

# Advanced analysis of LIB and Related Materials

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## 1 Abstract

To develop advanced Li-ion batteries, elucidating the influence of functional components on battery performance is crucial. However, Li-ion batteries are very difficult to analyze, because they comprise various organic-inorganic or liquid-solid materials. Accordingly, in this study, we developed a new analytical method to elucidate the two- and three dimensional nanostructure and crystalline distribution as well as a method to visualize the quantified dispersion state of the ingredient for Li-ion batteries.

## 2 Key Features of Analytical Techniques

- New advanced analytical technique applicable to an analytical area.
- Clarification of unidentified performance characteristics and the beneficial effect of lithium-ion batteries and battery related materials.

As specific successful case examples:

- 1) 3D image-surface observation: Visualization of coating formation, acicular structure and fibrous (mesh) structure in nanoscale.
- 2) Visualization of images starting from the spatial distribution of functional groups to the distribution of binders inside the electrode.
- 3) In-situ observation of the microstructure inside a battery used for analytical study during charge/discharge cycles using a high-resolution X-ray CT apparatus.

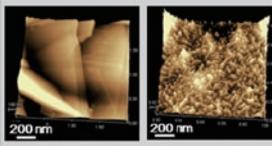
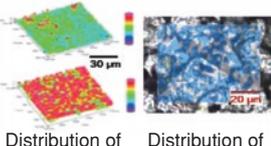
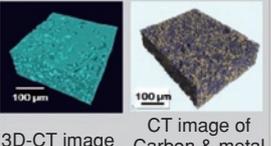
Field	Nano level / 2D & 3D	Micro level / 2D	Micro level / 3D
Target	Material, Surface	Component, Electrode	Device, Structure
Equipment	 Hybrid SPM	 Raman Microscope	 High definition X-ray CT
Point of View	3D-observation of LIB electrodes in nano scale	Mapping of crystalline state and chemical state in sub-micro scale	In-situ, 3D-observation of LIB electrodes in sub-micro scale
Output	 Height image of anode material for LIB	 Distribution of crystal on anode surface Distribution of binder polymer on electrode	 3D-CT image of voids in electrode CT image of Carbon & metal composite anode

Figure 1 New analytical technologies suitable for various fields

## 3 Development Background

LIB needs a breakthrough to boost energy density, service life and safety while reducing costs, hence the need to develop new technology, including new material. As well as overcoming current technological challenges, various development works are ongoing to realize characteristics tailored for specific applications. However, it is difficult to analyze LIB performance by traditional analytical methods alone because the analytical target is an organic/inorganic and solid/liquid composite body, the main components of which are complex active material particles with a textured surface, and which also include many unidentified areas. Accordingly, we tried a new analytical approach to clarify advanced performance characteristics in this field.

## 4 Technical Content

During this research, we newly developed complex analytical methods allowing the 2D- and 3D-nanostructure of inorganic/organic and solid/liquid composite bodies, crystalline distribution and clarification of the dispersed state of specific components (visualization, quantification) to be clarified as follows:

- ① 3D image-surface observation: Visualization of the coating formation, acicular structure and fibrous (mesh) structure in nanoscale.
- ② Visualization of images starting from the spatial distribution of functional groups to the distribution of binders inside electrodes.
- ③ In-situ observation of microstructures inside batteries used for analytical study during charge/discharge cycles and using a high-resolution X-ray CT apparatus. Leveraging these new analytical approaches, the manifestation mechanism of the characteristic features of LIB and battery materials was clarified as follows:

1) By visualizing nanoscale morphological differences, we determined essential key factors to improve battery characteristics. Composition of active material, difference in 3D surface morphology, namely, difference in 3D image of smooth structure, acicular structure and fibrous (mesh) structure, were elucidated using scanning probe microscope. This approach could make capturing of 3D morphological image over large area easier while conventional cross-section image by Transmission Electron Microscope (TEM) was difficult to create the same; thereby, capturing nanoscale surface morphology of active material, which is high impact factor for battery performance, was made easier.

2) The difference in the dispersed state of binders caused by differences in the composition of active materials was confirmed by microscopic images, and it was also clarified that the area occupancy rate of the binder was one of factors behind lower resistivity. Figure 2 shows the correlation between the dispersed state inside the electrode and the AC resistance for each binder type. For each binder type, we confirmed that the lower the area occupancy rate, the lower the AC resistance of the electrode. The dispersion state of the binder as an electrical insulating material should occupy the minimum area in the electrode to reduce resistance. By utilizing dispersion state assessment techniques inside the electrode, optimization between the composition of binder/additive and the dispersed state can be successfully implemented and thus help boost electrode performance.

3) Swelling inside the battery during the charging cycle was confirmed by in-situ observation of its internal microstructure during the charging/discharging cycle. Figure 3 shows analytical images of the electrode swelling/contraction inside the battery, as identified by non-destructive X-ray CT scanning. The negative electrode layer swelled by 12 to 14  $\mu\text{m}$  after charging and approximately in line with the theoretical expansion coefficient of graphite. From now, higher durability and lower swelling of electrodes should be essential to extend LIB service life. We expect that the non-destructive visualization measurement technique will play a greater role in microscale observation inside LIB during the charging/discharging cycle.

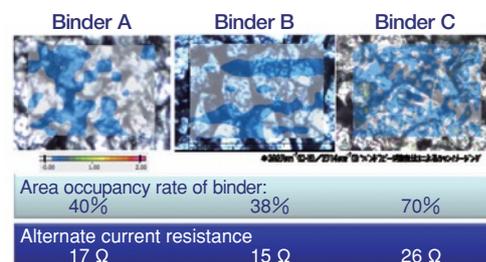


Figure 2 Relationship between distribution in electrode and AC resistance based on the difference in binder type

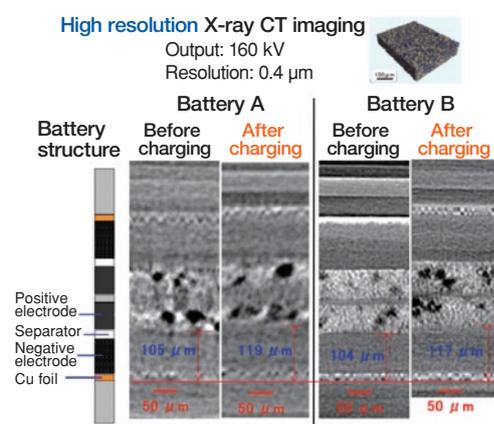


Figure 3 Expansion / constrictive analysis of the battery electrode by X-ray CT

## 5 Future Business Development

- Technical developments in the field of various battery energy-storage device types and their related materials by applying this analytical approach
- Detailed elucidation of the characteristics and manifestation mechanism of inorganic/organic and solid/liquid composite materials in infocommunication and life science fields

### 【References】

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- 2) 2010, Fact-Finding General Survey of Battery & its Related Market (second volume), Fuji Keizai (2009)