

## Application News

# No. S44

### Surface Observation

## SPM Observation of Rechargeable Lithium-Ion Battery Separator

### ■ Introduction

Rechargeable lithium-ion batteries are a type of storage battery in which a lithium transition metal oxide compound, represented by lithium cobalt oxide, is used in the positive electrode, graphite, black lead, or other carbon material is used in the negative electrode, and an organic solvent is used in the electrolyte. Taking advantage of their high energy density and cell voltage, rechargeable lithium-ion batteries are widely used as rechargeable batteries for small consumer electronic products such as smartphones and other IT devices. In recent years, development to plugin hybrid vehicles and electric vehicle applications has also been remarkable, and even higher output and performance are expected.

On the other hand, because rechargeable lithium-ion batteries are unstable due to short circuit, overcharging/overdischarging and impact, various measures have been taken to enhance safety. The separator is one critical part that contributes to safety. Separators play the roles of preventing short circuit between the positive and negative electrodes, while simultaneously allowing smooth passage of lithium ions. However, when a battery short circuit or other problem causes heat generation, they also have the function of breaking the electrical current. Micro porous polyolefin films are used as general separator materials. When a high temperature condition occurs, these films have a shutdown function that prevents passage of ions by closing the pore structures at temperatures near the melting point of the material.

This article introduces an example of observation of separators with a scanning probe microscope (SPM).

E. Iida, S. Moriguchi, C. Kawai

### ■ Instrument Used in Observation

The SPM is a microscope which observes at high magnifications the 3-dimensional shape and local physical properties of samples by scanning the sample surface with a microscopic probe (cantilever). It can also support observation under controlled atmospheres and vacuum environments by using an environment controlled chamber. The appearance of the SPM and an environment controlled chamber are shown in Fig. 1 and Fig. 2, respectively.



Fig. 1 SPM-9700HT™ Scanning Probe Microscope



Fig. 2 Environment Controlled Chamber

### ■ Separators

The samples observed in this experiment were three types of separators (separators A to C) taken from commercially-available rechargeable lithium-ion batteries for small electronic products. The main material is polyolefin.

General rechargeable lithium-ion batteries have a structure in which the positive and negative electrodes are immersed in the electrolyte in a coiled condition while separated by the separators, as illustrated in Fig. 3.

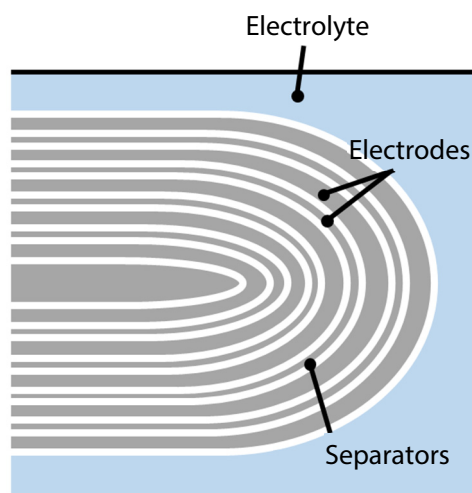


Fig. 3 Schematic Diagram of Internal Structure of Rechargeable Lithium-Ion Battery

### ■ Surface Shape Observation of Separators

Fig. 4 shows images of the surface shapes of separators A to C. The field of view is  $5\ \mu\text{m} \times 5\ \mu\text{m}$ , and the vertical direction in each image corresponds to the longitudinal direction of the separator. The porous structure which is a distinctive feature of separators can be seen. The pore diameter of A is large in comparison with those of B and C, and the pore shape is also different in B and C.

### ■ Observation of Pore Contraction Under Heating

Separator A was heated from room temperature to  $125\ ^\circ\text{C}$  and  $140\ ^\circ\text{C}$  using a sample heating unit, and its surface shape was observed at each temperature. Fig. 5 shows images of the surface shape. The field of view is  $5\ \mu\text{m} \times 5\ \mu\text{m}$ , and the vertical direction in each image corresponds to the longitudinal direction of the separator. As the temperature rises, gradual contraction of the pores due to the effect of melting of the separator can be seen. In this experiment, observation was conducted under a vacuum environment using an environment controlled chamber to suppress the effects of adsorption of oxygen and water onto the separator and gas discharge from the separator due to heating.

### ■ Conclusion

The surface shapes of separators of rechargeable lithium-ion batteries were observed and evaluated by SPM. The changes in the pore shape under a heating environment were successfully captured by sample heating. This experiment demonstrated that evaluation of battery materials in a condition close to the real environment can be realized by using an SPM that enables environmental control including heating.

SPM-9700HT is a trademark of Shimadzu Corporation in Japan and/or other countries.

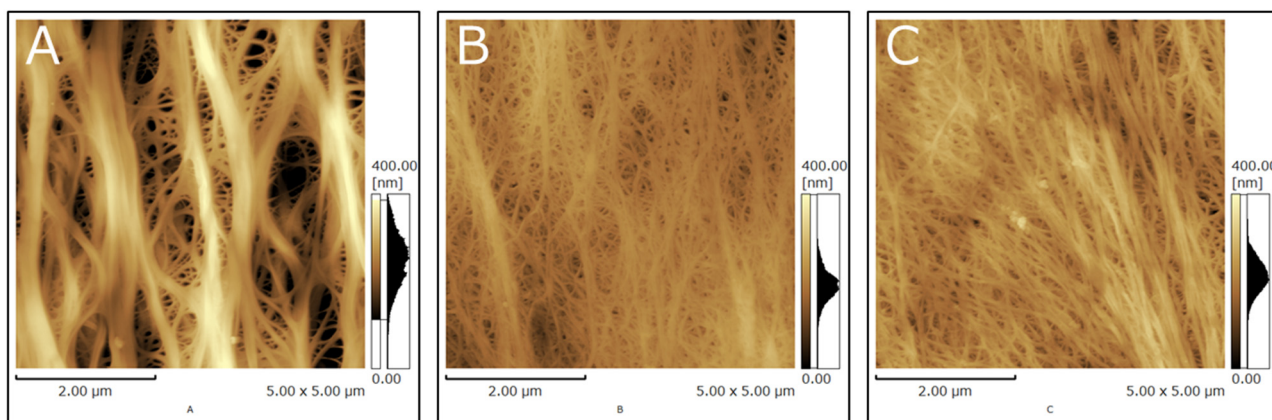


Fig. 4 Images of Surface Shapes of 3 Types of Separators

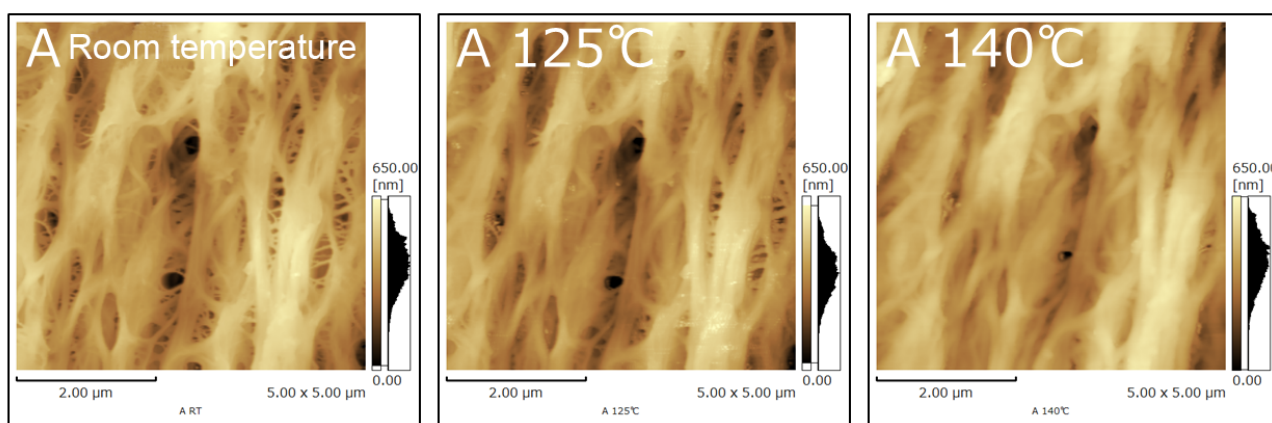


Fig. 5 Images of Surface Shape of Separator A Under Heating Environment