozens of industries and research fields, serving customers in academia, government, and industry. Our TEMs, DualBeam™ FIB-SEMs, comprehensive portfolio of SEMs, XPS, and microanalysis solutions, combined with software suites, take customers from questions to usable data by combining high-resolution imaging with physical, chemical, elemental, mechanical, and electrical analysis across scales and modes.

Financial and Leasing Services

At Thermo Fisher Scientific, we will not let budgetary constraints stand between you and your next great discovery.

We are your one-stop partner for the best laboratory products and analytical technologies available, plus the unique financing options you need to accelerate success in science or industry.

Cost-effective financing designed for each individual customer is key to any successful capital equipment solution.

We understand not just your advanced technology and application requirements, but the business challenges you face when financing your critical equipment assets. For decades, we have worked closely with businesses, hospitals, universities, and municipalities to provide flexible financing terms to support their successful operations.

If you are looking for off-balance sheet financing, accelerated ROI, technology protection, or cash flow management, our innovative financing options can help meet your company's budgetary needs and bottom-line goals.

We also offer instrument maintenance and training services.

[Explore equipment leasing and financing options](#)

Thermo Scientific™ Avizo™ Trueput Software for Battery Quality is an archive of workflows for the quality inspection of battery samples.

Learn more at thermofisher.com/batteryresearch

thermo scientific

For research use only. Not for use in diagnostic procedures. For current certifications, visit thermofisher.com/certifications

© 2024 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific and its subsidiaries unless otherwise specified. BR0190-EN-11-2024