The Battery Scientist's Characterization Toolkit

Analytical Workflows for Battery Development, Manufacturing, and Failure Analysis

COVALENT
Material Insights

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Why Battery Characterization Matters

The Race to Electrify Everything

Few industries are evolving as rapidly as energy storage, and batteries sit at the center of that growth. Electric vehicles, consumer electronics, grid-scale storage systems, aerospace platforms, and defense technologies all depend on batteries that are safer, lighter, cheaper, faster-charging, and longer-lasting than the generation before them.

Battery developers are tasked with balancing a complex set of competing requirements:

- Energy density
- Power delivery
- Charge rate
- Cycle life
- Calendar life
- Safety
- Manufacturing yield
- Cost
- Sustainability and recyclability

The challenge is that improving one battery attribute often comes at the expense of another. Increasing energy density may reduce cycle life. Faster charging can accelerate degradation. New materials may improve performance but introduce manufacturing challenges or safety concerns.

Success depends on understanding how materials behave at every stage of development—from raw material synthesis through manufacturing, testing, and eventual failure. This is where battery characterization becomes essential.

Batteries Are More Complex Than They Appear

At first glance, a lithium-ion battery seems simple. A typical cell consists of four primary components:

- Cathode
- Anode
- Separator
- Electrolyte

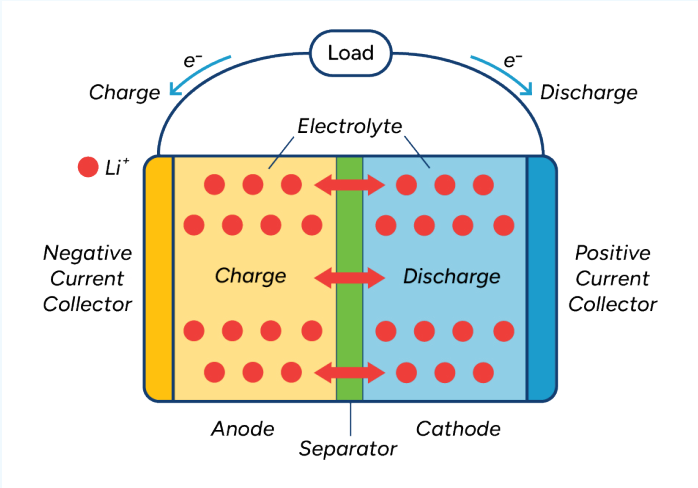


Figure 1: Basic operating principle of a lithium-ion battery. During charging, lithium ions (Li^+) move through the electrolyte from the cathode to the anode while electrons travel through the external circuit. The separator is an electronically insulating porous membrane that allows lithium-ion transport but does not allow electrical contact between the anode and cathode materials. During discharge, the process reverses: lithium ions migrate through the separator and back to the cathode while electrons flow through the external circuit to power the connected device.

In reality, each of these components is a sophisticated engineered system composed of multiple materials working together. Each component requires a unique set of characterization methods to answer very different questions.

A modern battery cell is a complex assembly of interacting materials and components. The cathode, anode, separator, and electrolyte are each engineered systems in their own right, combining active materials, additives, coatings, and binders that are tuned to work together. Small changes in chemistry, particle size, crystal structure, porosity, or contamination can meaningfully affect how a cell performs and how long it lasts, and those effects don't stop at the component level. They propagate into how the assembled device behaves under real operating conditions. Understanding a battery, then, requires characterization at every scale, from individual materials to the finished cell.

The challenge facing battery scientists is determining which variables matter, how they interact, and why performance changes over time.

Characterization Connects Materials to Performance

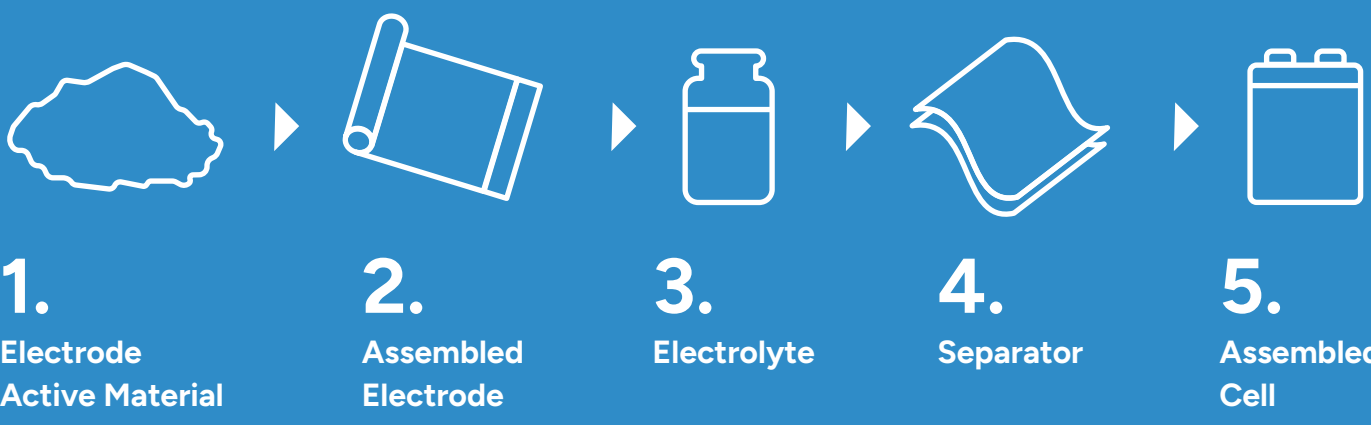
Component-level materials characterization is the process of measuring the chemistry, structure, morphology, and behavior of the materials inside a battery. It's a foundational subset of the broader practice of battery characterization. Modeling and data science can predict a lot of cell behavior efficiently, but predictions are only as good as the assumptions and data behind them. When a cell doesn't behave the way a model says it should, someone still has to find out why at the physical level. That's the role component-level characterization plays: validating model assumptions against real materials, uncovering degradation mechanisms the models didn't anticipate, and explaining the outliers modeling alone can't resolve.

For example:

- Why is capacity fading faster than the model predicted?
- Why did internal resistance increase in a way the data doesn't explain?
- Why did a cell swell during cycling despite passing simulation?
- Why does one supplier's material underperform another's, even when their specs look identical on paper?
- Why did a battery fail in the field when nothing in the model flagged it?

Answering these questions requires much more than measuring voltage and current. Engineers often need to examine particle morphology, crystal structure, elemental composition, porosity, interface chemistry, mechanical integrity, and degradation products simultaneously. No single analytical technique can provide all these answers. Effective battery characterization therefore relies on combining multiple techniques into integrated workflows.

As the battery industry grows and evolves, materials characterization becomes increasingly important. The companies that succeed are not necessarily those with the largest number of analytical instruments. They are the organizations that know how to ask the right questions, select the right characterization tools, and connect materials behavior to real-world performance. In the chapters that follow, we will explore how characterization supports battery innovation at every stage of development—from raw materials and active materials to cell assembly, performance testing and failure analysis.



Characterization Throughout the Battery Manufacturing Process

Before a battery is ever assembled, manufacturers must verify the quality of incoming materials. The problem is compounded by the fact that there are often multiple suppliers for the same product which need to be verified to be “the same” despite appearing so on paper.

Questions commonly include:

- How pure is the material?
- Is the particle size distribution correct?
- Does the crystal structure match specifications?
- Is the surface area within acceptable limits?
- Are there trace contaminants present?

Materials characterization helps researchers get to the bottom of these questions.

Electrode Active Material: The Engine of the Battery

The active material is where energy is stored and released. Just as an engine converts chemical energy into kinetic energy, the active material converts chemical energy into electrical energy by reversibly storing and releasing lithium ions. Every improvement in battery performance—higher energy density, faster charging, or longer cycle life—ultimately depends on chemical processes occurring in microscopic particles that constitute the active material. When we characterize electrode active materials, we allow for the optimization of the engine before assembling the battery.

The active materials in both the cathode and anode start as fine powders, and even in the finished battery they remain powders that have been mixed into a coating and attached to a metal foil. Active materials are produced as powders in part because particle size and pore structure together determine how much surface area is available for the electrochemical reactions that drive charge transfer. That surface area is a real lever on performance, but it isn't simply "more is better": the same surface that enables faster reaction kinetics also drives side reactions with the electrolyte, so particle size and porosity are tuned to balance reactivity against degradation rather than to maximize surface area outright.



Figure 2: Raw Lithium Iron Phosphate powder cathode material

While powder particle morphology influences how much surface area is available for electrochemical reactions, crystal structure determines how lithium ions move and are stored within each particle. The crystal structure of the active material acts as a network of atomic pathways that lithium ions use during charging and discharging. Together, these properties strongly influence battery capacity, charging rate, cycle life, and overall performance.

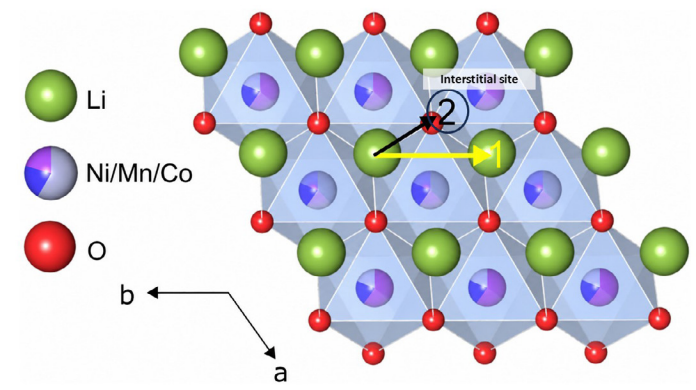


Figure 3: Schematic of two possible local Li^+ jump pathways within the Li layer of layered NMC: (1) between neighboring Li sites and (2) from a Li site to an interstitial site.

Cathode active material powders such as Nickel Manganese Cobalt (NMC), Nickel Cobalt Aluminum (NCA), Lithium Iron Phosphate (LFP), and Lithium Manganese Iron Phosphate (LMFP) continue to evolve as researchers seek the ideal balance of energy density, safety, cycle life, and cost. These active materials form the heart of the cathode and strongly influence battery performance throughout its lifetime. On the anode side, efforts are being made to develop silicon-rich and silicon-carbon active material powders to replace conventional graphite and increase energy density.

Characterization plays a critical role in the development of these next-generation materials. Battery engineers rely on a suite of analytical tools to understand crystal structure, particle morphology, elemental composition, purity, surface area, and electrochemical behavior.

For example, X-ray Diffraction (XRD) is commonly used to characterize the crystal phases present in battery powder materials. When X-rays hit a crystalline material, they scatter from the regularly spaced planes of atoms inside the crystal. In an XRD pattern, the x-axis (2θ, in degrees) is the angle at which the X-ray detector measures diffracted X-rays, which directly relates to the spacing between atomic planes in the crystal. XRD is frequently used to verify phase purity and confirm that a material has crystallized into the structure required for its intended function. In other words, XRD can be used to verify that the crystalline pathways used by lithium are structured correctly.

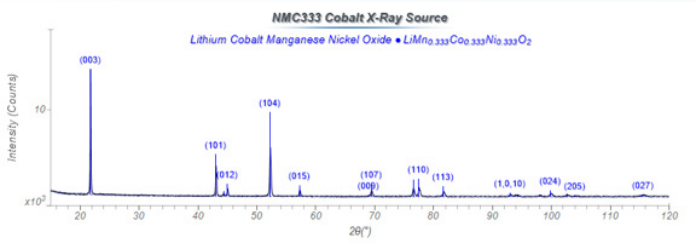


Figure 4: XRD pattern of NMC333 cathode powder. In the example shown, Rietveld refinement of a commercial NMC333 powder confirmed a single, phase-pure layered oxide (R-3m, PDF# 04-014-7636) with no detectable impurity phases. The refined lattice parameters (a = 2.858 Å, c = 14.222 Å) and clearly resolved (006)/(102) and (108)/(110) peak doublets indicate a well-ordered layered structure, and a refined crystallite size of 179 nm reflects a well-crystallized powder, properties that directly influence the lithium-ion transport and cycling performance of the finished cathode.



Figure 5: At Covalent, we use the Rigaku SmartLab for XRD work. The instrument's 9kW rotating anode allows for higher intensity X-rays than most other systems.

Laser diffraction particle size analysis (LD-PSA) and specific surface area measurements such as BET analysis of gas adsorption data help quantify particle size distributions and available surface area, both of which influence lithium transport and reaction kinetics. LD-PSA measures particle size by passing a laser beam through a dispersed sample and analyzing the angle-dependent pattern of scattered light. Larger particles scatter light at narrow angles relative to the beam, while smaller particles scatter at wider angles. Applying Mie scattering theory to this diffraction pattern, the volume-weighted particle size distribution is calculated.

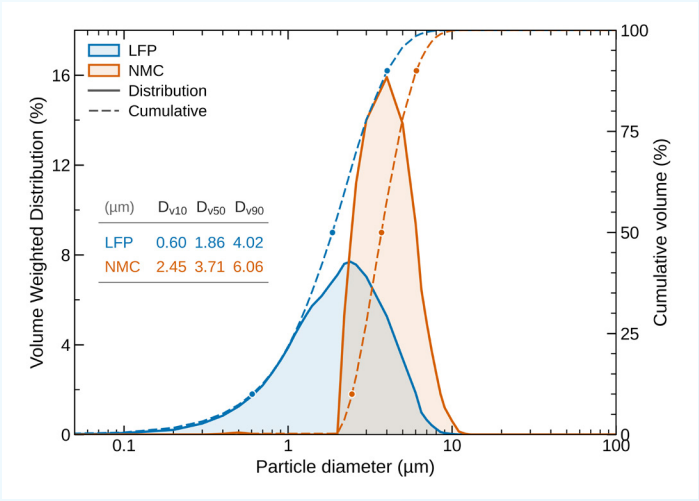


Figure 6: Example output of laser diffraction particle size analysis comparing the particle size distribution of two commercially sourced common battery cathode materials: Lithium Iron Phosphate (LFP), and Lithium Nickel Manganese Cobalt Oxide (NMC). In this example, NMC shows a narrower size distribution and slightly larger average particle sizes, while LFP shows a broader distribution with a tail of very fine particles down to sub-micron sizes.



Figure 7: At Covalent, we use an Anton Paar PSA-1190 for LD-PSA work. This instrument allows for measurements of particles in a range of 0.04 µm to 2500 µm and incorporates dry jet dispersion to prevent agglomerate formation and improve accuracy for dry samples.

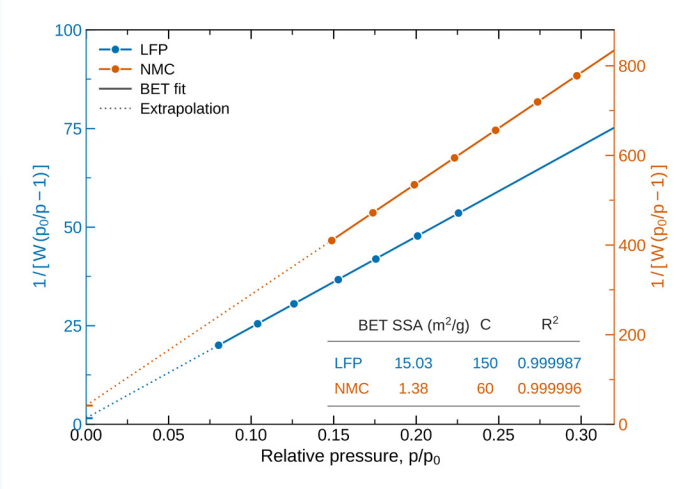


Figure 8: BET surface area analysis performed on nitrogen gas adsorption data at 77.3 K from commercial LFP and NMC powders. The linearity of the plot and positive C constants demonstrate good agreement with the BET model over the selected relative pressure range for both materials and are requirements for physically meaningful and reliable specific surface area measurements. BET specific surface areas are calculated from the slope and intercept of the linear function fitted to the presented transformed data — an ~11x difference measured between LFP and NMC reflects the differences in particle morphology related to both particle size and pore structure.

The BET method measures specific surface area by cooling the sample to liquid nitrogen temperature and then exposing it to controlled doses of high purity inert gas, typically nitrogen. As nitrogen forms a monolayer on the surface of each particle, changes in pressure are used to determine how much gas adsorbs onto the surface. The BET model allows us to predict the surface area because every nitrogen molecule occupies a known area (0.162 nm^2). Surface area is an important property for battery materials because it influences electrolyte contact, lithium-ion transport, reaction kinetics, and the formation of interfacial layers such as the solid electrolyte interphase (SEI).



Figure 9: At Covalent, we use Anton Paar's Nova 800 for BET analysis. This tool has integrated degassing stations and multi-sample parallel analysis capability with a reproducibility specification of less than 2% and can measure surface areas down to 0.01m2/g with no known upper limit.

Once the active material has been coated onto the electrode foil, mercury intrusion porosimetry (MIP) can be used to characterize the finished electrode itself rather than the loose powder. While particle size analysis and BET surface area describe the raw material before coating, MIP measures the total pore volume, pore size distribution, and porosity of the assembled electrode. In addition to intraparticle porosity, it can be used to characterize interparticle porosity (the void space between particles). This structure determines the medium through which lithium ions move once the cell is built. MIP is prized for its resolution and range, capable of resolving pore diameters from roughly a millimeter down to a few nanometers, but its reliance on liquid mercury has made it difficult to run routinely: mercury is toxic, and handling it requires dedicated safety infrastructure, waste protocols, and specialized training that many labs choose to avoid.

To address this, Anton Paar has introduced eGaIn (eutectic gallium-indium alloy) intrusion porosimetry. eGaIn is a non-toxic liquid metal that behaves similarly to mercury under pressure, and when measured on Anton Paar's PoreMaster with a dedicated eGaIn filling station, which loads the metal into the sample cell without exposure to oxygen, it reproduces mercury's pore size range and resolution without the associated hazards. Side-by-side testing on certified reference materials and on battery anode graphite sheets shows that eGaIn and mercury produce comparable pore size distributions and total intruded pore volumes once eGaIn's higher surface tension and contact angle are accounted for in the Washburn equation. This allows for a safer, more sustainable path to the same porosity data MIP has long provided, and though we don't currently offer this novel instrument in house, it's one that Covalent feels is worthy of highlighting.



Figure 10: eGaIn is a non-toxic, environmentally benign liquid metal that poses no risk to lab personnel or the environment. It requires no protective gear, special ventilation, or hazardous material handling procedures. This makes high-performance porsimetry possible in any standard laboratory environment



Figure 11: Image of Anton Paar's PoreMaster eGaIn Filling Station.

While XRD can give us information about crystal structure in active powders and PSA and BET can provide information about particle size and surface area, we also need information about the stoichiometry of the powders. Battery materials rely on precise elemental ratios. Even tiny changes in elemental composition can dramatically affect battery performance, lifetime, and safety. ICP-OES and ICP-MS provide sensitive elemental analysis for impurity detection and stoichiometry verification.

Element	Measured Concentration (wt%)
Li	7.15
Ni	48.82
Mn	5.90
Co	5.99
Al	<0.01
Fe	0.004
Cu	0.002

Table 1: Representative ICP-OES data on raw NMC powder. ICP-OES confirmed that the cathode matched the target NMC811 stoichiometry while showing only trace levels of metallic contaminants (Al, Fe, Cu), indicating successful synthesis and high material purity. The results yield stoichiometry of $Li_{0.99}$ $Ni_{0.80}$ $Mn_{0.10}$ $Co_{0.10}$.



Figure 12: At Covalent, we use a Thermo Fisher Scientific iCAP-7400 Duo for ICP-OES. This instrument measures wavelengths from 166 to 847 nm for flexible multi-element analysis.

Scanning Electron Microscopy (SEM) can be utilized to gather images revealing particle morphology, size, and microstructural features.

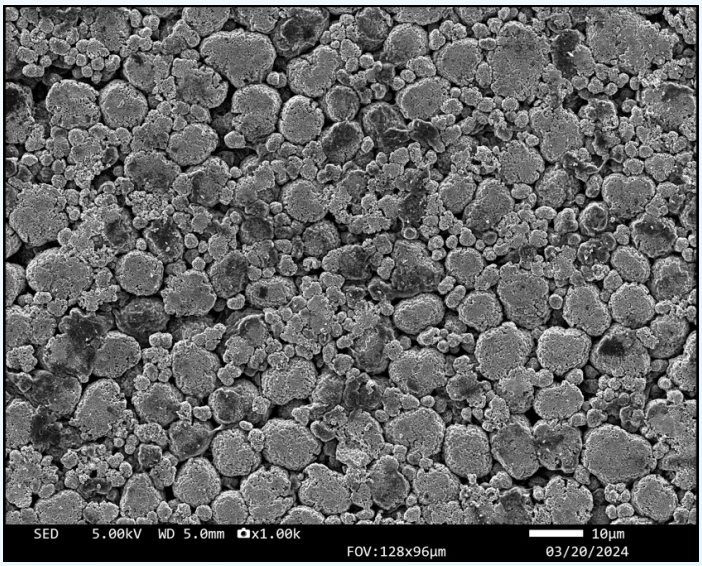


Figure 13: SEM image of NMC-811 powder. We have a large fleet of SEM instrumentation at Covalent from several different brands.

Together, these techniques allow researchers to correlate material composition and structure with battery performance, enabling faster optimization of new active material formulations.

Assembled Electrodes:
The Battery's Road Network.

The active material may be the engine, but the electrode is the transportation network that delivers traffic to every destination.

Once active materials are produced, they are combined with conductive additives, polymer binders, and solvents to form a slurry. This slurry is coated onto a metallic current collector and dried to create an electrode.

Electrode manufacturing is one of the most important steps in battery production. The electrode microstructure determines how efficiently lithium ions and electrons can move through the system. Even when using identical active materials, differences in electrode manufacturing can lead to dramatically different battery performance. Characterization at this stage often focuses on understanding electrode architecture and ensuring manufacturing consistency.

Once active materials are incorporated into electrodes, engineers need to understand:

- Coating uniformity
- Porosity
- Particle distribution
- Binder distribution
- Cracking
- Delamination

These factors strongly influence ion transport, mechanical stability, and ultimately battery performance. At this stage, characterization often includes electron microscopy and requires sample preparation with broad ion beam (BIB) polishing. The BIB is a gentle argon beam that smooths surfaces after they have been cut with a blade in preparation for electron microscopy. This ensures that the analyst is looking at features in the battery itself rather than artifacts induced by mechanical cutting. At Covalent, we have an air-free transfer unit allowing analysts to cut samples in a glove box, BIB-polish, and SEM-image electrodes without exposing the sample to atmosphere and thus contaminating air-sensitive materials commonly found in batteries, such as lithium.

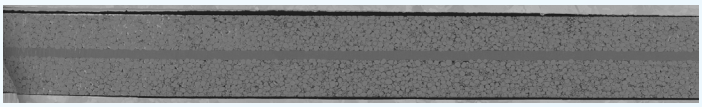


Figure 14: SEM image of a BIB-polished cathode cross section.



Figure 15: At Covalent, we use a JEOL IB-19520CCP for most of our BIB needs. This instrument has cryo cooling capabilities allowing us to mill through temperature sensitive materials such as polymers and adhesives without causing thermal damage to the sample.

For more high-resolution analysis, a focused ion beam (FIB) can be used to cross-section individual particles within the SEM and investigate internal structure.

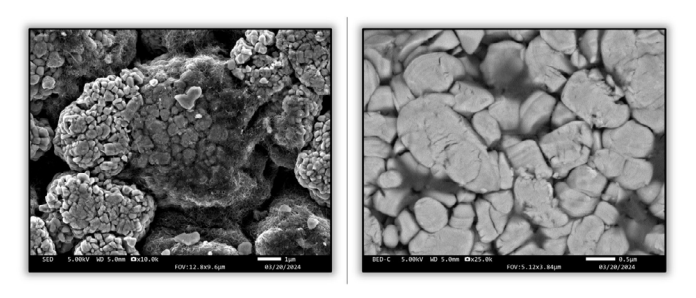


Figure 16: SEM images showing FIB cross section of a single NMC-811 particle agglomerate. In the cross sectioned face (right) we observe moderate cracking. Cracking between primary particles indicates mechanical fatigue caused by repeated lithiation and delithiation, allowing electrolyte to penetrate deeper into the particle and accelerating parasitic reactions, impedance growth, and capacity fade. These observations provide valuable insight into the structural integrity of the cathode and its expected electrochemical performance.

SEM imaging can also be used to visualize delamination between the current collector and the electrode. Delamination is generally a serious degradation mechanism because it reduces the electrical contact between the active material and the current collector. Even if the electrode is still capable of storing lithium, it becomes much harder (or impossible!) to access electrically.

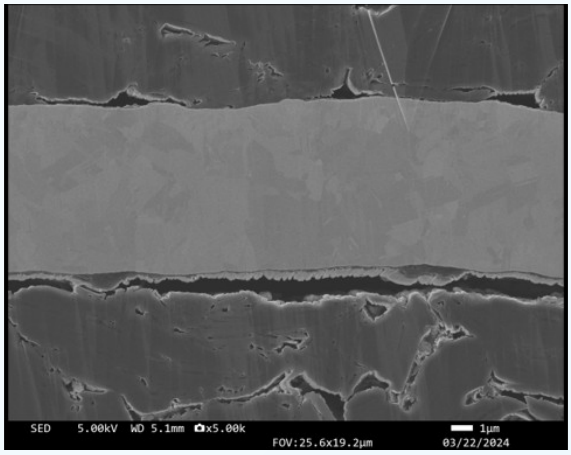


Figure 17: SEM image of a BIB polished graphite anode revealing delamination between the anode and the current collector.

Electron dispersive spectroscopy (EDS) can be used within the SEM to complement high-resolution imaging by revealing the elemental distribution throughout an anode cross section. Rather than simply identifying which elements are present, elemental maps show where they are located, allowing engineers to evaluate coating uniformity, detect contaminants, examine SEI formation, and assess the interface between the active material and current collector. These insights help identify manufacturing defects and degradation mechanisms that can lead to increased resistance, capacity loss, and reduced battery lifetime.

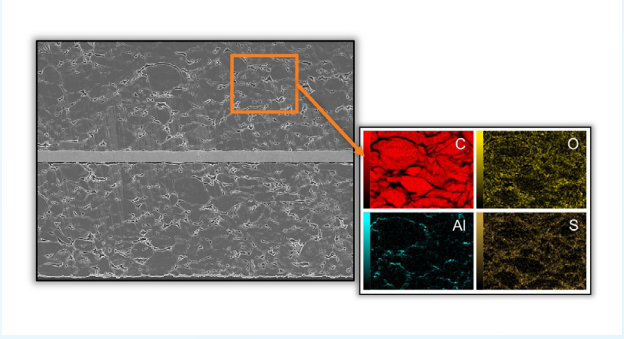


Figure 18: SEM image and EDS maps of a BIB polished anode cross section in this image, we can see that there is trace aluminum localized to particle surfaces.



Figure 19: At Covalent, we run a fleet of EDS detectors, including the Oxford X-Max 150 and Ultim Max 100. These detectors are capable of resolving carbon signal from graphite anodes and conductive carbon additives at speeds up to 200,000 counts/second, without losing spectral resolution.

Raman spectroscopy is widely used to evaluate the degree of graphitization in battery anodes by distinguishing highly ordered graphitic carbon from disordered or amorphous carbon. It does this by measuring how carbon atoms vibrate within the material, since those vibrations depend on how ordered the atomic structure is. Highly ordered graphite produces a different Raman spectrum than defective or amorphous carbon, allowing researchers to assess the quality and structural integrity of the anode. Because the degree of graphitization directly influences electrical conductivity, lithium-ion transport, and cycle life, Raman spectroscopy is an important tool for both quality control and failure analysis in batteries.

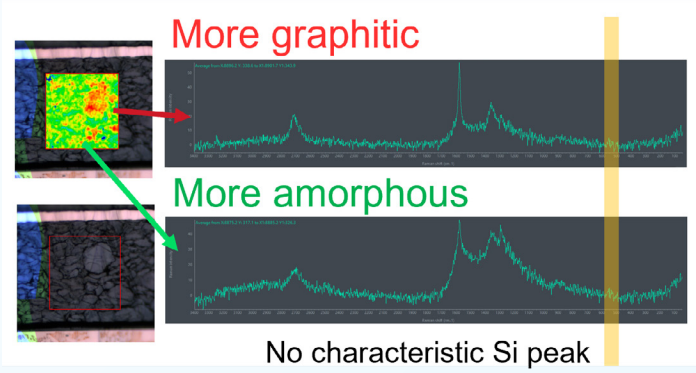


Figure 20: Raman spectra collected from two regions of a graphite anode reveal differences in carbon ordering. The graphitic peak (G peak), found near 1580 cm^{-1}, is produced by carbon atoms vibrating within an ordered graphitic lattice. The upper spectrum exhibits a stronger, sharper G peak relative to the defect peak (D peak), indicating a higher degree of graphitization, while the lower spectrum shows a larger D peak and broader features consistent with increased structural disorder. The absence of a characteristic silicon peak indicates that the analyzed regions are primarily carbon-based.



Figure 21: One of the Raman systems we use at Covalent is the Thermo Scientific DXR3xi Raman Imaging Microscope, equipped with a 532 nm laser, for carbon characterization in battery electrodes. Unlike a single-point Raman measurement, the DXR3xi's imaging camera enables spatial resolution better than 0.5 µm and hyperspectral Raman mapping across areas up to 101.6 mm × 76.2 mm, letting us visualize how graphitization and structural disorder vary spatially across an electrode rather than relying on one spectrum from one spot.

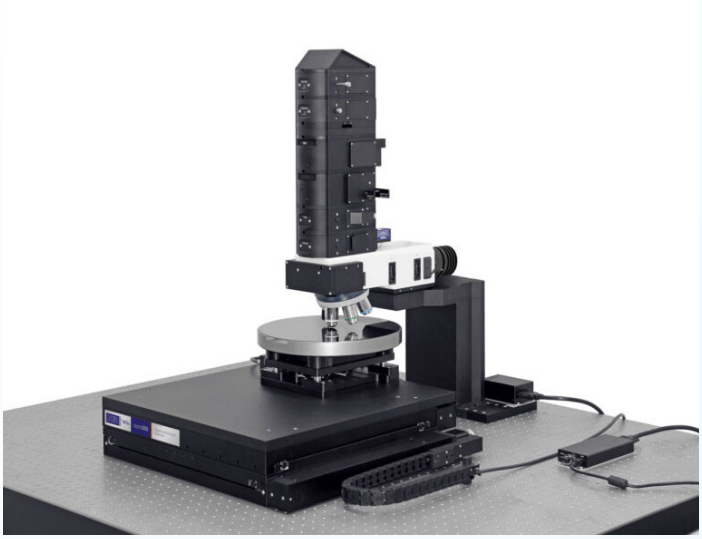


Figure 22: Covalent also uses the Oxford Instruments WITec360. This instrument can handle larger sample formats and has a UV laser for photoluminescence.

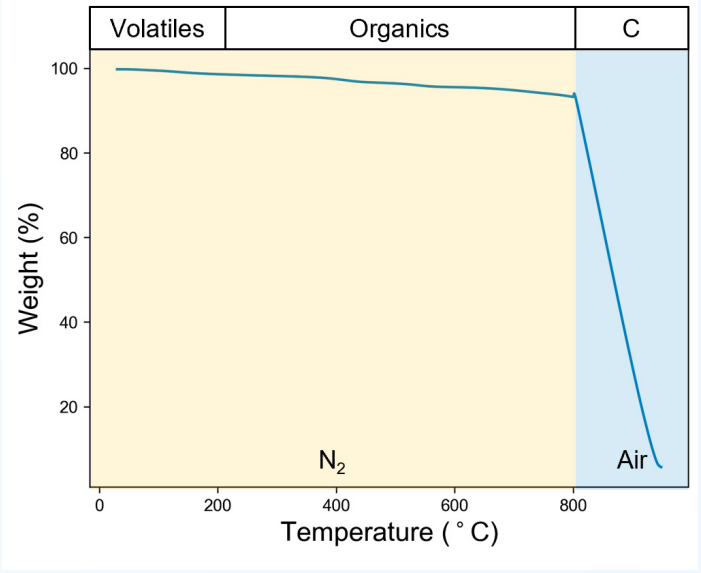


Figure 23: TGA results on anode material. In this example, a small weight loss at lower temperatures corresponds to volatile compounds and organic binder materials, while switching from nitrogen to air causes the remaining carbon to oxidize rapidly, producing a sharp decrease in mass.

Thermogravimetric analysis (TGA) measures how the weight of a sample changes as it is heated. Because different battery materials decompose or oxidize at different temperatures, the resulting weight-loss curve can be used to determine the composition of an electrode. By analyzing these weight-loss events, researchers can quantify the relative amounts of moisture, binder, conductive carbon, and active material, making TGA a valuable tool for quality control, reverse engineering, and failure analysis.



Figure 24: At Covalent, we use TA Instruments TGA 5500, the most advanced TGA, offering 0.1 µg balance resolution.

The Electrolyte:
The Chemical Highway of a Battery

The electrolyte provides the medium through which lithium ions travel between the electrodes. It doesn't store energy itself, but it determines how quickly, safely, and efficiently ions can move throughout the battery. Like a highway system, it must remain open, clean, and stable throughout the battery's lifetime.

Modern lithium-ion electrolytes are typically composed of a lithium salt dissolved in a mixture of organic carbonate solvents, along with carefully selected additives that improve safety, extend cycle life, and promote the formation of stable interphase layers on the electrodes. The most common electrolyte salt is lithium hexafluorophosphate ($LiPF_6$), dissolved in mixtures of solvents such as ethylene carbonate (EC), dimethyl carbonate (DMC), diethyl carbonate (DEC), or ethyl methyl carbonate (EMC). Small changes in composition can significantly influence ionic conductivity, charging rate, low-temperature performance, gas generation, and long-term battery stability.

The electrolyte continually evolves throughout the battery's lifetime. Electrolyte molecules participate in the formation of the solid electrolyte interphase (SEI) on the anode and the cathode electrolyte interphase (CEI), while also slowly decomposing during cycling, high-temperature storage, overcharge, and abuse conditions. These reactions consume electrolyte, generate gases, alter salt concentration, and produce degradation products that increase internal resistance and reduce battery life.

For battery developers, electrolyte characterization can answer several fundamental questions:

- Is the electrolyte formulation correct?
- Are the intended solvents, salts, and additives present at the proper concentrations?
- Has the electrolyte degraded during manufacturing or cycling?
- Are contaminants present that could reduce performance or accelerate failure?
- How does the electrolyte chemistry evolve throughout the battery's lifetime?

At Covalent, we offer an electrolyte characterization package which combines Gas Chromatography–Mass Spectrometry (GC-MS), Nuclear Magnetic Resonance (NMR) spectroscopy, and ICP-OES/MS to help our customers get to the bottom of these questions. Each technique provides a different piece of the puzzle, and together they provide a comprehensive understanding of electrolyte composition, purity, degradation, and performance.

For fresh electrolytes, GC-MS confirms the identity of the intended solvent blend. During battery aging studies, it becomes even more valuable by revealing decomposition products generated during cycling or thermal abuse. The appearance of new chromatographic peaks or changes in peak intensity often indicate solvent breakdown, additive consumption, or unwanted side reactions occurring inside the cell. GC-MS is therefore commonly used to verify electrolyte formulations, compare competing electrolyte chemistries, investigate battery failures, and monitor electrolyte degradation throughout cycling.

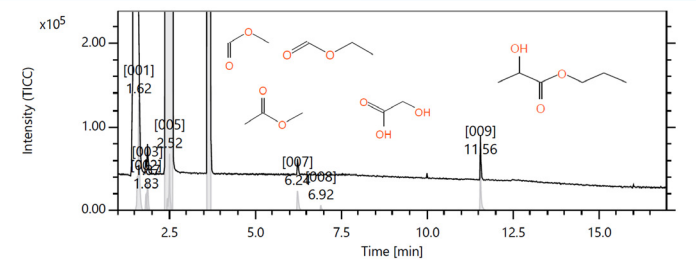


Figure 25: GC-MS separates volatile compounds within the electrolyte before identifying each by its unique mass spectrum, giving an effective fingerprint of electrolyte chemistry. Beyond the primary DMC and EC solvent peaks, library matching identifies several trace-level peaks as electrolyte decomposition byproducts — methyl formate, ethyl formate, methyl acetate, glycolic acid, and propyl lactate — each present at less than 0.6% area, consistent with early-stage solvent oxidation.



Figure 26: At Covalent, we use the JEOL JMS-T2000 AccuTOF GC-Alpha GC-MS. This instrument has very high sensitivity, with detection limits down to 18.7 femtograms.

While GC-MS excels at identifying volatile organic compounds, NMR spectroscopy provides highly quantitative information about both organic solvents and dissolved lithium salts.

NMR identifies molecules by measuring the unique magnetic environments of their atomic nuclei, producing characteristic peaks for each chemical species. ^{1}H NMR is commonly used to determine the relative concentrations of electrolyte solvents, allowing researchers to verify that the electrolyte formulation matches its intended design. Because NMR signal intensity is directly proportional to the number of nuclei present, solvent ratios can be measured with high accuracy.

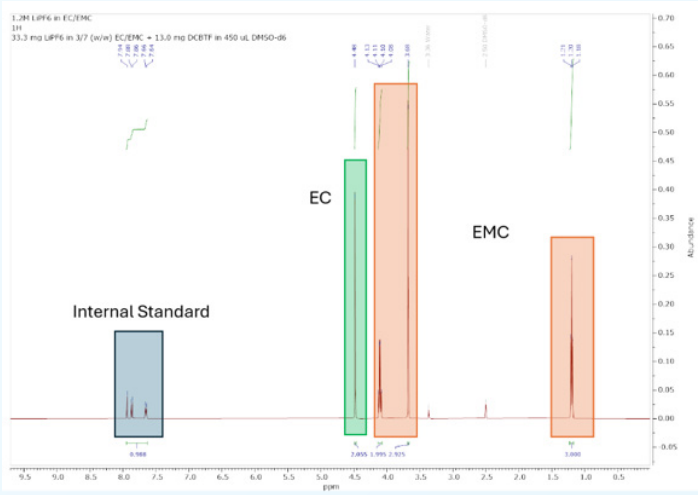


Figure 27: Example ^{1}H NMR spectrum of a lithium-ion battery electrolyte. In this example, the spectrum confirms the presence of ethylene carbonate (EC) and ethyl methyl carbonate (EMC), while the peak integrals are used to accurately determine the relative concentration of each solvent in the electrolyte by comparing against an internal standard.

NMR can also be used to solve molecular structure. Understanding the molecular structure of electrolyte components is important because the exact molecules present determine how the electrolyte behaves inside

the battery. Unlike a metal or ceramic, where crystal structure dominates performance, the electrolyte's properties are governed by the identity and arrangement of its molecules.



Figure 28: At Covalent, we use the JEOL ECZL-G 500 MHz NMR spectrometer which can support multi-nuclear analysis including 1H, 13C, 15N, 19F, and 31P.

Ultimately, the purpose of the electrolyte is to transport lithium ions efficiently. Electrochemical Impedance Spectroscopy (EIS) provides a direct measurement of how effectively the electrolyte performs this role. By measuring the battery's response to small alternating electrical signals across a range of frequencies, EIS separates contributions from electrolyte resistance, charge-transfer resistance, and diffusion processes. As electrolytes age, decomposition products accumulate and the SEI thickens, producing measurable increases in impedance. These changes often correlate with capacity fade, power loss, and reduced charging performance.

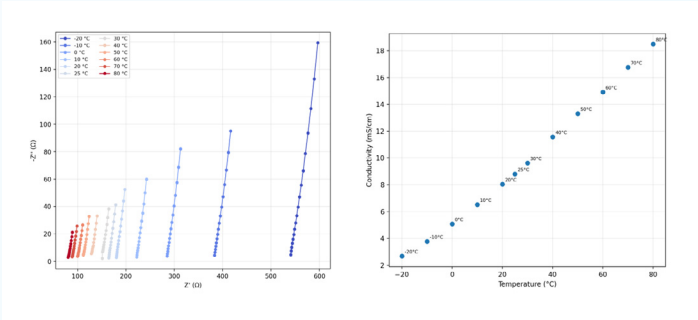


Figure 29: Example Nyquist plots obtained by electrochemical impedance spectroscopy (EIS). The x-axis (Z′) represents the battery's resistive component, while the y-axis (Z″) represents the reactive component associated with electrochemical processes such as charge transfer and lithium-ion diffusion. Each point corresponds to a different measurement frequency. As temperature increases, the curves shift to lower impedance, indicating improved ion transport and faster electrochemical reactions.



Figure 30: At Covalent, one of the tools we use for EIS is the Autolab PGStat302N. This instrument pairs a wide-bandwidth potentiostat (±10 V, 1 MHz) with an optional FRA32M module for EIS, giving the low-current resolution and high-frequency reach needed to resolve bulk ionic conductivity from interfacial effects in electrolyte characterization.



Figure 31: For temperature-controlled cell testing, Covalent uses the RHD Microcell HC — Peltier-based temperature-controlled cell fixture (–40°C to +100°C). This instrument enables automated temperature control during conductivity, cyclic voltammetry, and battery/supercapacitor cycling measurements.

The Separator: The Battery's Highway Median

A battery separator may be one of the thinnest components in a lithium-ion cell, but it plays one of the most critical roles. If the active materials are the engine that stores energy, the electrodes are the local road network, and the electrolyte is the chemical highway that carries lithium ions, then the separator is the battery's highway median. It's a physical barrier separating the positively charged cathode and negatively charged anode. The separator does not allow electrons through, thus preventing dangerous short circuits, while its network of microscopic pores allows lithium ions to move freely between the electrodes during charging and discharging. The separator must strike a delicate balance: it needs to be mechanically strong enough to withstand manufacturing and battery operation, chemically stable in a harsh electrolyte environment, and porous enough to enable rapid ion transport. Even small defects or changes in its structure can reduce battery performance, accelerate degradation, or, in the worst cases, compromise battery safety.

When characterizing battery separators, Fourier Transform Infrared Spectroscopy (FTIR) is commonly used to identify the chemical composition and any functional coatings that influence battery performance. FTIR works by shining infrared light on a material and measuring which wavelengths are absorbed. Because every type of chemical bond vibrates at characteristic frequencies, the resulting spectrum acts as a molecular fingerprint that can be used to identify polymers, solvents, coatings, and other chemical compounds. Attenuated Total Reflectance (ATR) is a surface-sensitive version of FTIR that analyzes only the outer few micrometers of a material. By collecting spectra from each side of a battery separator, ATR-FTIR can identify surface coatings, polymers, and other chemical features that may not be distinguishable using conventional transmission FTIR alone. This information is valuable for quality control, reverse engineering, and failure analysis because the separator chemistry directly affects thermal stability, mechanical strength, electrolyte wettability, and battery safety.

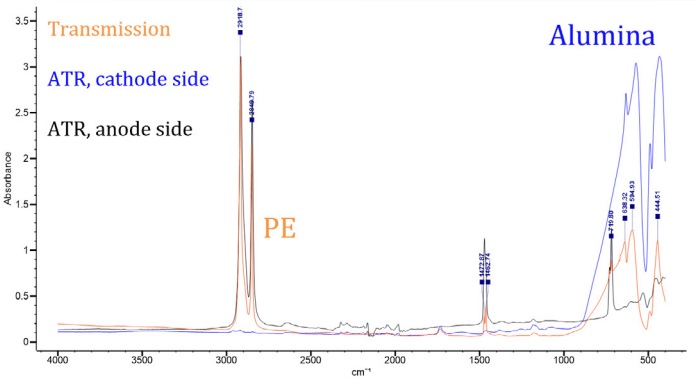


Figure 32: FTIR results on a battery separator. In this example, strong C–H stretching peaks confirm that the separator is composed primarily of polyethylene (PE), while characteristic Al–O absorption bands reveal an alumina ceramic coating on the cathode-facing surface. By comparing spectra collected from each side of the separator, ATR-FTIR confirms the layered construction of the material and verifies that the ceramic coating is present where intended.



Figure 33: At Covalent, we run a Thermo Fisher Scientific Nicolet Continuum infrared microscope for this kind of separator analysis. Its ATR objective probes only the outer few micrometers of each surface, and with a spectral range extending down to 450 cm^{-1}, it captures the low-wavenumber Al–O bands needed to confirm a ceramic coating alongside the polyethylene backbone. This lets us verify a separator's layered construction from each face independently.

Tensile testing provides battery manufacturers with quantitative data to optimize material selection, improve manufacturing yield, and ensure the separator remains mechanically robust enough to prevent internal short circuits throughout the battery's lifetime.

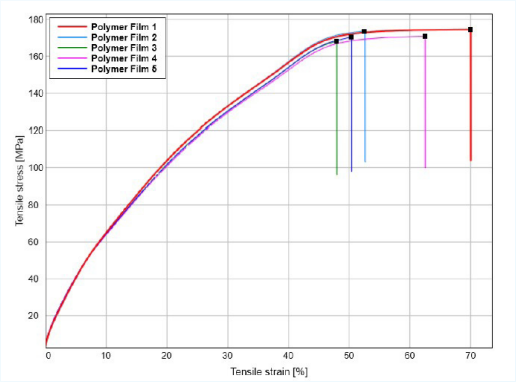


Figure 34: Tensile testing results on a polymer battery separator. The results showed that the separator could elongate by more than 50% before failure while maintaining a tensile strength of approximately 172 MPa, demonstrating the flexibility needed to withstand battery manufacturing processes such as winding, stacking and calendaring. Read the full case study on this example here.



Figure 35: At Covalent, we use an Instron 68SC-5 Universal Testing Machine for tensile testing of battery separators. This instrument supports high resolution testing with ±0.5% accuracy to 1/1000 of capacity.

Cell Performance Testing:
Putting the Infrastructure to the Test

By this point, we've examined each of the battery's critical components individually—the engine (the active material), the road network (the assembled electrode), the chemical highway (the electrolyte) and the safety barrier (the separator). But a high-performance infrastructure system isn't judged by its individual components alone. It's judged by how well every part works together. Once assembled into a complete cell, the interactions between these components play a significant role in determining the battery's capacity, power, efficiency, lifetime, and safety. Characterizing the finished cell allows engineers to evaluate the battery as an integrated system before investigating individual components to identify the root cause of any performance issues.

Within many commercial cells, the electrode stack is wound into a structure commonly referred to as a jelly roll before being packaged inside the final cell housing.

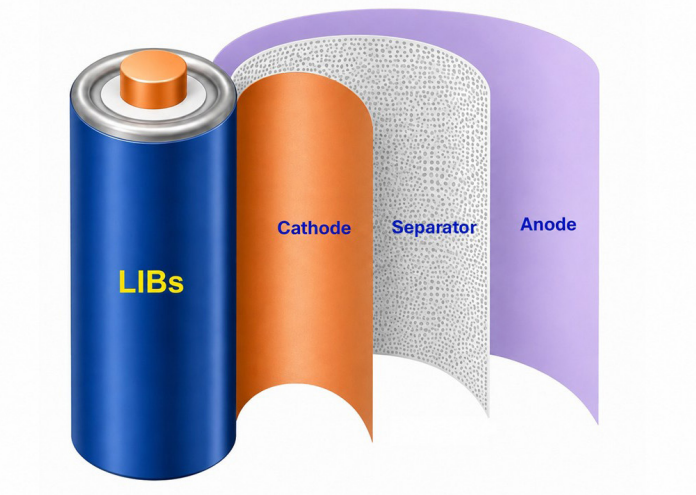


Figure 36: Illustration of a jelly-roll style battery.

Once assembled, the battery enters the testing phase. At this point, developers seek to understand how the cell behaves under realistic operating conditions.

Typical questions include:

- How much energy can the battery store?
- How quickly can it be charged?
- How much power can it deliver?
- How many cycles will it survive?
- How does temperature affect performance?
- What degradation mechanisms emerge over time?

Covalent can perform controlled abuse and aging studies on assembled cells by cycling batteries under stressful conditions, such as low temperature, elevated temperature, or accelerated charge/discharge profiles. During testing, battery performance can be monitored through capacity retention and EIS, then paired with teardown and post-mortem analysis to connect electrical degradation to physical and chemical failure mechanisms such as lithium plating, SEI growth, electrolyte decomposition, particle cracking, delamination, and separator damage using any of the techniques mentioned in this eBook.

Additionally, non-destructive techniques such as 2D X-ray analysis, micro computed tomography (μCT) and Scanning Acoustic Microscopy (SAM) are commonly used before and after cell cycling in order to baseline the state of the battery as received and compare results after abuse testing.

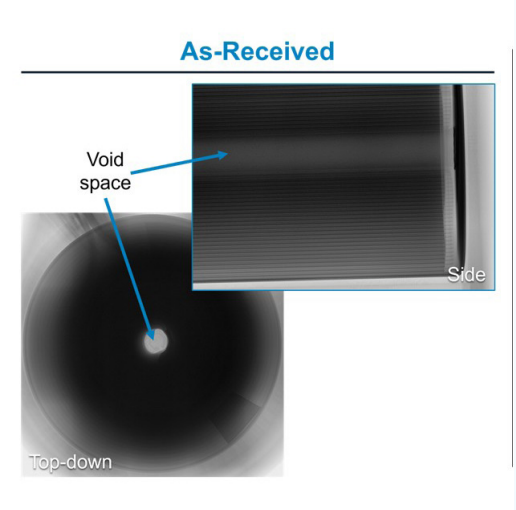


Figure 37: 2D X-ray imaging provides a rapid, non-destructive way to assess the structural integrity of an assembled battery cell before it is disassembled for further analysis. In this example, the as-received cylindrical cell exhibits a uniform jelly roll with a well-defined central void, indicating that the electrode layers are evenly wound. After cycling to end-of-life, however, the jelly roll has mechanically collapsed at its core, eliminating the central void and creating a localized deformation. This type of collapse is a well-known failure mode caused by repeated mechanical stresses during battery operation, including electrode expansion and contraction over many charge-discharge cycles. By identifying defects such as jelly roll collapse early in the failure analysis workflow, 2D X-ray helps investigators rapidly determine whether a battery has experienced internal mechanical degradation and guides subsequent characterization techniques toward the most relevant areas of the cell.



Figure 38: All of the CT data in this ebook was collected on Covalent's Nordson DAGE Quadra 7 Pro, a 3D X-ray inspection system operating from 30–160 kV with automatic dual-mode switching between high-resolution and high-flux imaging. It's the workhorse behind the electrode and separator imaging you've seen throughout this guide, giving us the flexibility to inspect both delicate and dense battery components without swapping instruments.

While CT gives us a full 3D picture of a cell's internal architecture, it isn't always the fastest or most sensitive way to catch certain defect types, particularly delamination, voids, and disbonding at material interfaces. That's where scanning acoustic microscopy (SAM) comes in: by sending sound waves through the sample and reading back the reflections, SAM detects these interfacial defects nondestructively, often faster and at lower cost than a full CT scan. This isn't limited to assembled cells. We've applied the same approach to detect voiding and delamination within battery thin-film stacks, such as between current collector foils and their active-material coatings. Much of that work has been customer-proprietary, so we can't share specific data here, but the physics is the same as the pouch-cell example below: any buried interface with an air gap or disbond shows up as a bright acoustic reflection.

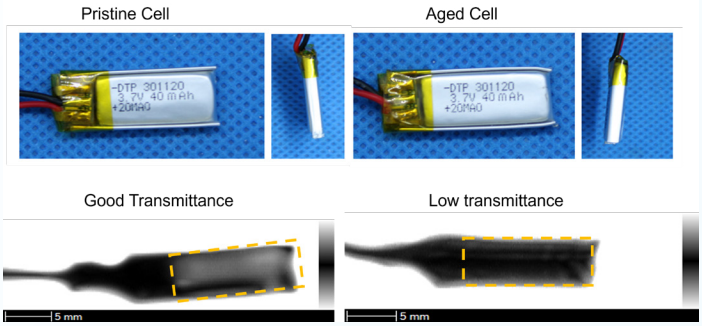


Figure 39: Scanning Acoustic Microscopy (SAM) data on a pouch cell battery that compares a pristine cell to a cell that was cycled until it no longer worked. The pristine cell's uniform, good transmittance indicates that the jellyroll/electrode stack is in tight mechanical contact throughout, fully wetted by electrolyte, with no delaminations or trapped gas anywhere along that scan line. The aged cell going to essentially zero transmittance in the same region means gas has appeared somewhere in that through-thickness path — an interlayer void, a blister, or delamination between electrode/separator layers (or between the stack and the pouch) and it's thick enough to fully block the beam.

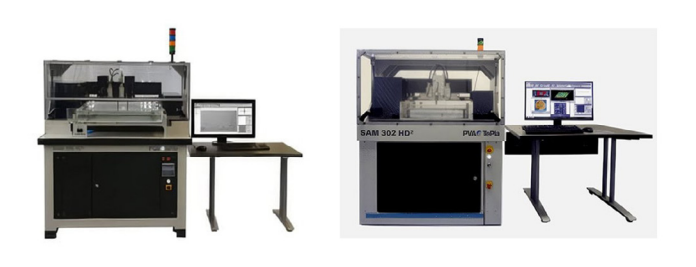


Figure 40: Covalent's SAM lab pairs two PVA TePla scanning acoustic microscopes, the SAM 302 HD2 and the SAM 501 HD, sized for different scales of work: the compact 302 HD2 handles smaller components and cells, while the larger-format 501 HD accommodates bigger samples like full battery packs and modules. Both work the same way, pulsing sound waves through the sample and reading back the echoes at material interfaces, so we can nondestructively map voids, delamination, and cracks anywhere from a single pouch cell to a full assembled pack.



Figure 41: In addition to the 302 HD2 and 501 HD, Covalent's SAM lab includes a PVA TePla Macrovue Nexgen 1000-P, which handles larger-format samples and adds rotational scanning capability. The rotational stage is particularly useful for cylindrical cells, since it can image the full curved surface in one continuous scan rather than stitching together flat sections. Cylindrical samples do need to be confirmed compatible with water immersion first, as SAM relies on a liquid couplant to transmit the acoustic signal.

Failure Analysis

No battery lasts forever.

Even successful products eventually degrade. In some cases, batteries fail unexpectedly during development, manufacturing, qualification testing, or field use. When this happens, the goal shifts from measuring performance to understanding root cause. Failure analysis often begins with the non-destructive inspection techniques mentioned in the last section of the eBook. This allows investigators to look inside a battery before opening it.

Using SAM and µCT, engineers may search for internal shorts, metal inclusions, swelling or voids. If additional investigation is required, the battery can be carefully disassembled and each component analyzed individually. The cathode, anode, separator, electrolyte, and evolved gases may all be characterized to determine what changed and why. The objective is not simply identifying what failed. The objective is understanding the mechanism responsible for failure so future designs can be improved.

A combination of non-destructive and destructive techniques is often required to reveal the true failure mechanism.

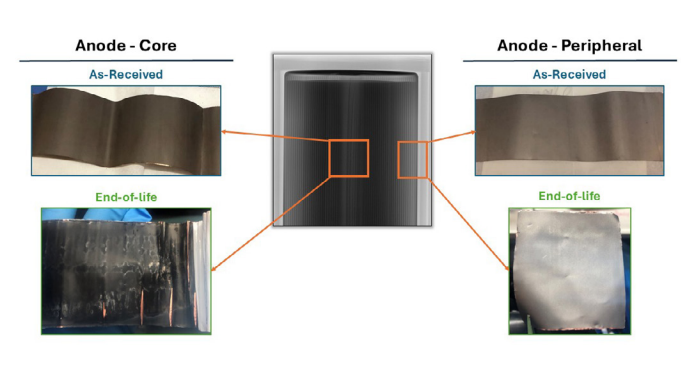


Figure 42: Following non-destructive X-ray inspection, the jelly roll was disassembled to compare the anode before and after battery aging. The as-received electrode exhibits a smooth, uniform surface at both the core and the outer regions of the cell. After cycling to end-of-life, the anode shows pronounced mechanical and chemical degradation, including wrinkling, discoloration, and localized surface damage that are most severe near the core where the jelly roll had collapsed. Comparing different regions of the electrode helps identify how aging and mechanical stresses vary throughout the cell and provides insight into the failure mechanisms responsible for performance degradation.

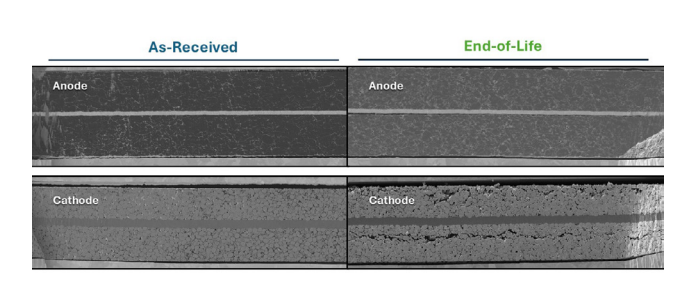


Figure 43: SEM images of a BIB-polished cross section on both the anode and the cathode of the battery as received and at end-of-life. In this example, the end-of-life images show significant cracking on the cathode, and the anode is delaminated from the current collector.

Different Companies, Different Questions

One of the most interesting aspects of the battery industry is that companies operate at different points along the value chain.

For example:

- A materials company may only produce cathode powder.
- An anode developer may focus exclusively on silicon materials.
- A separator manufacturer may specialize in polymer films.
- A cell manufacturer may purchase components from multiple suppliers and assemble complete batteries.
- An automotive company may only be interested in the performance of finished cells and packs.

Each organization asks different questions. A cathode developer may care about crystal structure and elemental composition while an electrode manufacturer may focus on porosity and coating quality. A battery manufacturer may care about cycle life and degradation mechanisms. A failure analysis engineer may need to determine why a cell failed after 1,000 cycles. The characterization strategy must be tailored to the specific question being asked.

Characterization as a Continuous Feedback Loop

The battery development lifecycle is often depicted as a linear process. In reality, it is a continuous feedback loop. Characterization results obtained during cell testing frequently drive changes in electrode design. Electrode characterization may reveal issues that require modifications to active material synthesis. Failure analysis findings often influence future manufacturing processes. At every stage, analytical data informs the next development decision. The organizations that move fastest are typically those that can generate high-quality characterization data, interpret it correctly, and rapidly translate insights into action.

At Covalent, we are proud to offer both the expertise and the instrumentation to help companies at every point along the battery value chain accelerate their research and design better batteries.



Instruments and techniques used at every stage of battery development

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Do you have a battery characterization problem we can help with?

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