SHIMADZU

Multilateral Evaluation of Lithium-ion Batteries and Materials

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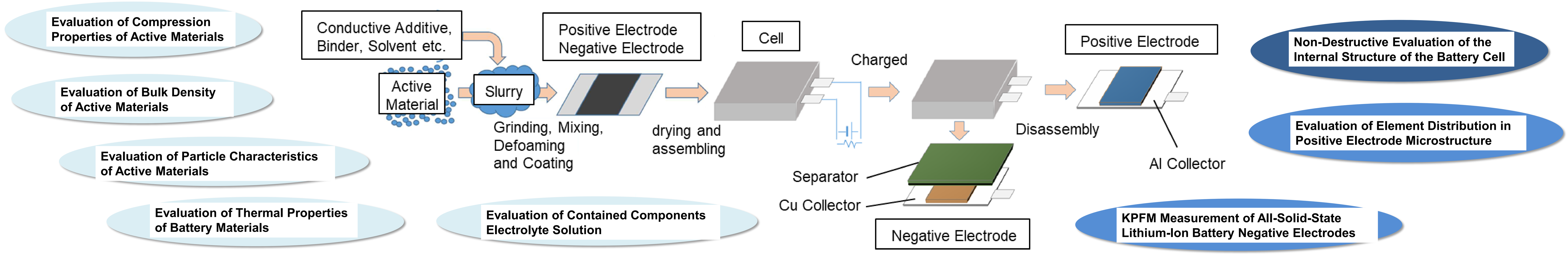
The applications for lithium-ion batteries are expanding dramatically. Increasing capacity, extending life, reducing cost, and improving the safety of lithium-ion batteries are important areas of research. The components of LiB are roughly divided into the positive electrode, negative electrode, separator, and electrolyte solution. This poster introduces the analysis technology for each manufacturing process.



Evaluation of Compression Properties of Active Materials

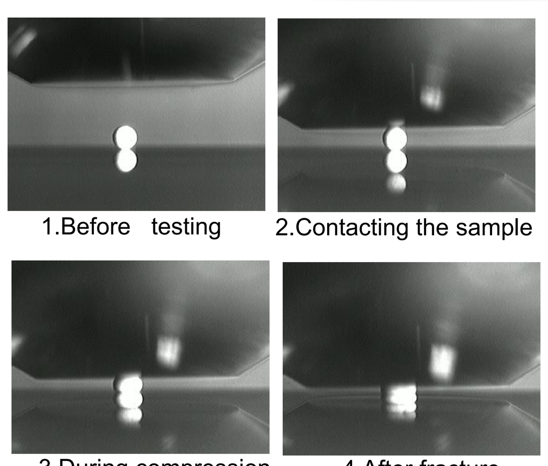


Fig. 1 Side Monitor image

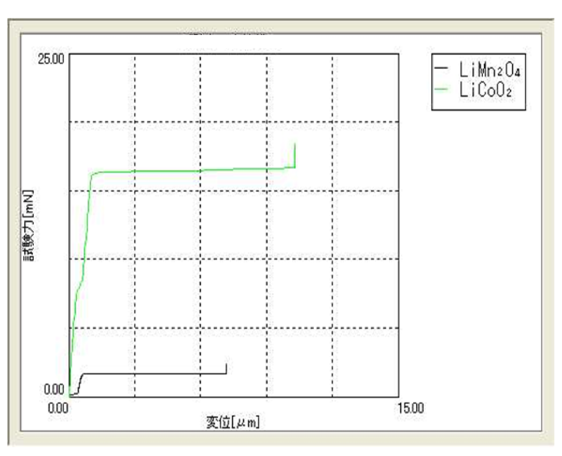


Fig. 2 Test force-displacement graph


Micro Compression Tester

MCT™ Series

Table 1 Compression Test Results

Sample Name	Fracture strength [mN]	Particle size [μm]	Strength [MPa]
$LiMn_2O_4$	1.67	13.0	7.79
$LiCoO_2$	16.23	13.3	72.75

- Evaluation of the compressive strength of a single particle
- Consideration of conditions for battery packaging and restraint pressure
- Examination of manufacturing process conditions (change in strength during heating)

Evaluation of Particle Characteristics of Active Materials

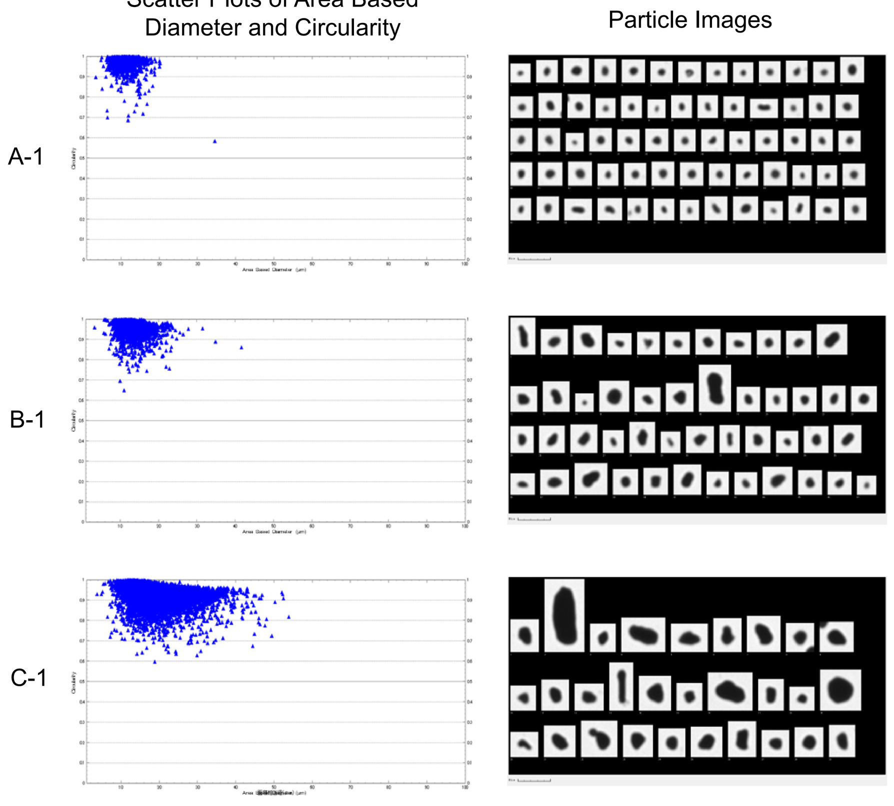


Fig. 3 Evaluation of Coarse Particles in Active Materials

We would like to express our deep gratitude to Dainen Material Co., Ltd. for providing the samples.


Dynamic Particle Image Analysis System

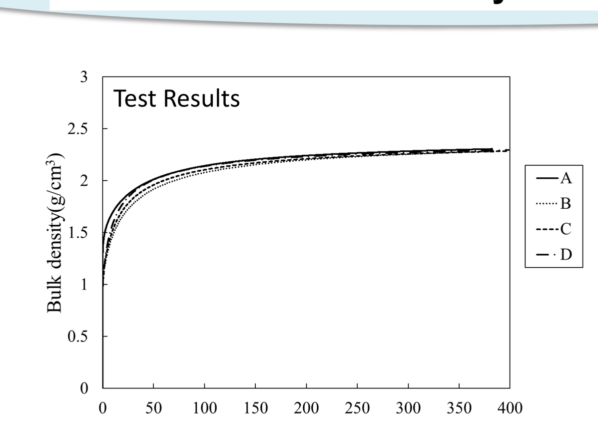
iSpect™ DIA-10

Table 2 Evaluation of Median Diameter and Counts

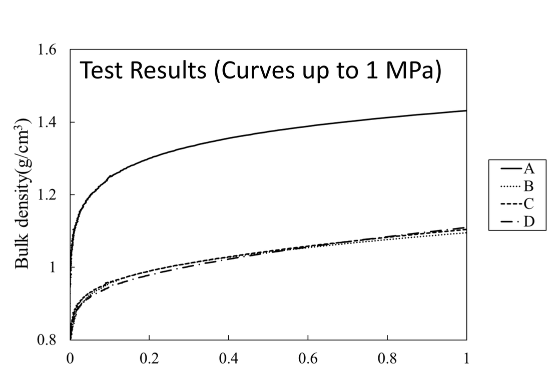
Sample	A-1	B-1	C-1
Median Diameter (μm)	10.174	16.035	22.187
Circularity (Average)	0.972	0.952	0.927
Circularity (Standard Deviation)	0.037	0.044	0.051

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

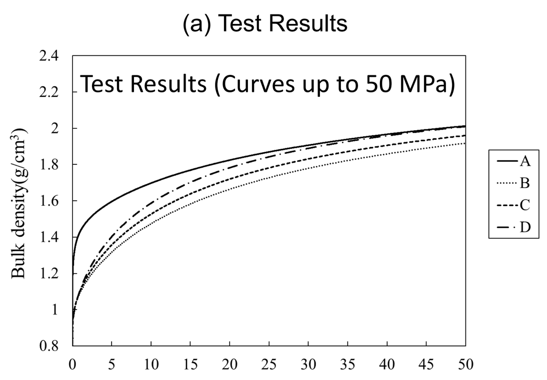
Evaluation of Bulk Density of Active Materials



(a) Test Results



(b) Test Results (Curves up to 1 MPa)



(c) Test Results (Curves up to 50 MPa)


Precision Universal Testing Machine AGX™-V2

Fig. 4 Evaluation of Bulk Density of Graphite Powder

We would like to express our deep gratitude to Dainen Material Co., Ltd., for providing the samples.

- Bulk density, an important property of battery materials for improving battery performance, can be measured.
- By using a precision universal testing machine, bulk density can be evaluated over a wide load range, from low to high.

Evaluation of Thermal Properties of Battery Materials

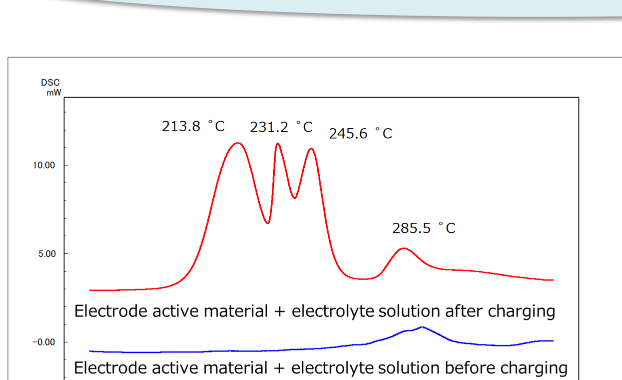


Fig. 5 DSC Measurement of Electrode Active Materials

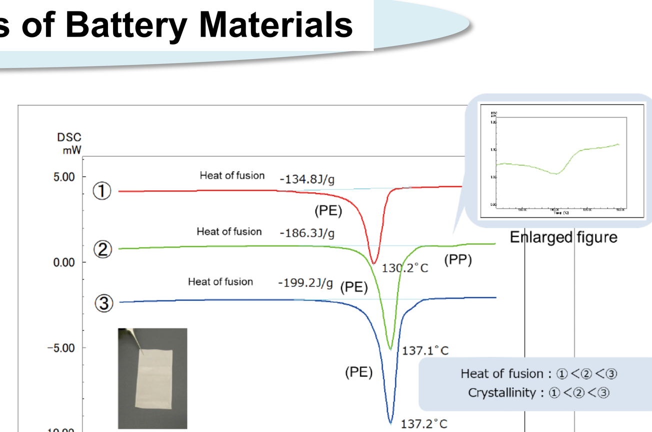


Fig. 6 DSC Measurement of Separators


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

Evaluation of Contained Components in Electrolyte Solution

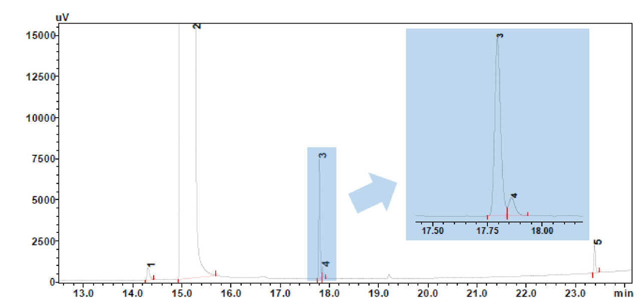


Fig. 7 Chromatogram of N-Methyl-2-Pyrrolidone (NMP) Solution for Purity Testing

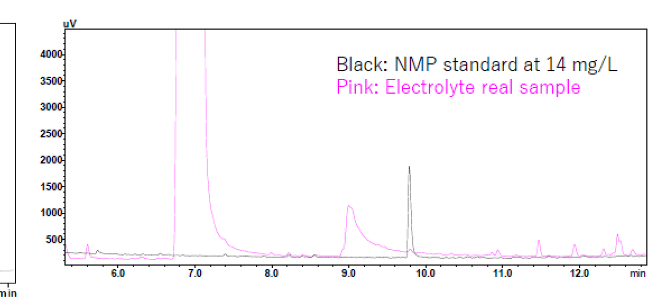


Fig. 8 Chromatogram comparison of NMP standard solution at 14 mg/L and the electrolyte real sample


Nexis™ GC-2030 gas chromatograph equipped with FID-2030 detector and HS-20 NX headspace autosampler

- Analysis of battery electrolytes and N-methyl-2-pyrrolidone (NMP) without tedious sample preparation using HS-20NX
- Automatic switching of the carrier gas for full flexibility and gas consumption control can be realized using the gas selector for Nexis GC-2030

Evaluation of Element Distribution in a Positive Electrode Microstructure

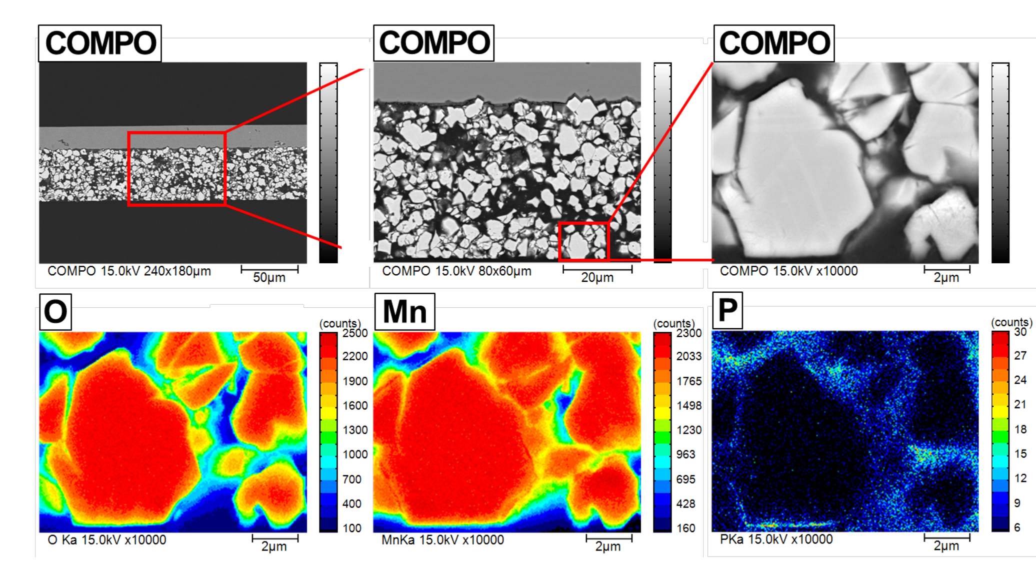


Fig. 9 Mapping Analysis of Active Material on Surface Side of Positive Electrode Cross Section

We would like to express our deep gratitude to the National Institute of Advanced Industrial Science and Technology for providing the samples.


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

KPFM Measurement of All-Solid-State Lithium-Ion Battery Negative Electrode

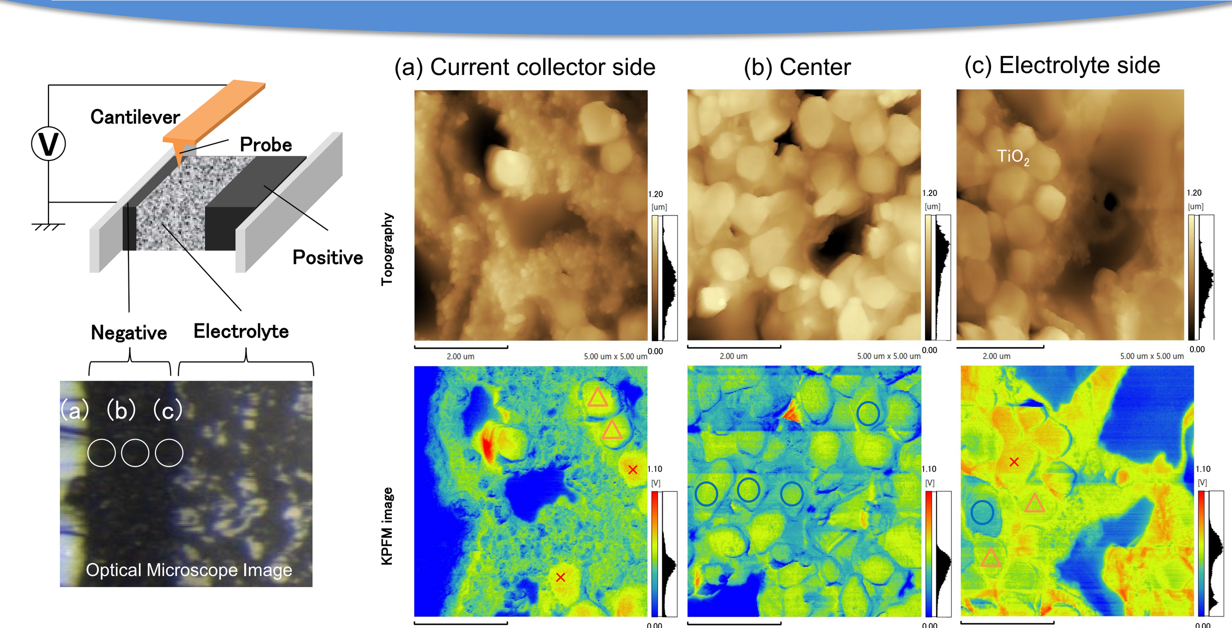


Fig. 10 All-solid-state LiB measurement site and KPFM measurement results


Scanning Probe Microscope (Atomic Force Microscope) SPM-Nanoa™

○ Charging very well
△ Charging well
× Not charging well

(a)(c) Not charging well
(b) Most particles can be charged

- Observation and measurement of the cross section of an all-solid LiB negative electrode
- Enables SPM (AFM) observation and analysis of a charged battery in an inert atmosphere without exposure to ambient air
- Topography: Distribution of active material TiO_2
- KPFM: Imaging the state of charge of TiO_2

Reference: E. Iida et al., "SPM/AFM Evaluation of Interface of All-Solid-State Lithium-Ion Batteries", IVC-22, Sapporo, Japan (September 13, 2022)

Non-Destructive Evaluation of the Internal Structure of the Battery Cell

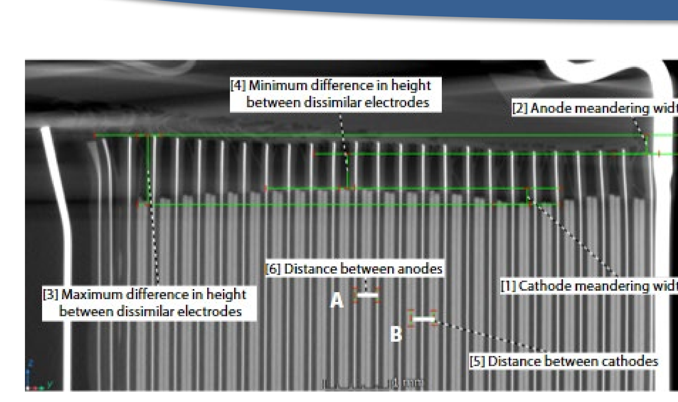


Fig.11 Result of the Distance Measurement between Electrodes

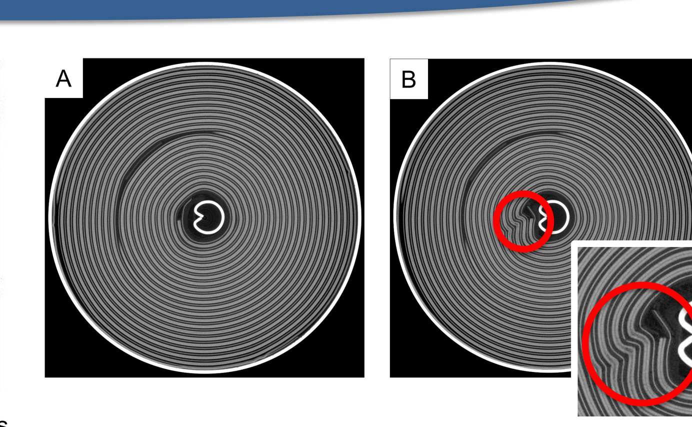


Fig.12 Cross-Sectional Image of an 18650 Type LIB Cell -A before charge/discharge -B after charge/discharge


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

Table 3 Result of the Height and Width Measurement between Electrodes

Measurement points	Result (mm)
[1] Cathode meandering width	0.248
[2] Anode meandering width	0.293
[3] Maximum difference in height between dissimilar electrodes	1.067
[4] Minimum difference in height between dissimilar electrodes	0.532
[5] Distance between cathodes	0.356
[6] Distance between anodes	0.348

- X-ray CT can observe internal structure nondestructively
- Can create cross sectional 2D and 3D images
- Can be used to evaluate LiB current failure, foreign matter electrodes, charge/discharge degradation evaluation, etc.
- Analysis time from several 10 seconds to several tens of minutes