Battery Testing and Materials Characterization

A Selection Guide for Analytical Solutions

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Unlocking the Full Potential of Batteries

Cutting-Edge Analytical Solutions for the Next Generation of Battery Development

The race for the next generation of battery technology is well underway. In just a few decades, rechargeable batteries have grown well beyond their roots in consumer electronics and now support sustainable transportation and renewable energy storage. Tremendous advancements in battery efficiency, power, output, and safety have primed batteries, especially lithiumion batteries, for these demanding applications.

Now, battery developers are tasked with optimizing their designs for both current and new applications. In addition to building safer, low-cost electric vehicles, batteries must reach even higher energy density for use in freight trucks and buses. Similarly, batteries with higher capacity and lower costs will be the formulations of choice for grid energy storage. And above all else, battery developers must meet demand for sustainable, affordable material sourcing in the face of lithium and cobalt scarcity.

The frontier for battery innovation is vast but the challenges are steep. Success will be determined by small margins in energy, power output, cycle life, safety, and cost – all of which require superior optimization of battery materials and electrochemical reactions. Leading labs rely on TA Instruments for analytical characterization and testing of their batteries.

Using the right techniques and instruments gives you the upper hand in efficiently advancing battery technology with proven quality and performance. From R&D and material selection to manufacturing and quality control, TA Instruments' solutions support the full breadth of battery innovation. This guide explains our comprehensive battery offerings by battery component, so you can find the right technique to enhance your battery development.

Full Battery Cell

Testing battery cells is an important step in optimizing battery chemistries and evaluating changes before scaling designs to larger formats. Battery scientists need to determine cells' efficiency as well as degradation during cycling. When [isothermal microcalorimetry \(IMC\)](https://www.tainstruments.com/products/microcalorimetry/isothermal-calorimetry/) is paired with a cycler or potentiostat, critical insights on lifetime predictions, cell performance rankings, and heat management evaluation can be developed.

Numerous notable battery labs, including the Jeff Dahn group and 3M, have [published research](https://www.tainstruments.com/lithium-ion-battery-quality-and-performance-testing-with-isothermal-microcalorimetry/) using TA Instruments isothermal microcalorimeters paired with battery cyclers for full cell testing. The [new Battery Cycler](https://www.tainstruments.com/battery-cycler-microcalorimeter/) [Microcalorimeter Solution](https://www.tainstruments.com/battery-cycler-microcalorimeter/) is built with this application in mind. The solution pairs TA Instruments' state-of-the-art TAM IV Microcalorimeter to measure the thermal behavior of samples with the BioLogic VSP-300 Potentiostat, a research-grade electrochemical analysis tool to probe the electrical properties of materials. The result is an end-to-end, in-operando measurement tool for elucidating the thermodynamic and electrochemical details of battery cells in real time, in a flexible and intuitive system with pre-programmed methods.

Battery Cycling Resources

Application Note: [Determination of Parasitic Power in Lithium-ion Batteries using the Battery Cycler Microcalorimeter Solution](https://www.tainstruments.com/applications-notes/determination-of-parasitic-power-in-lithium-ion-batteries-using-the-battery-cycler-microcalorimeter-solution/) Application Note: [An Overview of Isothermal Microcalorimetry in Battery R&D and QA - TA Instruments](https://www.tainstruments.com/applications-notes/an-overview-of-isothermal-microcalorimetry-in-battery-rd-and-qa/) [Learn more about the Battery Cycler Microcalorimeter Solution.](https://www.tainstruments.com/battery-cycler-microcalorimeter/?utm_source=TAWebsite&utm_medium=Homepage&utm_campaign=BCMS-Tile)

Battery Materials **Electrodes**

Electrodes connect battery terminals to the electrolyte and house the chemical reactions for charging and discharging. Electrodes therefore must demonstrate good electrochemical stability with the electrolyte as well as hold large amounts of ions without changing their structure. Cathode materials have been at the forefront of battery innovation in recent years, with anode materials now gaining attention as the next area of development.

Electrode Manufacturing

All batteries require cathode and anode coatings on a current collector. Modern industrial battery manufacturing uses slotdie coating, in which the electrode slurry is deposited onto the current collector to form a smooth, uniform coating. In order to speed up battery manufacturing, researchers are optimizing slurry formulations and testing different drying temperatures.

Rheology enables engineers to produce consistent slurry viscosities that result in uniform coatings for high performing and safer batteries. A rheological profile measurement can help ensure a uniform, defect-free coating that leads to production of consistent, high-quality electrodes with high batch-to-batch repeatability and low scrap rates. Engineering new slurry materials, formulations, or processing methods all require thorough rheological characterization to verify the slurry's quality.

Using accurate, reliable rheometers gives labs the upper hand in predicting slurry behavior before application, saving valuable resources and time. Screening incoming raw materials or new formulations helps users detect unexpected behavior and avoid large-scale production issues. While viscometers measure basic viscosity and flow, rheometers add shear or stress measurements that provide crucial information on slurry structure and behavior.

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The ideal slurry has a low viscosity for optimum mixing and coating (high shear rates), but a high enough viscosity for good levelling during drying and for minimizing particle settling and agglomeration during storage (low shear rates).

Since slurries are made up of solid particles of active materials, conductive additive, polymeric binder, and solvent, battery labs benefit from rheometers capable of testing all materials separately as well as combined in a slurry. The [Discovery Hybrid Rheometer](https://www.tainstruments.com/products/rheology/discovery-hybrid-rheometers/) is uniquely capable of switching between solid, liquid, and powder samples in less than 10 seconds with industryleading results and intuitive operation.

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Application Note: [Rheological Evaluation of Battery Slurries with Different Graphite Particle](https://www.tainstruments.com/applications-notes/rheological-evaluation-of-battery-slurries-with-different-graphite-particle-size-and-shape/) [Size and Shape](https://www.tainstruments.com/applications-notes/rheological-evaluation-of-battery-slurries-with-different-graphite-particle-size-and-shape/) [Shear and Flow Properties of an NMC Based Dry Cathode Powder](https://www.tainstruments.com/applications-notes/shear-and-flow-properties-of-an-nmc-based-dry-cathode-powder/) [Characterization of LIB Cathode Slurries Using Simultaneous](https://www.tainstruments.com/applications-notes/characterization-of-lib-cathode-slurries-using-simultaneous-measurements-of-rheology-and-impedance-spectroscopy-rh137/) [Measurements of Rheology and Impedance Spectroscopy](https://www.tainstruments.com/applications-notes/characterization-of-lib-cathode-slurries-using-simultaneous-measurements-of-rheology-and-impedance-spectroscopy-rh137/)

eBook: [Essential Battery Slurry Characterization Techniques](https://www.tainstruments.com/essential-battery-slurry-characterization-techniques/)

Binder and Additive

In addition to ensuring proper electrode coating, the binder and additive in electrode slurries greatly contribute to the overall performance. Binders require high thermal stability, good adhesion, and flexibility to facilitate proper battery performance. Low quality binders and additives can cause cracking and chipping on the electrode coating.

Battery developers typically test binder and additive at two key stages:

- To verify incoming materials' quality
- To measure the binder and additive's composition and electrode performance

[Thermogravimetric Analysis \(TGA\)](https://www.tainstruments.com/products/thermal-analysis/thermogravimetric-analysis/) measures weight change (loss or gain) and the rate of weight change as a function of temperature, time, and atmosphere. Loss of mass indicates possible decomposition or vaporization, while a gain in mass indicates possible oxidation, sorption or that the material is reacting with the purge gas.

In batteries, TGA quantifies the amount of binder and additive in the electrode. This measurement helps ensure the same amount of active material, binder and additive are in each batch of electrode. On the Discovery TGA, the high sensitivity Tru-Mass Balance is ideal for binder and additive testing as it ensures accurate measurement of each component in the electrode. Insufficient amounts of binder will affect the active the electrode material's

adhesion to the metal collector; too much binder will reduce the active material's content and affect the electrochemical reaction. Optimization of binder/additive ratios is essential for optimal battery performance and improvement of battery life.

TGA also measures the thermal stability, degradation temperatures, and content and composition of binder and additive. Thermal stability can help identify the right composition and types of the binder to ensure proper performance during electrode manufacturing.

[Differential Scanning Calorimetry \(DSC\)](https://www.tainstruments.com/products/thermal-analysis/differential-scanning-calorimeters/) measures binder melting and crystallization as well as glass transition. Crystallinity is related to a binder's mechanical properties, so this measurement can be used as a diagnostic to predict binder behavior. Glass transitions are related to the binder's flexibility, and DSC also reveals melting of the binder which will impact the thermomechanical properties. Lithium-ion batteries typically operate at temperatures of -20 °C to 60 °C. Higher temperatures can disrupt the solid-electrolyte interface (SEI), or even lead to decomposition. Thermal analysis enables researchers to understand the thermal stability of the electrode while optimizing slurry composition and solvent drying for improved batteries.

Characterization for Binders and Additives

Active Cathode and Anode Materials

A battery's cathode, or positive electrode, is usually made of a metal oxide; the anode, or negative electrode is usually made of graphite. The cathode and anode are capable of intercalating lithium ions during charge and discharge. The electrode must hold lithium ions without changing its structure, offer good electrochemical stability with the electrolyte, and be a good electrical conductor and diffuser of lithium ions. Additionally, the thermal stability and rate capability of the entire battery is largely dependent on the cathode and anode material.

Battery researchers are investigating cathodes and anodes with higher specific capacities while maintaining structural, chemical, and thermal stabilities along with low cost. Thermal analysis enables researchers and engineers to understand the thermal stability (phase transition, melting, and decomposition) of electrode and binder materials for safer and longer lasting batteries across all operational temperatures.

Dry Electrode Processing

Dry battery electrode processing is a growing technique that removes the toxic solvent and lowers the cost of electrode manufacturing. Dry battery electrode processing can be improved with powder rheology coupled with temperature control. Powder rheology measures the flowability, shear, compressibility, and wall friction of dry electrode mix to help optimize mixing and processing conditions. Plus, new temperature control features for powder rheology on the Discovery Hybrid Rheometer allow users to obtain crucial powder insights under application-specific conditions.

Cathode and Anode Resources

Application Note: [Safety Evaluation of Lithium-ion Battery Cathode and Anode Materials Using Differential Scanning](https://www.tainstruments.com/applications-notes/safety-evaluation-of-lithium-ion-battery-cathode-and-anode-materials-using-differential-scanning-calorimetry/)

Electrode Problem Solving

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Separators

Battery separators are porous membranes that separate the anode and cathode while allowing transport of ions during charge and discharge. Battery separators play a critical role in lithium-ion battery performance, including the prevention of thermal runaway. Some of the requirements for a battery separator include:

- Good electronic insulator
- Minimal electrolyte resistance
- Mechanical and dimensional stability
- Chemical resistance to the electrolyte
- Prevent contact between the electrodes
- Readily wetted by electrolyte
- Uniformity in thickness and properties

A number of analysis techniques are useful for determining separators' quality, stability, and integrity.

Separator Problem Solving

Thermal analysis of separators includes TGA, DSC, and TMA.

[Thermogravimetric Analysis \(TGA\)](https://www.tainstruments.com/products/thermal-analysis/thermogravimetric-analysis/) measures weight change and the rate of weight change as a function of temperature, time, and atmosphere. In separators, TGA helps researchers determine the decomposition temperatures of separators. That data can then help them determine the composition or percentage of individual compounds in the separator, a key step for quality control.

TGA curves of coated separator (blue) and uncoated separator (green)

Thermogravimetric data is critical to setting proper temperature limits for Differential Scanning Calorimetry method development. [Differential Scanning Calorimetry \(DSC\)](https://www.tainstruments.com/products/thermal-analysis/differential-scanning-calorimeters/) measures temperatures and heat flows associated with thermal transitions in a material. DSC helps battery separator researchers analyze the melting temperature, impact on melting, and further understand properties of the polymer content determined from the TGA results. DSC data is crucial in predicting how different materials will impact the separators' final properties.

[Thermomechanical Analysis \(TMA\)](https://www.tainstruments.com/products/thermal-analysis/thermomechanical-analyzers/) measures changes in the dimensions of a sample as a function of time, temperature, and force in a controlled atmosphere. TMA measures battery separators' shrinkage onset and rupture temperature. This data helps researchers confidently characterize separators and investigate the potential impact of added coating formulations. High reproducibility in TMA is especially important when measuring safety critical properties such a melt integrity, as a ruptured separator can lead to internal short circuit in a battery that may lead to thermal runaway.

When thermal analysis is combined with mechanical testing, it's possible to understand

dimensional stability of a polymer's length and shape, e.g., the polymer separator. This thermal and dimensional insight can help prevent separator failure and ensure battery safety.

For mechanical analysis of separators, researchers use [Dynamic Mechanical Analysis \(DMA\)](https://www.tainstruments.com/products/rheology/dynamic-mechanical-analyzers/) which measures the mechanical properties of materials as a function of time, temperature, and frequency. One particular challenge is preventing undesired elongation of separator material, which leads to deformation. DMA helps researchers understand tensile strength, tensile strain, and rupture point to identify material capabilities and limitation. This data empowers labs to optimize separators for specific applications, choosing adequate materials to offer the desired thermal and mechanical stability at end-use conditions.

Storage modulus, loss modulus, and tan delta curves for uncoated and coated separators

Example Experiments

See how TGA, DSC, TMA, and DMA contribute to efficient and effective separator characterization in these application notes with real experimental data:

[Thermal Analysis of Battery Separator Film](https://www.tainstruments.com/thermal-analysis-of-battery-separator-film/)

[Battery Separator Film Development: Impact of Coating](https://www.tainstruments.com/applications-notes/battery-separator-film-development-impact-of-coating/)

Electrolytes

One of the most critical battery components is the electrolyte, which transports Lithium ions between the cathode and anode during the charge and discharge process. Lithium-ion battery electrolytes are traditionally made of highly flammable solvent with highly reactive lithium salts.

Gel polymer electrolytes are a new formulation of interest as they offer strong electrochemical properties and improved safety over traditional solvent-based electrolytes. Despite these advantages, gel polymer electrolytes still require significant improvements to fully contend with liquid electrolytes.

When electrolytes reach their thermal degradation temperature, they will degrade and generate a highly exothermic reaction. When this reaction happens inside a battery pack, the highly exothermic degradation leads to thermal runaway if the heat is not removed from the battery.

How can you maintain battery temperature to avoid electrolyte degradation and optimize electrolytes for improved battery safety?

Electrolyte thermal stability can be investigated with Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). Diagnostic thermal stability testing saves time and resources as the safest, most stable electrolytes can be selected early in development.

TGA elucidates the decomposition temperature and solvent loss profile of electrolytes at high temperatures. This data contributes to understanding the overall thermal stability of the electrolyte. Additionally, DSC measurements identify the onset temperature and heat of reaction during exothermic decomposition. The heat of reaction can be used to estimate the heat released during the battery runaway. Combining TGA and DSC helps researchers evaluate the safety hazards of their batteries under chemical abuse and design effective thermal management systems.

How can you optimize the gel polymer electrolyte's mechanical properties?

Gel polymer electrolytes uniquely act as both the electrolyte and the separator, so the mechanical concerns of a separator's ability to separate the electrodes also apply to this electrolyte formulation. Dynamic Mechanical Analysis (DMA) measures the electrolyte's temperature-dependent mechanical response, offering key insights into the electrolyte's tensile strength, tensile strain, and rupture point under specific conditions. DMA helps battery scientists identify their electrolyte's material capabilities and limitations, facilitating stages of electrolyte development through to QA/QC of electrolyte formulation. Gel polymer electrolytes are a new formulation of interest as they offer strong electrochemical properties and improved safety over traditional solvent-based electrolytes. Despite these advantages, gel polymer electrolytes still require significant improvements to fully contend with liquid electrolytes.

How can you facilitate efficient electrolyte manufacturing with effective formulations?

A key area of electrolyte research involves optimizing formulations for ideal pumpability during manufacturing. This step offers ample opportunity for reduced cost and increased efficiency. Rheology enables engineers to measure viscosity and produce electrolyte solvents with optimal pumpability during manufacturing.

Components in lithium-ion batteries, such as electrolytes, lithiated materials and solid-electrolytes interface on electrode, are highly sensitive and reactive materials with atmosphere. Testing and handling of such materials must be done in an inert environment. It can be challenging but necessary to analysis those samples under an inert testing environment to prevent sample integrity is compromised by a brief exposure to nitrogen, oxygen, or water.

TA Instrument Discovery TGA and Discovery Hybrid Rheometers enable glovebox operation for Argon and Nitrogen environments, providing reliable material data of atmosphere-sensitive samples.

Resources

[Application Note: Thermogravimetry of Air Sensitive Materials - TA Instruments](https://www.tainstruments.com/applications-notes/thermogravimetry-of-air-sensitive-materials/)

Module Pack Materials

Thermal Management System

Battery thermal management systems (TMS) offer the opportunity to improve battery life and reduce overall battery cost – however, they must be carefully designed for this purpose. Maintaining temperature uniformity within a cell, and from cell to cell, requires proper understanding of the thermal conductivity and cooling capacity of the materials used in the TMS.

Optimizing thermal management systems with DSC, thermal conductivity meter and laser flash will reduce the overall cost of the battery pack, plus ensure battery safety and efficiency.

Thermal Interface Materials

Thermal adhesive and phase change materials (PCM) are used to regulate the battery pack temperature. The heat produced by the battery during use poses a threat of inducing thermal events and is detrimental to the performance of the battery itself. The advantage of PCM is they can maintain stable temperature when materials undergo phase changes. [Differential](https://www.tainstruments.com/products/thermal-analysis/differential-scanning-calorimeters/) [Scanning Calorimetry \(DSC\)](https://www.tainstruments.com/products/thermal-analysis/differential-scanning-calorimeters/) is especially helpful in optimizing PCM through measuring phase transitions, heat of fusion, and heat capacity to evaluate the materials cooling capacity.

Thermally conductive adhesive successfully holds the battery in place despite heat and offsets heat that could otherwise impede performance. Laser Flash and thermal conductivity meter are used to measure thermal diffusivity and thermal conductivity. This data helps battery developers select the best materials to dissipate heat.

Successful thermal interface materials must offer excellent thermal conductivity and thermal stability. All of these parameters can be evaluated using material analysis techniques.

Thermal Interface Material Problem Solving

Casing and Enclosure

The battery, which commonly weighs 300 kilograms (661 pounds), is mounted to the casing or enclosure. Battery casings are integral for overall battery performance, as they protect all internal components against contamination, adverse atmospheric changes, and damage from impact. Casings are commonly made of steel, aluminum or polymers composite. Battery casings must be molded or formed into their required shapes while providing protection for the inner components, which can be achieved through detailed material analysis and testing.

Casing and Enclosure Problem Solving

Technique Cheat Sheet

[Thermogravimetric Analysis \(TGA\)](https://www.tainstruments.com/products/thermal-analysis/thermogravimetric-analysis/) measures weight change (loss or gain) and the rate of weight change as a function of temperature, time, and atmosphere. Thermogravimetric data is critical to setting proper temperature limits for Differential Scanning Calorimetry method development. Other common usages include measuring:

- Thermal and oxidative stability of materials
- Moisture and volatile contents
- Composition of multi-component materials
- Decomposition kinetics and estimated lifetime of a product
- Effects of reactive or corrosive atmospheres

[Differential Scanning Calorimetry \(DSC\)](https://www.tainstruments.com/products/thermal-analysis/differential-scanning-calorimeters/) measures temperatures and heat flows associated with thermal transitions in a material. Common usage includes investigation, selection, comparison, and end-use performance evaluation of materials in research, quality control and production applications. Properties measured by TA Instruments' DSC techniques include:

- Phase changes
- Glass transition point
- Melting and crystallization
- Heat of reaction (enthalpy)
- Product stability
- Reaction kinetics

[Dynamic Mechanical Analysis \(DMA\)](https://www.tainstruments.com/products/rheology/dynamic-mechanical-analyzers/) measures the mechanical properties of materials as a function of time, temperature, and frequency. In addition to quantifying viscoelastic properties of materials, DMA can also quantify finished component and product characteristics, reflecting the important contribution that processing has on end-use product performance. DMA is commonly used to measure:

- Mechanical properties
- Modulus and stiffness
- Creep and stress relaxation
- Glass transition temperatures (Tg) and secondary transitions
- Cure and processing optimization
- Filler effects in composites

[Rheology](https://www.tainstruments.com/products/rheology/discovery-hybrid-rheometers/) is the study of flow and deformation of materials under the influence of an external force or stress. In batteries, rheology measurements are used to ensure successful material processing, optimize product performance, gain insights into complex microstructures and develop novel materials. While viscometers are limited to measuring viscoelasticity, rheometers provide more comprehensive information for battery development, including:

Non-Newtonian Behaviors

- Shear thinning
- Thixotropy
- Yield stress

Viscoelasticity

- Storage Modulus
- Loss Modulus
- Tan Delta

Rheology is especially crucial in forming battery slurries that form uniform, defect-free coatings required for production of consistent, high-quality electrodes with high batch-to-batch repeatability and low scrap rates. Additionally, rheology is used to optimize the manufacturing and performance of gel polymer electrolytes and adhesives.

[Isothermal Microcalorimetry \(IMC\)](https://www.tainstruments.com/products/microcalorimetry/isothermal-calorimetry/) is a non-specific and non-destructive technique for measuring the smallest reactions in a material during a physicochemical process. This is done by measuring the heat flow from the sample at a constant temperature. Isothermal microcalorimetry offers key battery insights:

- Heat management
- Entropy changes
- SEI formation
- Full cycle parasitics
- Narrow cycle parasitics

For whole battery testing, the **[Battery Cycler Microcalorimeter](https://www.tainstruments.com/battery-cycler-microcalorimeter/)** [Solution](https://www.tainstruments.com/battery-cycler-microcalorimeter/) is able to measure heat flows during battery cycling on a fully integrated, flexible system. Data from the TAM IV Isothermal Microcalorimeter data are both collected and analyzed in one user interface, making it easy to run experiments and analyze results. The Battery Cycler Microcalorimeter Solution is revolutionizing whole cell testing with the highest throughput, fastest data aggregation, and best experimental efficiency of any testing solution available.

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