

Micro X-ray Diffraction Study of Three-dimensional Spherulites of Bacterial Polyester

Taihei MUKAIDE¹, Takashi NOMA¹, Kazuhiro TAKADA¹, Atsuo IIDA²
Takeshi IMAMURA¹, Takashi KENMOKU¹

¹Canon Research Center

² KEK-PF, Tsukuba, Ibaraki 305-0801, Japan

Introduction

Poly(3-hydroxyalkanoates), PHAs were known to be synthesized by various bacteria and biodegradable thermoplastic polyesters. In general, polymers show high ordered structures through the various crystallization methods. One of them is called spherulites. Many researchers have investigated about structure of spherulites, but most of them have studied only about two-dimensional spherulites [1]. In this study, the internal structure of three-dimensional spherulites of poly(3-hydroxy-5-phenoxyvaleric acid), PHPxV, homopolymer was investigated. The position-dependent of the molecular orientation was measured. The micro X-ray diffraction method was very useful for the investigation of partially ordered materials.

Experimental

Micro X-ray diffraction measurements were carried out with synchrotron radiation source at BL4A. The schematic diagram of experimental system is shown in Fig. 1. The incident X-ray beam was monochromated by W-Si multilayer monochromator. Its energy was 8keV. The monochromated X-ray beam was formed into a micro X-ray beam by the Kirkpatrick-Baez system consisting of a pair of elliptical mirrors made of platinum-coated fused quartz. The beam's size was about $5 \times 5 \mu\text{m}^2$ at the sample position [2]. Two pinhole collimators, which were 200 μm and 30 μm in the diameter, and made of tantalum and tungsten, were inserted between the Kirkpatrick-Baez system and the sample in order to cut off the unnecessary scattering X-ray. A CCD camera was used as a detector and its distance from the sample was 100 mm. The diameter of the sample was about 100 μm . The sample was stuck on the top of glass fiber (Fig. 2(a)) and then it was set in a sample holder.

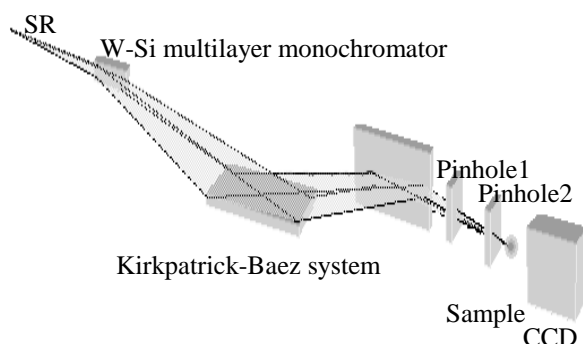


Fig. 1. The schematic diagram of experimental system.

Results and Discussion

The fiber diffraction patterns of the sample are shown in the Fig. 2. The upper part of the sample was measured in horizontal steps of 20 μm . PHPxV belong to orthorhombic space group $P2_12_12_1$ with cell dimensions of $a = 8.369 \text{ \AA}$, $b = 21.301 \text{ \AA}$ and $c = 5.593 \text{ \AA}$ [3]. The fiber diffraction patterns show that crystals were oriented with the b axis along the radius.

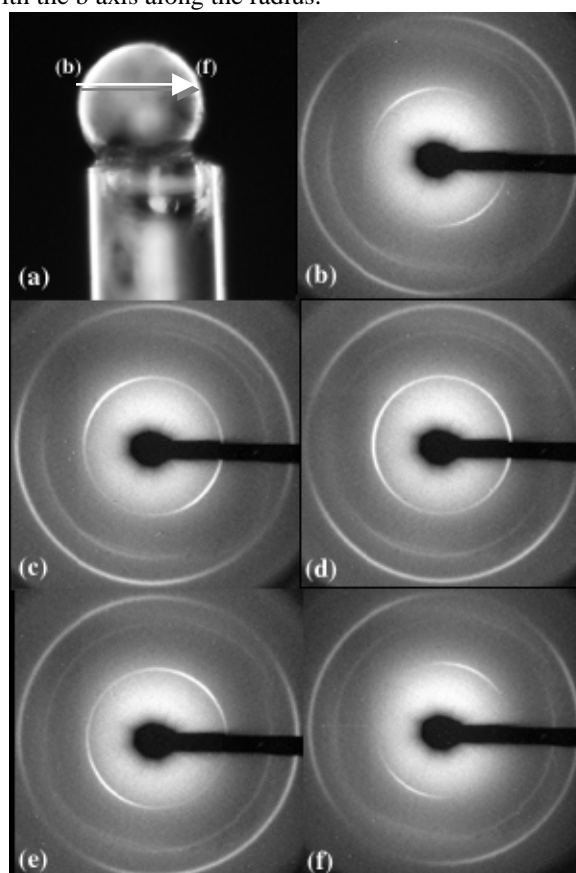


Fig. 2. The assemblage (a) and the fiber diffraction patterns (b) - (f) of the sample. The fiber diffraction patterns from (b) to (f) were measured in horizontal steps of 20 μm .

References

- [1] A. Mahendrasingam et al., J. Synchrotron. Rad. 2, 308 (1995).
- [2] A. Iida et al., Nucl. Instrum. Methods B. 82, 129 (1993).
- [3] T. Mukaide et al., Polymer Preprints, Japan (English Edition, Vol.50, No.1)

* noma.takashi@canon.co.jp