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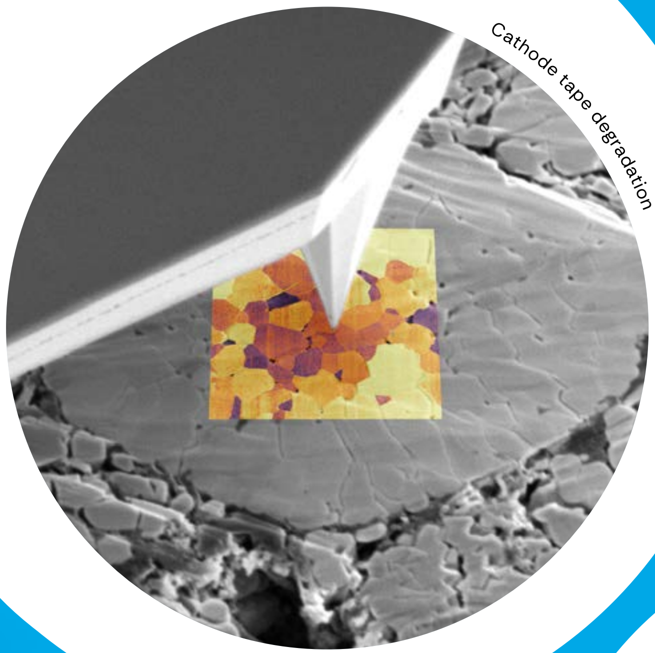

Battery component materials analyses using AFM-in-SEM LiteScope™

application note

Key added values

- Site-specific local resistivity of particles
- In-situ performance and degradation of battery components
- No air exposure of sensitive surfaces
- Complementary to SEM analytical capabilities (FIB, EDX, etc.)

Application areas

- Cell and battery components
- Raw active materials
- Cathodes
- Anodes
- Solid electrolytes
- Liquid electrolytes

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Battery components analyses using AFM-in-SEM LiteScope™

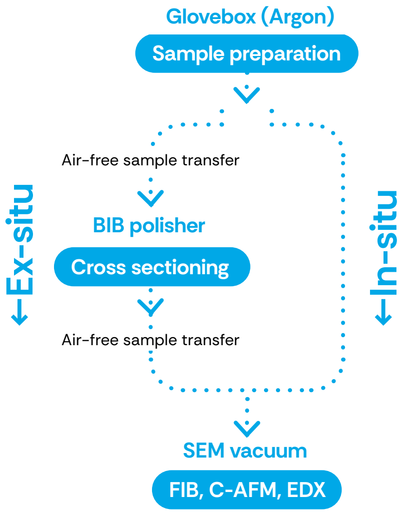
Energy storage devices are crucial to today's technology. Understanding their features, performance, aging, and capacity is vital. Most high-end batteries are **Li-ion**, with **active materials extremely susceptible to atmospheric contamination and degradation**.

Our battery materials solution with **LiteScope** provides a **complete hardware and measurement workflow approach**. It allows for an **air-free sample transfer system** that prevents contamination by oxygen and humidity, while ensuring uncompromised sample surface preparation. The imaging power of **AFM-in-SEM provides** a **complex** and **site-specific** understanding of the **electrical and chemical properties** of battery components.

Measurement workflow

1. Ex-situ sample preparation using Sample Transfer System

This option allows **larger cross-section preparation** in specialized instruments while maintaining the **sample surface in the necessary environment**, even during transfer and AFM-in-SEM measurement.



2. In-situ sample preparation using Focused Ion Beam

LiteScope enables safe **AFM probe positioning** for large stage tilts, **preserving the tip from redeposition** and allowing the **use of FIB to cut a trench anywhere on the sample**. This allows measurement of the uncovered surface using **AFM-in-SEM technology**.

Cathode Tape Inspection

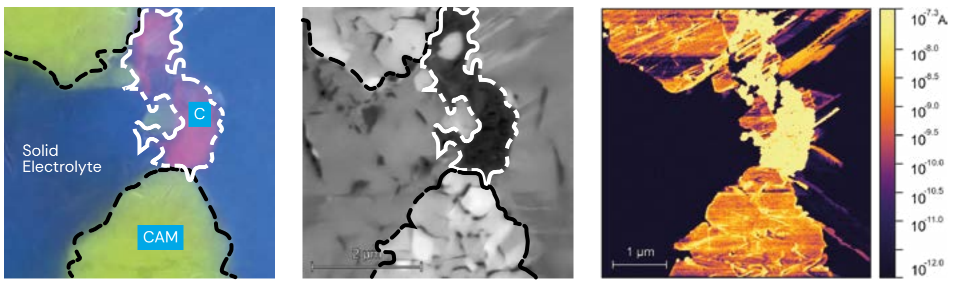
Sample prepared by Thermo Fisher Scientific.

Solid State Batteries (SSBs) show promise over Li-ion batteries with **higher energy density, longer lifespan, and improved safety**. A **cathode tape** composed of Lithium Nickel Manganese Cobalt Oxide (NMC) particles was opened after 200 cycles in a glovebox, cross-sectioned ex-situ, and measured using **AFM-in-SEM** (Measurement Workflow 1).

SEM imaging identified the polished area of interest. The **EDS map** showed the **cathode tape's composition**, including the solid electrolyte, NMC particles, and carbon additive. **C-AFM confirmed the carbon additive's conductivity** and its role in connectivity. This **AFM-in-SEM** correlation aids in identifying material functions or failures **without altering the sample surface**.

Correlated EDX, SEM, C-AFM of interface NMC particles-solid electrolyte

/ EDX composition map / Secondary electron image / C-AFM current map



Key benefits and features

+ Fast and easy sample transfer: Small evacuation time is needed to exchange the samples.

+ No air or humidity exposure: Air-sensitive samples are protected from surface contamination or oxidation in a vacuum or inert gas atmosphere at all times.

+ Complete measurement workflow: A comprehensive procedure for the cross-sectional analysis of complex battery components and materials testing.

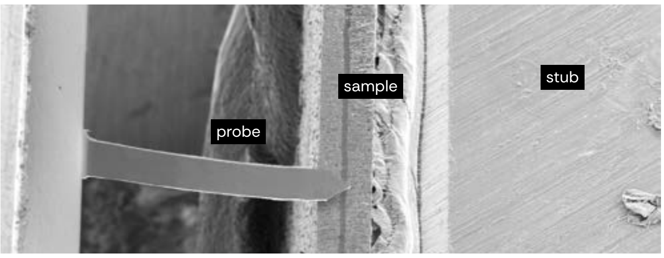
+ Upgrade to SEM solution: Extended possibilities are enabled using FIB/GIS prep, AFM, C-AFM, KPFM, phase imaging, SE, BSE, EDX, Fz spectroscopy, ...

IntraParticle conductivity

Sample prepared by Thermo Fisher Scientific.

The **cycling of batteries** involves the transport of lithium ions from the cathode to the anode and vice versa, which causes the **electrode particles to expand and contract**. Over many cycles, the battery performance decreases. **Understanding these degradation mechanisms** is crucial for **longer battery lifetime** and **better performance**.

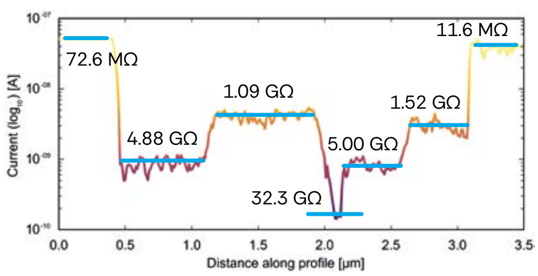
SEM overview: probe navigation to cross-sections



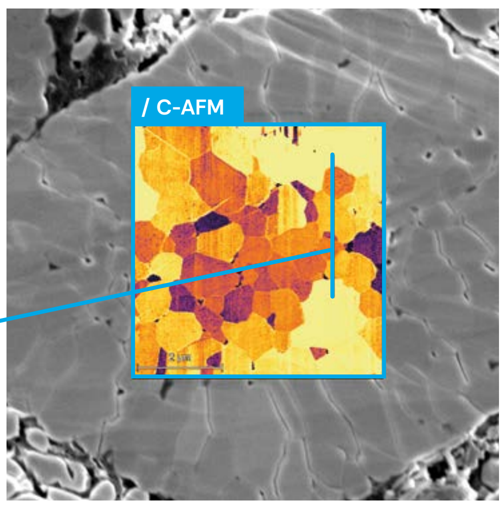
We employed the **C-AFM** to image a secondary particle from an **NMC cathode of a liquid electrolyte (LE) battery** after 200 charge-discharge cycles. It revealed an interesting **conductivity structure** of primary particles and the **cracks formed** between them during cycling. While there is an overarching tendency for the primary particles to be less conductive closer to the particle center, another effect causes seemingly random conductivity variations. Local C-AFM proves, that **some** of the **primary particles** lost electrical contact with their neighbors, probably due to cracks, and **became disconnected**. The effect is not negligible, as the current profile taken across a few grains shows differences in orders of magnitude, which then **influences (decreases) overall battery performance**.

Complex conductivity distribution inside the secondary NMC particle is revealed by C-AFM

/ Current profile with calculated resistivity



/ SEM



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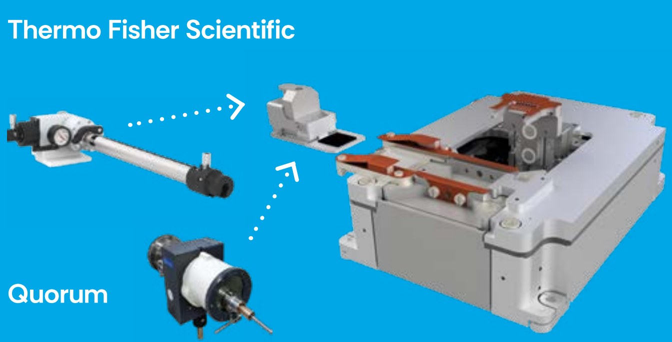
LiteScope™ solution for battery components analyses

Complete workflow

We deliver a complete workflow for sample surface preparation, air-free sample transfer, site-specific electrical and chemical properties measurements, and data post-processing.

Battery components and materials testing

This setup enables the characterization of performance and failures, understanding of cycling effects, active material degradation, inter/intraparticle connectivity, and delamination.



Sample Transfer Module

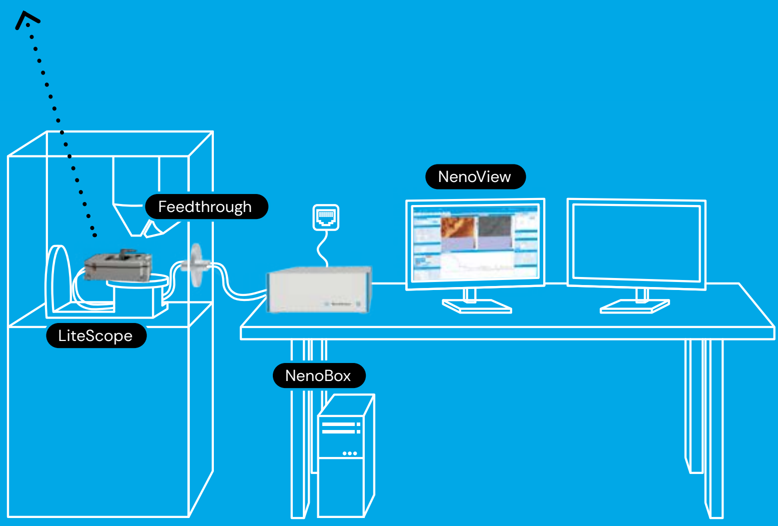
Dedicated Module for AFM-in-SEM LiteScope provides an effective solution for safe sample transfer under a controlled environment.

Our module of compact design is compatible with most already existing sample transfer systems and their accessories.

LiteScope Setup →

NenoVision combines tradition and expertise with unique solutions in nanoscale AFM-in-SEM microscopy.

Thanks to its optimized design, the AFM LiteScope seamlessly integrates into most SEM systems produced by Thermo Fisher Scientific, TESCAN, ZEISS, Hitachi, Jeol, and their accessories.



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