# Change in Fine Structure of Nylon 6 Gut Yarn in Twisting, Annealing, and Untwisting Processes. II. Observation by the Microbeam X-Ray Technique

ISHIO TSUJIMOTO, TERUO KUROKAWA, TOSHISADA TAKAHASHI, and KENSUKE SAKURAI, *Laboratory of Textile Physics, Fukui University, Fukui, Japan*


### Synopsis

The local structure in the radius direction perpendicular to the fiber axis in nylon 6 gut yarn subjected to twisting and heat setting was investigated by the microbeam x-ray technique. The observations revealed that the *b*-axis (or chain axis) in a crystal gradually inclines within a tangential plane parallel to the fiber axis and away from it with increase of twist, not only in the outer region but also in the center region, i.e., in the vicinity of the center axis. This local behavior of the *b*-axis could not be distinguished in the previous work,[1] when the usual collimator was employed in the WAXD technique.


## INTRODUCTION

In the previous paper[1] it was shown that the angle of streak line on the surface measured by the electron microscope is in good agreement with that of the twist measured by the optical microscope in nylon 6 gut yarn treated by twisting and annealing. With regard to the effect of the twisting on the fine structure, the outer region of the sample is more influenced. Furthermore, we have made clear the relation between the fine structure and the crimpness as a characteristic of false-twisted yarn. These results were obtained mainly from the usual wide- and small-angle x-ray diffractograms.

In this work it is our purpose to study the local structure in the radius direction perpendicular to the fiber axis in more detail. For this purpose the microbeam x-ray technique was employed.

## EXPERIMENTAL

### Sample

A commercial nylon 6 gut yarn (Toray Co., 7000d, density of 1.1472 g/cm$^3$) was used. For adequate lamellar structure, the sample was annealed at 190°C under no tension. This controlled sample was subjected to twist and heat set. Details of the treatment are given in the previous paper.[1] The direction of the incident beam was normal and parallel to the fiber axis and the three positions within a sample, i.e., the center and the intermediate and outer regions, were exposed as shown in Figure 1. The sample diameter was about 1 mm. When the incident beam is parallel to the fiber axis, three positions in a cross section are perpendicular to the fiber axis shown in Figure 1(i), where their positions are denoted

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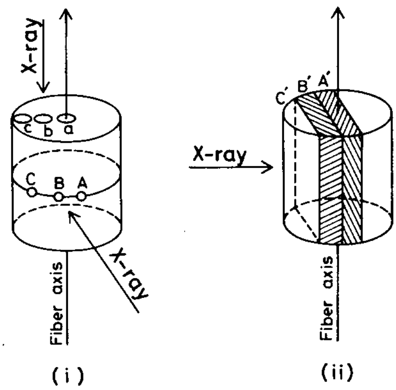


Fig. 1. Schematic representation of the relationship between the incident beam and the exposed positions in the sample.

as a, b, and c corresponding to the center and the intermediate and outer regions, respectively. Two different geometrical conditions on the sample were adopted when the incident beam is normal to it. One is the case when the sample is cylindrical as it is shown in Figure 1(i), where the positions of A, B, and C are denoted in the same manner. Another case is as follows: the sample is cut out parallel to the fiber axis corresponding to the three regions, the three pieces of which are similarly denoted as A′, B′, and C′ as shown in Fig. 1(ii) and the incident beam is normal to the surface of the cut pieces. The sample was cut out as carefully as possible so that it should not be distorted.

### Microbeam X-Ray

Microbeam wide-angle x-ray photographs were obtained with a Toshiba Denki model XC-40H generator equipped with a microcamera, Rigaku Denki model B-3. The x-ray source was nickel-filtered $CuK_\alpha$ radiation (40 kV, 20 mA). The pinhole collimator with a diameter of 100 $\mu$ as well as the sample holder and the camera are set within a vacuum box. The distance between the sample and the camera was 10 mm, and the exposure time was 3 hr. The position to be exposed in a sample was deliberately adjusted by use of the microscope to the direction of the incident microbeam x-ray.

## RESULTS AND DISCUSSION

Microbeam wide-angle x-ray photographs of the heat-setting samples with twisting histories of $0t/m$, $150t/m$, and $250t/m$ are shown in Figures 2, 3, and 4, respectively. The two Debye Scherrer rings shown in Figures 2(a) and 2(c) correspond to the reflections of the (200) and (002) planes in the $\alpha$-form crystal of nylon 6. These rings show that the distributions of the $a$- and $c$-axes are the cylindrical symmetries around the fiber axis not only in the center region but also in the outer region. As is seen from Figures 2(A), and 2(C), the reflections of the (200) and (002) planes are observed on the equator, which indicate that both planes are oriented parallel to the fiber axis and the weak reflection of the (020) plane observed on the meridian suggests that this plane is oriented normal to the fiber axis.

The patterns shown in Figures 2(A′) and 2(C′) are similar to those observed

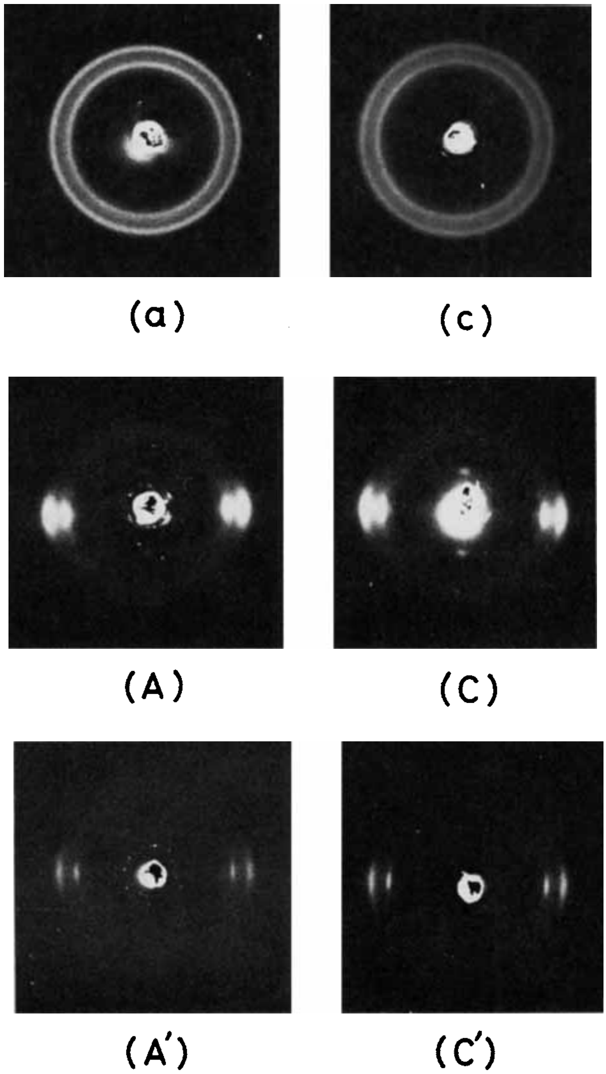


Fig. 2. Microbeam WAXD photographs for the samples with 0*t/m* twist. (a), (c), (A), (C), (A′), and (C′) correspond to the exposed positions shown in Fig. 1.

in Figures 2(A) and 2(C), except for the sharpness of the reflection, which may be due to the effect of the thickness of exposed samples but not due to the different structure resulting from the positions in a sample, i.e., the former is thicker than the latter. Consequently, it is concluded that the *a*- and *c*-axes are oriented normal to the fiber axis at any positions within a sample in the case of 0*t/m* twist.

In the cases of 150*t/m* and 250*t/m* twists, Figures 3(a), 3(b), 3(c), 4(a), 4(b), and 4(c) show that a part of the reflection rings disappears. In addition, the uniformity of the intensity on the ring deteriorates as the exposed position shifts outward and the twist number increases. Particularly, it can be seen in Figure 4(c) that this markedly occurs in the outer region of the sample with the 250*t/m* twist.

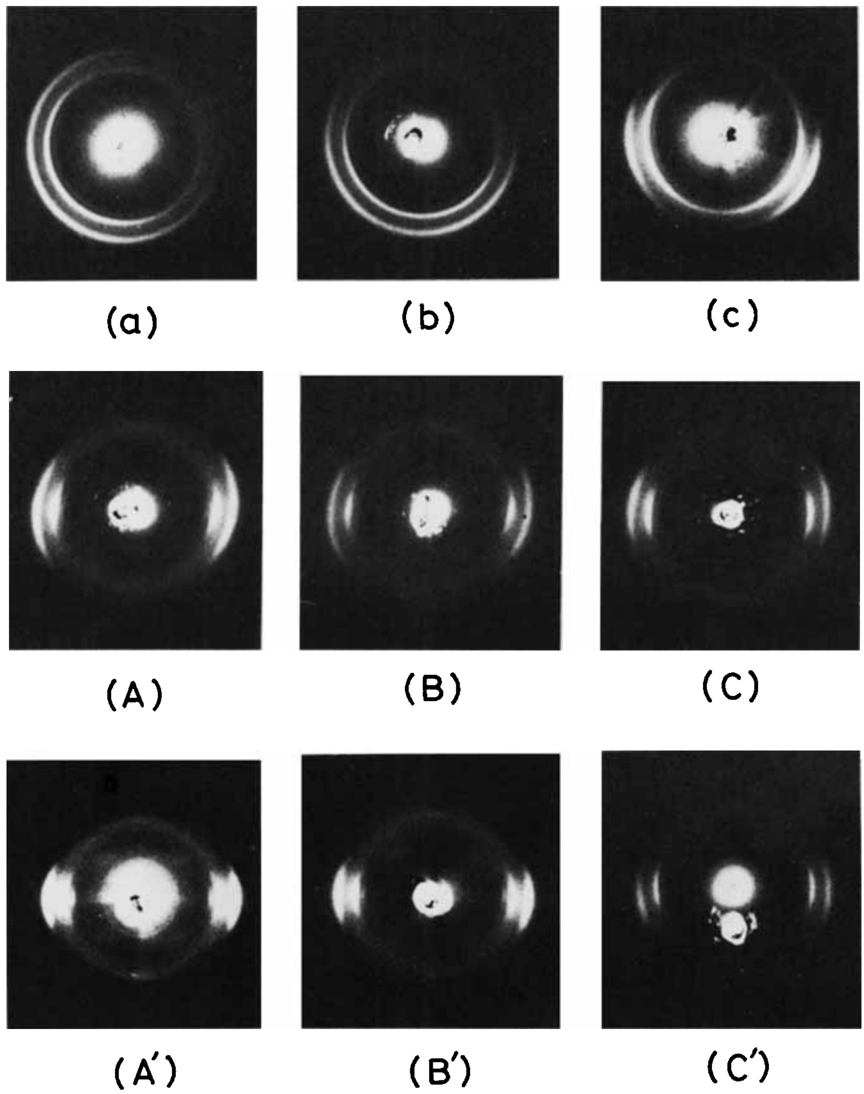


Fig. 3. Microbeam WAXD photographs for samples with $150t/m$ twist. (a), (b), (c), (A), (B), (C), (A′), (B′), and (C′) correspond to the positions shown in Fig. 1.

Considering the relation between Ewald's sphere and the position sphere of the reciprocal vector of the sample,[2] if the chain axis of crystals in the uniaxial oriented fiber does not incline from the fiber axis, the ring pattern is to be obtained when the incident beam is parallel to the fiber axis (known as "end pattern"). On the other hand, if the chain axis inclines, the ring wanes and this is the reason for the appearance of the patterns as observed in Figures 3(a), 3(b), 3(c), 4(a), 4(b), and 4(c). In particular, Figures 3(c) and 4(c) show that the intensity distribution on the ring is likely to segregate into two parts so that they might gather on the equator. Besides, the pattern in Figure 4(c) is rather similar to that in Figure 4(C′).

These indicate that in the outer region the *b*-axis remarkably inclines away from the fiber axis toward the direction with an angle of 45° to the incident beam.

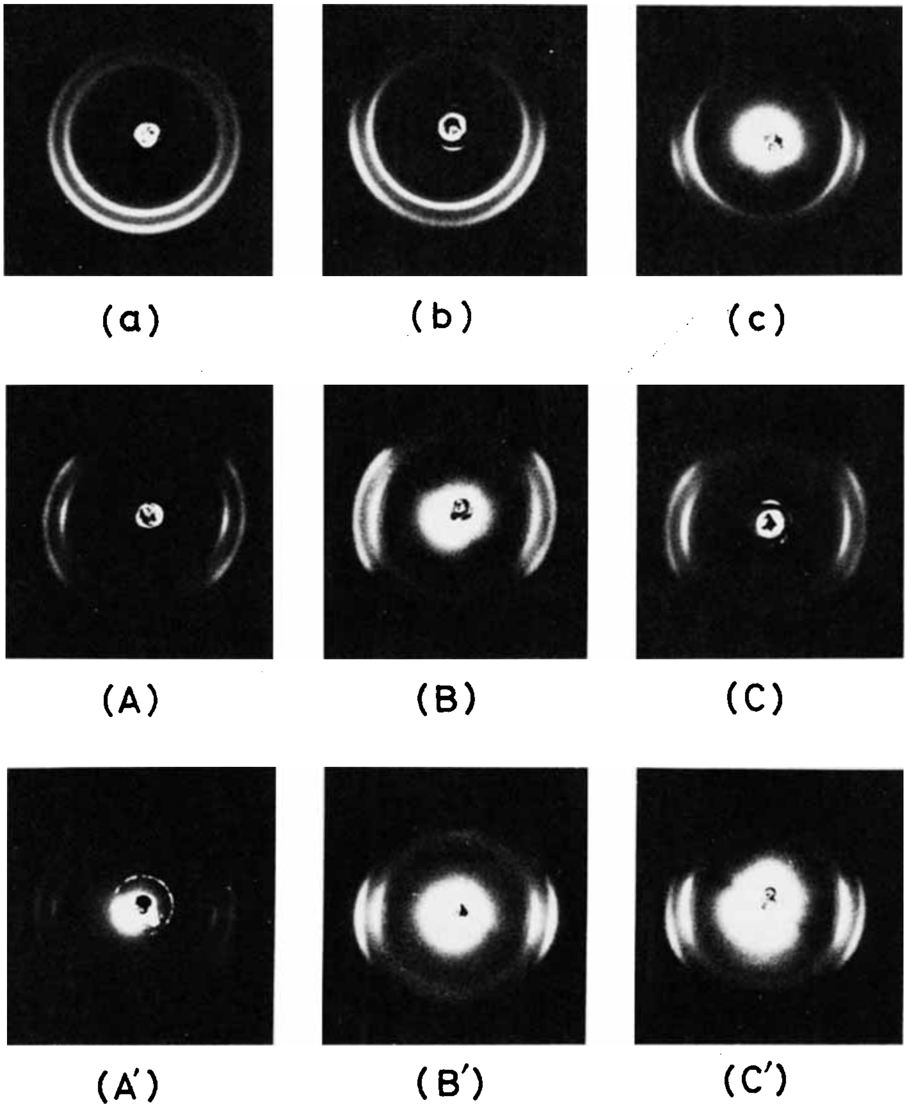


Fig. 4. Microbeam WAXD photographs for samples with 250*t*/*m* twist.

It can be concluded that the *b*-axis slightly inclines from the fiber axis even in the center region, i.e., in the vicinity of the center axis, of the twisted sample. At the same time, the *b*-axis inclines more remarkably in the outer region than in the center region.

WAXD photographs, which were obtained by use of the usual x-ray beam with a diameter of 500 $\mu$, are shown in Figure 5, when the incident beam is perpendicular to the fiber axis. Naturally, the arcs of the (200) and (002) planes broaden along the azimuthal direction compared with those observed by the microbeam, since the reflections are attributable to the overall regions of the sample (containing the three positions defined above) when the usual x-ray beam is used. However, it is clear that the degree of orientation of the *a*- and *c*-axes decreases as the twist number increases, whether the microbeam or the usual beam was

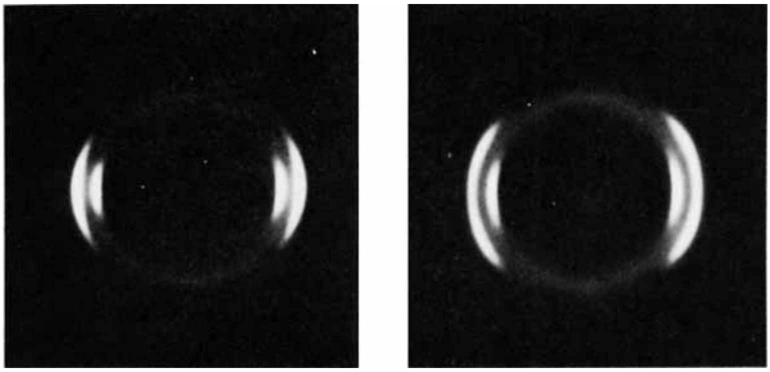


Fig. 5. WAXD photographs for samples with 150$t/m$ and 250$t/m$ with use of the usual collimator, i.e., incident beam diameter of 500 $\mu$.

used, as shown in Figures 3, 4, and 5. This means that the reduction of the degree of orientation takes place in any local region with increase in twist.

It should be noted that a four-point pattern of the (002) reflection is observed in Figure 4(C) and that the patterns of the (200) and (002) reflections appear to shift below the equator, shown in Figures 3(C′) and 4(C′). It is also noted that in Figures 3(A), (B), and (C) and Figures 4(A), (B), and (C) the reflection patterns are distorted to be oblique to the equator.

These observations can be explained as follows: an asymmetric pattern as regards the equator can be obtained when the $b$-axis is inclined toward the direction of the incident beam, as is seen in the inclined x-ray photographs. With regard to the four-point pattern and the oblique pattern, it should be due to the effects of the thickness of exposed samples and the reverse orientation of the $b$-axis in the front side and in the back side portions within a circular sample through the direction of the incident beam.

First taking into account the effect of the sample thickness on the reflection pattern in the x-ray photograph, the reflection spot due to the front side of the sample appears farther than that due to the back side, because of the difference in distances between the scattering point and the camera. Second, let us consider the change in the distribution band of the reciprocal lattice vectors on the position sphere of the (200) and (002) planes. Before twisting, these reciprocal vectors are distributed on the equator of the position sphere. On twisting the $b$-axis inclines spirally and the inclined direction is opposite between the front side and the back side regions of the sample. At the same time, the position sphere rotates, so that the $b$-axis should turn out and thus the distribution band slants away from the horizontal plane. When considering all regions of the exposed sample from the front side to the back side, the slant of the distribution band changes continuously. Consequently, a reasonable interpretation for the observed patterns could be obtained from the relation between Ewald's sphere of reflection and the condition of the distribution band of reciprocal lattice vectors.

Here, we did not consider the effect of the distortion of crystal lattice, but it will be necessary to study this in more detail.

In summary, the $b$-axis gradually inclines away from the fiber axis and spirals in correspondence to the twist angle on the surface of the sample as the twist number increases. At the same time, the degree of orientations of the $a$- and $c$-axes decreases at any local regions of the sample. Particularly, it should be noted that the high twisting causes the $b$-axis to incline considerably in the outer region of the sample. In the center region of sample this behavior could also be recognized by use of the microbeam x-ray technique although it could not be done in the previous work,[1] where the usual WAXD technique was employed.

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