실시간 X-선 산란을 이용한 배향된 PET 섬유의 구조 형성 메카니즘

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Investigation of Structure Formation in Amorphous Cold-Drawn Poly(ethylene terephthalate) Yarn using Time Resolved X-ray Scattering

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Using a time resolved X-ray scattering technique at the Pohang Accelerator Laboratory(PAL) we were able to investigate the structural changes of cold-drawn poly(ethylene terephthalate) yarn based on the variation of (001) reflection patterns upon increasing temperature(Fig. 1, 2). The X-ray results revealed the appearance of weak but sharp transient diffraction peak in the direction of meridian with a period of 10.46Å at 75℃. This structure can be identified with the mesophase structure proposed by a number of workers to occur in the earlier stage of the development of triclinic crystal. We also found that this transient peak intensifies at temperatures below 75℃ but weakens above 75℃ with the increase of crystallinity. We indexed this transient peak in the meridian as $(001)_m$ reflection, because this transient meridional reflection has the spacing of 10.46Å nearly corresponding to the length of the c-axis (10.75Å) in poly(ethylene terephthalate) crystal lattice. This reflection then completely disappeared at temperatures above 100℃ and a new reflection at off-axis, which is assumed to be a (001) reflection from a triclinic unit cell, appeared when the temperature reached above 150℃. We assigned this reflection (9.46Å in spacing, 62° in azimuthal angle) as $(001)_t$. The results imply that the oriented PET forms a metastable structure before forming a thermodynamically more stable triclinic crystal structure. The orientation factors of amorphous (F_{on}) and crystalline component (f_c) at different temperatures are shown in Fig. 3. The decrease of Fon observed above 75℃ can be attributed to the transformation of the oriented chains to the crystalline fraction by heat treatment rather than the relaxation. On the other hand, the increase of the crystalline orientation factor is related to the viscoelastic property changes in the direction of fiber axis with the temperature increase. $\phi_{c,z}$(angle between the fiber axis and the crystalline c-axis) estimated by azimuthal distribution of (010) reflection at 75℃ was 13.34°. This value coincided with that can be derived from the d-spacing of $(001)_m$ reflection (12.73°)and the c-axis of triclinic lattice, indicating that the angle between the oriented chains and the fiber axis is ~13° at the transient state. The time resolved X-ray structures of the PET yarn led to a conclusion that the PET yarn initially with a oriented amorphous structure (nematic-like structure) at room temperature transforms into a transient smectic C-like structure at ~75℃, then finally to a thermodynamically stable triclinic structure at higher temperatures.

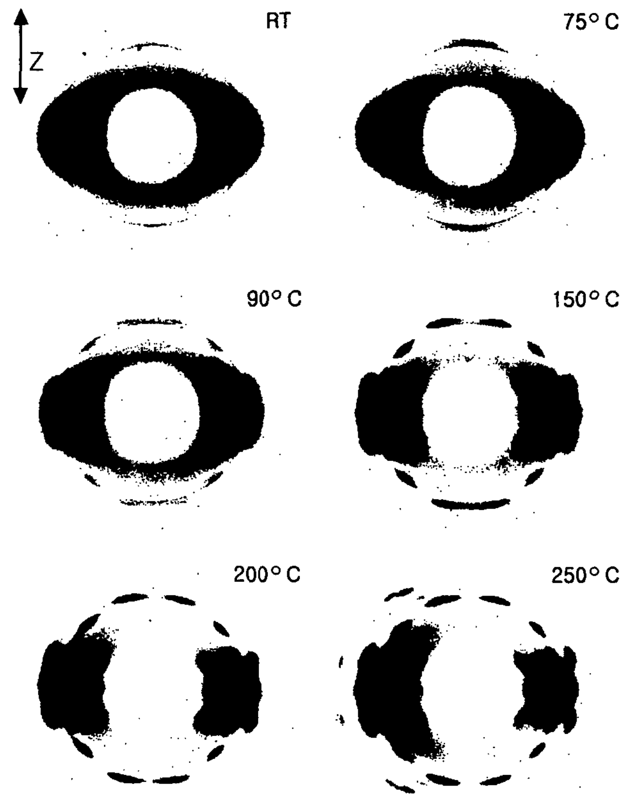

Figure 1. Selected two dimensional WAXS patterns of a cold-drawn oriented PET yarn at various temperatures

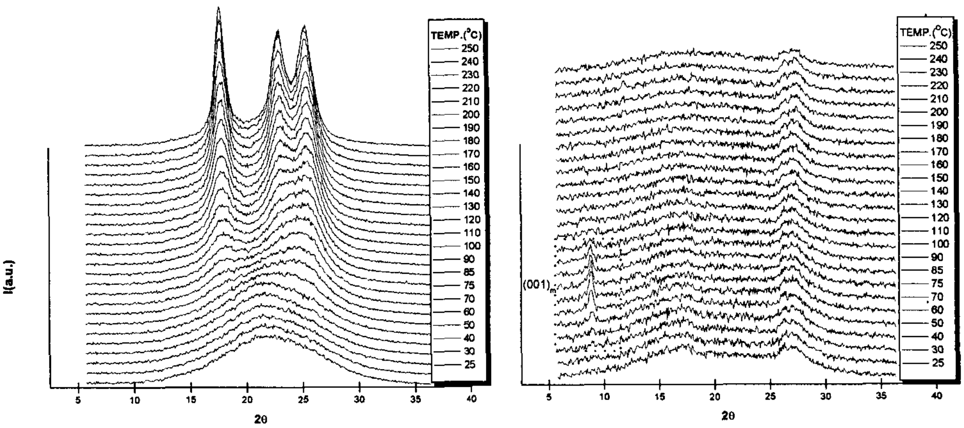

Figure 2. WAXS profiles in a) equator and b) meridian at various temperatures

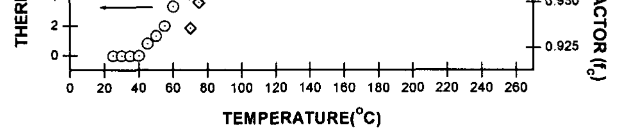

Figure3. Variation of crystalline orientation factor and thermal relaxation rate at different temperatures

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