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Three Dimensional Orientation Measurements in Liquid-Crystalline Polymers by FT-IR ATR Dichroism

by

A. Pirnia and C.S.P. Sung

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University of Connecticut Institute of Materials Science Department of Chemistry Storrs, CT 06268

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THREE-DIMENSIONAL ORIENTATION MEASUREMENTS

IN

LIQUID CRYSTALLINE POLYMERS BY FT-IR ATR DICHROISM

A. Pirnia and C. S. P. Sung Institute of Materials Science Department of Chemistry University of Connecticut Storrs, CT 06268

ABSTRACT

FT-IR ATR dichroism technique using double-edged crystal and speciallybuilt attachment was used for a comprehensive characterization of molecular orientation in three dimension on an injection molded liquid crystalline copolyester plaque. This copolymer contains 75% of hydroxybenzoic acid (HBA) and 25% of 2,6-hydroxynaphthoic acid (HNA). Orientation functions were estimated averaging about a 10 μ thick layer as a function of the location from the gate as well as the thickness direction. Orientation profiles are similar for both HBA vibrational peak and HNA vibrational peak, which is not inconsistent with the proposed random copolymer structure by x-ray studies. Orientation, however, is found to be a strong function of the location from the gate as well as the thickness direction. In general, greater orientation has been observed on the skin than the intermediate or the core surface, with molecules lying on the surface rather than being tilted along the thickness direction.

INTRODUCTION

Comprehensive characterization of the solid state molecular structure and orientation in molded liquid crystalline polymers is necessary for better understanding of the physical and mechanical properties. One such

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example is injection molded thermotropic liquid crystalline copolyesters (LCP"s) made of p-hydroxybenzoic acid and 6,2-hydroxynaphthoic (HBA-HNA), which are known to have mechanical properties equivalent or superior to fiber reinforced engineering plastics(1). However, the physical properties of the fabricated moldings of LCP's are highly anisotropic; they vary from location to location as well as along the thickness direction often resulting in several layered structures (2,3). This type of anisotropic structure is due to the complex flow histories at each location, but to date, no theoretical prediction based on flow analysis is available. Experimentally, various techniques such as SEM, (2,4) TEM (5) and x-ray diffraction (6) have been used to characterize their solid state structure and have provided useful insights in understanding the complex morphology of this class of LCP's.

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In this paper, we report the experimental results of three dimensional orientation measurements on an injection molded thermotropic copolyester as a function of the distance from the gate and of the thickness. The technique used is FT-IR attenuated total reflection (ATR) dichroism which has been developed in our laboratory, using a special ATR crystal and ATR attachment (7,8). The important parts of this ATR set-up are a double-beveled symmetrical crystal and sample holder to allow the sample to be rotated without disassembling and reassembling operations which change the contact area between the polymer and the ATR crystal.

EXPERIMENTAL

A wholly aromatic copolyester consisting of 75 mol% p-hydroxybenzoic acid (HBA) and 25 mol% 6,2-hydroxynaphthoic acid (HNA) was used in this study. The end-gated injection molded part had the dimensions 203 mm x

- 2 -

51 mm x 2 mm. The melting point was 300° C and the material was processed at a melt temperature of 305° C with a mold temperature of 100° C. The plaques were sectioned into 16 squares (15 mm x 15 mm) using a band saw. Pairs of these squares are mirror images of each other and were labelled from A to H as a function of distance from the gate, A being the closest to the gate. Different pairs were separated from each other by 10 mm spacing.

The skin macrolayer was studied after wiping the surface with acetone. The core was reached with an end cutter on a milling machine from the opposite skin macrolayer. Subsequently, the sample was milled from the core side to reach the intermediary region. The position of the core and the intermediary region were 50% and 25% of the thickness respectively from the studied skin macrolayer. All surfaces were wiped with acetone and the edges smoothed out with a scalpel for intimate contact with the crystal.

FT-IR spectra was obtained with a Nicolet 60 SX instrument. A liquid nitrogen cooled MCT-A detector was used. Groups of 50 scans were co-added for a total of 200 scans at a spectral resolution of 4 cm⁻¹.

RESULTS AND DISCUSSION

Fig. 1 shows representative FT-IR ATR spectra obtained from the surface of injection molded LCP's. It represents the four spectra from the core of the A section (closest to the gate), obtained by rotating the sample (A_{TEX} , A_{TEY}) as well as the polarizer (A_{TMX} , A_{TMY}) at 45° incident angle. The following equations have been used to estimate three spacial absorbances, A_x , A_v and A_z from the four ATR intensities.(9)

$$A_{\text{TEX}} = \alpha A_{\chi} \tag{1}$$

$$A_{TMX} = BAy + \gamma A_{Z}$$
(2)

$$A_{\text{TFY}} = \alpha A y \tag{3}$$

$$A_{\text{TMY}} = B A_{\chi} + \gamma A_{Z}$$
 (4)

- 3 -

We define X, Y and Z as the flow direction, transverse to the flow and the thickness direction, respectively. In the above equations, α , β and γ are constants determined by the refractive indices of the sample and the reflection crystal(9). The refractive index of this copolymer was estimated by simple addition of functional group contribution, (10) to be 1.67. Based on this rough estimate, α β γ are calculated to be 59.10, 2.31 and 115.74. For our analyses, we used three well-separated IR bands at 1504 cm⁻¹, 1470 cm⁻¹ and 1410 cm⁻¹, which are known to be parallel bands associated with the aromatic ring vibration in the polymer (11).

Of these three bands, one at 1470 cm^{-1} is only due to the HNA component while the other two bands are mostly due to HBA with slight contribution from HNA.(12) This assignment has been confirmed in our laboratory by transmission IR spectra of homopolymers (HBA or HNA) via KBr pellet method.

Orientation functions $F_{\chi y}$ and $F_{\chi z}$ have been calculated, by the following equations:

$$F_{xy} = \frac{D_{xy} - 1}{D_{xy} + 2}$$
(5)
$$F_{xz} = \frac{D_{xz} - 1}{D_{xz} + 2}$$
(6)

where $D_{xy} = A_x/A_y$ and $D_{xz} = A_x/A_z$.

We also assumed the angle between the transition moment vector for the three bands and the chain axis to be zero, in deriving Eqn. (5) and (6). The penetration depth under our experimental conditions are about 10 μ , at the three bands, assuming a perfect contact.

Fig. 2 illustrates the orientation functions F_{xy} and F_{xz} as a function of distance from the gate for the core macrolayer based on the band at 1410 cm⁻¹. F_{xz} is always greater than F_{xy} .

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Fig. 3 and 4 illustrate the orientation profile, for the band at 1470 cm^{-1} , both as a function of the distance from the gate and of the thickness (skin, intermediate and core of the plaque). Between core, intermediate and skin, we connected the orientation profiles by smooth lines.

From F_{yy} curves in Fig. 3, the following trends are observed:

(a) In the skin layer, molecules are more oriented along the flow (x) direction in comparison to the transverse to the flow, even though its extent is a strong function of the location from the gate. The large orientation value at position G may be due to the pronounced fountain flow (13), while little orientation is observed near the end of the closed mold (position H).

(b) In the core layer, smaller orientation along the flow direction is observed, especially in the middle locations (C to G) where the orientation even appears to be slightly transverse to the flow. Similar transverse orientation has been observed at the core of another HBA-HNA copolymer (2). Intermediate regions give values between the skin and the core.

The other bands at 1410 cm⁻¹ and 1504 cm⁻¹ (mostly due to HBA) show very similar profiles as Fig. 3. These results are not inconsistent with the suggested structure of the random copolymer by x-ray studies. (6)

From F_{xz} curves in Fig. 4, orientation along the flow is shown to be quite high in comparison to the thickness direction, regardless of the position along the thickness. This result means that even at the core where these rigid molecules are randomly oriented in the xy plane (cross-section plane) as inidcated by low F_{xy} values, they tend to lie flat on the plane rather than tilted toward the thickness direction. This tendency was also observed in shearinduced LCP's (14).

- 5 -

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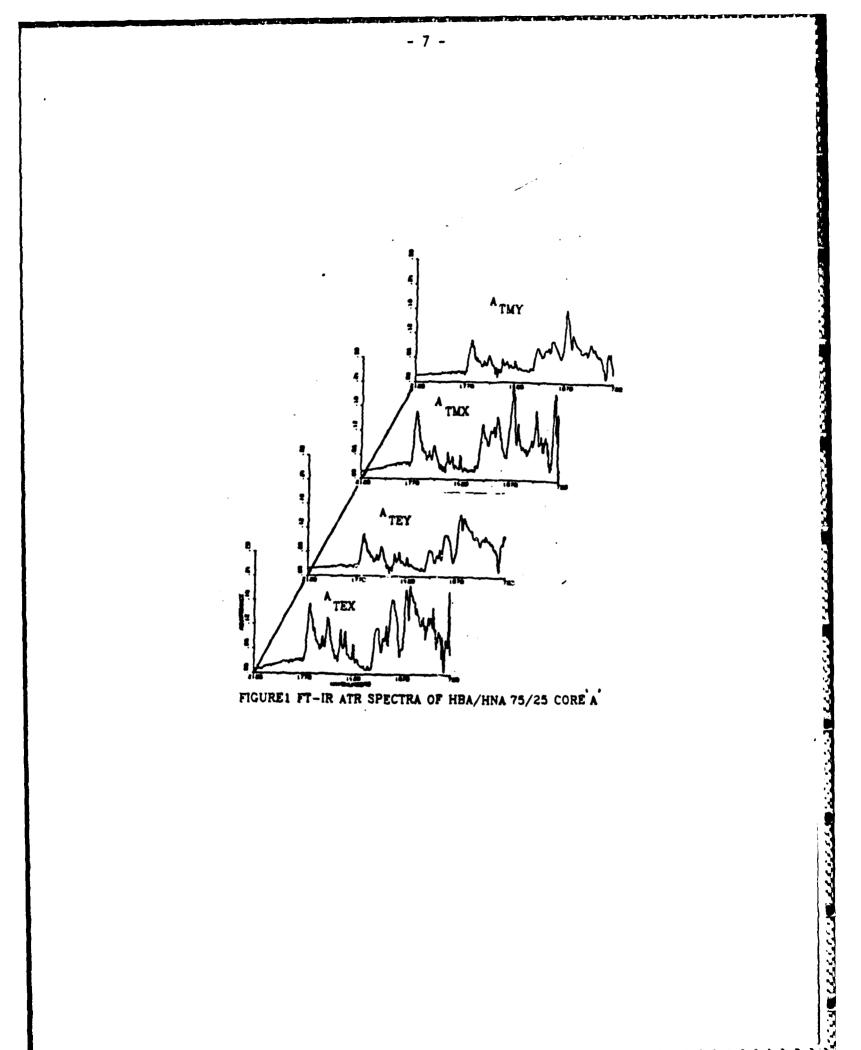
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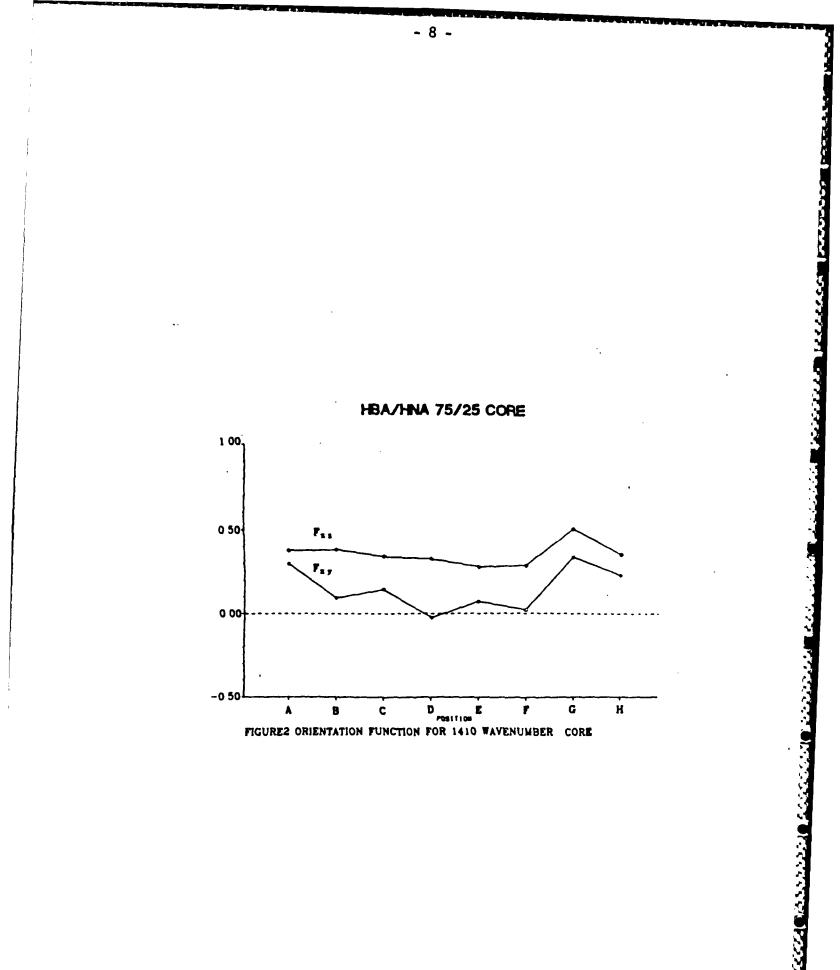
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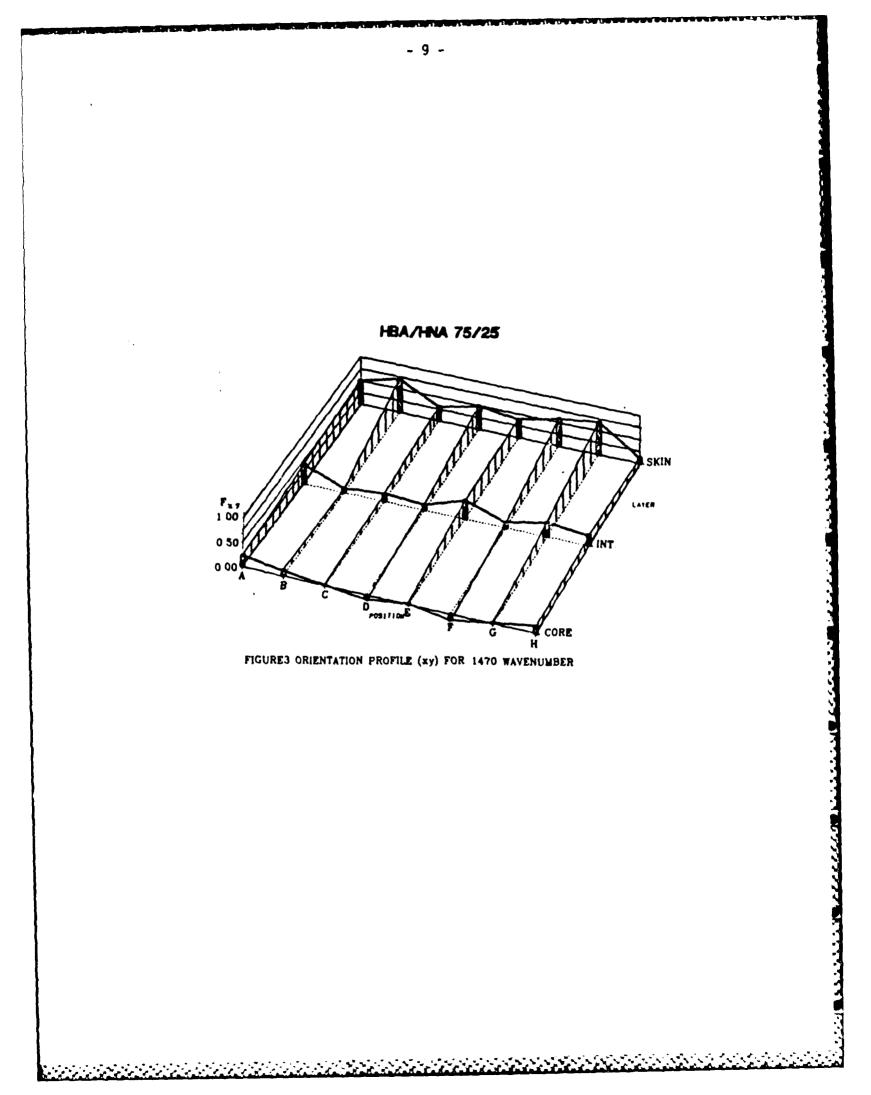
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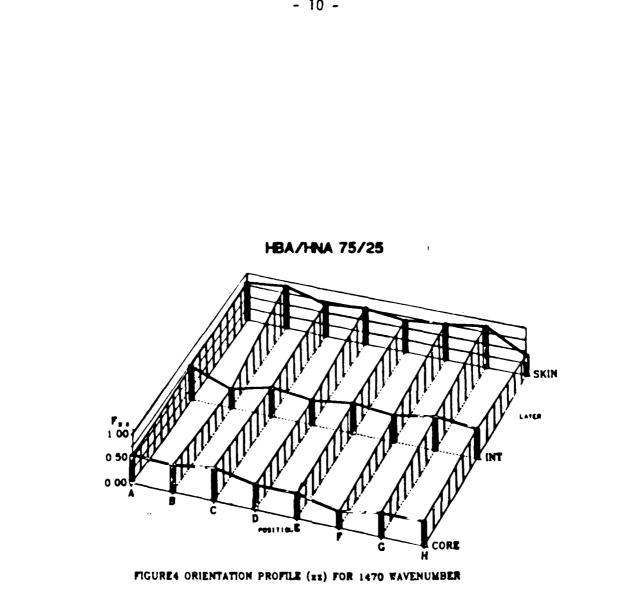
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