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X-RAY AND THERMAL STUDIES OF NYLON 5-7.(U)  
MAY 80 J LIN, M H LITT, G FROYER

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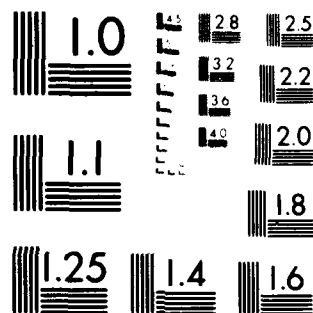
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Task No. NR356-362

TECHNICAL REPORT NO. 2

X-RAY AND THERMAL STUDIES OF NYLON 5-7

by

Ju-Chui Lin, M. H. Litt and Gerard Froyer

Prepared for Publication

in the

Journal of Polymer Science

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ELECTE  
JUN 2 1980  
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Department of Macromolecular Science  
Case Western Reserve University  
Cleveland, Ohio 44106

May 14, 1980

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| 1. REPORT NUMBER<br>Technical Report #2                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | 2. GOVT ACCESSION NO.<br>AD-A084 942                   | 3. RECIPIENT'S CATALOG NUMBER                                            |
| 4. TITLE (and Subtitle)<br>X-RAY AND THERMAL STUDIES OF NYLON 5-7                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | 5. TYPE OF REPORT & PERIOD COVERED<br>Technical Report | 6. PERFORMING ORG. REPORT NUMBER                                         |
| 7. AUTHOR(s)<br>Ju-Chui Lin, M. H. Litt, Gerard Froyer                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | 8. CONTRACT OR GRANT NUMBER(s)<br>N00014-77C-0508      | 9. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS<br>NR 356-362 |
| 10. CONTROLLING OFFICE NAME AND ADDRESS<br>Office of Naval Research (Code 472)<br>Arlington, Virginia 22217                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 11. REPORT DATE<br>May 1980                            | 12. NUMBER OF PAGES<br>22                                                |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 15. SECURITY CLASS. (of this report)                   | 16a. DECLASSIFICATION/DOWNGRADING SCHEDULE                               |
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| 18. SUPPLEMENTARY NOTES                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                        |                                                                          |
| 19. KEY WORDS (Continue on reverse side if necessary and identify by block number)<br>Nylon 5,7, x-ray, unit cell, pyroelectricity, DSC measurements, melting point, crystallinity.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                        |                                                                          |
| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>The unit cell and probable space group of Nylon 5,7 has been determined. The unit cell is monoclinic with the dimensions $a = 0.483$ nm, $b = 0.935$ nm, $c = 1.662$ nm and $\gamma = 58.9^\circ$ . The space group is probably $P_2$ , which is non-centrosymmetric. Rolled, annealed samples show three dimensional orientation. The melting point peak of a rapidly cooled sample is about $213^\circ\text{C}$ when it is heated at $20^\circ\text{C}/\text{min}$ . Slow cooling, $< 1^\circ/\text{min}$ generates a higher melting species, $T_m = 228^\circ\text{C}$ . Crystallinities are in the normal nylon range, up to |                                                        |                                                                          |

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50% for a slow-cooled sample.

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### ABSTRACT

The unit cell and probable space group of Nylon 5,7 has been determined. The unit cell is monoclinic with the dimensions  $a = 0.483$  nm,  $b = 0.935$  nm,  $c = 1.662$  nm and  $\gamma = 58.9^\circ$ . The space group is probably  $P_b$  which is non-centrosymmetric. Rolled, annealed samples show three dimensional orientation.

The melting point peak of a rapidly cooled sample is about  $213^\circ\text{C}$  when it is heated at  $20^\circ\text{C}/\text{min}$ . Slow cooling,  $\leq 1^\circ/\text{min}$  generates a higher melting species,  $T_m = 228^\circ\text{C}$ . Crystallinities are in the normal nylon range, up to 50% for a slow cooled sample.

## INTRODUCTION

Nylon 5-7 could be pyroelectrically and piezoelectrically active (1). Knowledge of its structure is therefore important; however it has never been studied thoroughly. Kinoshita (2) postulated that all polyamides with odd numbers of methylene groups on the backbone have a pseudohexagonal unit cell or the one very close to it. He called it the  $\gamma$ -form, and suggested a pleated sheet structure similar to that proposed by Pauling and Corey (3) for polypeptides. Magill (4,5) confirmed Kinoshita's hypothesis by obtaining a pseudohexagonal electron diffraction pattern from a Nylon 5-7 single crystal. Up to the present time no x-ray data are available for Nylon 5-7 except from Slichter (6) who reported a single Bragg spacing of  $4.14\text{\AA}$  at room temperature and Dreyfuss (7) who reported its chain repeat as  $15.6\text{\AA}$  and its lamellar long spacing as  $76\text{\AA}$ .

This study was made to obtain structural information on Nylon 5-7 in order to conduct dielectric and pyroelectric studies properly.

## EXPERIMENTAL

Synthesis of Nylon 5-7

The procedure was adapted from that of Beck and Magat (8). Pimelic acid (0.2 mole, 32.0 gm) and 1,5-pentane diamine (0.202 mole, 20.6 gm) were dissolved in 250 ml of isopropanol and chilled in an ice bath to precipitate the salt. It was filtered and dried under vacuum at room temperature to avoid oxidation. In order to get a pure salt, the starting materials have to be purified and the salt recrystallized from isopropanol. Melting point of the salt is  $191^{\circ}\text{C}$ .

4.5 gm of salt was placed in a 2 mm thick glass polymerization tube which

was evacuated to  $10^{-3}$  mm Hg and then sealed. The sealed tube was placed in a stainless steel screw capped container into which water was poured in order to balance the pressure inside and outside the sealed tube when the temperature was raised. The metal container was then placed in a  $240^{\circ}\text{C}$  oil bath for 3 hours. After cooling the sealed tube was opened and the polymer was heated at  $260^{\circ}\text{C}$  under vacuum in an appropriate flask for about 1 hour to increase the molecular weight.

The 1% reduced viscosity of resulting polymer was measured in m-cresol at  $25^{\circ}\text{C}$  using an Ubbelohde dilution viscometer.  $\eta_{sp}/c$  found to be 2.1. The melting point of the as-synthesized Nylon 5-7 is  $214^{\circ}\text{C}$ . All melting point measurements were done using a Perkin-Elmer DSC-II.

#### Film Preparation and Orientation

Films of Nylon 5-7 were produced by the following procedures:

(A) Polymer was placed between two pieces of Teflon coated aluminum foil, with a  $100\ \mu$  brass spacer. It was pressed at  $232^{\circ}\text{C}$  and 25,000 psi for 5 minutes, cooled to  $180^{\circ}\text{C}$  over 17 minutes and annealed at this temperature for 1 hour, it was then removed from the press and cooled to room temperature within 5 minutes. The resulting film was about  $100\ \mu\text{m}$  thick. It was then sandwiched between two  $150\ \mu\text{m}$  thick sheets of high density polyethylene and rolled uniaxially at room temperature. The final oriented film was  $37 \pm 5\ \mu\text{m}$  thick. The film was then annealed under tension at  $180^{\circ}\text{C}$  for 45 hours at 1 mm Hg.

(B) The film was prepared using exactly the same procedure as described in (A) except one gold shadowed aluminum foil and one Teflon coated aluminum foil was used to sandwich the sample. The resulting film was about  $100\ \mu\text{m}$

thick without rolling.

#### X-ray Diffraction

X-ray diffraction patterns were obtained using Ni filtered copper K $\alpha$  radiation in a Statton camera. Sample to film distances were determined by dusting the sample with calcium fluoride or shadowing the film with gold. Samples were prepared by gluing several pieces of polymer film together. X-ray diffraction patterns were obtained with the pressed or rolled surfaces of the specimen parallel to and perpendicular to the x-ray beam.

#### Density Measurement

Densities of samples of Nylon 5-7 were determined by the flotation method. Chloroform and benzene were used as the flotation media. A film of Nylon 5-7 was put into benzene, then chloroform was added drop by drop with appropriate stirring until the sample sank. The density of the medium was determined by weighing a 5.00 ml sample in a 5.00 ml volumetric flask.

### RESULTS AND DISCUSSION

#### X-ray Diffraction Pattern

Figure 1(a) shows the x-ray diffraction pattern of Nylon 5-7 film prepared by procedure (A), taken perpendicular to the film; the roll direction is vertical. Figure 1(b) shows the x-ray diffraction pattern of the same polymer film with the beam parallel to the roll direction. Figure 2(a) and 2(b) show sketches of the respective diffraction patterns with the diffractions indexed. One can see that the rolled annealed film, Figs. 1(b) and 2(b) shows good three dimensional orientation. Surprisingly, the  $b^*$  axis is perpendicular to the film surface.

A monoclinic unit cell was found with the following least-square refined

parameters:  $a = 0.483(2)$  nm,  $b = 0.935(2)$  nm,  $c = 1.662(5)$  nm and  $\gamma = 58.9(1)^\circ$ .

This is quite close to a pseudohexagonal unit cell which agrees with the results reported by Kinoshita (2) and Magill (4,5). The Miller indices of all the 17 diffractions found in Figures 1(a) and (b) are listed in Table I. The unit cell can be converted into a metrically orthorhombic one with  $a = .483$ ,  $b = .802$  and  $c = 1.662$  nm. The agreement between observed and calculated d-spacings is better than 0.6% in each case. There are two monomer repeats per unit cell and the calculated crystalline density is 1.168 gm/cc. This is lower than the single crystal density found by Magill (5) of 1.2. The measured density of the rolled, annealed sample (from procedure (A)) is 1.131 gm/cc while the density of the sample processed from procedure (B) is 1.133 gm/cc. Magill (5) reported the density of drawn Nylon 5-7 fiber as 1.139 gm/cc, which is within experimental error of our result.

The x-ray diffraction pattern of the film obtained from procedure (B) was taken with the beam parallel to the surface of the pressed film, Figure 3. It is seen that this film was nucleated by the gold at the surface and has directionally crystallized to some degree with the chain axis parallel to the surface. The d-spacings found were all in good agreement with the measured d-spacings from the rolled, annealed film. The indices of the major diffractions are also shown in the Figure. We cannot say which is the growth direction as indexing, other than for  $c$ , is ambiguous. The  $c$  axis value of  $16.62\text{\AA}$  shows that this chain has the  $\gamma$  conformation. The fully extended all trans structure would have a repeat of  $17.3\text{\AA}$  (10).

From observation of Table I, the following systematic absences were observed:  $hk0$ :  $k = 2n + 1$ , and  $00l$ :  $l = 2n + 1$ . The monoclinic unit cell

could belong to the space group  $P2_1/b$  or  $P_b$  (11).

While the  $P2_1/b$  space group accounts for all the systematic absences, it is impossible to pack two nylon chains, even in the  $\gamma$  form, into such a unit cell. This space group has a center of symmetry, thus each amide group must have a counterpart pointing  $180^\circ$  from the first. Due to the  $b$  glide, this implies that each chain twists through  $180^\circ$  as it goes from one amide bond to the next. This has not been found in any nylon. The unit cell dimensions are normal for Nylon 5,7 implying that its packing is similar to the normal nylon packing.

The space group  $P_b$ , generates the expected chain packing; H-bonding is parallel to the  $b$  axis and all amide groups can H-bond well because they point in the same direction. Nylon 5-7 has an approximate plane of symmetry. In order to generate appropriate H-bonding; the  $c$  reflection must be between the NH and CO groups, which is midway between the planes of symmetry in the molecule (the center  $CH_2$  group of each  $(CH_2)_5$  sequence). Thus, the chain nature plus the requirements for H-bonding and the  $b$  glide means that for any atom at  $c$ , there is an equivalent atom at  $\bar{c}$ , and two approximately equivalent atoms at  $\frac{1}{2}-c$  and  $\frac{1}{2}+c$  due to the chain symmetry. This is shown schematically in Fig. 4. Thus one would expect meridional reflections with  $l$  odd to be absent.

The proposed structure is anisotropic and thus should be electrically active. Our initial poling attempts show that Nylon 5-7 can be poled easily to a medium degree of pyroelectric activity,  $\sim 10^{-10}$  C/cm $^2$  (12). This tends to confirm the conclusion that the unit cell is anisotropic and is probably  $P_b$ .

### Thermal History

The melting temperature of the rolled, annealed film was found to be 215°C and that of gold nucleated crystallized film 228°C, (literature (9) value: 183°C.) The difference between the melting temperatures considering that both samples have the same crystal form may be because the rolling of a film sheared the large, well crystallized lamellae to thinner lamellae, which melt lower.

The effect of thermal history on crystal perfection was also studied. Nylon 5-7 was first heated to 238°C. Then the following treatments were made: (I), The melt was quenched to room temperature at 320°C/minute; (II), The melt was cooled to room temperature at 20°C/minute; (III), The melt was held one hour at 215°C, then cooled to room temperature at 1.25°C/minute; (IV), The melt was held half an hour at 220°C, then cooled to room temperature at 0.31°C/minute. All these treatments were done in the DSC-II sample chamber. X-ray diffraction studies of all four samples show no changes in the crystal structure.

The melting behavior of the samples described above was studied by heating them at 20°C/minute in the DSC. The quenched sample showed a single melting endotherm at 215°C, Figure 5 (endotherm I). Sample IV, cooled at lowest rate gives a high, single melting endotherm at 228°C. Samples II and III show two melting peaks. The slower the cooling (endotherms III and IV), the more material fused at the higher temperatures. The melting peaks tend to stay at two temperatures, ~ 213° and 228°. This is understandable, as the necessity for H-bonding requires that the lamellae have well defined thicknesses (7), which tends to lead to well defined melting points.

Table II gives the melting points, the heats of fusion and the estimated crystallinities of the samples. These crystallinities are probably lower bounds.  $\Delta H_f$  may be lower for Nylon 5-7 than for Nylons 6-6 and 6, since we are comparing a  $\gamma$  form with  $\alpha$  forms of the other nylons.

#### CONCLUSIONS

The x-ray diffraction studies of Nylon 5-7 support Kinoshita's postulate that odd and odd-odd nylons have the  $\gamma$  form crystal structure. There is only one crystal form of Nylon 5-7 which can be generated by mechanical treatment-film rolling, by thermal treatments, or by gold nucleated crystallization. The crystal structure was found to be monoclinic with unit cell dimensions of  $a = .483$  nm,  $b = .935$  nm,  $c = 1.662$  nm and  $\gamma = 58.90^\circ$ . With the space group,  $P_b$ , the H-bonding direction of both chains must be in the same direction; Nylon 5-7 has a net dipole in the unit cell and can be electrically active.

#### ACKNOWLEDGEMENT

This work was supported by the Naval Research Foundation under Grant No. N00014-77C-0508. We also like to express our gratitude to Mr. Stephen Weinhold for the encouraging discussions throughout this work.

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TABLE I. Observed and Calculated d-Spacings and Indexing of the X-ray Diffraction Pattern of Oriented Nylon 5-7<sup>a</sup>

| Miller Index<br>h k l | Intensity <sup>b</sup> | d <sub>obs</sub> (nm) | d <sub>calc</sub> (nm) | % Difference |
|-----------------------|------------------------|-----------------------|------------------------|--------------|
| 100                   | sh, s                  | 0.4146                | 0.4133                 | 0.32         |
| 120                   | sh, v                  | 0.4146                | 0.4134                 | 0.30         |
| 220                   | sh, m                  | 0.2402                | 0.2413                 | -0.47        |
| 020                   | sh, vs                 | 0.3998                | 0.4002                 | -0.10        |
| 240                   | sh, vvw                | 0.2067                | 0.2067                 | 0.00         |
| 200                   | sh, vvw                |                       | 0.2066                 | 0.04         |
| 120                   | sh, vw                 | 0.2341                | 0.2355                 | -0.25        |
| 140                   | sh, vw                 |                       | 0.2355                 | -0.25        |
| 101                   | b, w                   | 0.4016                | 0.4011                 | 0.12         |
| 121                   | b, w                   |                       | 0.4011                 | 0.12         |
| 211                   | sh, vw                 | 0.2303                | 0.2290                 | 0.57         |
| 231                   | sh, vw                 |                       | 0.2290                 | 0.57         |
| 002                   | sh, s                  | 0.8262                | 0.8309                 | -0.57        |
| 102                   | sh, w                  | 0.3713                | 0.3700                 | 0.35         |
| 122                   | sh, w                  |                       | 0.3701                 | 0.33         |
| 103                   | sh, w                  | 0.3321                | 0.3312                 | -0.26        |
| 123                   | sh, w                  |                       | 0.3313                 | -0.25        |
| 004                   | sh, m                  | 0.4142                | 0.4154                 | -0.30        |
| 104                   | sh, w                  | 0.2943                | 0.2930                 | 0.45         |
| 124                   | sh, w                  |                       | 0.2930                 | 0.45         |
| 105                   | sh, w                  | 0.2579                | 0.2590                 | -0.43        |
| 125                   | sh, w                  |                       | 0.2590                 | -0.43        |
| 006                   | sh, w                  | 0.2763                | 0.2770                 | -0.24        |
| 106                   | b, m                   | 0.2300                | 0.2301                 | -0.04        |
| 126                   | b, m                   |                       | 0.2301                 | -0.04        |
| 017                   | b, m                   | 0.2280                | 0.2275                 | 0.22         |
| 017                   | b, m                   |                       | 0.2275                 | 0.22         |
| 107                   | sh, vw                 | 0.2062                | 0.2058                 | 0.17         |
| 127                   | sh, vw                 |                       | 0.2059                 | 0.16         |

a. Calculated with  $a = .483$  nm,  $b = .935$  nm,  $c = 1.662$  nm,  $\gamma = 58.90^\circ$ .

b. sh--sharp; vs--very strong; s--strong; m--medium; w--weak; vw--very weak; b--broad; vvw--very very weak.

Table II. Heat of Fusion and Degree of Crystallinity of Nylon 5-7 as a Function of Treatment<sup>a</sup>

| Sample                                     | I          | II         | III        | IV         |
|--------------------------------------------|------------|------------|------------|------------|
| Weight (mg)                                | 3.73       | 3.02       | 3.63       | 4.24       |
| Peak Melting Temperature <sup>c</sup> (°C) | 215        | 212<br>221 | 210<br>228 | 228        |
| $\Delta H(\text{cal/gm})^b$                | 14.4       | 15.2       | 18.0       | 21.3       |
| % Crystallinity <sup>c</sup>               | $33 \pm 5$ | $35 \pm 5$ | $41 \pm 6$ | $48 \pm 6$ |

a. See text for the thermal treatment.

b. There is  $\pm 5\%$  error depending on how the baseline was chosen.

c. As no heat of fusion is known for pure Nylon 5-7, we use the values from Nylon 6 and Nylon 6-6.  $44 \pm 7$  cal/gm for 100% crystallinity (10).

#### FIGURE CAPTIONS

- Figure 1(a). X-ray diffraction pattern of a rolled, annealed Nylon 5-7 film. The beam is perpendicular to the film surface.
- Figure 1(b). X-ray diffraction pattern of a rolled, annealed Nylon 5-7 film. The x-ray beam is parallel to the film surface and the c-axis.
- Figure 2(a). A sketch of the x-ray diffraction pattern of Nylon 5-7 film (see Figure 1(a)). Reflections are indexed.
- Figure 2(b). A sketch of the x-ray diffraction pattern of Nylon 5-7 film (see Figure 1(b)). Indices of reflections are shown. The  $h^*$  axis is perpendicular to the film surface.
- Figure 3. X-ray diffraction pattern of a directionally crystallized Nylon 5-7 film. The x-ray beam is parallel to the film surface.
- Figure 4. Schematic representation of the bc projection of the Nylon 5-7 unit cell.
- Figure 5. Endotherms of melting peaks of Nylon 5-7 at four different thermal histories.

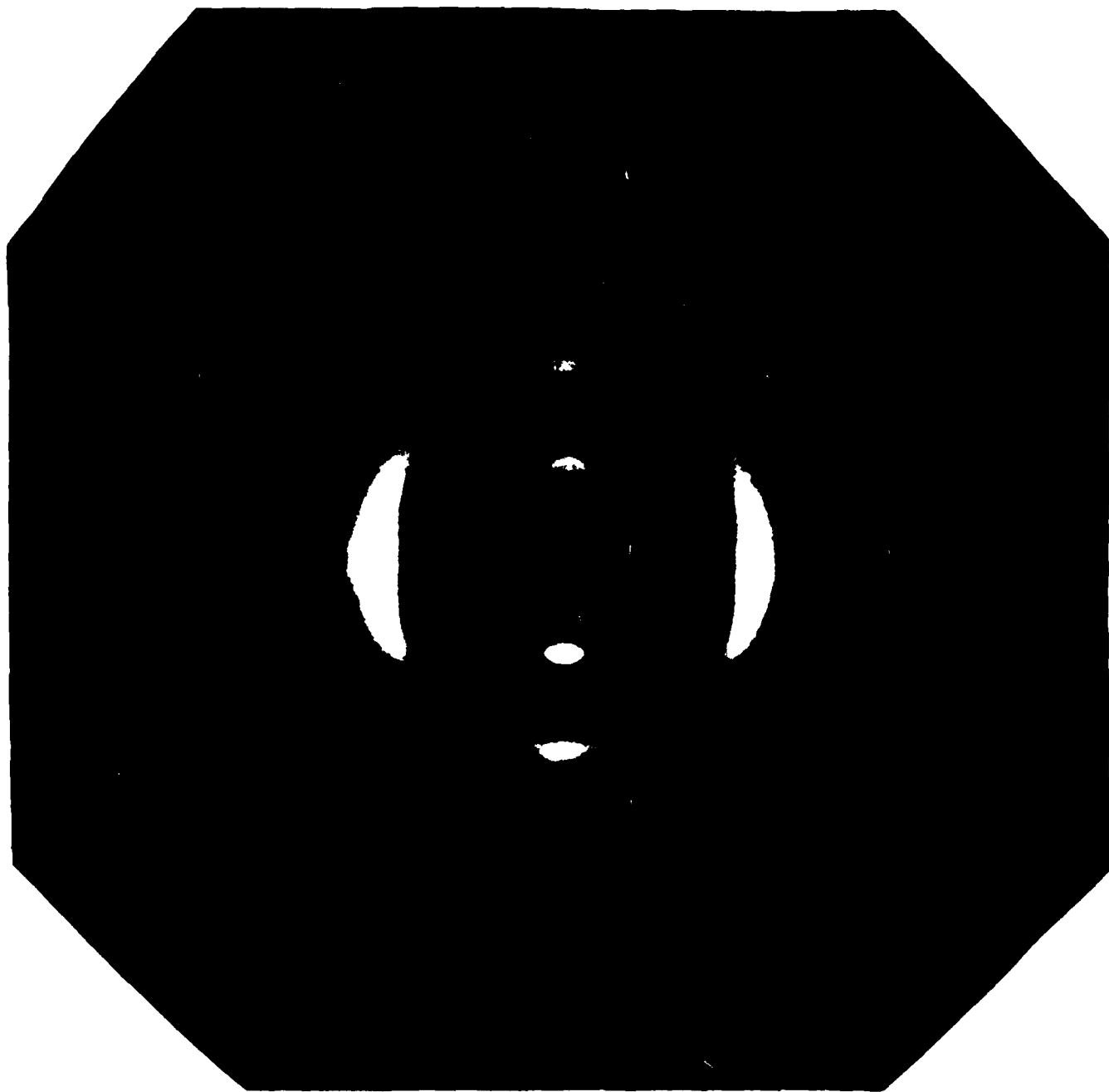


Figure 1(a)

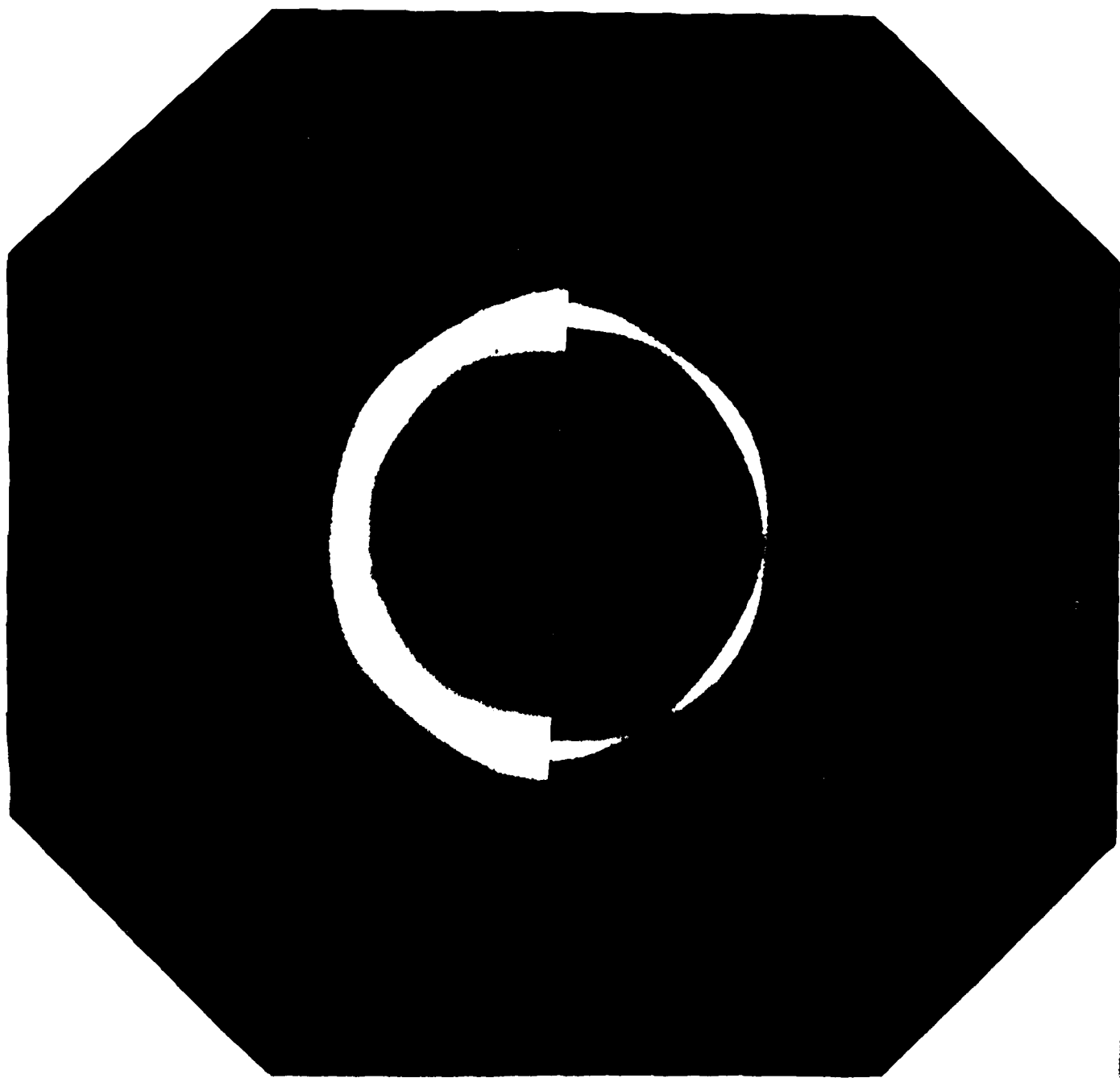


Figure 1(b)

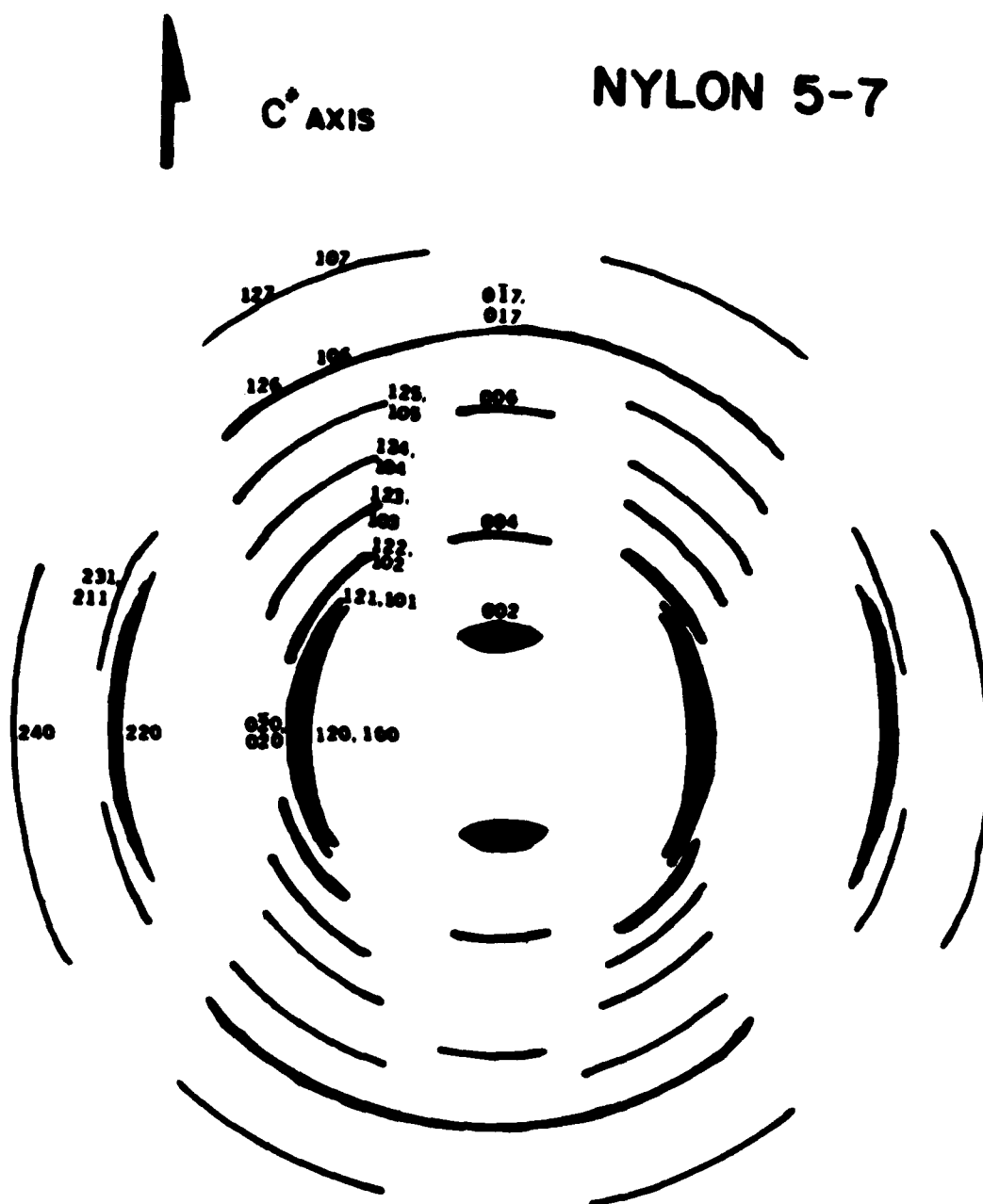


Figure 2(a)

NYLON 5-7

$b^*$  axis

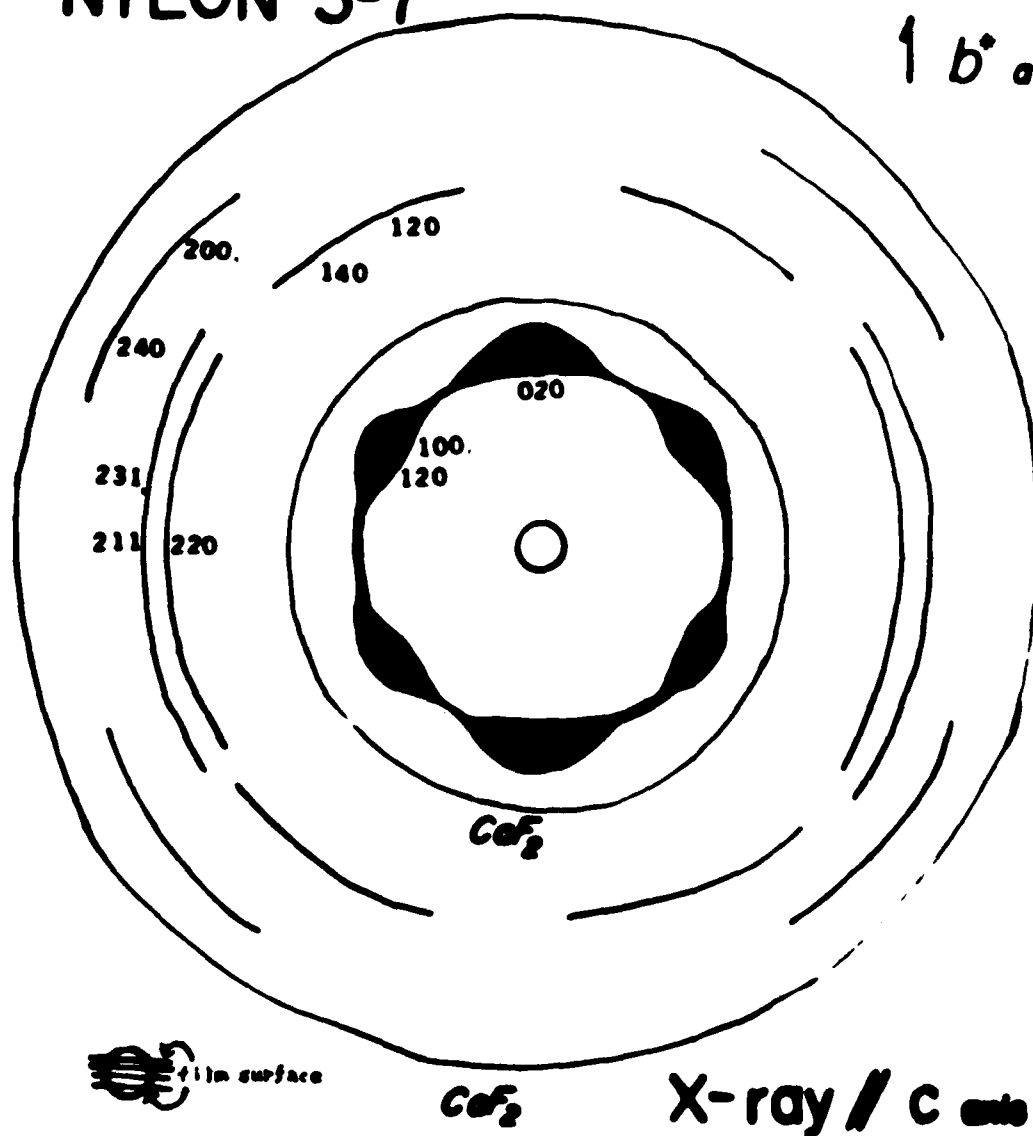
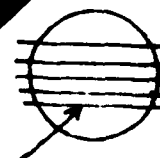
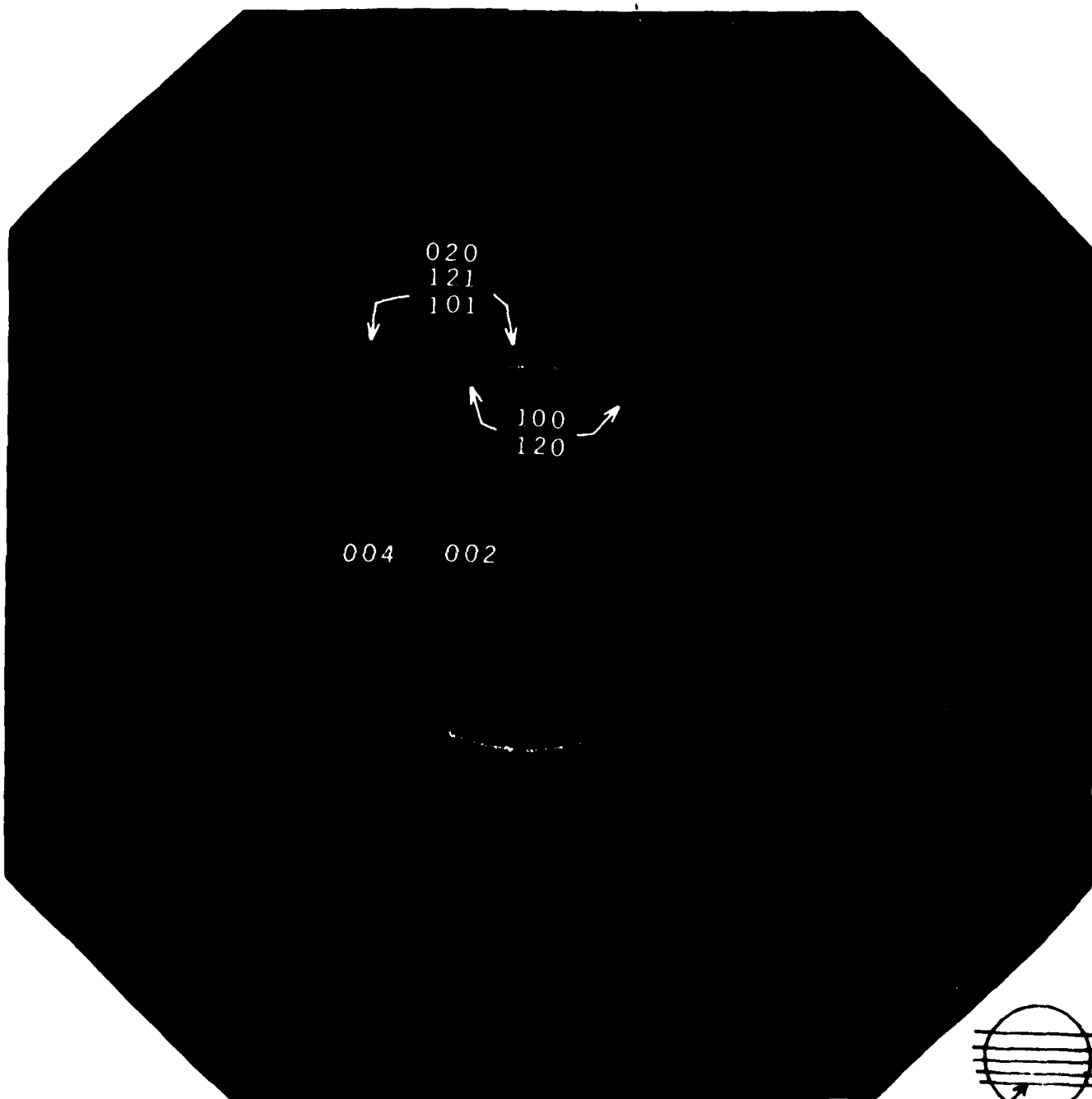


Figure 2(b)



film surface

Figure 3

# NYLON 5-7

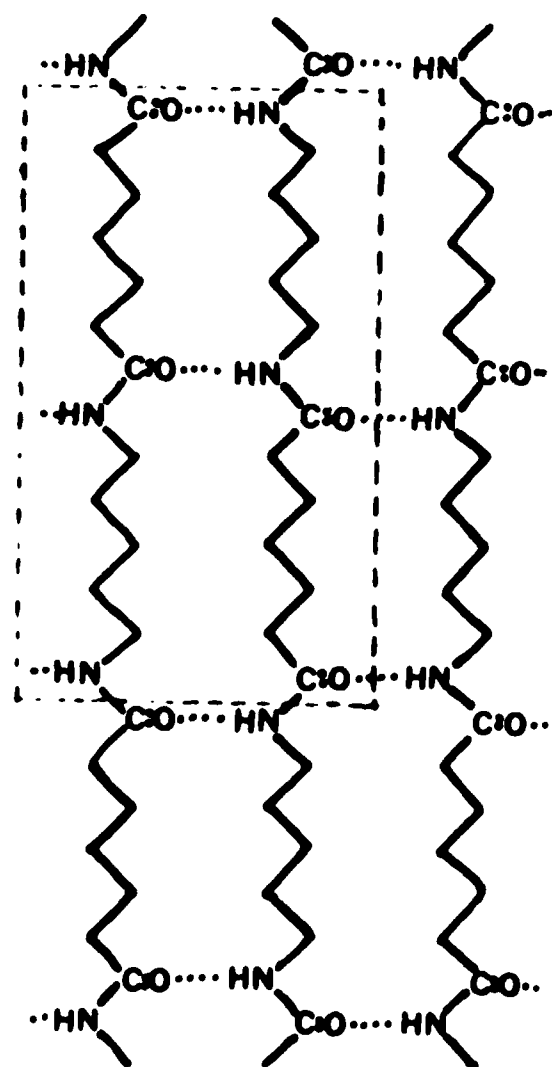


Figure 4

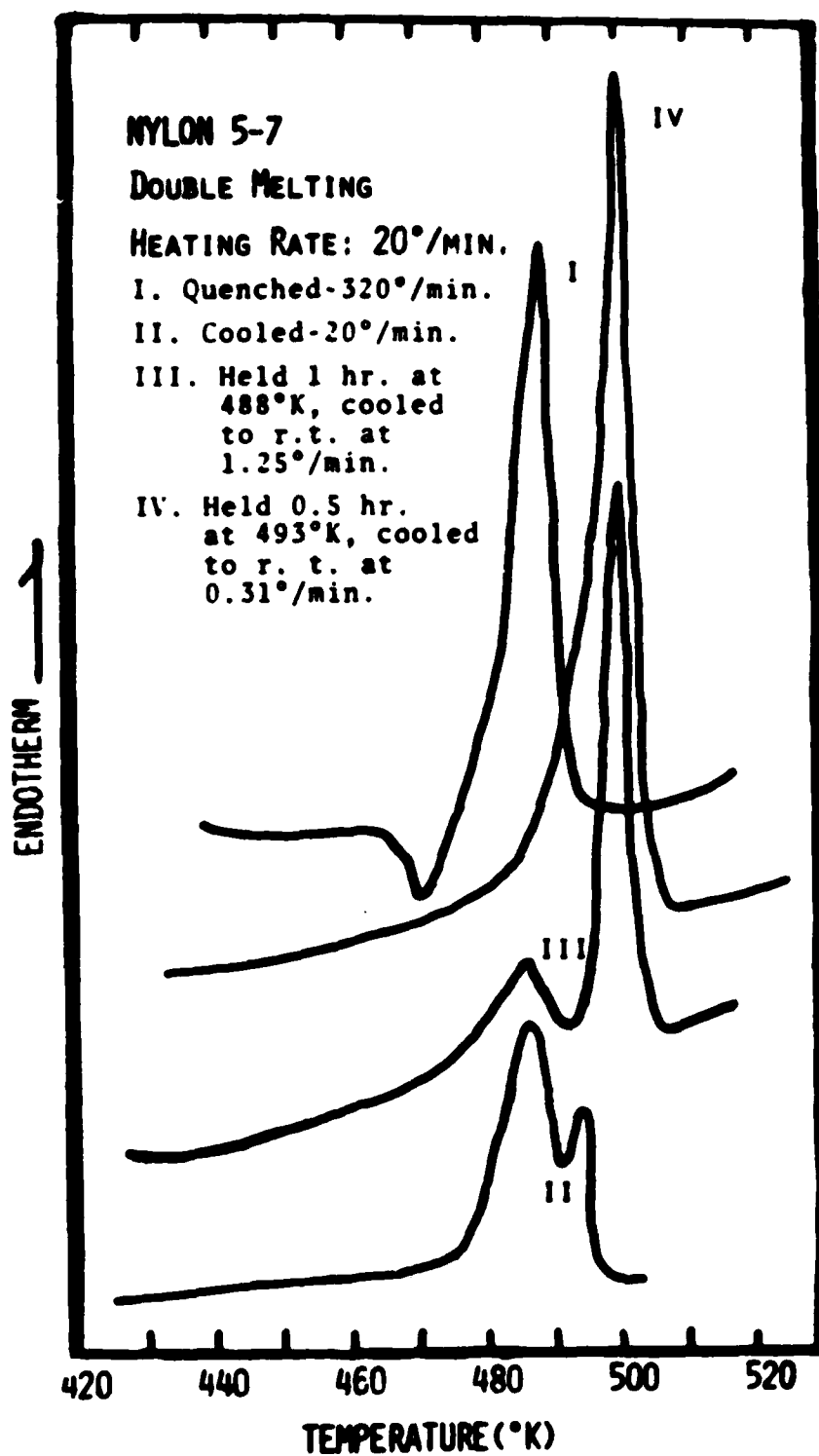


Figure 5