

Guide to Lithium-ion Battery Solutions



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Material	Instrument	Application Example	
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Negative Electrode	Active Material / Conductive Additive / Binder	Dynamic Particle Image Analysis System DIA Particle size measurement and image analysis of electrode materials P33	

Material	Instrument	Application Example
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		Battery cell
Confirmation of changes in gas composition due to deterioration P21		

Material Testing

Compression test of various materials that construct a Lithium-ion Battery



Micro Compression Tester MCT Series

- Consideration of conditions for battery packaging and restraint pressure
- Examination of manufacturing process conditions (change in strength during heating)

Inner structural materials of a Lithium-ion Battery are subjected to external force during production processes and to pressure during use. Therefore, evaluating the strength of each structural material is important to maintain consistent quality. Below are the results of compression tests performed on Lithium-ion Battery materials using the Micro Compression Testing Machine.

Purpose

Compression test of positive electrode active materials

Data

Compression Test Results

Sample Name	Fracture strength [mN]	Particle size [μm]	Strength [MPa]
LiMn ₂ O ₄	1.67	13.0	7.79
LiCoO ₂	16.23	13.3	72.75

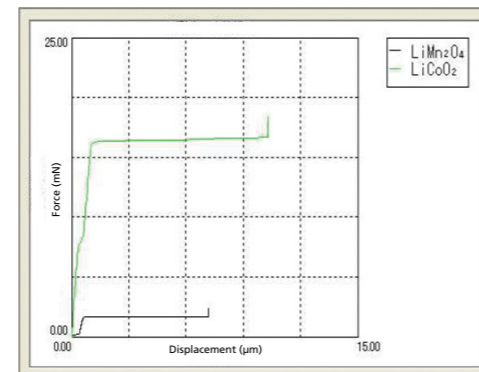
Result

The force is on the vertical axis, the displacement is on the horizontal axis, and fracture occurs at the inflection point where the displacement becomes horizontal. The fracture strength of the lithium cobalt oxide LiCoO₂ particle was measured to be 72.75 MPa compared to 7.79 MPa for the lithium manganese oxide, LiMn₂O₄.

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Samples

Two types of positive electrode active materials

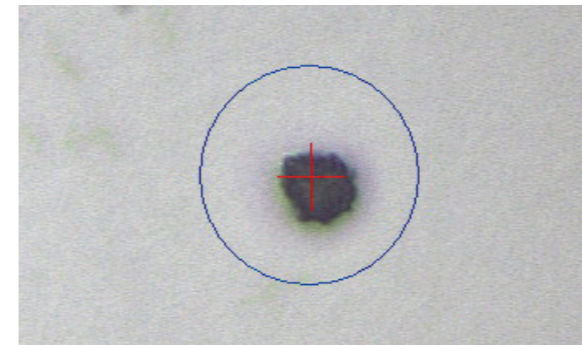


Test force-displacement graph

Purpose

Compression test of solid electrolyte raw materials

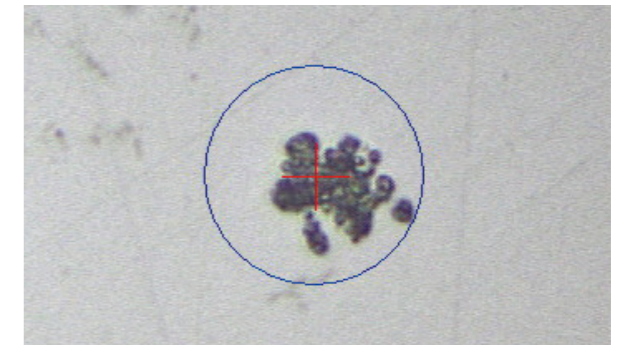
Data



Before

Samples

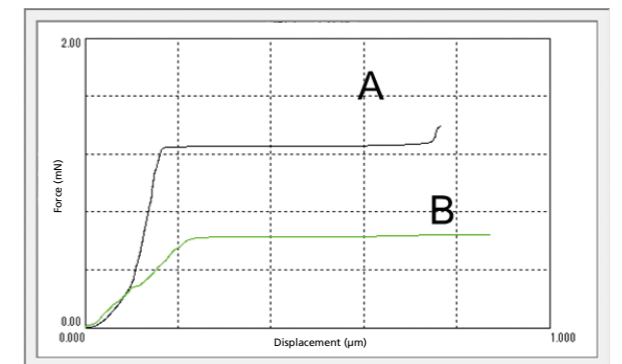
Two types of solid electrolyte raw materials



After

Compression Test Results

Sample Name	Fracture strength [mN]	Particle size [μm]	Strength [MPa]
A	1.25	1.765	315
B	0.63	4.265	27



Test force-displacement graph

Result

By measuring the fracture strength, we can compare the correlation with the ease of molding as an electrolyte. Comparing particles A and B shows that the fracture strength of particle B is about 1/10 weaker. This indicates that the particles adhere well to each other during the molding process.

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Strength measurement and strain characteristic evaluation of a separator at high temperature



AUTOGRAPH Precision Universal Tester AGX-V

- Various mechanical strength measurements of Lithium-ion Battery materials
- Evaluation of the piercing strength of a separator packed with high density
- Visualization of strain distribution by DIC analysis

The separator is installed so that it is in contact with the positive and negative electrodes. Since the temperature rises during charging, it is necessary to maintain mechanical strength even as the temperature changes. The following shows an example of measuring how the strength of a separator in the tensile test and the piercing test changes with respect to temperature changes, and an example of evaluating the strain characteristics after piercing damage.

Purpose

Tensile test of separator

Data

Tensile Test Results

Specimen	Lithium Battery Separator		
Specimen Name	A	B	C
Elastic Modulus (MPa)	902	1856	1376
Tensile Strength (MPa)	165	118	101
Break Point Strain (%)	27.6	31.7	29.1

Result

From the measurement results, it can be seen that the separator with high tensile strength is A.

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Purpose

Tensile strength measurement of a separator
(Condition : 25 °C, 60 °C, and 90 °C)

Samples

Two types of separators, prepared in dumbbell shapes along both the length and width direction

Data

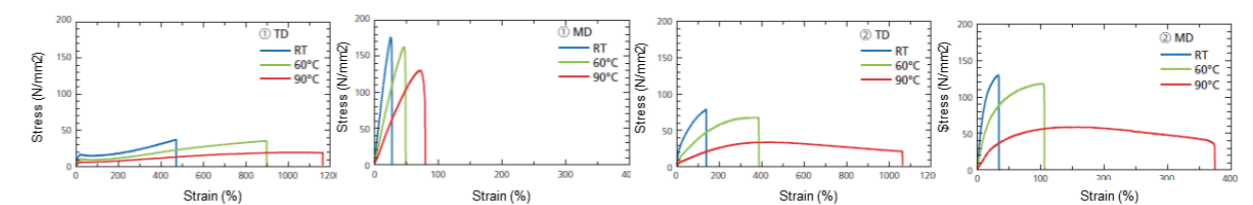
Mechanical properties of separator (1) and (2) in the short side and long side directions

Long side: Machine Direction
Short side: Transverse Direction

Sample	25 °C		60 °C		90 °C	
	Tensile strength (MPa)	Rupture strain (%)	Tensile strength (MPa)	Rupture strain (%)	Tensile strength (MPa)	Rupture strain (%)
(1)Short side	36.9	471.4	35.4	898.8	19.3	1044.0
(1)Long side	175.6	26.8	162.5	57.0	129.9	76.7
(2)Short side	78.2	138.5	68.8	347.6	33.8	427.9
(2)Long side	129.5	34.1	118.3	105.3	58.7	367.2

Separator (1)

Separator (2)



Stress-strain graph

Result

Separator (1) is produced by near uniaxial stretching in the machine direction (MD), and separator (2) is produced by biaxial stretching at a low stretching ratio. Despite the increase in elongation properties at 60°C, excellent mechanical strength is maintained.

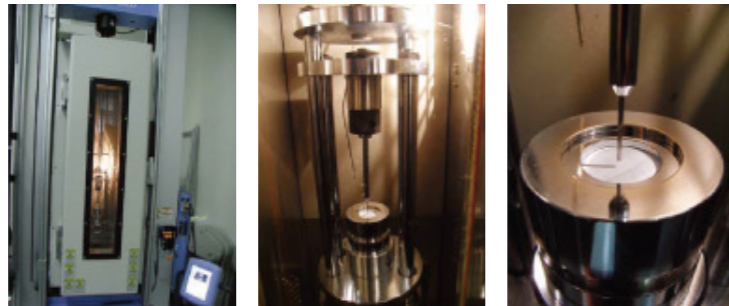
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Purpose

Puncture strength measurement of a separator
(Under environment of 25 °C, 60 °C, 90 °C)

Samples

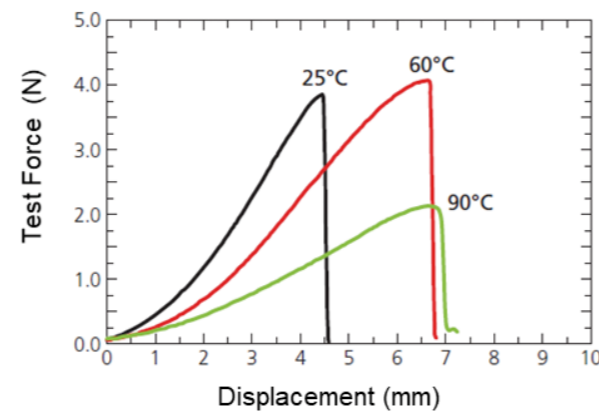
Separator



Data

Maximum Test Force / Displacement for Each Temperature

Test Temperature (°C)	Maximum Test Force (N)	Maximum Displacement (mm)
25	3.85	4.45
60	4.07	6.63
90	2.13	6.68



Test Force-Displacement Curve

Result

Comparing the results at 25 °C and 60 °C, we can see that the maximum test force is the same, but the maximum displacement value is higher at 60 °C. Next, comparing the characteristic values at 60 °C and 90 °C, we can see that the maximum test force decreases at 90 °C, but the maximum displacement is the same. From the above, it can be seen that the lithium-ion battery separator used in this test has an increase in elongation characteristics but no decrease in strength at 60 °C.

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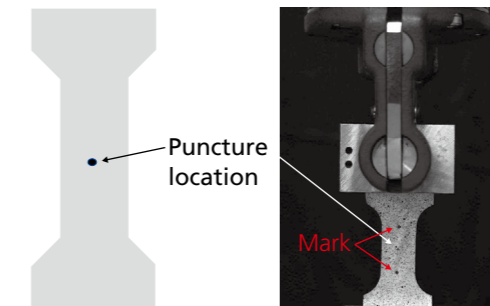


Purpose

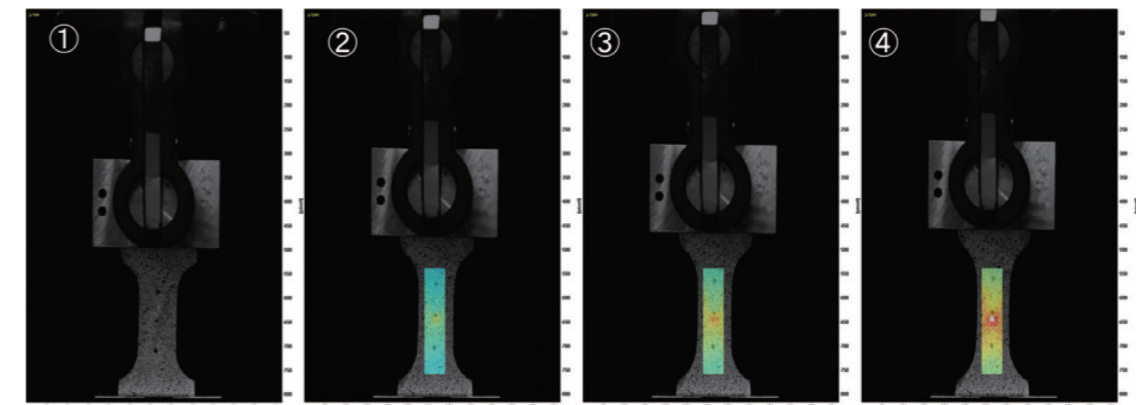
Evaluating the strain characteristics of separators after puncture damage

Samples

Separator after puncture test



Data



Strain distribution under tensile load

Result

We evaluated the strain characteristics of a separator after puncture to investigate how a damaged separator may act under stress. DIC analysis is a method of comparing random patterns on the surface of an object before and after the object is deformed to determine the amount of pattern movement and measure strain. Using the DIC analysis method, we can visually see that the area around the damaged area in the center turns red where stress is concentrated and strain is the largest.

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Thermal Analysis

Evaluate the decomposition characteristics and thermal stability of battery materials during heating



Differential Scanning Calorimeter DSC-60 Plus

- Examination of Lithium-ion Battery safety
- Selection of compound for electrode material and selection of compounding conditions
- Evaluation of physical properties of polymer materials by crystallinity

In some cases, lithium-ion batteries may generate abnormal heat due to overcharging, which may lead to problems such as ignition, in the worst case. To ensure the safety of the battery, it is important to evaluate the decomposition characteristics and thermal stability of each component by DSC during heating.

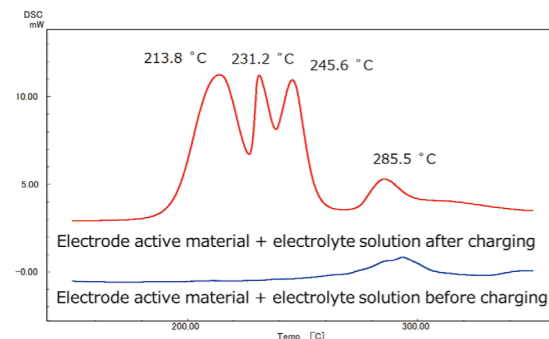
Purpose

Evaluation of decomposition characteristics and thermal stability of each component

Samples

Charged/Uncharged electrode active material and electrolyte solution

Data



DSC Measurement of Electrode Active Materials

Result

The upper curve shows the active material after charging. The active material becomes unstable due to charging, and a large exothermic peak due to decomposition is observed from around 200 °C. The exothermic peak around 290 °C is thought to be from the electrolyte.

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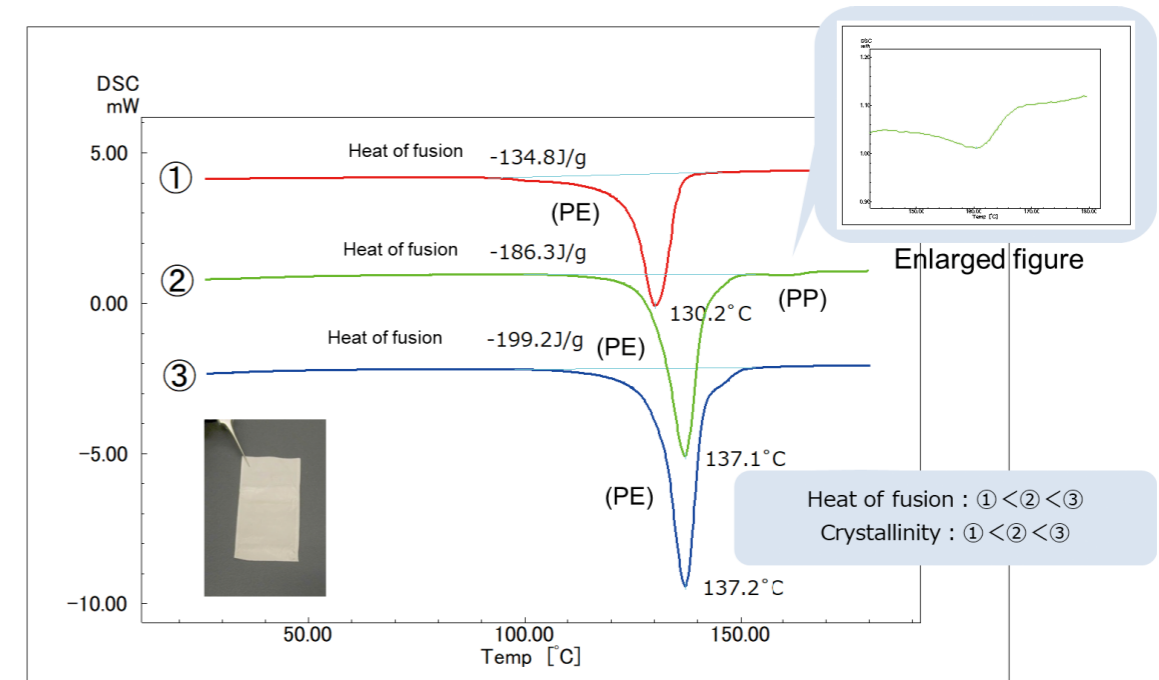
Purpose

Determining the melting temperature and calorimetry of separators

Samples

Three types of separators

Data



DSC Measurement of Separators

Result

The endothermic peaks were observed at around 100 °C to 140 °C, which is expected to be the melting point of polyethylene. When the separator is exposed to high temperature, it shrinks near the melting temperature, which affects the insulation. For safety reasons, it is necessary to know the temperature at which the separator contracts, and the measurement of the melting temperature by DSC is an indicator of the contraction temperature.

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Moisture evaluation during heating of electrode active material



Thermogravimetric Analyzers TGA-50 series

- A small amount of water content can be detected
- Evaluation of connectivity by measuring the dehydration process temperature
- Measurement is possible in the atmosphere of air or nitrogen

It is important to control the moisture content in lithium-ion batteries because it affects their lifetime. The moisture content of a material can be measured using a thermogravimetric analyzer (TGA).

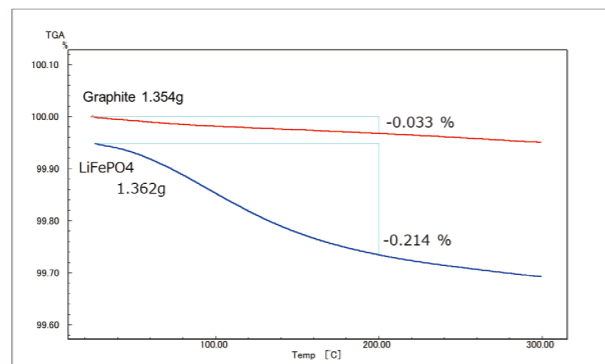
Purpose

Measurement of moisture content in the active material

Samples

Positive/Negative electrode active material

Data



TGA Measurement of Electrode Active Materials

Result

A weight loss rate at 200 °C was determined, and the moisture content was 0.033% and 0.214%, respectively.

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Evaluation of shrinkage amount when the separator is heated



Thermomechanical Analyzer TMA-60

- It is possible to measure the amount of shrinkage and shrinkage stress at each temperature
- Continuous recording of a sample's dimensional changes as temperature changes or over time in the order of μm
- Tensile measurement, expansion measurement, and needle insertion measurement are possible with one unit

In general, the higher the shrinkage temperature and the smaller the amount of shrinkage, the safer it is. Thermo-mechanical analysis (TMA) was used to measure mainly the shrinkage of the separator.

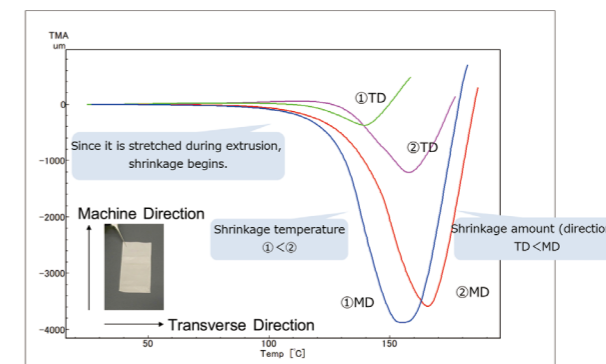
Purpose

Measure the shrinkage of the separator

Samples

The two directions of the separator called the MD (machine direction) and TD (transverse direction)

Data



TMA Measurement of Separators

Result

According to the data, the start of shrinkage begins approximately between 80 °C and 100°C. In terms of the amount of shrinkage, the amount of shrinkage is larger in the MD direction than in the TD direction for both No.① and ②.

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Evaluation of electrolyte solution in an inert atmosphere



Fourier Transform Infrared Spectrophotometer IRSpirit™

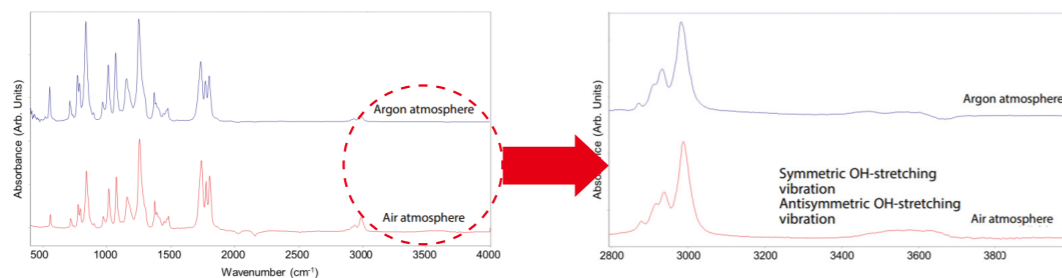
- Raw material confirmation, foreign matter screening analysis
- Small design allows it to be used in glove boxes and draft chambers

It is desirable to handle and characterize the cell components under an atmosphere not affected by water vapor or oxygen. The IRSpirit, a compact FTIR, can be installed in a glove box, thus enabling the evaluation of cell components in a high-purity argon atmosphere with low dew point and low oxygen concentration.

Purpose

Comparison of spectra under argon and air atmospheres

Data



Infrared Spectra of EC+DEC (3 : 7) Electrolyte Solution Containing 1M LiPF₆

Result

Although the optical properties of argon and air are very different, the left image shows that the infrared spectra acquired under both atmospheres were almost identical, appearing to be unaffected by the difference in atmosphere. Note that in the right image, the broad absorption of symmetric and antisymmetric OH-stretching vibrations from water molecules were observed between 3400 and 3700 cm⁻¹ in the air atmosphere. Meanwhile, the influence was negligible in the infrared spectrum in the argon atmosphere.

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Purpose

Spectrum comparison in the presence and absence of LiPF₆

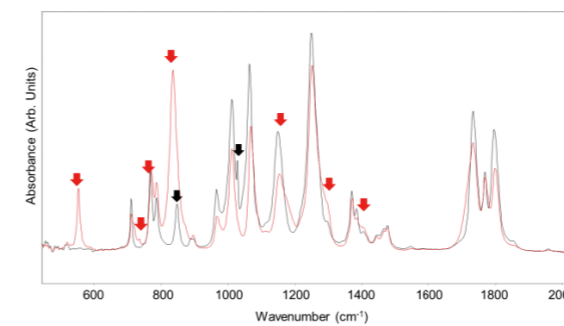
Samples

EC + DEC (3 : 7) Electrolyte Solution (Containing 1M LiPF₆)
 EC + DEC (3 : 7) Electrolyte Solution (Not containing 1M LiPF₆)
 1M LiPF₆ : Lithium Hexafluorophosphate
 EC : Ethylene Carbonate
 DEC : Diethyl Carbonate

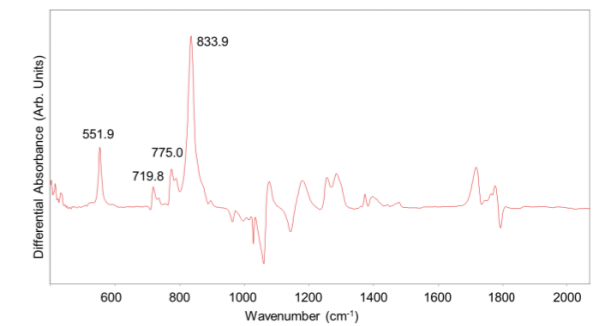


*Since IRSpirit can be controlled wirelessly, a sample can be measured while the FTIR unit is installed in a glove box (custom order required).

Data



Infrared Spectra of EC+DEC (3 : 7) Electrolyte Solution Containing 1M LiPF₆ (red) and EC+DEC (3 : 7) Solution (black)



Difference Spectrum between EC+DEC (3 : 7) Electrolyte Solution Containing 1M LiPF₆ and EC+DEC (3 : 7) Solution

Result

The red and black arrows on the left are characteristic absorptions of each component. To clarify the difference between the two spectra, the red line minus the black line is shown on the right. In the frequency range of 700 ~ 1000 cm⁻¹, characteristic vibrational modes in which EC or DEC and lithium ions are solvated are observed. Therefore, it can be assumed that the four absorption lines shown in the right figure are characteristic absorption lines in which EC or DEC and lithium ions are solvated.

The data shown above was provided by Associate Professor Takashi Ito at Frontier Research Institute for Interdisciplinary Sciences, Tohoku University. We would like to take this opportunity to express our sincere gratitude. Please also see the article in FTIR TALK LETTER Vol. 35.

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Comparison of components between new and deteriorated electrolyte solutions



Ion Chromatograph HIC-ESP

- Evaluation of products generated by decomposition of electrolyte
- Shorten analysis time by utilizing a column switching system

Lithium hexafluorophosphate is commonly used as the electrolyte solution. It is hydrolyzed by the trace amount of water contained in the electrolyte solution. Fluoride ions generated by this decomposition affect battery performance, so analysis of decomposition is important in the quality control process.

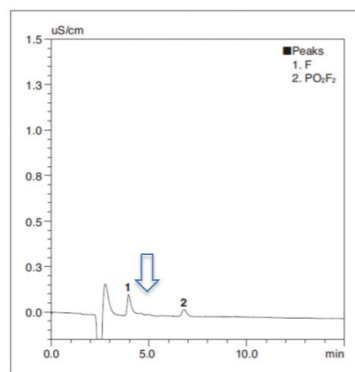
Purpose

Evaluation of Lithium Hexafluorophosphate Decomposition in Electrolyte Solution

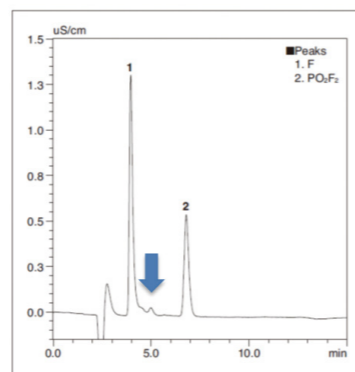
Samples

Electrolyte Solution
(New, deteriorated: accelerated deterioration test)

Data



Chromatogram of Electrolyte Solution (New)



Chromatogram of Electrolyte Solution (Degraded Product)

Result

A peak was not detected in the new electrolyte solution (New), but only in the deteriorated electrolyte solution (Degraded Product).

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Qualitative analysis of solvents and trace additives used in electrolyte solutions



Single Quadrupole GC-MS GCMS-QP™2020 NX

- Evaluation of reaction products generated by deterioration
- Qualitative analysis of unknown components that cannot be identified only by retention time information

The electrolyte solution in a Lithium-ion Battery is composed of organic solvent (mainly carbonate-based), electrolyte and additives. It is important to evaluate the electrolyte solution and the deterioration state of the electrolyte solution due to charging and discharging. GCMS is useful for the analysis of those constituents.

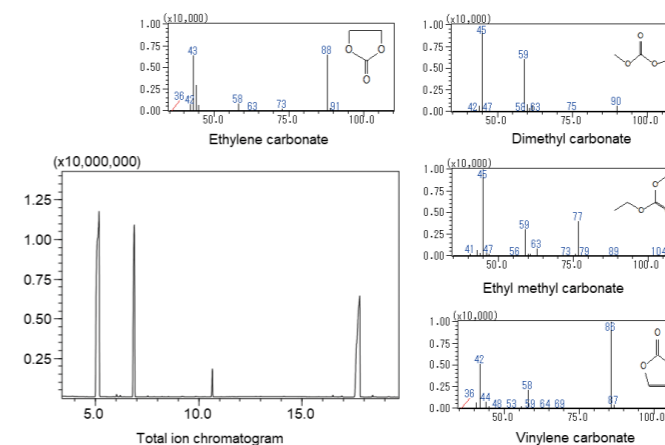
Purpose

Solvents used in electrolyte solution and component analysis of additives

Samples

Electrolyte Solution

Data



Result

Dimethyl carbonate, ethyl methyl carbonate, and ethylene carbonate, which are used as solvents, were identified from library search results. Vinylene carbonate, which was used as an additive, was also identified.

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Component analysis of internally generated gas and deterioration evaluation due to charging and discharging



Gas Chromatograph Nexis™ GC-2030

- BID (Barrier-discharge Ionization Detector) is more sensitive than TCD, and can deal with objects that cannot be detected by FID
- Detect inorganic gases and hydrocarbons simultaneously
- Qualitative analysis when combined with GCMS

It is necessary to analyze the internally generated gas when conducting a deterioration evaluation of a Lithium-ion Battery. We will introduce an example of simultaneous analysis of internal gas using a system equipped with the unique BID detector. It eliminates the need for carrier gas switching and the combined use of multiple devices, which was required in the past, and enables easier and faster measurement.

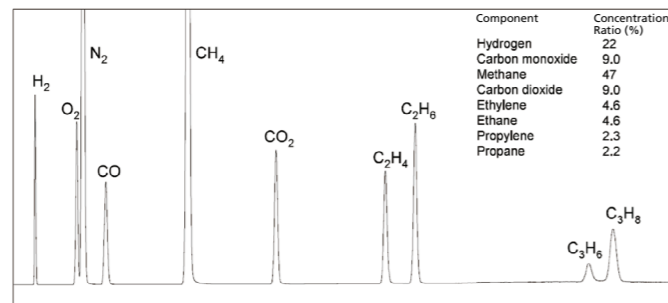
Purpose

Simultaneous analysis of internal gas

Samples

Internal gas

Data



Simultaneous analysis of internal gas

Result

The GC-BID enables simultaneous analysis of inorganic gas components (H₂, O₂, N₂, CO, CO₂) and lower hydrocarbon components (CH₄, C₂H₄, C₂H₆, C₃H₆, C₃H₈) in the battery.

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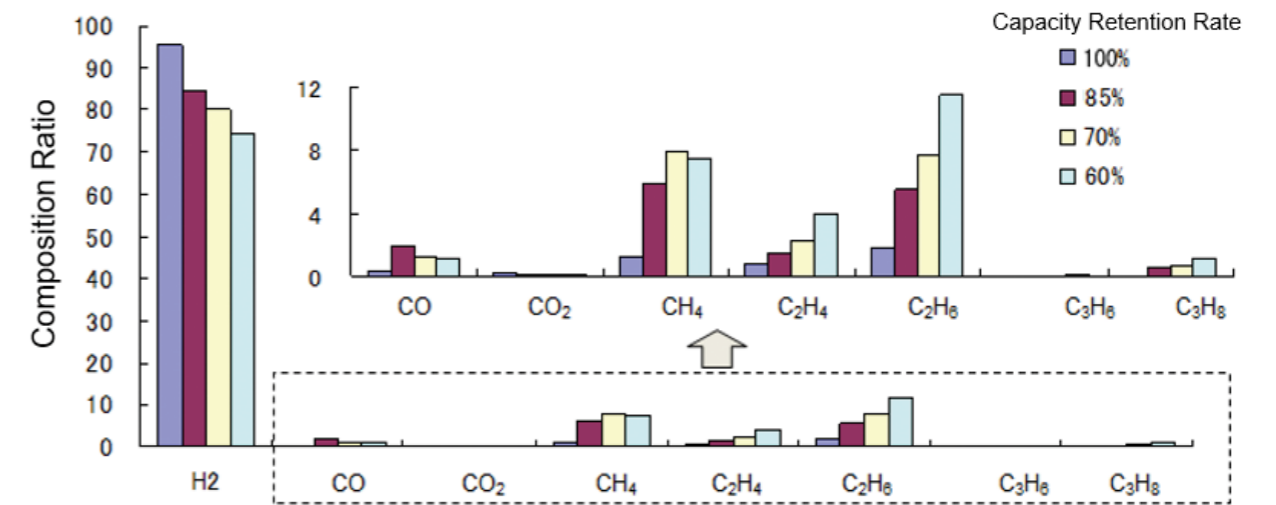
Purpose

Confirmation of changes in gas composition due to deterioration

Samples

Internal gas obtained from four batteries with different capacity retention rates

Data



Changes in the composition of internal gas due to deterioration

Result

As the capacity retention rate decreases, the ratio of hydrogen decreases and the ratio of hydrocarbon increases.

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Internal Structure Evaluation

Non-destructive observation of internal structures and deterioration evaluation after charging and discharging



Microfocus X-Ray CT System inspeXio™ SMX™-225CT series

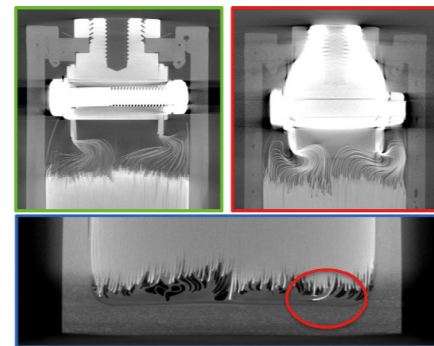
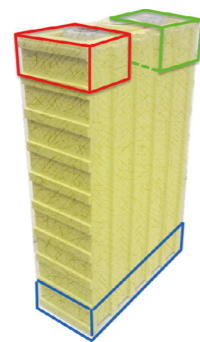
- Non-destructive internal observation is possible
- Data can be confirmed immediately after shooting by high-speed calculation
- Confirmation of detailed parts with a high-resolution detector

The X-ray CT device can observe the internal structure non-destructively. Therefore, it is used for analysis of defective products, comparison of non-defective products / defective products, comparison before and after charging / discharging, and observation of internal structures in cycle tests. In many cases, it is used for finished battery products. It is also used for observing the three-dimensional structure of minute parts such as electrodes.

Purpose

Non-destructive observation of the current collector and of the lower part of the battery

Data



The X-ray CT image of a large Lithium-ion Battery for a vehicle

Result

The green frame is the positive electrode current collector, the red frame is the negative electrode current collector, and the blue frame is the X-ray CT image of the lower part of the battery. You can see in detail how the negative electrode is bent.

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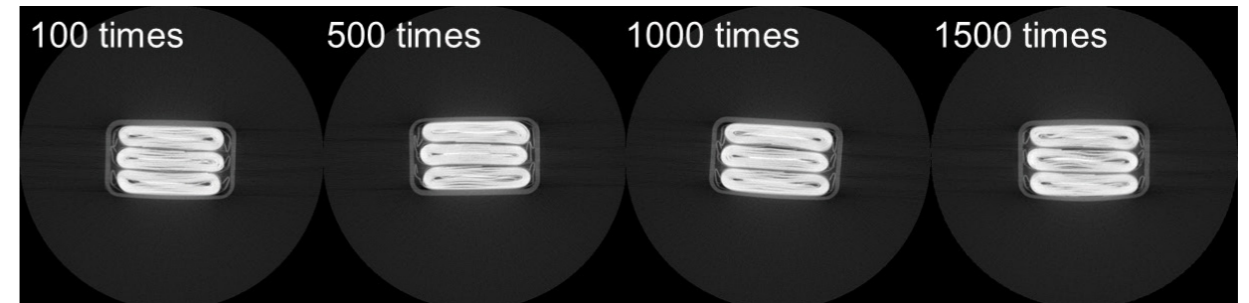
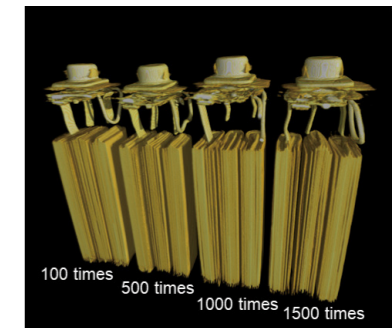
Purpose

Internal observation of cells that have been charged and discharged 100 to 1500 times

Samples

Small-capacity Lithium-ion Battery that has been charged / discharged

Data



Cross-sectional image of a Lithium-ion Battery in a charge-discharge cycle test (100 ~ 1500 times)

Result

This data shows results from repeatedly charging and discharging the Lithium-ion Battery and scanning at 100 times, 500 times, 1000 times, and 1500 times. Because X-ray CT can observe the inside non-destructively, it is not necessary to prepare a lot of samples for a cycle test. Therefore, it is possible to track the state changes over time for the same sample.

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Internal Structure Evaluation

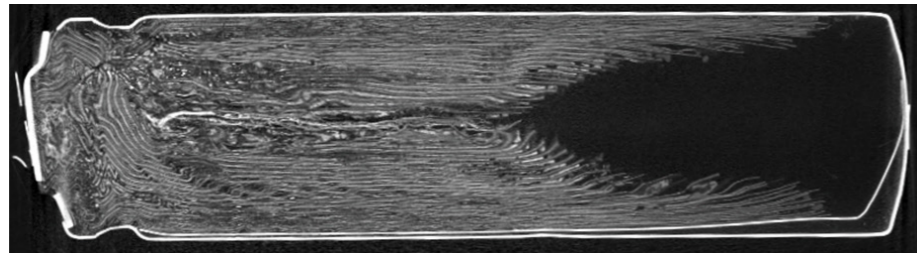
Purpose

Non-destructive observation of the internal structure of an exploded Lithium-ion Battery

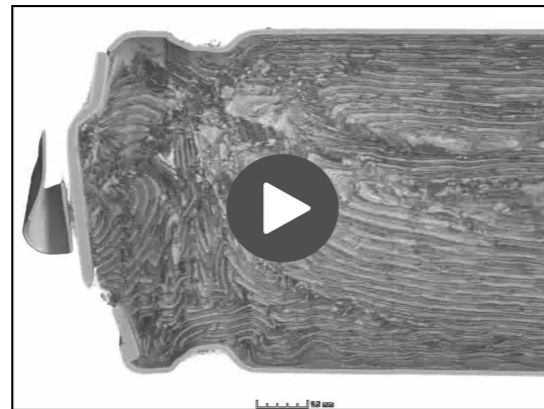
Samples

An 18650 type Lithium-ion Battery that was overcharged, heated, and exploded

Data



Cross-sectional image



3D image (movie)

Result

You can see that the inside is greatly deformed by the explosion. With X-ray CT, it is possible to observe the condition without destroying it, so it is possible to exactly confirm what kind of deformation has occurred from a load test.

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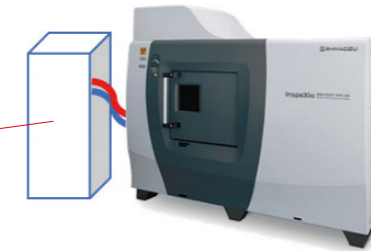
Purpose

Non-destructive observation of an internal structure before and after a charge / discharge test

Samples

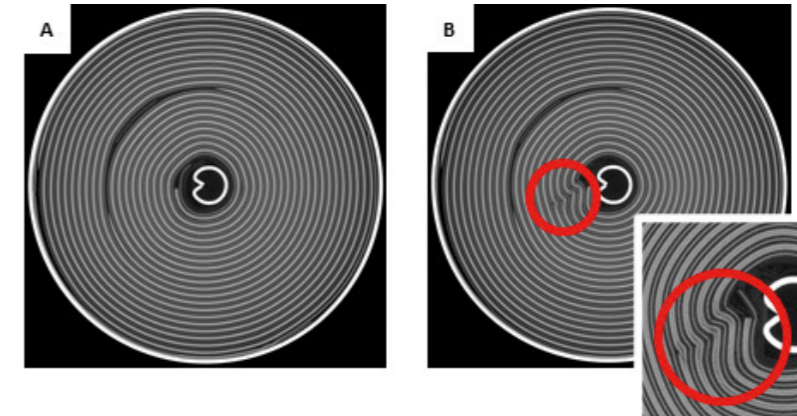
An 18650 type Lithium-ion Battery

Charge / discharge instrument



inspeXio™ SMX™-225CT FPD HR Plus with attached charge / discharge system

Data



Cross-sectional image of 18650 type Lithium-ion Battery before and after a charge / discharge test

Result

The cross-sectional image of an 18650 type Lithium-ion Battery before and after a charge / discharge test is shown. As shown in the red circles, you can see a deformation near the center of the battery due to expansion and contraction of the inside caused by charging and discharging. Even if the battery has no problem in appearance, it's possible to observe any internal deformations using X-ray CT.

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Microanalysis

Observation of minute parts of the positive electrode, elemental analysis and chemical bond state analysis



Electron Probe Microanalyzer EPMA™-8050G

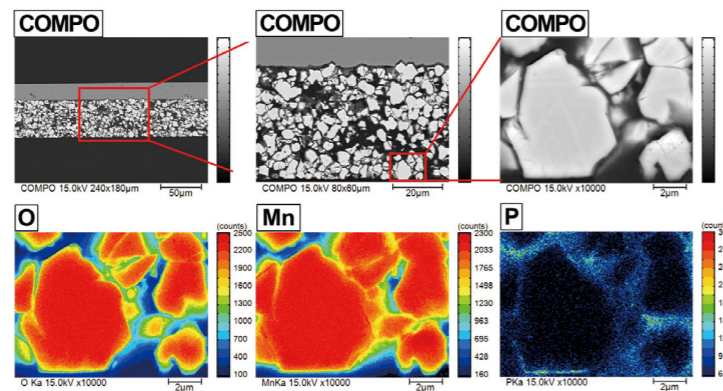
- Both high sensitivity and high spatial resolution are achieved due to excellent electron probe characteristics
- Chemical bond state analysis of minute parts is possible
- Wide area mapping by stage scan

A positive electrode has a structure in which a mixture of the active material, binder, and conductive additive are coated on a collector made of aluminum foil. Evaluation of the distribution of these components is important to improve cell performance and quality control, and when conducting failure analysis.

Purpose

Observation and elemental analysis of minute parts of a positive electrode's cross section

Data



Element mapping analysis of the cross section of a positive electrode

Samples were provided by the National Institute of Advanced Industrial Science and Technology

Result

Above data is the result of a mapping analysis of the positive electrode cross section. Not only coarse particles with sizes of several μm , but also fine particles with sizes of less than $1 \mu\text{m}$, the condition of boundaries and the element distribution can be observed.

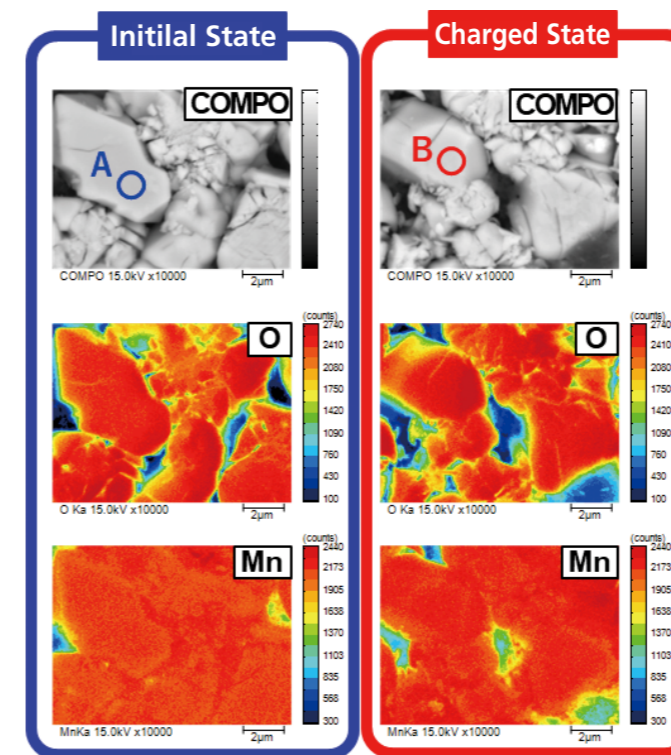
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Purpose

Evaluate the chemical bonding state of the active material of a positive electrode

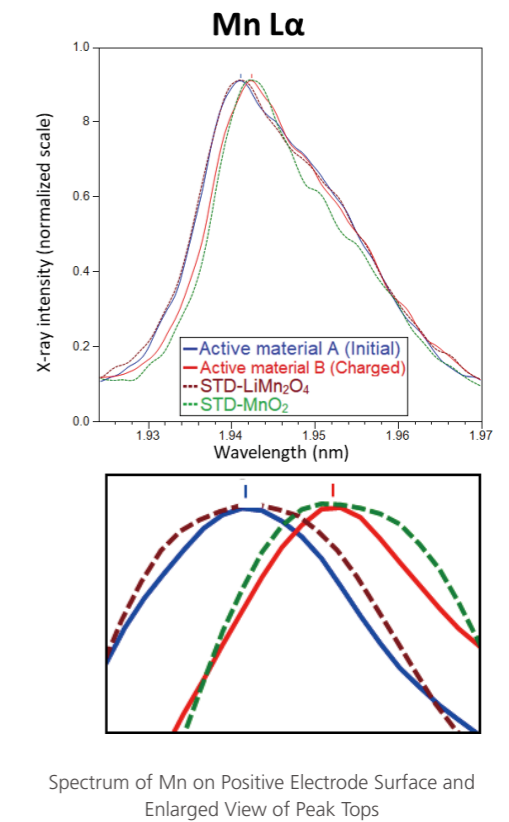
Data



State analysis of the positive electrode surface

Samples

Comparison of initial state and charged state



Spectrum of Mn on Positive Electrode Surface and Enlarged View of Peak Tops

Samples were provided by the National Institute of Advanced Industrial Science and Technology

Result

The difference shown in the spectrum is due to a peak shift between the initial state and the charged state. EPMA can detect changes in the state of chemical bonds aimed at small parts.

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Observation of the shape of minute areas of battery material and measurement of conductive distribution



Scanning Probe Microscope SPM-Nanoa™

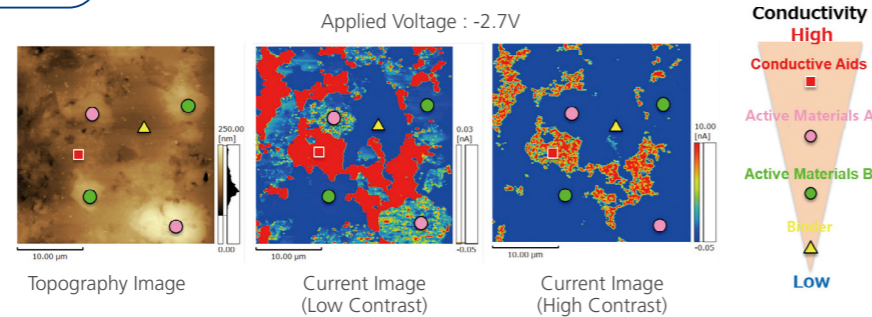
- Observation near the interface and grasp of physical properties
- Confirmation of the conductivity of minute parts
- Observation of minute parts and evaluation of physical properties are possible the air atmosphere

Positive and negative electrodes contain active materials, conductive aids, and binders. In order to improve the conductivity between the active materials, a conductive aid is added, and each is bound and held by a binder. Evaluating how these elements are distributed in the positive electrode and how conductive paths are formed helps improve the performance of Lithium-ion Batteries. We observed the surface shape and current distribution by measuring the minute amount of current flowing through the conductive cantilever while applying a voltage to the positive electrode.

Purpose

Observation of surface shape and measurement of potential distribution of minute parts of a positive electrode

Data



Result

We observed the surface shape and current distribution by measuring a minute amount of current flowing through the conductive cantilever while applying a voltage to the positive electrode. From the topography image, it is not possible to determine where conductive aids, active materials, and binders are. However, in the current image, you can see the three areas of red, yellow-green, and blue in order of conductivity. It can be estimated that the red areas are conductive additives, the yellow-green areas are active materials, and the blue areas are binders.

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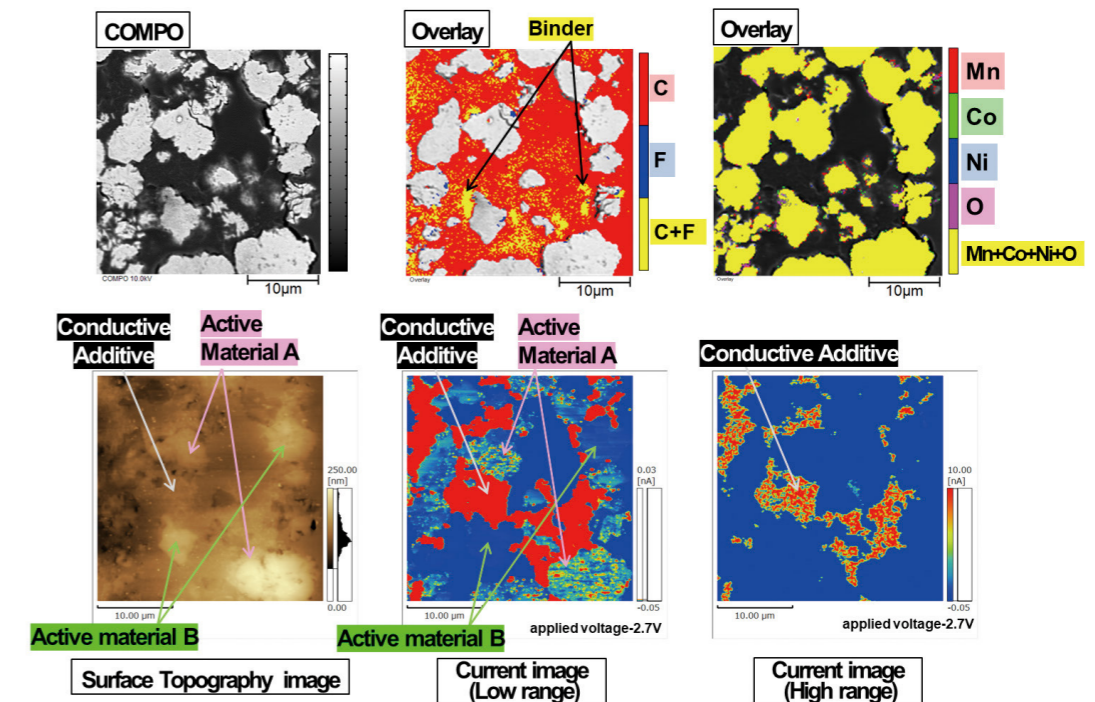
Purpose

Evaluation of element distribution and conductivity distribution in the same field of view

Samples

Positive electrode sheet of the NCM
(lithium nickel manganese cobalt oxide)

Data



Result

This data compares EPMA and SPM data within the same area. EPMA evaluates the distribution of elements whereas SPM evaluates the distribution of shape and conductivity. By analyzing the state of materials from multiple instruments, it is possible to evaluate the uneven distribution and isolated state of a battery's material components, which can help with quality improvement and product development.

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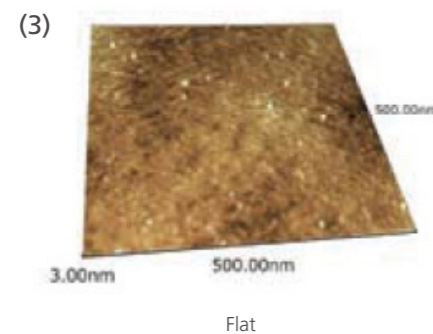
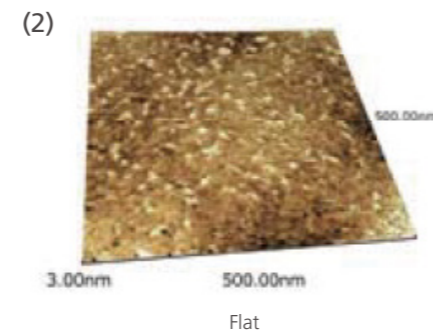
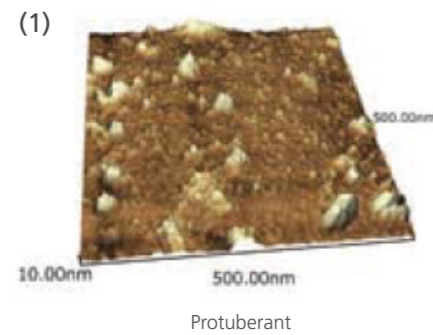
Purpose

Surface shape observation in electrolyte solution

Samples

Binder for negative electrode

Data

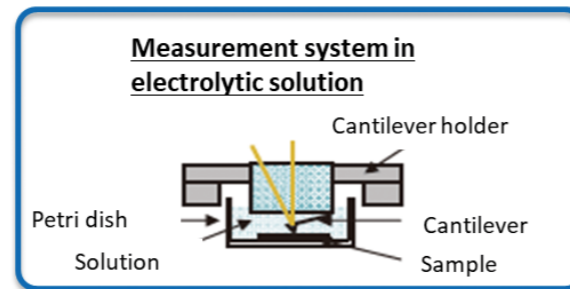


Sample Provision: Komaba Lab of Tokyo University of Science

Result

It was confirmed that the flat binders (2) and (3) were uniformly gelled in the electrolyte. SPM enables observation of an electrolyte in a real-world environment!

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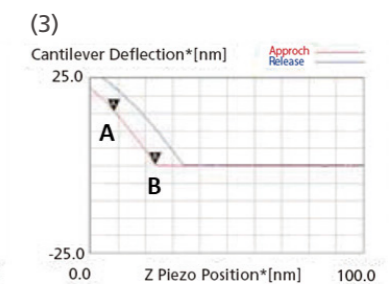
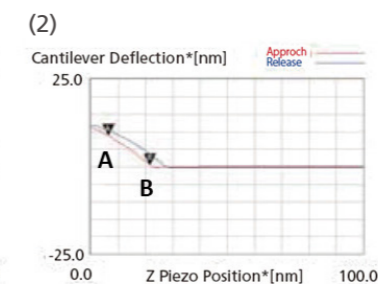
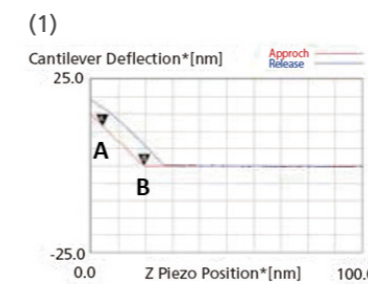
Purpose

Force curve measurement in electrolyte solution

Samples

Binder for negative electrode

Data



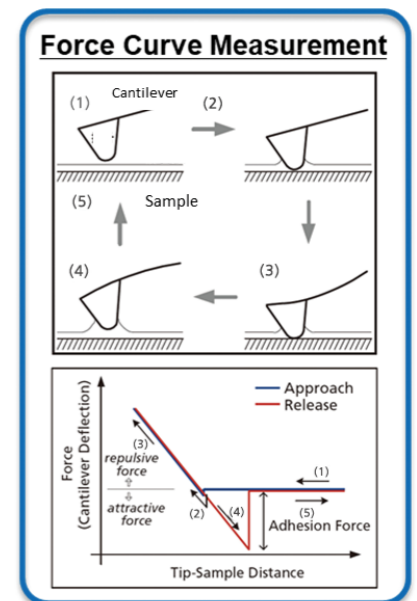
Deflection of the Cantilever [nm]	11.5	8.4	15.2
Amount of Binder Deformation[nm]	3.7	6.8 Low Rigidity	0.0 High Rigidity

Sample Provision: Komaba Lab of Tokyo University of Science

Result

The deflection of the cantilever was measured when the probe was pushed into the sample by about 15 nm, and a force curve was obtained. Point B on the force curve indicates the start of the depression, and point A indicates a 15 nm depression from point B. The amount of binder deformation can be obtained from the difference between the amount of indentation and the amount of deflection from point B to point A. From the data obtained, the amount of binder deformation indicates that (2) is the least rigid and (3) is the most rigid.

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Particle Characterization

Carbon black particle size and dispersion / aggregation evaluation



Laser Diffraction Particle Size Analyzer SALD-2300

- Black samples that easily absorb light can be detected
- Analysis while maintaining the state of slurry
- Monitoring of aggregation and dispersion status

The grain size and grain size distribution of carbon black have a great influence on an electrode's characteristics and the quality and yield of the final product. For proper evaluation, it is important to know the dispersion / aggregation state of the target sample. SALD-2300 realizes highly sensitive measurement of scattered light, and can perform highly reliable measurement even with weak scattered light from primary particles after dispersion processing.

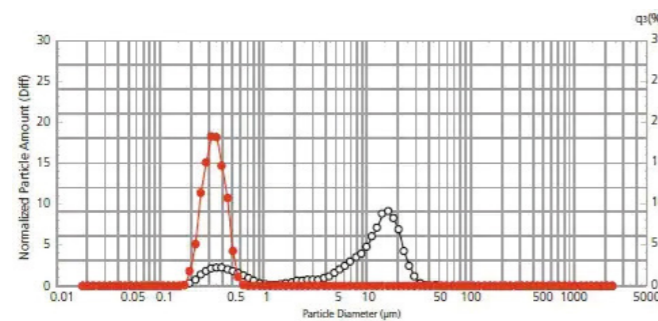
Purpose

Particle size distribution measurement of particles with different dispersed states

Samples

Carbon black particle

Data



Measurement of dispersed solvent

Result

It can be seen that by the homogenizer dispersion treatment, aggregates of about 10 µm are dispersed in the fine particles in the submicron region.

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Particle size distribution measurement and shape confirmation of active material particles



Dynamic Particle Image Analysis System iSpect™ DIA-10

- Abnormal particles (foreign matter, agglomeration) are detected
- Acquire images of individual particles and check the shape
- Detect trends and abnormal values by statistical analysis

DIA can detect trace amounts of coarse particles. By detecting coarse particles in the positive electrode material powder, it is possible to prevent deterioration of Lithium-ion Battery performance and improve safety.

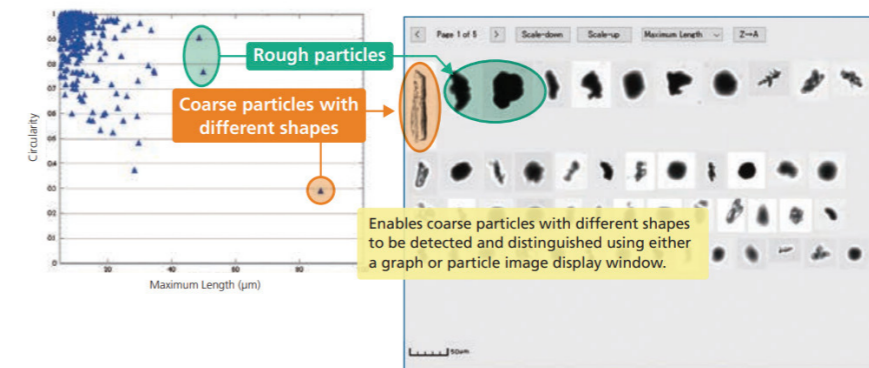
Purpose

Detection of coarse particles contained in positive electrode material

Samples

Positive electrode material powder

Data



Result

Coarse particles in powder used for a positive electrode can lead to performance issues and degradation of the material. These particles can be detected and distinguished either through the image analysis window or a plot based on specific parameters. This allows the quality of the powdered raw material to be easily verified.

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