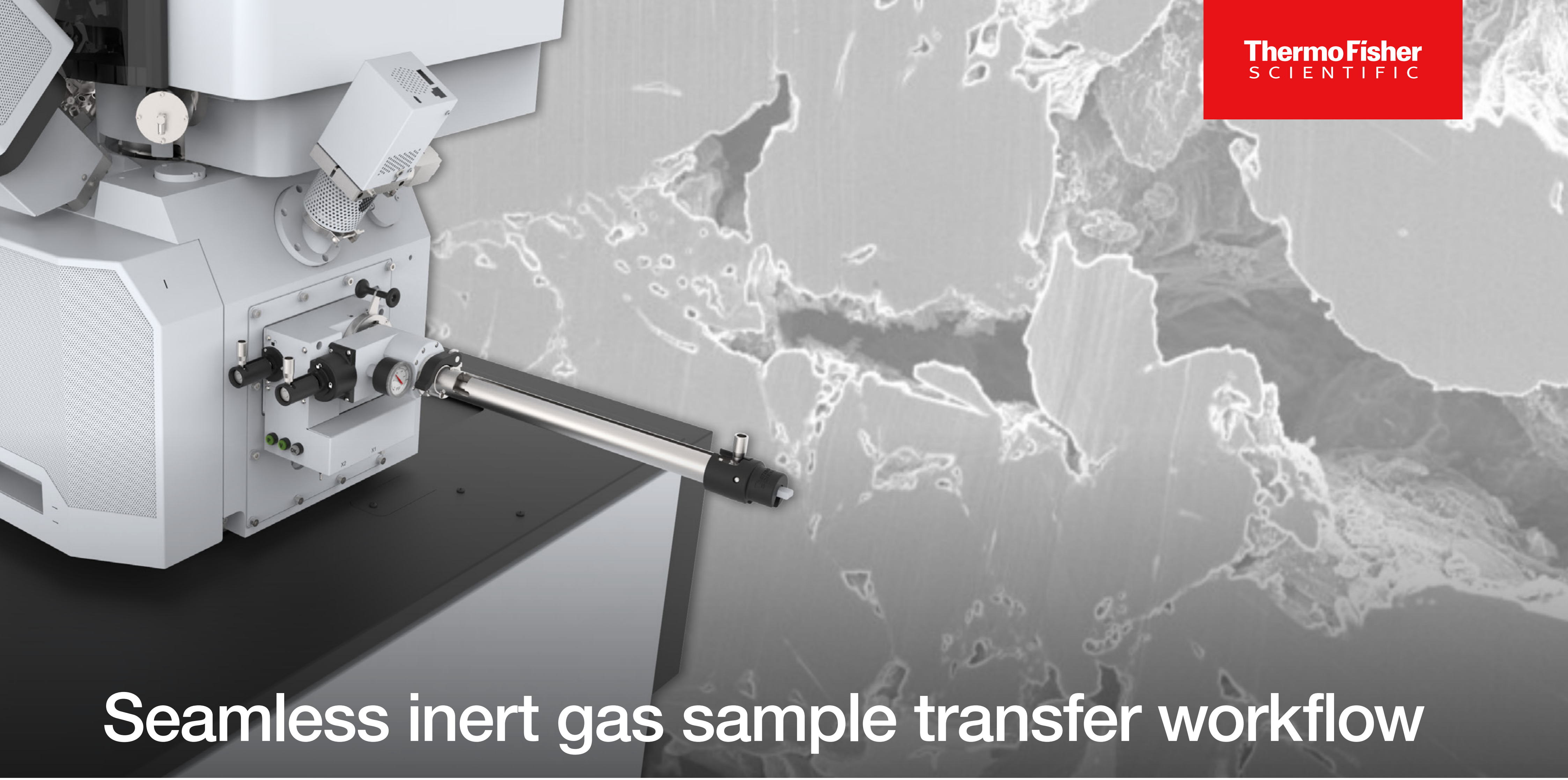

Seamless inert gas sample transfer workflow

Analyze air-sensitive materials in their native state

thermo scientific

Exploring new possibilities

Transitioning to a net-zero world is one of the greatest challenges humankind has ever faced. The energy sector is one of the biggest sources of greenhouse gas emissions and holds the key to averting the worst effects of climate change. However, today's energy storage devices are limited by the performance of their constituent materials. Overcoming these limitations requires deeper understanding of the physical and chemical properties of the materials. The material research becomes even more challenging if the material is air-/moisture-sensitive.

Introducing the Thermo Scientific™ Inert Gas Sample Transfer (IGST) Workflow, designed to enable new insights into the world of air-sensitive materials. With the right tools and techniques, the IGST Workflow can enable you to push the boundaries of your research and step into the unexplored world of air-sensitive materials.

Introduction

Researchers want answers to their material research without thinking about limitations on equipment connectivity.

An optimum sample preparation and sample transfer between various instruments without compromising sample integrity is crucial for any microscopic material analysis.

Sample preparation for scanning/transmission electron microscopy (S/TEM) analysis is one of the most critical and time-consuming tasks. It becomes even more challenging if the sample material is air- and/or moisture-sensitive.

The Thermo Scientific™ IGST (inert gas sample transfer) Workflow uses tools like the Thermo Scientific CleanConnect™ Sample Transfer System to allow you to focus on your research rather than worry about contaminating the sample.

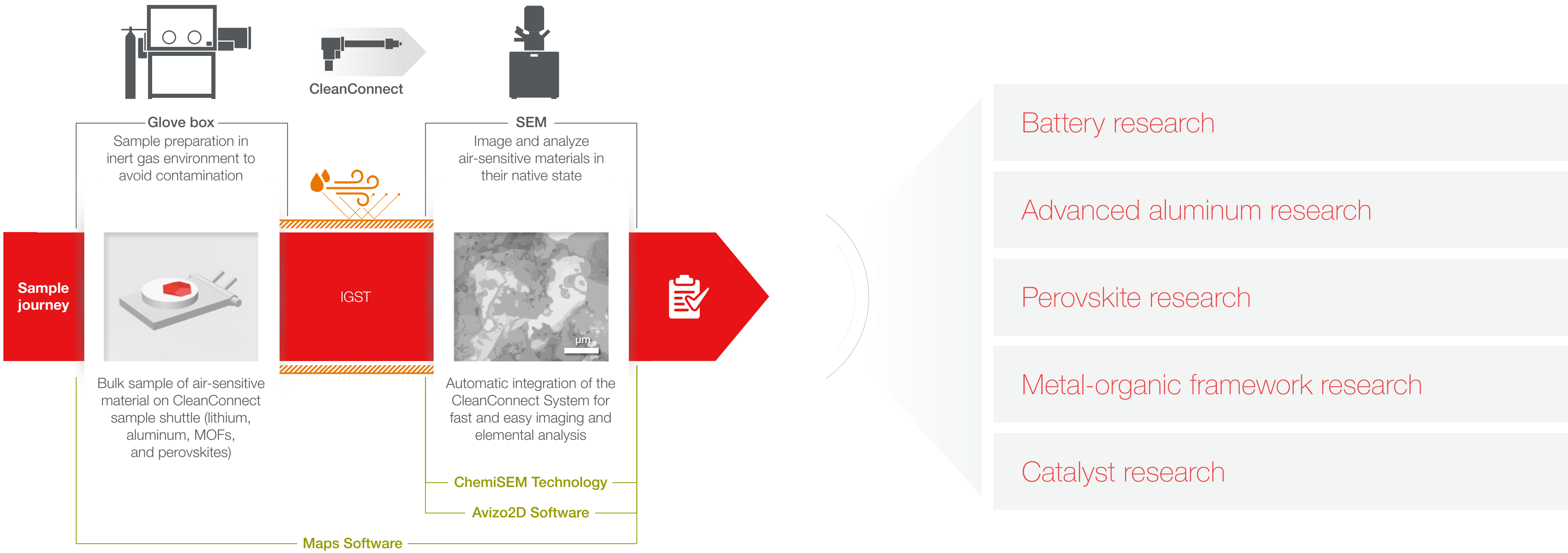

Figure 1. Inert gas workflow using Thermo Scientific scanning electron microscopes.

Key benefits

- Sample integrity is preserved, resulting in high-end material characterization in its native state
- Ergonomic and modular design of CleanConnect System enables simple sample handling
- In-house design of CleanConnect System enables seamless connectivity and automatic integration with a variety of SEMs and DualBeams
- Cost savings with no modification to existing glove box
- Compatibility with cryo-stage allows sample integrity while milling against high temperatures

Whether you are conducting cutting-edge materials R&D at atomic level or simply interested in microanalysis of air-sensitive material like lithium, the CleanConnect System is compatible with a variety of SEMs and DualBeam™ systems to enable results-focused workflows.

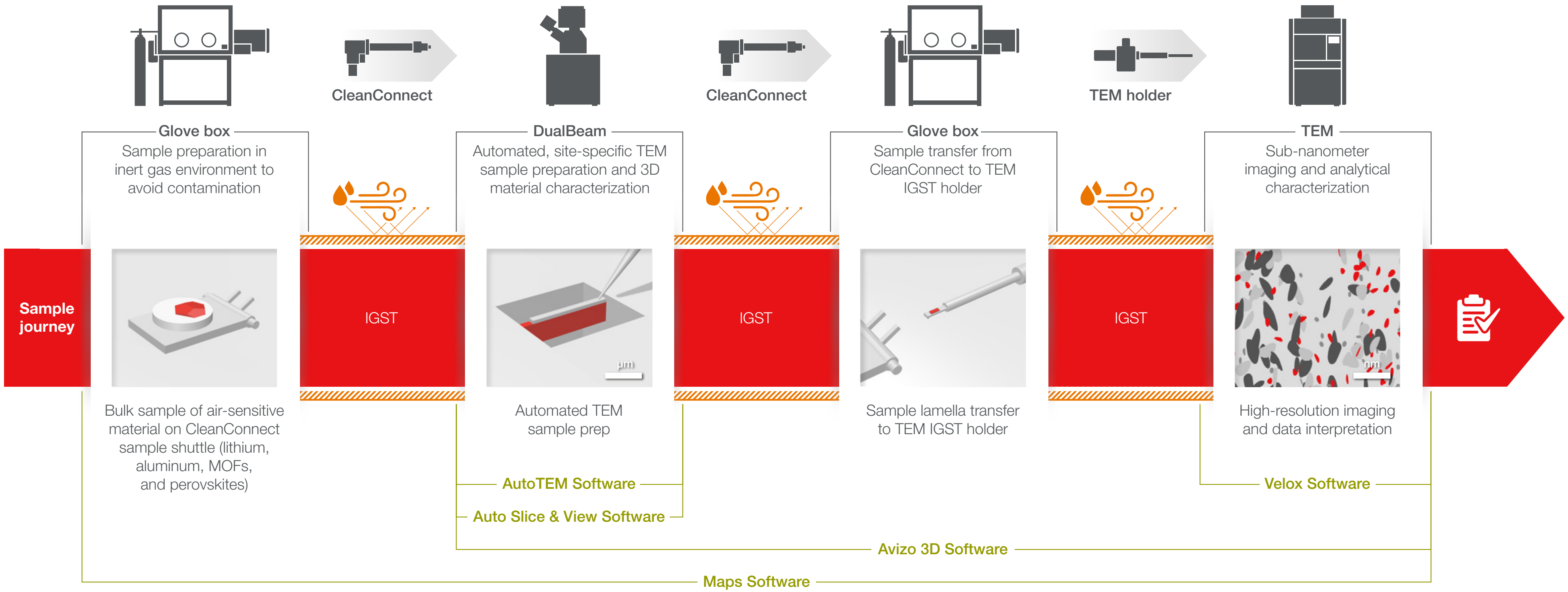

Figure 2. Inert gas workflow for nanoscale analysis using Thermo Scientific DualBeam and TEM systems.

Comparison of workflows

With the ultimate goal to reduce the greenhouse gases that trap heat in the atmosphere and warm the planet, material scientists and industrial researchers are accelerating the efforts to build better batteries that will pave the path toward a clean-energy future.

To get deeper understanding of the future requirements and to be able to design cheaper, more efficient, more flexible batteries, they need to build strong fundamental battery chemistry science. Our analytical solution offers a variety of capabilities to address some of these challenges.

Figures 3 and 4 show a comparison of an inefficient sample transfer with its risk of lithium and charged graphite anode contamination versus a seamless sample transfer to SEM using the CleanConnect System. The pristine metal surface allows in-depth analysis of the surface features. Without the CleanConnect System, this is not possible, as the sample surface would be completely modified.

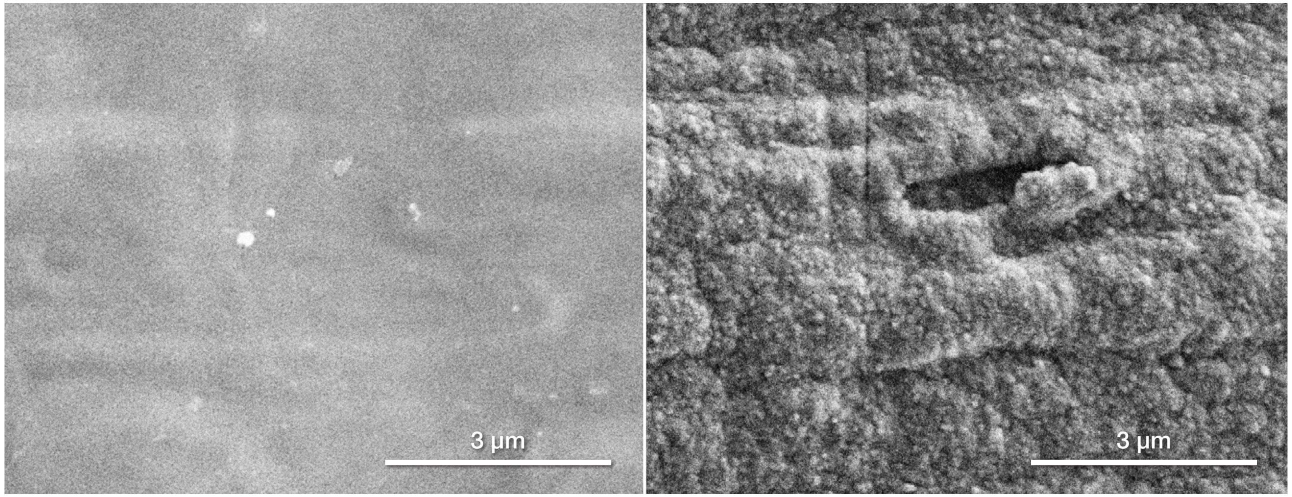

Figure 3. Lithium transfer using the CleanConnect System (left) versus lithium transfer in air, ~2 minutes (right).

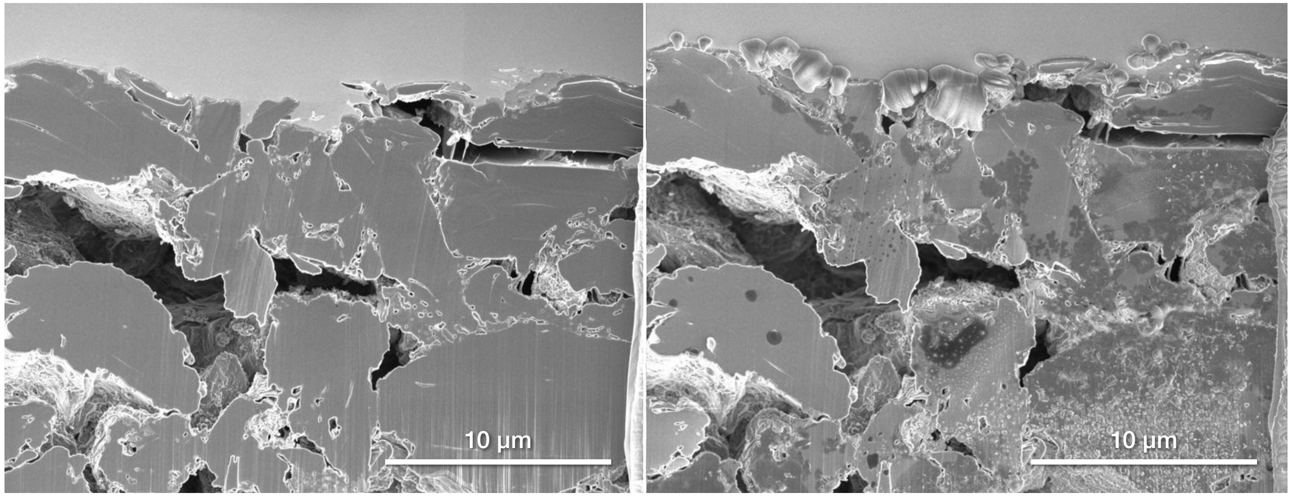

Figure 4: Charged graphite anode transfer using the CleanConnect System (left) versus transfer in air, ~2 minutes (right).

Battery characterization

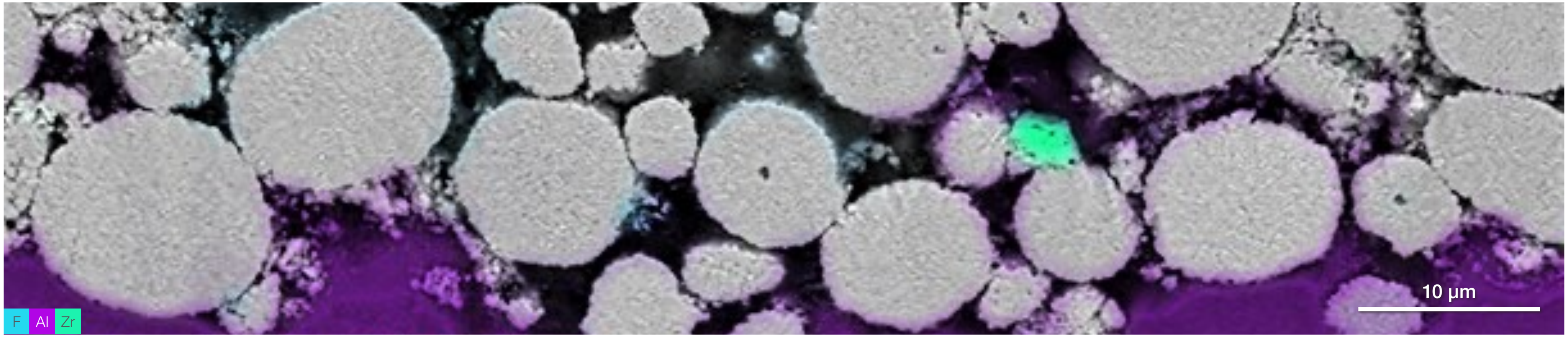

Figure 5. ChemiSEM cross-section image of an electrode active material.

A holistic characterization of batteries and energy storage materials extends beyond advanced SEM imaging. To understand and develop next-gen materials, researchers need to understand the structural and chemical composition of their sample and be able to easily visualize and analyze their data.

Our software packages, like Thermo Scientific ChemiSEM™ Technology for live, integrated EDS (energy dispersive X-ray spectroscopy) and Thermo Scientific Avizo™ Software for unmatched data processing, enable faster access to data without compromising accuracy.

Elemental analysis with traditional EDS runs on a dedicated hardware and software system. This requires constant switching between two separate operating platforms, usually navigating through complex and time-consuming workflows to achieve required results. ChemiSEM Technology overcomes these hurdles. SEM and EDS are integrated within a single interface, providing a streamlined user experience with all the tools needed to interpret the data in one place. Moreover, unique data processing algorithms provide elemental information up to 10 times faster than with conventional means. The intuitive implementation significantly reduces the need for user training.

Because ChemiSEM Technology is always on, you will discover features in your samples that might otherwise go unnoticed. In a ChemiSEM image, colors directly point to chemical elements, turning scanning electron microscopy into a live color imaging technique. An example of this in shown in Figure 5, where a ChemiSEM image of a cross section of an electrode active material shows the presence of zirconium-rich impurities and the distribution of the fluorine-rich binder.

Image analysis

Imaging analysis software like Avizo Software can turn the EM image into valuable data insights for researchers to quantitatively interpret the structure–performance correlation. It identifies and measures complex features in electrode materials and in entire cells with high accuracy.

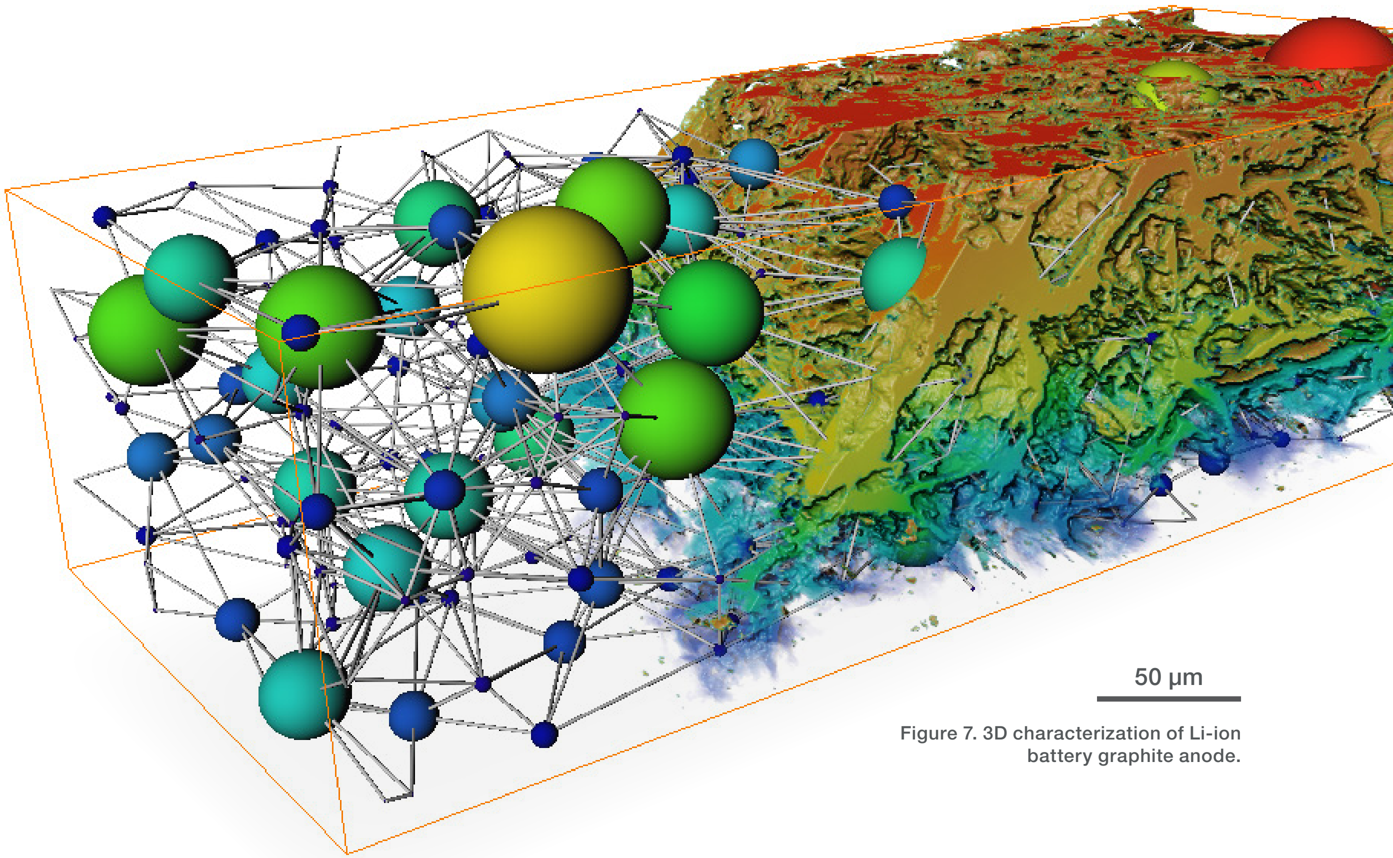

Figure 7. 3D characterization of Li-ion battery graphite anode.

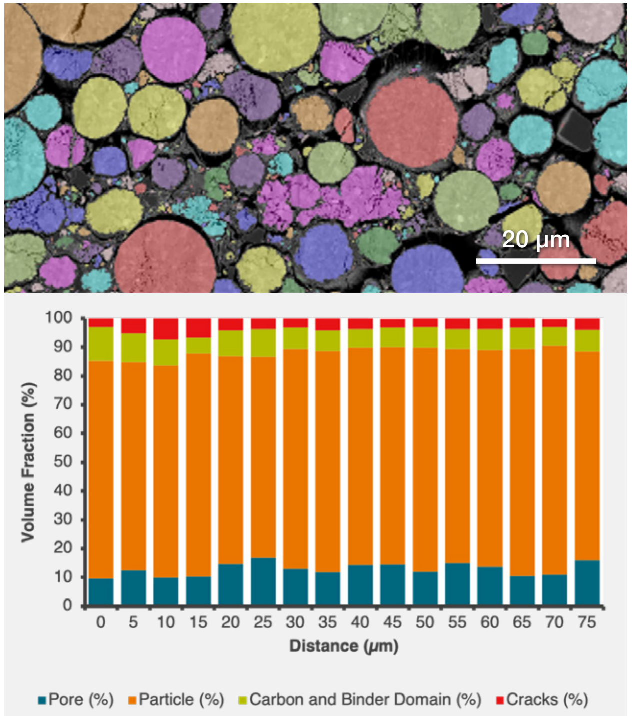

Figure 6: Phase fraction analysis on an NCM cathode along the calendaring direction via Avizo2D Software.

At the macro level, Avizo Software can be used to assess the quality of the battery manufacturing process by allowing you to investigate packaging, check solder points, and detect leakage or porosity and delamination. You can also examine the aging process and investigate foil, cathode, and anode morphological changes or core leakage.

At the microscopic level, Avizo Software allows you to estimate the tortuosity and permeability of the porosity structure of electrode and separator; you can then use effective transport parameters in the electrochemical performance simulation. You can characterize the cell's performance by quantifying the triple phase boundary (TPB), phase distribution, and connectivity.

Avizo Software is one package to visualize and analyze battery samples, from both 2D and 3D acquisition techniques. For a deeper understanding of structure–performance correlation and structural quantification, 3D imaging can provide a lot more additional information than 2D imaging. Structural characteristics such as connectivity and tortuosity that relates to the battery transport property can only be analyzed using a 3D imaging approach.

DualBeam systems

To achieve these exeellent results from 3D material characterization, it is cruicial to have enhanced imaging and detection techniques.

Our advanced Thermo Scientific Helios™ and Scios™ portfolio of DualBeam systems are able to characterize a wide range of materials for the battery research needs of today and tomorrow.

Figure 8 shows the use of different DualBeam technologies to study the battery at different length scales. By using the plasma FIB, you can collect the NMC cathode's 3D structure with the length scale of 100 µm. By using our Ga-FIB system, you can characterize a separator sample. If you are looking for large-volume reconstruction at sub-mm length scale, laser PFIB will be the choice to collect such data set. All data can then be analyzed via Avizo Software to extract all those microstructural parameters.

The distribution of lithium within the electrode is of interest to evaluate its electrode performance during cycling. However, elemental analysis techniques that are frequently used in the electron microscope, such as EDS or WDS, are not able to effectively detect lithium. Because time-of-flight secondary ion mass spectrometry (TOF-SIMS) has been incorporated into our DualBeam system, you can effectively analyze the lithium distribution within the sample. Figure 9 shows the SEM image of NMC cathode cross-section and its corresponding lithium distribution collected by TOF-SIMS. It provides insight into how the lithium is distributed within the electrode by correlating with the electrode performance.

Besides the electrode, you can also use a DualBeam to study structures at the particle level. Figure 10 shows how our DualBeam was used to study the interior structure of a single NMC cathode particle. Different grain structures at different depths of the particle can be explored. You can also use EBSD to study the grain orientation of each primary particle.

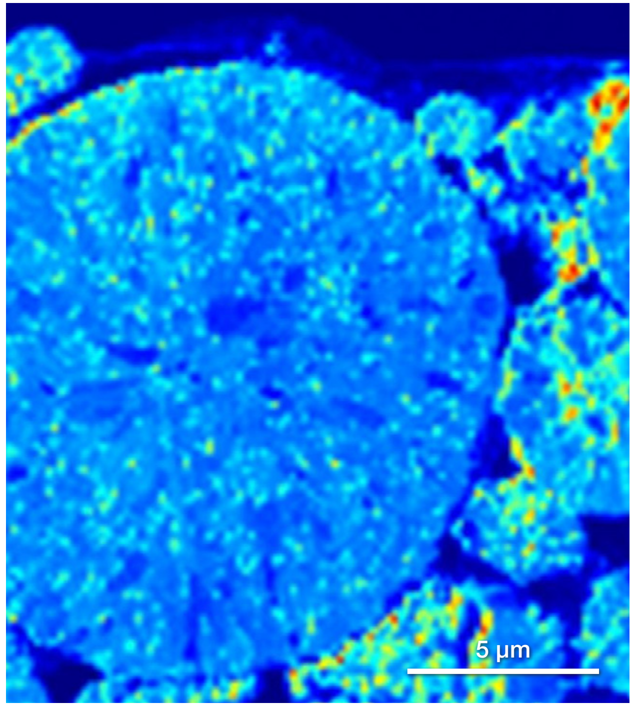

Figure 9. TOF-SIMS map of NMC cathode cross-section showing lithium distribution.

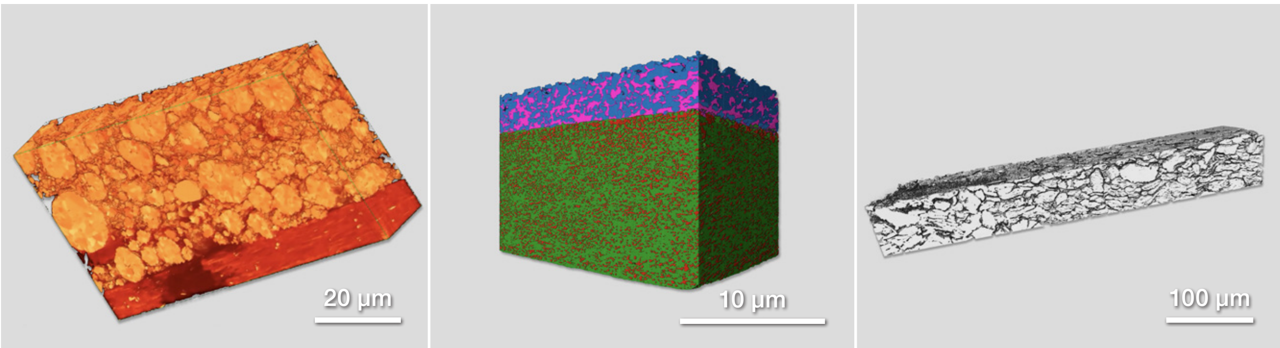

Figure 8. Different DualBeam technologies used to characterize battery elements at various length scales. PFIB: NMC cathode (left), Ga-FIB: separator (middle), and laser PFIB: graphite anode (right).

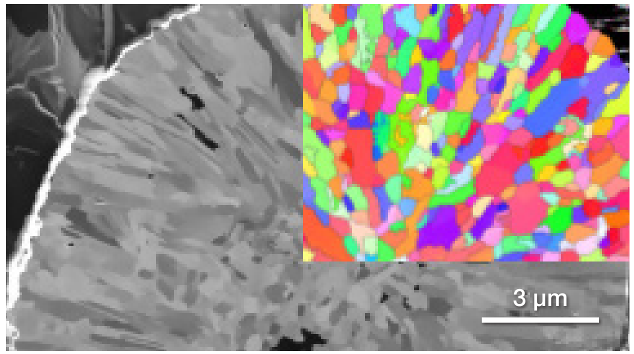

Figure 10. DualBeam used to study the interior structure of a single NMC cathode particle.

TEM sample preparation

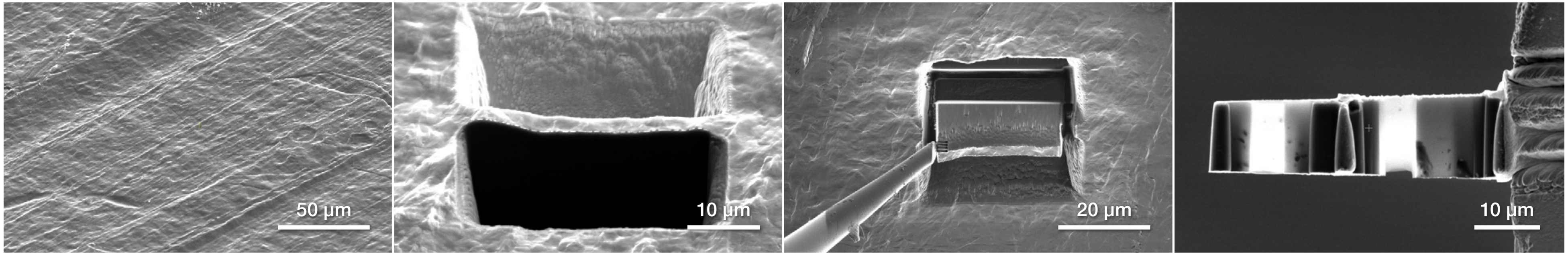

Figure 11. TEM sample preparation of lithium using a DualBeam system.

Our DualBeam instruments with AutoTEM Software automates TEM sample preparation, making formerly manual, error-prone procedures much faster and reproducible, resulting in more accurate results. Figure 11 shows the TEM sample preparation of lithium using a Thermo Scientific DualBeam instrument.

Strong understanding of fundamental science—how materials perform at the atomic and molecular levels—translates into superior system performance. Atomic and molecular insights are important in every step of the technology development process, not just the early stages.

Higher resolution imaging and sample prep with our DualBeam systems is followed by atomic-scale analysis in a transmission electron microscope (TEM). When combined, these technologies provide a complete understanding of sample structure and composition in its native state, accelerating development of novel materials.

Figure 12 shows high-resolution TEM images of lithium, acquired in Thermo Scientific Velox™ Software, that prove the sample is well-protected during the entire workflow.

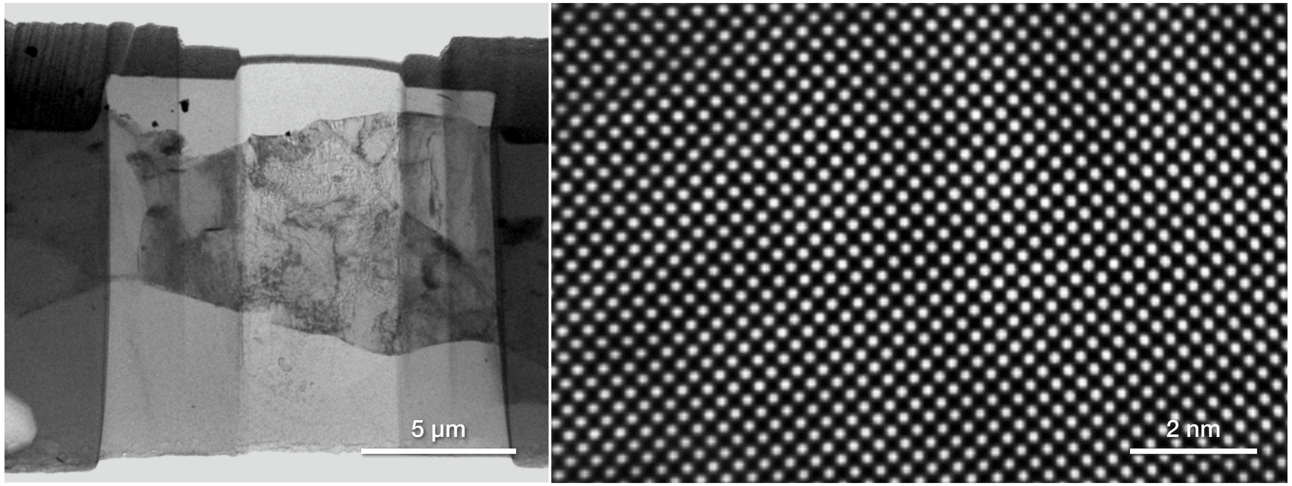

Figure 12. TEM lamella image of lithium (left), high-resolution TEM image of lithium (right).

Future challenges

Although the market for lithium-ion batteries continues to grow, the challenge is developing batteries that are safer and longer-lasting with higher energy density.

To help with this research, many scientists are continuing the quest to explore new materials to achieve battery breakthroughs. Our analytical techniques and battery workflows further support these researchers. An example is the advanced material characterization of solid-state batteries: from visualization, image analysis, and in situ studies to addressing challenges like lateral growth of dendrites in SEI (solid electrolyte interphase) layers and instability of the SEI caused by the volume changes of the Si material or just to observe interactions between materials at the nanometer level. These are just a few ways in which electron microscopy is helping to advance battery research.

Whether you are exploring alternative energy sources or developing stronger, lighter materials and sophisticated nanodevices, the right instruments and optimum workflows can help take your materials to the next level so you can discover the solutions of tomorrow.

Our IGST Workflow allows you to develop materials that meet the demands of today's and tomorrow's social and economic challenges. This unique workflow offers vast exploration and experimentation capabilities without compromising material integrity.

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.

Step ahead. Step beyond. Duration 1.33

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.

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Explore equipment leasing and financing options

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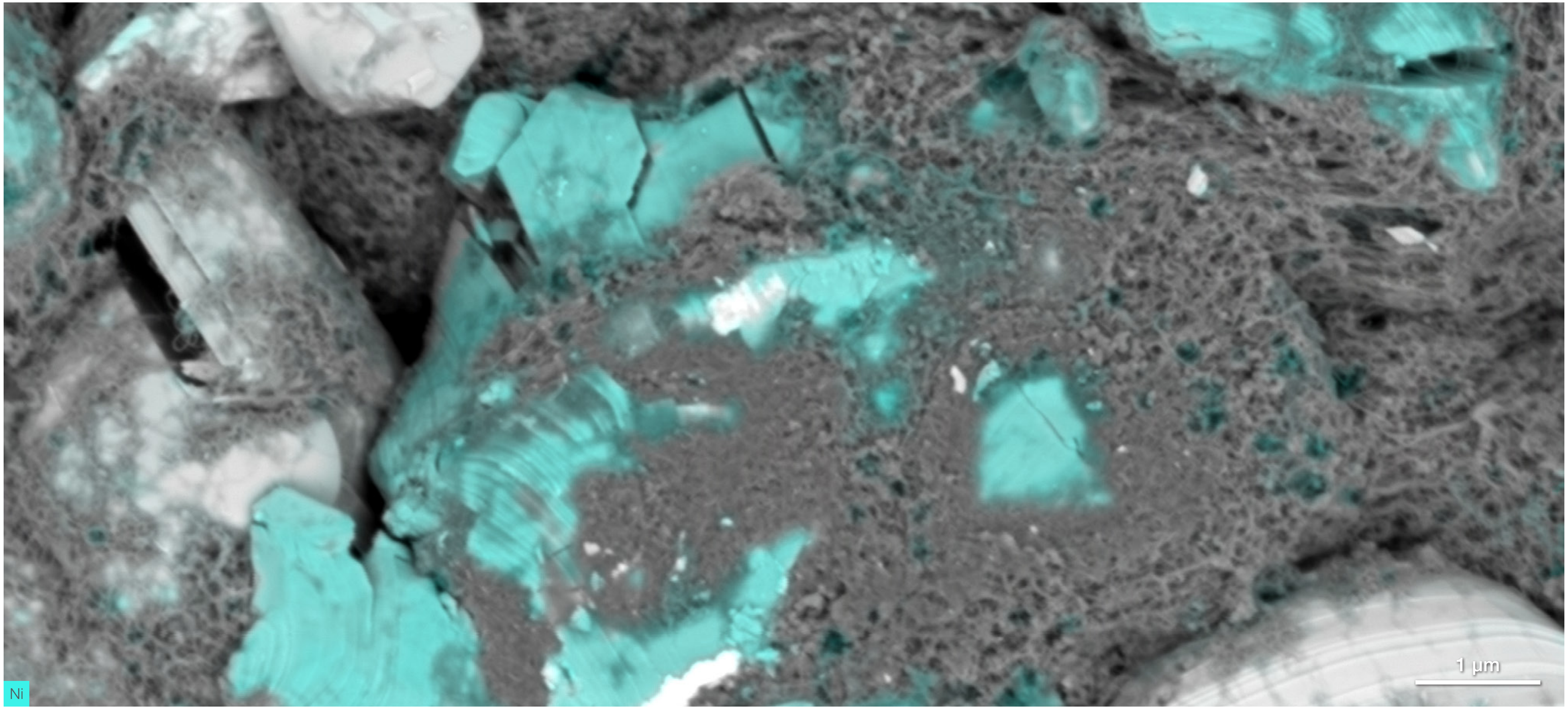

Figure 13. Study of the materials' distribution of a Li-ion battery cathode via ChemiSEM Technology at 2 keV.

Learn more at **thermofisher.com/battery-research**