Study in Ultra-Small Angle X-ray Scattering for Fiber Structure

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Ultrahigh molecular weight polyethylene fiber (UHMWPE fiber) having an excellent tensile strength higher than 3 GPa is very useful for industrial usages. It is crucial to elucidate the relation between the tensile property of the fiber and its inner structure to improve the tensile property. However, it is still not clarified. To elucidate the detailed structure of the fiber, we carried out ultra-small angle X-ray scattering at BL19B2. In the previous experiment at 2004A, we showed that the available q range of the ultra-small angle X-ray scattering in BL19B2 is 0.005 $nm^{-1} < q < 0.15$ nm^{-1}. Here amplitude of scattering vector **q** is defined as $q=(4\pi/\lambda)\sin(\theta/2)$, being λ of wave length and θ of scattering angle.

UHMWPE fiber contains characteristic fibrous structure, so-called "shish-kebab", aligning parallel to the fiber axis. The structure is thought to be a key structure for improving the fiber property. However, there is a problem should be solved to elucidate this structure by the ultra-small angle scattering. Very intense reflection of X-ray generates from the surface of the fiber and this reflection superposes the scattering from the shish-kebab structure. To solve the problem, the fiber was immersed in a liquid having the same electron density with the fiber. We found that the electron density of the 40/60 mixture of hexane and paraffin chloride exhibits good match with that of the fiber.

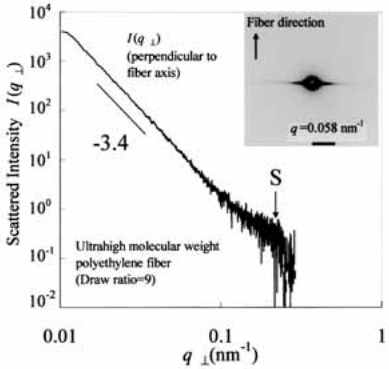

Figure 1. ultra-small angle X-ray scattering pattern of ultrahigh molecular weight polyethylene fiber and scattered intensity perpendicular to fiber direction.

Figure 1 shows a scattering pattern of an ultrahigh molecular weight polyethylene fiber and scattered intensity perpendicular to the fiber axis as a function of q. A streak like scattering appears perpendicular to the fiber axis. The scattered intensity perpendicular to the fiber axis monotonically decays from q=0.01 nm^{-1} to 0.1 nm^{-1} and the power of the profile is 3.4. It should be noted that a shoulder is observed at higher q than 0.1 nm^{-1} indicated by an arrow marked with "S". We are going to analyze this scattering profile according to a scattering theory based on cylindrical objects.

Observation of the electrodes in the laminated cell by refraction contrast imaging method

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Recently, a number of studies for the lithium ion batteries have been conducted in order to improve energy density and reliability of batteries, which are used for mobile devices. For above-mentioned purpose, it is important that morphological change caused by electrochemical reaction on the electrode surface in the assembled batteries can be observed in process. Refraction contrast method is applied to this observation because it is able to distinguish minute change that shows small difference of absorption or density. In this study, we prepared two kinds of electrodes: the electrode without morphological change (sample A) and the electrode that has morphological change on it (sample B), put them into Al laminated cell and detected the difference between them.

We made use of zooming tube, beam monitor and CCD camera to detect X-ray images and the experiment has been conducted with energy 15keV at which the optimum refraction contrast images of electrode inside can be obtained most clearly. Each sample was in laminated cell to prevent it from degrading, and was attached in the sample stand.

Figure 1(a) shows the refraction contrast image of sample A. About 20-micron dark images and striped pattern are seen. The former are from Al lamination and the latter originates in the rolled mark of copper foil. No other structure was detected in this electrode. Figure 1(b) shows the refraction contrast image of sample B. Several contrast structures appear, which have relation with the change of either thickness or component of electrode through the Al laminated cell and the Cu collector by use of highly bright and parallel SPring-8 X-ray. In-situ refraction contrast observation will become possible by designing and assembling the battery that consists of low attenuation materials.

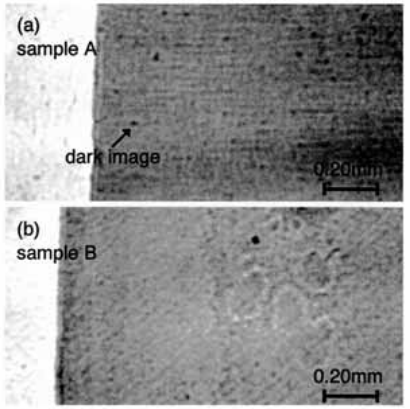

Figure 1. Refraction contrast images of :
(a) electrode without morphological change (sample A)
(b) electrode with morphological change (sample B)