

-From Materials to Cells and Modules-

Analytical Solutions for Lithium-Ion Batteries

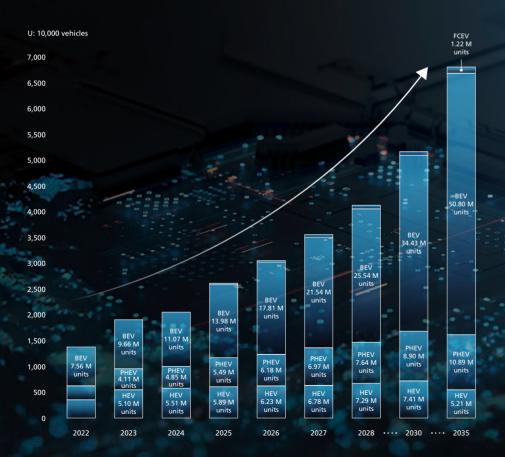


For a Future Enabled by Lithium-Ion Batteries

Important devices in terms of achieving a carbon-free society, lithium-ion batteries (LiB) have attracted heightened interest in mobility and energy fields, particularly in the area of electric vehicles. As a result, the demand for analytical solutions has expanded, not just for improving product performance, such as ensuring consistent product quality and improving environmental friendliness, but also for performing a wide variety of other analyses.

Shimadzu offers a variety of analytical and measuring instruments for lithium-ion battery applications ranging from R&D and evaluation of material characteristics to product quality control, degradation analysis, and recycled material evaluation. We are committed to achieving a sustainable society by offering optimal solutions for customers involved in all life cycle stages of lithium-ion batteries.

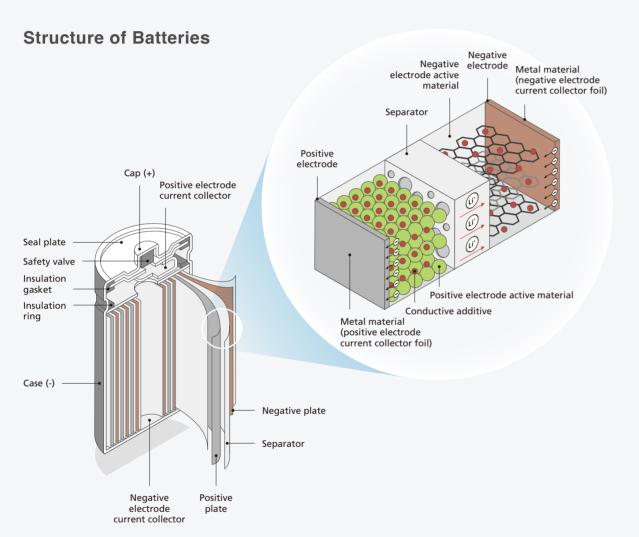
Global Adoption and Sales Outlook of Electric Vehicles (HEV, PHEV, BEV, and FCEV)



Reference: 2024年版電気自動車関連市場の最新動向と将来予測(株式会社総合プランニング)

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Lithium-ion batteries are composed of positive electrode materials, negative electrode materials, separators, and electrolyte solution. Individual completed batteries are referred to as cells and assemblies of multiple cells packaged together are referred to as modules. The evaluation of batteries requires an extremely wide variety of methods because batteries are assembled from a variety of materials, such as ceramics,

polymers, and an electrolyte solution, because they involve electrochemical changes due to charge-discharge cycles, and also because they are difficult to handle due to their sensitivity to air and moisture. Therefore, batteries require multifaceted evaluations using a wide variety of analytical instruments. For safety reasons, reliability testing and safety evaluations that involve physical destruction are essential.

Positive Electrode

Positive electrodes are typically created by applying a slurry mixture of positive electrode active materials, a binder, conductive additive, and other materials onto aluminum foil, and then drying and press forming the coated foil. Positive electrode materials typically consist of a ternary (NMC) or lithium iron phosphate (LFP) cathode material, polyvinylidene fluoride (PVDF) as a binder, carbon black or other such substance as a conductive additive, and n-methylpyrrolidone (NMP) as a solvent.

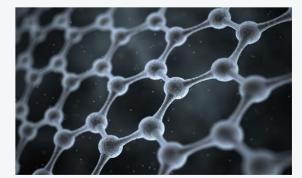
Because the raw materials can affect the energy density, safety, service life, and other performance characteristics of batteries, it is important to evaluate their particle properties, thermal properties, composition, and other characteristics.

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Negative electrodes are typically created by applying a slurry mixture of negative electrode active materials, a binder, conductive additive, and other materials onto copper foil, and then drying and press forming the coated foil. Negative electrode materials typically consist of graphite, styrene-butadiene rubber (SBR) or carboxymethylcellulose (CMC) as a binder, carbon black or other such substance as a conductive additive, and water as a solvent. Because the raw materials can affect the energy density, safety, service life, and other performance characteristics of batteries, it is important to evaluate their particle properties, thermal properties, composition, and other characteristics.

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Separator

Separators consist of a porous membrane used to separate positive electrodes from negative electrodes. Whereas ions can normally pass back and forth through the pores, the pores close up if the battery becomes hot. Thus, the separators serve to prevent uncontrolled heating. Separators are mainly made of polyethylene or other polyolefin polymer. It is important to evaluate the physical and thermal properties of separators and their composition, because separators must not obstruct lithium ion movement during charging-discharging and must be electrically insulative and mechanically strong in order to prevent short-circuiting between positive and negative electrodes.

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- > Evaluation of Organic Components PP.18-19

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Electrolytes Characteristics (Liquid and Solid Electrolytes)

The electrolyte is the substance located between the positive and negative electrodes that includes the ions that serve as a carrier. For liquid lithium-ion batteries, the electrolyte is referred to as an electrolyte solution, which consists of a mixture of ethylene carbonate (EC) or other organic solvent, lithium hexafluorophosphate (LiPF6) or other lithium salt, and vinylene carbonate (VC) or other additive. Because the electrolyte solution status can affect battery performance, it is important to evaluate its composition.

For all-solid-state batteries, three types of solid electrolytes, referred to as oxide-based, sulfide-based, and polymer-based electrolytes, are typically used. Because the status of solid electrolytes can affect battery performance, it is important to evaluate their particle, surface, and thermal properties as well as their chemical composition and other characteristics.

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Parameters for Evaluating Batteries and Battery Parts

Evaluation Parameters	Positive Electrode	Negative Electrode	Separator	Electrolyte Solution	Solid Electrolyte	Cell and Module
Evaluation of Particle Properties (Particle Size Analyzer, Pycnometer, and Specific Surface Area Analyzer)	~	~			~	
Evaluation of Inorganic Components (EDX, ICP, and AA)	~	~	~	~	~	
Evaluation of Physical Properties Universal and Hardness Testers	~	~	~		~	~
Evaluation of Thermal Properties (DSC and TG)	~	~			~	
Evaluation of Internal Structures (X-Ray CT)						~
Evaluation of Chemical Bond Status (XPS)	~	~				
Evaluation of Organic Components (LC, LCMS, GC, GCMS, and TOC)	~	~	~	~	~	~
Evaluation of Micro Areas (EPMA, SPM/AFM, and OLS)	~	~	~		~	

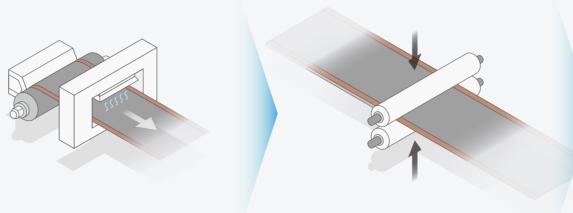
Step 1 Mix

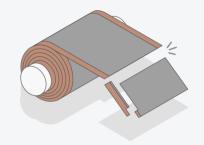


Step 3 Calendering



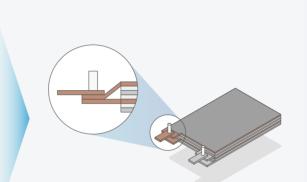
Slit





Process	Mix	Coat and Dry	Calendering	Slit
Process Description	Uniformly mixes respective raw materials used in battery positive/negative electrodes.	Applies the slurry mixture of respective positive/negative electrode raw materials onto current collector foil and dries the solvent/water.	Electrode materials are pressed to increase density and ensure consistent surface characteristics.	Electrode sheet material is cut to length based on the shape of the battery cell.
Applicable Samples	Respective raw materialsMixing status	Drying parametersSlurryCurrent collector foil	Pressing parametersElectrode sheet material	Cross sectionElectrode sheet material
Solutions	 > Evaluation of Particle Properties > Evaluation of Inorganic Components > Evaluation of Physical Properties > Evaluation of Thermal Properties > Evaluation of Organic Components PP.18-19	> Evaluation of Particle Properties > Evaluation of Physical Properties > Evaluation of Thermal Properties P.16	> Evaluation of Physical Properties > Evaluation of Micro areas P.19	> Evaluation of Physical Properties > Evaluation of Micro areas P.19

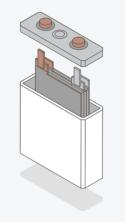
Step 1 Stack



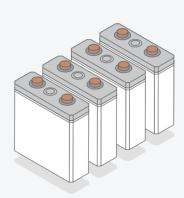
Step 2

Weld Tabs





Step 4 **Assemble Module**



Process	Stack	Weld Tabs	Assemble and Seal Cell	Assemble Module
Process Description	Stack the cut sheets of electrode material.	Weld the tabs used to remove electricity from the positive/negative electrodes.	Assemble the stack of battery components into a battery cell and fill it with electrolyte solution.	Assemble the cells into modules containing many cells for use in vehicles.
Applicable Samples	Stack status	Welding parameters	Sealing status	Heat dissipation
Solutions	> Evaluation of Internal Structures P.17	 Evaluation of Physical Properties (high-speed camera) Blue Diode Laser Technology Optimal for Cu-welding 	> Leak Detection Using a Helium Leak Detector	> Solutions for Automotive LINK



Recycling Lithium-Ion Batteries

Lithium-ion batteries contain rare metals such as Li. Co. and Ni. As the market for lithium-ion batteries expands, there are growing demands for using recycled materials in an effort to ensure a reliable supply of raw materials and achieve sustainability. Shimadzu offers various solutions for needs such as evaluating performance of battery using recycled materials, evaluating processes for minimizing process CO₂ emissions, and managing regulated substances emitted from manufacturing processes.



Inorganic Component Analysis

Analyze the components that could affect battery performance by accurately measuring Li, Ni, Co, MN, Cu, Fe, and other contaminants contained in precursor materials.

> Evaluation of Inorganic Components P.15

Battery Design

These solutions support the design of batteries using recycled materials.

> Evaluation of Chemical Bond Status P.17

> Evaluation of Micro Areas P.19

Powder Characteristics

Direct recycling can be considered or wet smelting process parameters can be optimized by measuring the particle size uniformity of black mass.

> Evaluation of Particle Properties P.14

Design for Easy Recycling

These solutions support developing thermal and mechanical designs that take into consideration how easy products are to disassemble and crush processes.

> Solutions for Automotive LINK

Evaluation of Organic Residues

The effects of refining processes can be minimized by analyzing carbon and binder residues.

> Evaluation of Thermal Properties P.16

Non-Destructive Inspection

The feasibility of secondary uses can be judged by evaluating X-ray CT scans.

> Evaluation of Internal Structures P.17

Evaluation of Liquid Waste and Gas Emissions

Analyzing the liquid waste and emission gas generated can help achieve clean manufacturing processes.

> Evaluation of Organic Components PP.18-19

Evaluation of Physical Properties

Physical properties involved in battery disassembly processes, such as how easy it is to dismantle assemblies or separate active substances, can be analyzed to optimize dismantling parameters or heat treatment parameters.

> Evaluation of Physical Properties P.16

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Evaluation of Particle Properties

Laser Diffraction Particle Size Analyzer

SALD-2300





- Measures a wide range of particle diameters from 0.017 to 2500 μm.
- Real-time measurements can confirm changes in the dispersion status as a
- From powders to concentrated pastes, a wide range of particle concentration levels, from 0.01 to 20 %, can be measured.



Application Example

Evaluation of Particle Properties of Lithium-Ion Battery Anode Materials —Particle Size Distributions and Shapes—

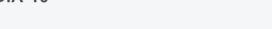
Dynamic Particle Image Analysis System

Particle detection system offers high reliability.

iSpect DIA-10







- Particle size, particle shape, and particle concentration can be evaluated
- Abnormal particles (foreign matter, aggregates, etc.) can be detected.



Application Example

Evaluation of Particle Properties of Lithium-Ion Battery Anode Materials —Particle Size Distributions and Shapes—

Evaluation of Inorganic Components

Multitype ICP Emission Spectrometer

ICPE-9820

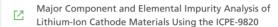


- Elemental impurities can be analyzed in electrodes, electrolyte solutions, etc.
- By measuring multiple elements at the same time, additional elements or wavelengths can be added after measurements are finished.
- Using a mini-torch helps reduce running costs.



Application Examples





Energy Dispersive X-Ray Fluorescence Spectrometer

EDX-7200





- Can measure samples in a variety of forms, including solids, powders, and
- Samples can be analyzed directly or with minimal pretreatment.
- Can analyze a wide range of concentrations, from ppm-levels to 100 %.

Application Example

Fast and Accurate Analysis of Black Mass Using EDX-7200 Facilitated by Using ICPE-9820

Infrared Raman Microscope

AIRsight





- Enables both FTIR and Raman measurements within the same field of view using a single software program.
- Information can be acquired from both organic and inorganic components using a single instrument.

Application Example

Analysis of Microplastics Using AlRsight Infrared/Raman Microscope



Evaluation of Physical and Thermal Properties

Precision Universal Testing Machine

Autograph AGX-V2 Series





- Can be used to evaluate a wide variety of physical properties.
- An extensive lineup of jigs is available, enabling a wide variety of tensile, compression, bending, penetration, and other testing.
- Strain distributions can be visualized using DIC analysis.



- Measuring the Bulk Density of Graphite Powder
- Evaluation of Temperature-Dependent Strength Properties of Lithium-Ion Battery Separator



EZ Test



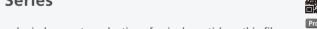
- Light weight and compact size make measuring easy.
- An extensive lineup of jigs is available, enabling a wide variety of tensile, compression, bending, penetration, and other testing.



Physical Properties Test of Battery-Related Components and Materials

Micro-Compression Testing Machine

MCT Series



- Enables physical property evaluation of a single particle or thin films.
- Enables the evaluation of pressing parameters for battery manufacturing.
- Supports heat testing (max. 250 °C).
- Includes extensive functionality for supporting measurements.

Application Examples

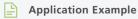
- Compression Test for Structural Materials of Lithium-Ion Batteries
- Compression Tests for Anode Material for Lithium-Ion Batteries

Simultaneous Thermogravimetric and Differential Thermal Analyzer

DTG-60 Series



- Enables evaluation of thermal decomposition and thermal stability characteristics during battery material heating.
- Can detect trace moisture content.
- Enables evaluations for reviewing battery stability.



Characterization of Lithium-Ion Battery Binders









Evaluation of Internal Structures

Microfocus X-Ray CT System

inspeXio SMX-225CT Series



- Enables non-destructive observation inside objects.
- Data can be checked immediately after scanning due to high-speed
- The detector offers the highest resolution in the industry for checking fine



Application Example



Analysis of the Cylindrical Lithium-Ion Battery by X- Ray CT System and Introduction to the Charge/Discharge Device Attached System

Evaluation of Chemical Bond Status

Imaging X-Ray Photoelectron Spectrometer

AXIS Supra+



- Enables analysis of elements and chemical bonds present within surface areas of about 10 nm.
- By differentiating between Ar gas clusters and a monatomic Ar ion source, a wide variety of samples can be sputtered.



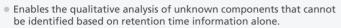


XPS analysis of solid-electrolyte interphase layer formed on a lithium-ion battery negative electrode



GCMS-QP2050

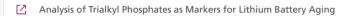






Application Examples

Residual NMP and Solvent Analysis in Lithium-ion Battery Cathode



Gas Chromatograph

Nexis GC-2030



 Proprietary Shimadzu BID detectors (barrier discharge ionization) detectors) enable evaluating inorganic gases and hydrocarbons at once.

Application Examples

Analysis of battery electrolytes and N-methyl-2-pyrrolidone (NMP) via headspace GC-FID

Analysis System for Gases in Rechargeable Lithium-Ion Batteries

Anion Chromatograph

HIC-ESP



- Evaluates reaction products not involved in electrolyte decomposition.
- Column switching shortens analysis times.

Application Example

Analysis of the Decomposition Products of Lithium Hexafluorophosphate in the Electrolytic Solution of Lithium-Ion Rechargeable Batteries by Column-Switching Ion Chromatography (Part 2)

Single Quadrupole Mass Spectrometer

LCMS-2050

- Enables component analysis and degradation evaluation of electrolyte solutions.
- High robustness and easy maintenance.









Fourier Transform Infrared Spectrophotometer

IRSpirit-X

- Wireless control enables measurements even within a glovebox (available by special-order).
- Enables reaction tracking, such as to confirm electrolyte solution
- Compact size is smaller than A3 paper.



Application Example

Measurement of Electrolyte Solutions for Lithium-Ion Secondary Batteries with IRSpirit Glove Box System

Evaluation of Micro Areas

Electron Probe Microanalyzer

EPMA-8050G

- Outstanding electron probe characteristics enable both high sensitivity and high spatial resolution.
- Analyzes chemical bond status in target micro areas.
- Elements can be mapped across large areas using stage scanning.

Application Examples

- State Analysis of Positive Electrode Active Material in Lithium Ion Battery
- Analysis of Positive Electrode (NCM) for Lithium Ion Battery by EPMA and SPM

Scanning Probe Microscope

SPM-Nanoa

- Enables micro-area observation, electrical property evaluation, and mechanical property evaluation in atmospheric air environments.
- Measures electrical currents within micro areas on positive and negative electrode surfaces.
- Surface shape can be observed and electrical properties evaluated at electrode—solid electrolyte interface surfaces.

Application Examples

- Surface Potential Measurement at the Electrode-Electrolyte Interface of a Charged All-Solid-State Lithium-Ion Battery
- Evaluating Electrochemical Activity and Electric Potential inside Cathodes in All-Solid-State Lithium-Ion Batteries







Related Information

Electrification Solutions

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