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Application News

No. P117

EPMA-8050G Electron Probe Microanalyzer
SPM-9700HT Scanning Probe Microscope / Atomic Force Microscope

Analysis of Positive Electrode (NCM) for Lithium Ion Battery by EPMA and SPM

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User Benefits

- Evaluation of uneven distribution and isolation of components of the positive electrode of lithium ion batteries is useful in quality improvement and product development.
- Multifaceted material analysis is possible by an integrated analysis by collaboration of EPMA and SPM.

■ Introduction

A lithium ion battery (hereinafter, LIB) is a rechargeable storage battery which is charged/discharged by desorption/insertion of Li^+ from the structure of the active material. In recent years, the applications of LIBs have increased exponentially, spurring active research aimed at increasing battery capacity and life, reducing costs, and improving safety. In those efforts, evaluation of the distribution states of the active material, binder, and conductive additive which are the principal components of the LIB positive electrode is important for improving performance and quality control.

Here, the distribution of elements in a LIB positive electrode was measured using an EPMA™ electron probe microanalyzer (EPMA-8050G), and the conductivity of the electrode was evaluated by comparing distribution images of the same fields acquired with the EPMA and an SPM scanning probe microscope (SPM-9700HT™).

■ Element Mapping of LIB Positive Electrode Cross Section

In this experiment, we analyzed the cross section of a positive electrode sheet of the NCM (lithium nickel manganese cobalt oxide; $(Li(Ni\text{-}Co\text{-}Mn)O_2$), which has become the main stream in positive electrode materials for LIBs in recent years. Fig. 1 is the result of a wide-area mapping analysis. As constituents of the active material, O, Mn, Co, and Ni show substantially the same distribution. It can be inferred that C shows the distribution of the binder and the conductive additive, and F indicates a component of the binder.

Fig. 2 is the result of a high magnification mapping analysis focusing on the active material in the same sample. When the cross section of the active material was enlarged, it was found that phases with different intensity ratios can be seen, especially from the X-ray images of Mn and Co. Moreover, around the active material, C and F, which are components of the conductive additive and binder, are distributed so as to enclose the active material, and the position with the particularly high F intensity is estimated to be a binder component.

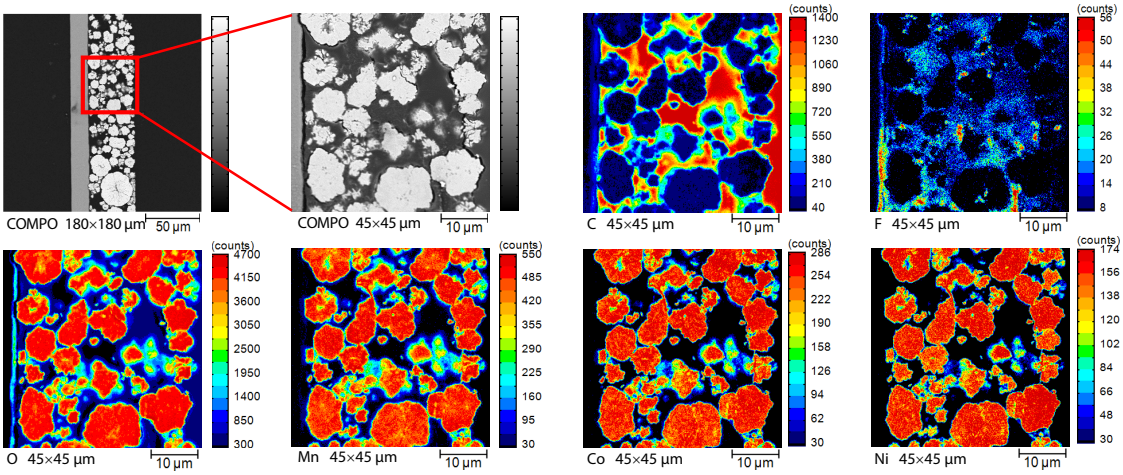

Fig. 1 Wide-Area Mapping Analysis of Cross Section of NCM Positive Electrode

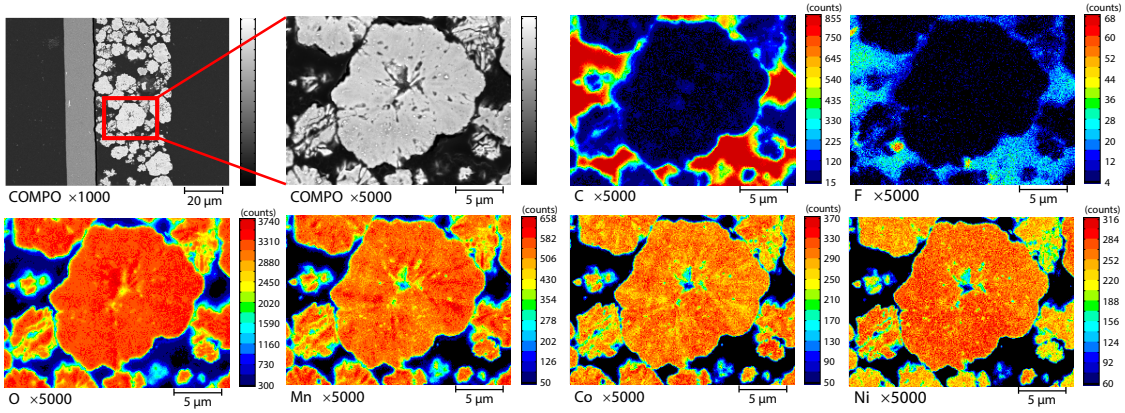

Fig. 2 Enlarged Mapping Analysis of Cross Section of NCM Positive Electrode

■ Distribution Images of Same View by EPMA and SPM

The results of an analysis of the same field of view by EPMA and SPM in the mapping range in Fig. 1 were compared. Fig. 3 to Fig. 5 show the EPMA data and Fig. 6 to Fig. 8 show the SPM data. Among the EPMA results, Fig. 3 is the composition image (COMPO), Fig. 4 is an overlay image of the mapping analysis of C and F, and Fig. 5 is an overlay image of the mapping analysis of Mn, Co, Ni, and O. The distribution of the elements can be understood from the EPMA results. It can be estimated that overlaid positions of C and F in Fig. 4 are the binder, other positions with high levels of C are the conductive additive, and the overlaid positions of Mn, Co, Ni, and O in Fig. 5 are the active material. In the SPM results, Fig. 6 is a surface topography image acquired with the SPM in the current mode, Fig. 7 is a low range current image, and Fig. 8 is a high range current image. Together with the topography of the extreme surface, the distribution of conductivity can also be understood by SPM by measuring the current that passes between the probe and the sample. For example, it can be inferred that the conductive additive is distributed at the position where a large current value was detected in Fig. 8.

Next, focusing on the active material, Fig. 7 shows that the current value of active material B is smaller than that of active material A. Furthermore, in the area around the active materials, the results revealed a tendency in which a larger number of areas with large current values exist around active material A, while comparatively few areas with large current values exist around active material B.

Based on these data, the distribution of the elements in the depth direction of the LIB positive electrode will be discussed using the schematic image of the analysis plane in Fig. 9. Generally, the conductive additive and active material are conductive, and the binder is not conductive. For example, in the case of the active material shown in pink in Fig. 9, the conductive path is connected with the aluminum collector by way of the conductive additive and other active materials, and a current can flow.

However, the active material shown in yellowish-green is surrounded by the nonconductive binder and is electrically isolated by voids caused by cracking, preventing a current flow. It can be estimated that results reflecting this internal state are obtained.

■ Conclusion

The distribution of the components of the active material, conductive additive, and binder of a LIB can be evaluated based on measurements of the elemental distribution by an EPMA element mapping analysis, and the surface state and distribution of conductivity can be understood by an SPM analysis. Moreover, a multifaceted analysis of the state of materials is possible by measuring the same field of view with multiple types of instruments. This approach can be utilized in quality improvement and product development by evaluating the uneven distribution and isolation state of the components of LIB materials.

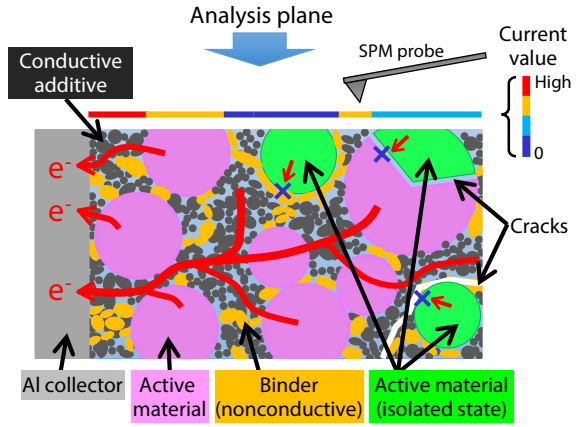

Fig. 9 Image of Component Distribution in LIB Positive Electrode

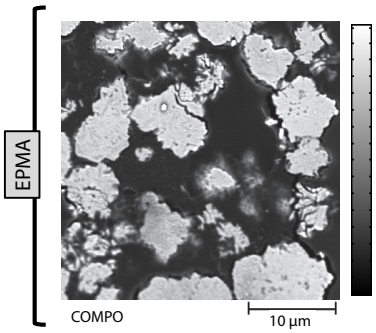

Fig. 3 Composition Image of Cross Section of NCM Positive Electrode

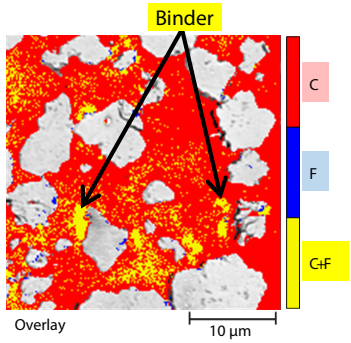

Fig. 4 Overlay Image of Cross Section of NCM Positive Electrode (C, F)

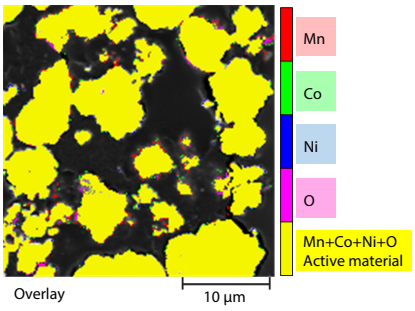

Fig. 5 Overlay Image of Cross Section of NCM Positive Electrode (Mn, Co, Ni, O)

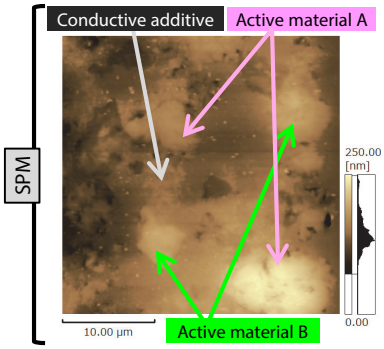

Fig. 6 Surface Topography Image by SPM

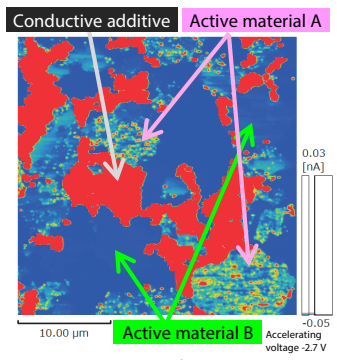

Fig. 7 Current Image by SPM (Low Range)

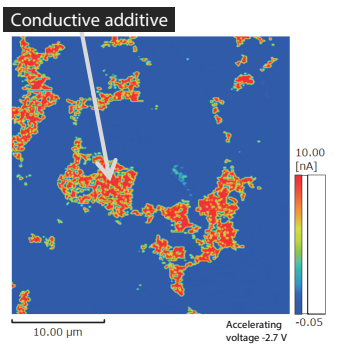

Fig. 8 Current Image by SPM (High Range)

First Edition: Feb. 2021

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