

User Benefits

- ◆ XRD enables nondestructive orientation measurement of crystals in polymers such as synthetic fibers.
- ◆ Measurement by the transmission method, which is necessary for investigation of the degree of orientation, is possible by using a Shimadzu rotational sample stage.
- ◆ Simple calculation of the degree of orientation is possible by using the orientation calculation software provided with the fiber sample attachment.

Introduction

The crystals of polymers are known to take a preferred orientation in a specific direction and form a fibrous structure when processed by heating, drawing, and rolling. The strength and flexibility of polymer products such as synthetic fibers can be confirmed by investigating their crystal orientation. Moreover, knowledge of the crystal orientation is useful in development to add new functions to resins and films. It is known that crystals also take a preferred orientation in natural wood fiber.

Orientation measurement is possible by using a Shimadzu XRD-6100 or XRD-7000 X-ray diffractometer in combination with the Shimadzu rotational sample stage and fiber sample attachment. This article introduces an example in which an X-ray was irradiated on a sample of packing string by the transmission method, and the degree of crystal orientation was measured during in-plane rotation of the sample to determine the peaks of particular angles.

Qualitative Analysis

A qualitative analysis of the packing string was carried out by the transmission method. A rotational sample stage was mounted on an XRD-6100 X-ray diffractometer, and a cut sample of the packing string was fixed and set in the fiber sample attachment. Photo 1 shows the condition of the fixed packing string, and Photo 2 shows the condition of the rotational sample stage and fiber sample attachment mounted on the XRD-6100 (transmission method). Table 1 shows the measurement conditions.

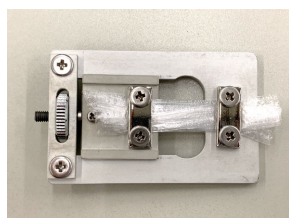


Photo 1 Condition of Packing String Fixed in Fiber Sample Attachment

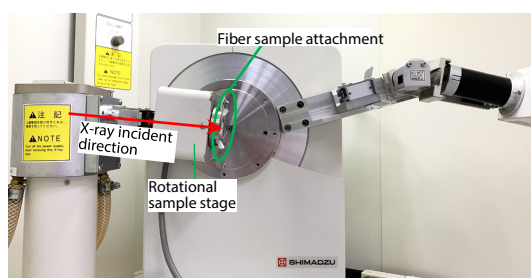


Photo 2 Condition of Attachments and Sample Mounted on XRD-6100 (Transmission Method)

Table 1 Qualitative Analysis Measurement Conditions

Instrument	: XRD-6100
X-ray tube	: Cu target
K β removal	: Counter monochromator
Detector	: Scintillation detector
Attachments	: Rotation sample stage, fiber sample attachment
Voltage - current	: 40 kV - 40 mA
Slit conditions	: DS = ϕ 4 mm, SS = 1°, RS = 0.3 mm
Measurement mode	: 2 θ continuous scan, θ fixed (transmission method)
Measurement speed	: 1°/min
Measurement step	: 0.02°
Measurement range	: 2 θ = 10° - 50° (θ = -90° fixed, transmission method)
Rotational speed	: 60 rpm

Results of Qualitative Analysis

Fig. 1 shows the diffraction pattern of the packing string. The sample was identified as polypropylene by a card search of the ICDD database. The Miller indices of the crystal planes of each peak are also shown in parentheses in the figure.

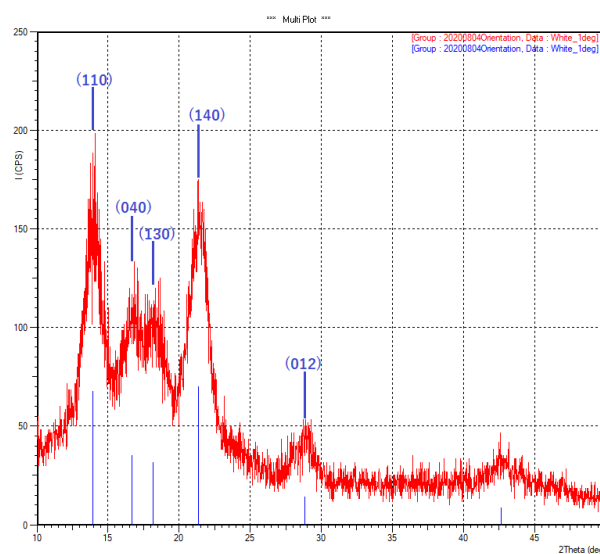


Fig. 1 Diffraction Pattern of Packing String and Results of Qualitative Analysis

Orientation Measurement

Based on the results of the qualitative analysis of the packing string, orientation measurements by the transmission method were carried out for the four main peaks of polypropylene. Table 2 shows the results for the measured peaks ① to ④.

As in the qualitative analysis, the rotational sample stage was mounted on the XRD-6100 X-ray diffractometer, and a cut sample of the packing string was fixed and set in the fiber sample attachment. Table 3 shows the analysis conditions. When the setting angle in the instrument is set to 90°, the machine direction (MD, machine axial direction) coincides with the measurement direction. Therefore, the orientation measurement was conducted over a range of 360° from 90° to 450° in the in-plane rotation (β rotation) of the sample.

Table 2 Peaks Measured in Orientation Measurements

Peak	2θ angle	Miller index
①	14.00°	(110)
②	16.70°	(040)
③	18.20°	(130)
④	21.36°	(140)

Table 3 Orientation Measurement Conditions

Instrument	: XRD-6100
X-ray tube	: Cu target
Kβ removal	: Counter monochromator
Detector	: Scintillation detector
Attachments	: Rotation sample stage, fiber sample attachment
Voltage - current	: 40 kV - 40 mA
Slit conditions	: DS = φ4 mm, SS = 1°, RS = 0.3 mm
Measurement mode	: β rotation, 2θ fixed (2θ = 14°, 16.7°, 18.2°, 21.36°), θ fixed (θ = -90°, transmission method)
Measurement speed	: 20'/min
Measurement step	: 1° step
Measurement range	: β = 90° - 450° (when β = 90°, the machine axis is vertical)

Results of Degree of Orientation Measurement (Profile)

Fig. 2 below shows the results of the degree of orientation measurement by the transmission method for peaks ① to ④ of polypropylene.

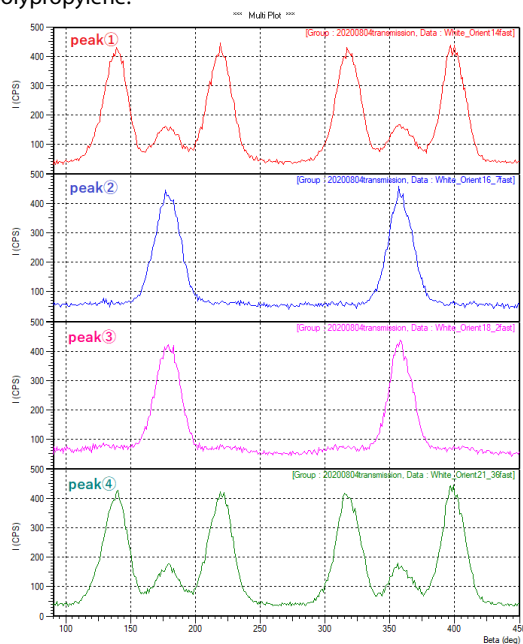


Fig. 2 Results of Degree of Orientation Measurement

Results of Degree of Orientation Calculation

The degree of orientation O (%) is calculated by the following equation.

$$O = \frac{360^\circ - \sum W_i}{360^\circ} \times 100 (\%)$$

* where, W_i is the half-width of each peak.

Fig. 3 shows the result of the calculation for peak ① by the orientation calculation software. Table 4 summarizes the results of the degree of orientation calculations for peaks ① to ④ by the same method.

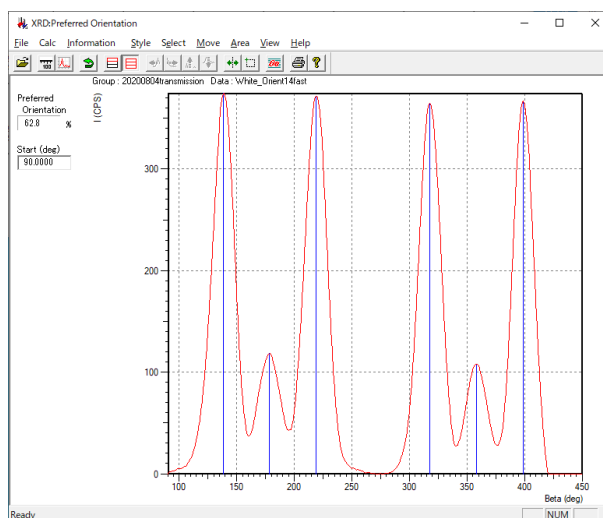


Fig. 3 Result of Degree of Orientation Calculation by Orientation Calculation Software (Peak ①)

Table 4 Results of Degree of Orientation Calculations

Peak	Degree of Orientation (%)
1	62.8
2	87.2
3	87.4
4	63.9

Conclusion

The packing string used as the sample was made of polypropylene. The results of the degree of orientation measurements by the transmission method of the four main peaks of polypropylene showed that the orientation direction and the degree of orientation differed depending on the crystal plane.