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Wide-angle X- ray diffraction and Small-angle X -ray scattering of polymer fluid under flow.

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Structure that is developed in polymer fluid under flow has been attracted in both academic and industrial points of view. Fiber spinning has been one of most important polymer processing and the process consists mainly of extensional flow of polymer fluid. Therefore, experiment of *in-situ* X-ray scattering in a spinline will lead to fruitful insight into structure developed in the process.

A spinning apparatus that is suitable for the *in-situ* X-ray experiment in BL40B2 was built by TOYOBO. The apparatus can control position of spinning fiber in horizontal direction with accuracy higher than 10 μm to adjust the position against the incident X-ray beam. Moreover, distances along the fiber ranging from 10 to 320 mm from the spinneret could be examined on the spinline.

Figure 1 shows series of wide-angle X-ray scattering (WAXS) patterns obtained from spinning fiber of high-density polyethylene (HDPE) in BL40B2. Flow direction is parallel to the vertical side of the figure. The numbers inserted in the figure indicate the distance from spinneret to the position where each pattern was taken. At the distance of 174 mm, the WAXS pattern consists of an amorphous halo (Figure 1a). Figure 1b obtained at the distance of 199 mm shows that (110) reflection appeared on equator as indicating by an arrow. At further downstream along the spinline, (110) reflection splits in off-equator direction (Figure 1d, at the distance of 274 mm from the spinneret). The off-equatorial splitting is well known in melt-spun HDPE fiber[1)], however, the prior development of (110) on equator at upstream of the spinline is brand-new data. This suggests that the heterogeneous development of crystal in the melt-spun HDPE fiber. Now we are analyzing the simultaneously obtained small-angle X-ray scattering (SAXS) patterns and detailed results will be shown elsewhere.

[Reference] 1) Schulz,J.M. et al *Macromolecules* **1999**, *32*, 8121

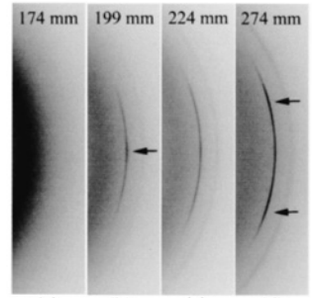

Figure 1. WAXS patterns (partial close-up)

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An attempt on high resolution x-ray structural study of the stable form of the CO complex of [NiFe]hydrogenase at ultra-low temperature II.

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Introduction

Hydrogenase from sulfate-reducing bacteria catalyzes the reversible oxidation of molecular hydrogen in conjunction with a specific electron transfer protein, cytochrome c_3. It plays an important role for hydrogen metabolism in various bacteria.

The active site is composed of two metal atoms (one Ni and Fe atoms). The Fe atom has four non-protein ligands. They have been reported as three diatomic ligands (SO, CO or CN) and one monatomic ligand (S or O) [1,2]. In addition, four cysteinyl sulfur atoms in the large subunit coordinate to the Ni atom, two of them also coordinate to the Fe atom, making a bridge between them.

Carbon monoxide has been known to be as a reversible inhibitor of substrate, dihydrogen. Its behavior on the Ni-Fe active site has been investigated by various spectroscopic methods. In our recent studies of the crystal structure analysis of CO-complex of [NiFe] hydrogenase, it has been proofed that the additional CO exclusively coordinates to the Ni atom instead of Fe. It has been also elucidated, however, that the coordination geometry of the Ni-CO structure is not stable but varies from crystal to crystal even at 100K. In order to see the stable Ni-CO structure, we have attempted to carry out the cryogenic crystallography at 30K on CO complex form of [NiFe] hydrogenase at beam line 40B2.

Experiments, Results and Discussions

Diffraction experiment was performed on one CO-complex crystal at 30 K using a Mar-CCD detector system. The complexed crystal was mounted in a thin capillary tube in the atmosphere of CO. The condition of the data collection was already determined by the former experiment at the same beam line. The crystal to cryo-gun distance was the most important parameter to avoid the generation of the ice-rings at ultra-low temperature when a capillary tube was used. One full data set was successfully collected at 1.2 A resolution with data completeness of 96.0%, and the quality of the reflection data is now under investigation.

References

1. Volbeda, A. *et al. Nature*.**373**,580 (1995)
2. Higuchi, Y. *et al. Structure* **5** 1681 (1997)