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3-Dimentional Observation of Lamellar Structure of a High Density Polyethylene Using Transmission Electron Micro-tomography

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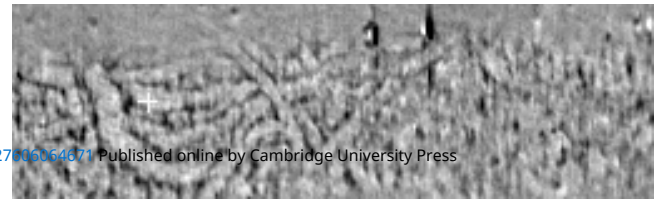
The morphology of crystallized lamellar structures has been considered by many researchers, large number of the optical and electron microscopy studies have been carried out. In case of observation, sample preparation techniques of polymer materials are very important in order to highlight or reveal certain parts of the total solid sample. Sano had established the treatment of the specimen with a heavy metal containing compound, such as ruthenium peroxides, RuO4 [1]. Direct observation of grown lamellar structure to b-axis direction in the spherulite of a high-density polyethylene (HDPE) was successful by his work [1]. However lamellar structure those who are visible in TEM images limit to situated in the edge-on of a specimen, although many lamellar structure are in existence potentially. By the way, transmission electron micro-tomography (TEMT) is interesting method to understand 3-dimentional morphology in many materials science field. In this study, in order to make progress in knowledge of the morphology of crystallized lamellar structure, TEMT of a melt crystallized HDPE was carried out.

The ultra thin section for TEMT was prepared using ultra microtome method after staining by RuO4 at 323 K. Gold particles of 10 nm diameter were deposited from aqueous specimen. A TECNAI G2 F20 TEM (FEI Company) operated at 200 kV was used. Bright field STEM observation was carried out. A projection was taken over the tilt angle from -60 degrees to 60 degrees with 1 degree increment. After the alignment of tilt series, 3D reconstruction was computed using the IMOD image pressing and modeling software [2].

Fig.1(a) and (b) show bright field STEM images at the tilt angle 0 degree and -40 degrees. Each image was obtained at the same area (arrows indicating the same gold particles as a marker). In Fig.1 (a) and (b), lamellar structures can bee seen as white lines, and have a thickness of 15 nm. In the center of Fig.1(a), randomly oriented lamellar structures were not visible, but in Fig.1(b). Hence, TEM and STEM images of crystallized polymer can not project total lamellar structures in the specimen. In order to reveal invisible lamella structure in Fig.1, TEMT was carried out. Fig.2 shows three orthogonal cross-sectional view of the 3D reconstruction at the same area of Fig.1. In Fig.2 x-y plane corresponds to upper surface of the section. Dashed lines in x-y plane represent position of other two orthogonal cross-sections. Considerable curvature of the lamella-structures can bee seen in Fig.2. In y-z plane (A), (B) and (C), stacking lamellar structures, which were including invisible ones at Fig.1(a), were clearly visible. The volume rendering of a single lamellar structure in FIG.2 was carried out. In this conference, a study of morphology of single lamellar structure in bulk specimen will be discussed.



We think that the technique of TEMT will be useful method for studies of lamellar morphology.

References

[1] Sano H., Usami T., and Nakagawa H. Polymer, **27** (1986) 1497-1504.

[2] The IMOD home page: http://bio3d.colorado.edu/imod/

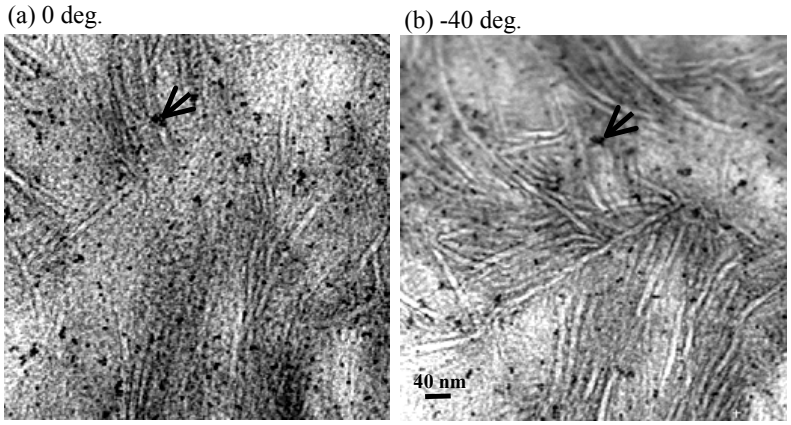


Fig.1. Bright field STEM images of a HDPE at the tilt angle 0 degree (a), and -40 degrees (b). White lines and black particles are lamellar structure and gold particles. Arrows in each figure indicate same gold particles as the marker.

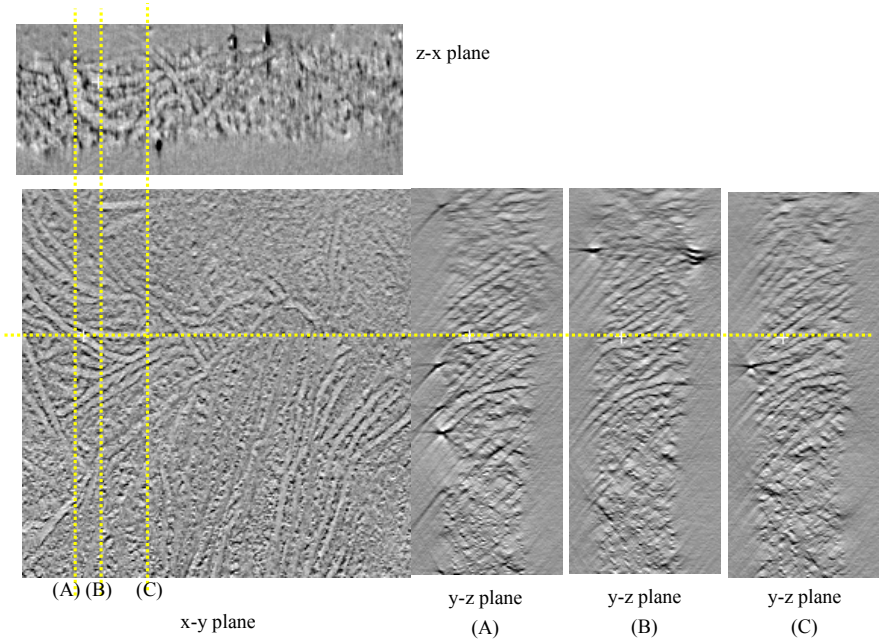


Fig.2. Orthogonal representation of 3D image obtained by dual-axis STEM-tomography, which was obtained at the same area of FIG..1.

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