## Three-dimensional Observation Techniques by Scanning Electron Microscopy

## Hiroaki Kadoura, Surface and Micro Analysis Lab.

For the enhancement of the functions of materials, control of the fine structure of the material is necessary. At the same time, an analytical method of clarifying the fine structure is necessary for the fine structure control.

Topics

Combining the oxygen plasma treatment that can selectively remove organic substances and scanning electron microscope (SEM) observation, we have established new techniques for threedimensional observation of the structure of organic/inorganic composite materials. Application examples and observation results are given below.

(1) In a film of a nylon-6/clay hybrid (NCH) which is created by polymerizing e-caprolactam in the intercalation of clay (montmorillonites), the clay is dispersed in the molecular state as found by cross-sectional observation using transmission electron microscopy (TEM). The change in the NCH surface as a result of the oxygen plasma treatment is shown in the SEM photographs (**Fig. 1**).

In the cross section of the film cut by a microtome before the treatment, it is impossible to distinguish nylon 6 from the clay (Fig. 1(a)). After the oxygen plasma etching for 30 seconds, nylon 6 is slightly removed from the surface and the clay appears, clarifying three-dimensionally the dispersion and orientation states of the clay (Fig. 1(b)). The shape of the clay becomes clearer after the etching for 60 seconds (Fig. 1(c)). The part in which the surface looks like butterfly wings corresponds to a single layer of the clay.

(2) In a coating film with pigment, dispersion of the pigment which influences the luster has been clarified by selectively removing the resin near the surface.

(3) The distribution of the binder in the negative electrode of a lithium ion secondary battery has been clarified by selectively removing the binder.

This observation technique is applicable also to morphology observation of polymer materials because oxygen plasma has different etching rates according to the type of organic substance.



(a) NCH surface before oxygen plasma treatment.



(b) NCH surface after 30 second oxygen plasma treatment.



(c) NCH surface after 60 second oxygen plasma treatment.

200nm

Fig. 1 SEM image of nylon-6/clay hybrid (NCH) film. The clay content in the NCH is 1.85 wt.%. The cross section of film was treated with oxygen plasma. The specimen had an inclination of 45 degrees to the horizontal plane.