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The Effect of Ultrasonic Waves on the Structure of Polyester Fibers (PET)

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Synopsis

The effect of ultrasonic waves at constant intensity of 20 W/cm^2 and frequency of 2×10^6 Hz on the microstructure of polyester fibers has been examined. Crystallinity, crystallite size, total orientation, and crystallite orientation have been estimated. It has been found that when subjected to different ultrasonic tests, the degree of fiber orientation decreases and crystalline perfection improves. The changes in crystallinity and crystallite size proved to be particularly difficult to determine with certainty.

INTRODUCTION

The present work constitutes part of a study on the ultrasonic field effect on the microstructure of fiber material. Polyamide (PA-6) fibers were subjected to ultrasonic waves.[1,2] The results obtained made it possible for us to try to delineate the mechanism of ultrasonic wave propagation in PA-6 fibers under given experimental conditions.

It has been found that at the same direction of the ultrasonic wave propagation in relation to fiber axis, the mechanism of conduction of vibration in the fiber material depends on its orientation. This phenomenon is accompanied by changes both in crystalline and noncrystalline regions. A fuller explanation of the problem requires wider examination of various fibers under different experimental conditions.

The present results of ultrasonic treatment of PET fibers are a further step on this way.

EXPERIMENTAL

Continuous raw polyester fiber yarn of draw ratios of 1;4;4,6, without any TiO_2, produced by the "Elana" Works (Poland) were the material under investigation. A MSGU-581 generator (made in Poland) was used for generating ultrasonic waves.

The ultrasonic treatment was carried out in a water bath, the frequency being $f = 2.0$ MHz and the intensity 20 W/cm^2. The initial mass of the samples and the volume of aqueous medium were constant in each experiment.

The fibers remained in a loose state while being treated with ultrasonic waves. The modifications shown in Table I were applied.

TABLE I
Conditions of Ultrasonic Treatment of PET Fibers

Sample no.	Arrangement of fibers in the samples	Time of ultrasonic treatment, sec	Temperature of bath, °C
1	fibers parallel to each other, the fiber bundle perpendicular to the wave direction	300	20
2	same as no. 1	3600	20
3	same as no. 1	300	80
4	fibers in random directions	3600	20

The physical microstructure of the fibers was analyzed by examining the internal orientation as well as the crystallinity and the crystalline structure of the polymeric material. The following magnitudes were measured:

1. The total orientation factor f_0,

$$f_0 = \frac{(n_\parallel - n_\perp)_w}{(n_\parallel - n_\perp)_k} \cdot \frac{d_k}{d_w} \tag{1}$$

where $(n_\parallel - n_\perp)_w$ = birefringence, determined in the striated field of the in-

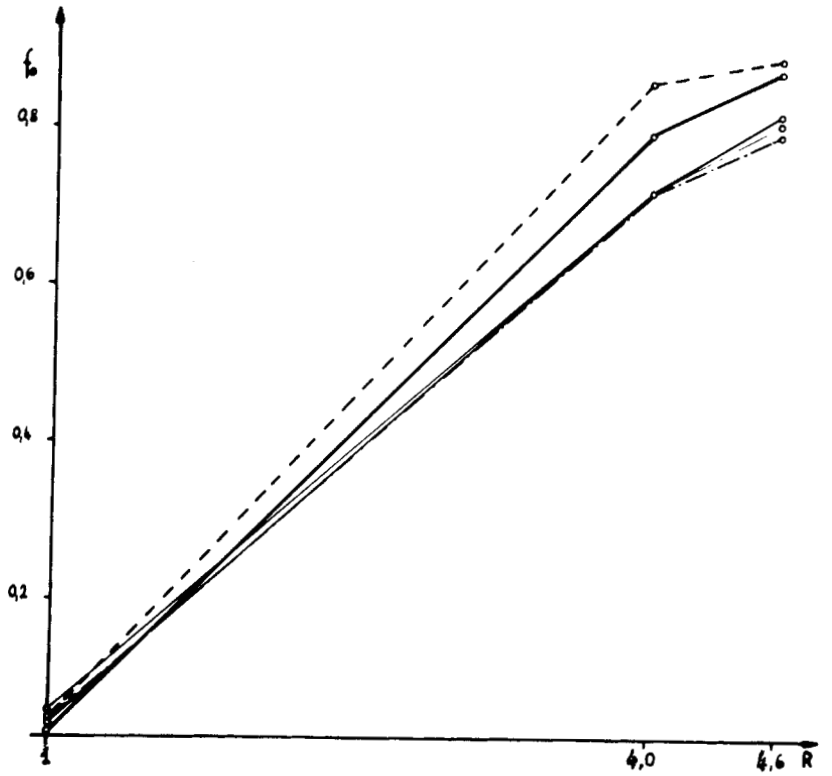

Fig. 1. Total orientation of polyester fibers treated with ultrasonic waves as a function of draw ratio: (—) case 1; (—) case 2; (—) case 3; (- - - -); (- - - -) untreated.

terference polarizing microscope[3]; d_w = density, measured in the gradient column filled with carbon tetrachloride and toluene[4]; $(n_\parallel - n_\perp)_k = 0.220$, birefringence of an ideal fiber,[5] explained in reference 9; and $d_k = 1.455$, density of crystalline fibers.[5]

2. The azymuthal half-angle was attained with the aid of the oblique-angle x-ray diagrams[6] from the formula

$$\cos \zeta_{H(003)} = \sin^2 \varphi \cos \delta_H + \cos^2 \varphi \tag{2}$$

where δ_H stands for the half-opacity angle of the (003) plane; and φ is the angle of inclination between the sample and the direction of the radiation beam.

3. Crystallinity ratio

a. $X_{d(\text{mass})}$ determined by means of the density technique:

$$x_d = \frac{(d - d_a)\, d_k}{(d_k - d_a) d_a} \tag{3}$$

where d_a, the density of amorphous fiber; assumes the value of 1.335[5]

b. X_r is obtained by using x-ray measurements and from the following equation[4]:

$$x_r = \frac{\Sigma |u - A|_{2\theta}}{\Sigma |c - A|_{2\theta}} \times 100. \tag{4}$$

A full description of this method is given in reference 7; $(U - A)$ shows the intensity difference of radiation passing through the examined sample and

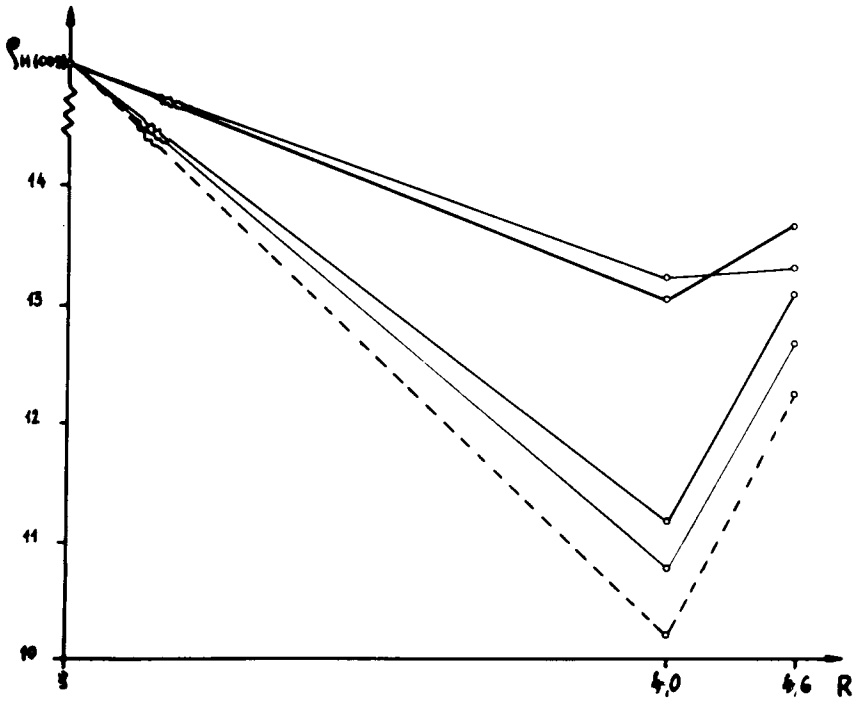

Fig. 2. Azymuthal half-angle of polyester fibers treated with ultrasonic waves as a function of draw ratio.

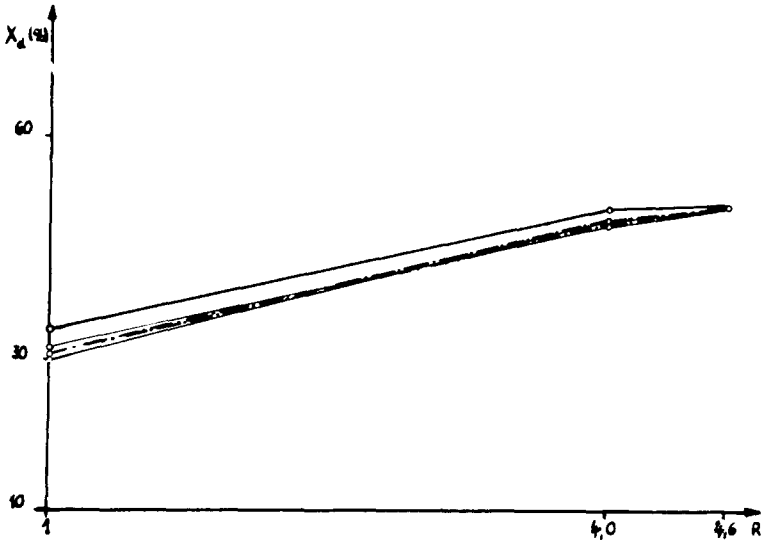

Fig. 3. Degree of crystallinity (using density measurements) of fibers treated with ultrasonic waves as a function of draw ratio.

the amorphous one, $(C - A)$ stands for the intensity difference of radiation passing through the crystalline sample and the amorphous sample.

4. Crystallite size

a. The long period l was obtained from the diffraction patterns made with a Geigerfleks Rigaku-Denki apparatus:

$$l = \frac{\lambda}{2 \sin \theta} \tag{5}$$

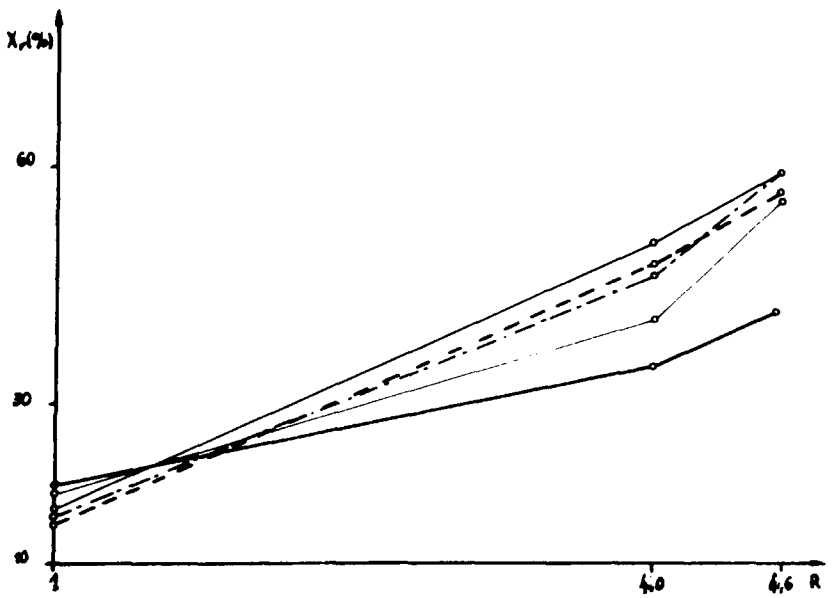

Fig. 4. Degree of crystallinity (using x-ray measurements) of polyester fibers treated with ultrasonic waves as a function of draw ratio.

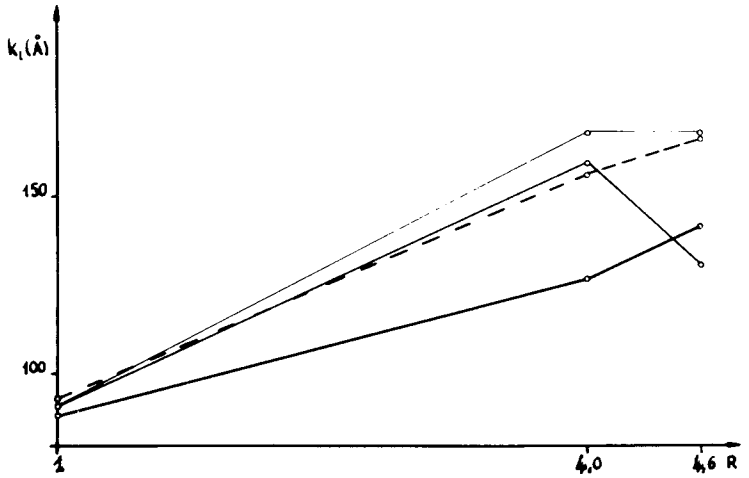

Fig. 5. Long period of crystals of polyester fibers treated with ultrasonic waves as a function of draw ratio.

where λ is the wavelength of CuK_α radiation equal to 1.54 Å, and θ denotes the reflection angle.

b. The lateral size was worked out from Debye diagrams made with a camera 57.3 mm in diameter, the diameter of the sample being 0.5 mm. The paratropic reflections (100) and (010) were analyzed by finding the size index D perpendicular to these planes from the Scherer formula:

$$D = \frac{\lambda}{l_{(\mathrm{wkl})} \cos \theta_{(\mathrm{hksl})}} \tag{6}$$

where b_{hkl} denotes the half-width of the spectral line expressed in radians. It should be noted that there is no correction used for the half-width and for the operating factors either, as the measurement conditions were identical in all cases and exposed to the same error.

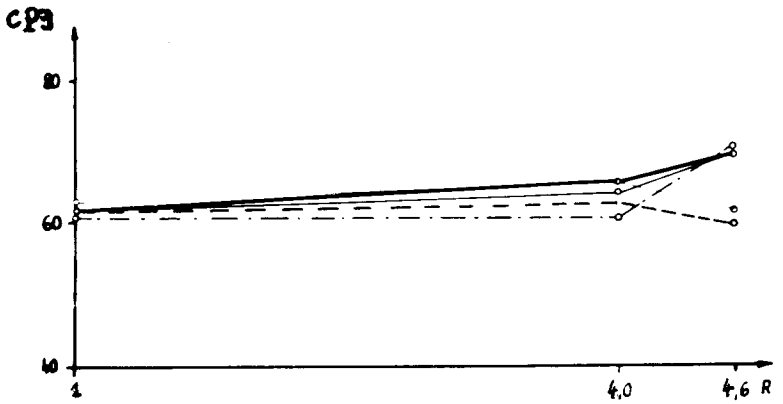

Fig. 6. Crystalline perfection index (CPI) of polyester fibers treated with ultrasonic waves as a function of draw ratio.

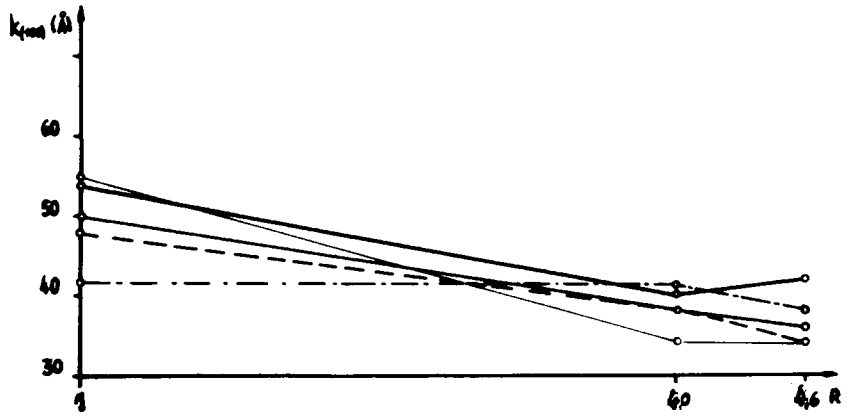

Fig. 7. Size of crystallinity perpendicular to the (100) planes of polyester fibers treated with ultrasonic waves as a function of draw ratio.

5. The crystalline perfection index was found from the Dismore and Statton[8] formula:

$$W_D = \frac{\frac{d_{(100)}}{d_{(010)}} - 1}{0.655} \times 100 \tag{7}$$

where $d_{(100)}$ and $d_{(010)}$, the interplanar spacings were calculated from Debye diagrams under conditions described under 4(b).

The value 0.655 for PET used in the denominator of the formula was calculated from the lattice parameters for PET according to the procedure described in reference 8.

RESULTS

The results obtained are shown in Figures 1–8 as functions of the draw ratio of the fibers and of ultrasonic treatment to which they were subjected.

The ζ_H angle and f_0 factor values (Figs. 1 and 2) reveal a decrease in material orientation in each case of ultrasonic treatment. The changes in general orientation are almost identical in all cases. The changes in ζ_H are most evident in cases 3 and 4 (Fig. 2).

Referring to Figure 2, the differentiation occurring in the treated original samples has not distorted the effect on the material of an ultrasonic field, which is testified to by the magnitude of δ_H.

The crystallinity of the material as estimated by density does not show any perceptible differences (Fig. 3), but some changes are seen in the case when x-ray measurements were made (Fig. 4). There is a tendency to decreased crystallinity at the first stage of ultrasonic treatment (case 1). It becomes more evident as the amount of energy, e.g., heat, is increased (case 3, Fig. 4). There is a slight increase in crystallinity at prolonged ultrasonic treatment when the fibers were perpendicular to the ultrasonic waves (case 2).

The long period of the orderly range shows a clear decrease as it is subjected to ultrasonic and heat treatments simultaneously (case 3, Fig. 5).

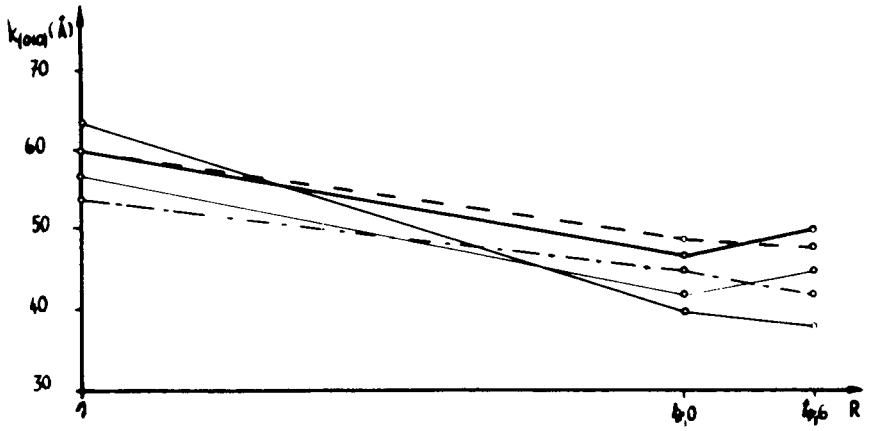

Fig. 8. Size of crystallinity perpendicular to the (010) planes of polyester fibers treated with ultrasonic waves as a function of draw ratio.

On the other hand, the remaining samples reveal rather small differences as compared the original values (Fig. 5). For technical reasons, the values for case 4 have not been indicated. The lateral sizes of the orderly range have resulted in an ambiguous system of changes.

There is only some regularity in the results with reference to the sizes perpendicular to the planes (010) (Fig. 8), viz., there is a tendency to an increase in crystallinity in this direction.

The crystalline perfection index of the orderly range shows some differences regarding the draw ratio functions of the treated fibers (Fig. 6).

There is no difference in the crystalline perfection index values for drawing up to the draw ratio of 4.0 independently of the kind of ultrasonic treatment, while an increase in the crystalline perfection index can be observed in all the cases of ultrasonic treatment in samples of draw ratio 4.6.

CONCLUSIONS

1. Ultrasonic treatment produces changes in highly oriented PET fibers. The changes tend to decrease the fiber orientation and depend on the conditions of treatment.
2. The changes of crystallinity and crystallite size cannot be explained in an unambiguous manner.
3. The crystalline perfection of the lattice is improved by subjecting it to ultrasonic treatment.
4. The difference of the mechanism of propagation of ultrasonic wave through the crystallite region perpendicular to the (100) and (010) planes (Figs. 7 and 8) is evident.
5. Conclusions 1 and 4 confirm the results of the work referred to in the introduction and seem to indicate a general regularity of the effect of ultrasonic tests on PET fibers, whereas conclusions 2 and 3 show a divergence from the previous results.

In order to clarify them, it is necessary to find a further explanation of the mechanism of changes taking place at the crystalline phase exposed to ultrasonic tests.

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