MICROWAVE RADIATIONS FOR HEAT-SETTING OF POLYESTER FIBERS

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ABSTRACT

The use of radio and microwave frequency is gaining importance for industrial applications such as heating, drying, and other processing. The most important advantage of using microwave is that it is non-contact or localized heating and the heat is produced within the material. This can be much more effective than indirect heating where the heat propagation is by heat conduction through the material. We have been investigating the influence of microwave radiation on different fibers for the last few years. In the present investigation we used microwave frequency of 2450 MHz to investigate its effect on polyester fibers. The polyester fibers were heat set in air as well as a liquid, which acted as a lossy substances. The liquid was chosen on the basis of earlier experiments, which showed the maximum effect. A comparative study was also carried out using conventional heating in silicone oil.

Using the method of X-ray Diffraction (XRD) we calculated the changes in % crystallinity and orientation. It was found that as the time of treatment under microwave radiation increased from 15 sec. to 120 sec. the order factor was found to increase from 0.32 to 0.71.The crystalline orientation as determined from the azimuthal scan was also found to increase. Such structural changes can be highly beneficial for the processing of fabric in industry. The microwave radiation process is fast, reliable and energy saving.

INTRODUCTION

PET is a low cost, high performance semi-crystalline thermoplastic polymer with excellent properties. PET is used in textiles, reinforcement of tyres and rubber goods, and food and beverage packaging [1]. However, for industrial applications PET should have superior mechanical properties and dimensionally stable structure. In other words the fiber should attain adequate crystallinity and orientation for its use in industrial application. Crystallinity and orientation are achieved by drawing and heat setting. In this study, we report the effect of microwave irradiations on yhe crystallinity of PET fibers.

Microwave irradiation has been successfully applied to a number of classical reactions[2-6]. The most important advantages of microwave irradiations are that it is a non-contact, localized, rapid, uniform, energy saving and pollution free heating process. The textile industry has extensively investigated uses of microwave energy for heating, drying, dye fixing, and finishing[7-10]. The Bombay Textile Research Association (BTRA) has also studied the applications of microwave energy in various areas of textile processing[11-12]. Heating materials using microwave radiations is limited to substances that can absorb this radiation and in turn dissipate this excess energy in the form of molecular oscillations. Materials, which absorb microwave radiations, are called “lossy”. Unfortunately, textile materials are not heated to any great degree when so irradiated, and must be surrounded by a medium (lossy) capable of generating heat when exposed to microwaves. In our earlier studies[13] we describe selection of lossy and their importance. The study mentioned in Reference 13 covers about 8 different liquids and on the basis of heat generated, glycerol was found to be the best. In the present work we exposed polyester filament yarn to microwaves for different time intervals and characterized them by X-ray diffraction (XRD). The results are compared with samples heat set in silicone oil for same time of intervals.

EXPERIMENTAL

Materials and Sample Preparation

The starting material was a partially oriented (POY) multifilament PET yarn of 126 denier, 34 filaments. This sample was supplied by M/s. Reliance Industries Ltd., and referred to as the parent. Experiments were conducted in a multiwave LG Microwave Oven at 2450 MH frequency and 850 W power output.

These PET-POY yarns were stretched to a draw ratio of 1.6x using a small rectangular stretch frame at 85°C in a water bath. The rate of stretching was 4.7 m. /min. which was kept constant through out the stretching process for all samples. These drawn yarns were referred to as the control sample. The drawn yarns were removed from the stretch frame and wound immediately on a test tube manually, to minimize the relaxation of molecular chains. This test tube was immersed in another test tube containing lossy liquid and exposed to microwave radiations for time intervals of – 15, 30, 60, 90, 105 and 120 seconds. For each sample fresh lossy was used and the amount of lossy was kept constant. The sample after microwave treatment was taken out and cooled to room temperature. After cooling, the samples were washed with water to remove lossy and dried in air. Another set of samples was prepared in silicone oil at 180 °C by conventional heating for same time intervals. After cooling, silicone oil from the surface was removed by using blotting paper, then washed with carbon tetrachloride (CCl_4) and dried in air. Conventional heating was done in an electrically heated silicone oil bath. Whenever we use microwave heating the liquid used was glycerol. For conventional heating the liquid used was silicone oil.

X-ray Diffraction Studies

The wide angle x-ray diffraction (WAXD) information was obtained on a Philips, XRD PW-1720 unit fitted with a texture goniometer. Samples were scanned using Nickel-filtered Cu Kα, X-rays of wavelength 1.5418 A°, generated at 35 kV / 20 mA current. The multifilament yarn samples were arranged parallel to each other and mounted on the sample holder. Equatorial scan was obtained from 2θ = 10° to 35 °. WAXD measurements were used to calculate the crystal size, lateral order (LO) or crystallinity and crystallite orientation angle for samples exposed to mw radiations and heat-set in silicone oil. For POY yarns where single broad peak occurs, WAXD was used to calculate the crystallinity index(CI).

Lateral Order Factor

The LO factor, which can be related to crystallinity, perfection and size of crystallites, was calculated from the resolution factor (RF) by using the equation [14]

$$RF = \frac{m_1 + 2m_2 \ldots\ldots\ldots\ldots + m_{n-1}}{h_1 + h_2 \ldots\ldots\ldots\ldots + h_n} \quad (1)$$

where m_1, m_2, mn are heights of minima and h_1,h_2 hn. are heights of maxima from the base- line. For PET the resolution factor can be written as

$$RF = \frac{m_1 + 2m_2}{h_1 + h_2 + h_3} \quad (2)$$

where m_1 and m_2 are the minima between the planes (010) and (110) and between the planes (110) and (100) respectively; h_1, h_2, h_3 are the observed maxima diffraction peaks of the planes (100), (110), and (010) respectively.

Order Factor (OF) was calculated by subtracting the RF from one.

$$O.F. = 1 - RF \quad (3)$$

Crystallinity Index

In the case of amorphous samples (POY yarn) where a single broad peak occurs the crystallinity index was calculated from the fixed count measurements at 28.6° and 26° using the equation [15].

$$Crystallinity\ Index = \frac{Intensity\ at\ 26° x\ 100}{Intensity\ at\ 28.6°} \quad (4)$$

Crystallite Orientation Angle

In the case of orientation studies the measurement of molecular orientation in the crystalline region was carried out by recording the Azimuthal intensity

distribution of equatorial plane (100). This was accomplished by fixing the glancing angle 2θ at major peak (100) and rotating the fiber bundle in a plane perpendicular to the direction of the X-ray beam[16] The width of the peak at half maximum intensity was calculated to find the crystalline orientation angle.

Crystallite Size

The half-width of the crystalline plane (100) was considered for the crystal size calculations from the Scherer's equation [17].

$$L_{(hkl)} = \frac{k\lambda}{\beta \cos\theta} \qquad (5)$$

where k is constant and a value of 0.9 was considered for our calculations, λ is the wavelength of the radiation used (1.542A^0), β is the half-maximum breadth in radians, and θ is the Bragg's angle.

RESULTS AND DISCUSSION

The experimental results obtained in the present study are presented and discussed below. *Figure 1* shows the XRD patterns of the control, the samples exposed to microwave radiations and heat-set in silicone oil. Order factor, orientation angle and crystal size, calculated on this basis, for parent and drawn polyester (control) samples are given in *Table I*

The presence of single broad X-ray peak and higher value of orientation angle indicates that the parent sample is amorphous and partially orientated. In case of the drawn sample the lower value of orientation angle with small improvement in order factor could be due to the drawing operation, which was carried out at 85°C slightly above the glass transition temperature. X-ray data for samples exposed to microwave radiations and heat-set in silicone oil for different time intervals are given in *Tables II and III* respectively.

Order Factor

The dependence of X-ray order factor on the time of treatment with microwave radiation and silicone oil is given in *Table II and III* respectively. In both sets of samples the order factor increases with increasing time of treatment.

TABLE I. Order Factor, Orientation Angle and Crystal Size calculated from X-ray diffraction for parent and control sample.

Sr. No.	Sample	X-ray data		
		Order factor/ (Crystalline Index)	Orientation Angle (deg.)	Crystal Size (A°)
1.	PET-POY (Parent)	(210)	24.7	-
2.	Drawn PET-POY (Control)	0.29	12.1	21.44

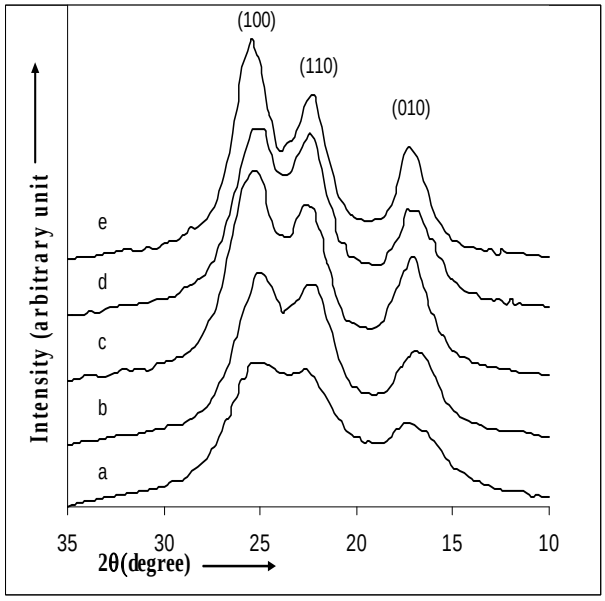

FIGURE 1 XRD patterns of polyester fibers, (a) drawn(parent sample), (b) heat-set in s. oil for 15 sec at 180°C ., (c) MW treated for 15 sec., (d) heat-set in s. oil for 60 sec. at 180°C and (e) MW treated for 60 sec.

Table II. Order Factor, Orientation Angle and Crystal Size calculated from X-ray diffraction for PET-POY control samples exposed to microwave radiations in Glycerol.

Sr. No.	Time of Treatment (seconds)	X-ray data		
		Order Factor	Orientation Angle (deg.)	Crystal Size (A°)
1.	15	0.32	12.0	22.61
2	30	0.53	10.5	25.45
3	60	0.64	7.9	40.74
4	90	0.68	7.1	40.74
5.	105	0.71	7.3	37.1
6.	120	0.68	6.0	37.05

It may be noted that the increase in order factor is observed initially for 15 seconds of treatment compared to the control sample but the increase in the case of silicone oil treatment is higher compared to the microwave treatment. This could be due to the fact that in the case of silicon oil the samples were kept directly at 180°C and microwave treatment was started at room temperature. From the data it is observed that in the case of the microwave treatment there is considerable increase in the order factor observed with time which increased from 0.32 to 0.71 for the time interval of 15 to 90 seconds. In the case of samples heat set in silicone oil there is marginal increase in order factor observed i.e. from 0.45 to 0.52 for the time interval of 15 to 90 seconds. In case of samples exposed to microwaves rapid increase in order factor is observed up to 90 seconds and then it is constant for higher time of exposure. The initial rapid increase may be due to enhancement in primary crystallization by microwave radiations, which is followed by secondary crystallization. Major structural reorganization is known to take place during primary crystallization, during which crystals grow around nuclei that already exist in drawn PET fiber[15].

Crystallite Orientation Angle

Data for the effect of microwave radiations on crystallite orientation for different durations of exposures are presented in *Table II and in Table III* for heat-setting with silicone oil. A decrease in orientation angle was observed with an increase in time for both sets of samples, which indicates that the crystalline orientation has increased. However, the level of increase in crystalline orientation in the case of samples exposed to microwaves is higher compared to the sample heat-set in silicone oil for the same time interval. The orientation angle decreased from 12.1 to 7.1 when the sample was exposed to microwaves for 90 seconds. While in the case of silicone oil it is 10.3 for the same time of interval.

Table III. Order Factor, Orientation Angle and Crystal Size calculated from X-ray diffraction for PET-POY control samples heat-set in silicone oil at 180°C.

Sr. No.	Time of Treatment (seconds)	X-ray data		
		Order Factor	Orientation Angle (deg.)	Crystal Size (A°)
1.	15	0.45	12.4	29.10
2	30	0.48	11.2	30.33
3	60	0.50	11.6	31.33
4	90	0.52	10.3	31.34
5.	120	0.51	12.0	29.00

Crystal Size

Crystal size was determined from the (100) plane for both sets of samples. The crystal size increased with an increase in time interval up to 60 seconds and afterwards more or less remained constant for longer time intervals in both cases. V.B. Gupta and et al[18] stated that defects in crystals can diffuse out with an increase in heat-setting time, and this lead to a reduction in the crystal width. However, the level of increase in crystal size is also very high in the case of samples exposed to microwave radiations compared to samples heat-set in silicone oil for same time interval. In fact the crystal size of 40.7A° attained by the sample exposed to microwave radiations for 60 seconds is quite high and is beyond expectation.

To confirm the effect of microwave radiations on the structure of PET we have carried out a study where the sample was heat set for 60 seconds in the same lossy by using conventional heating. The results compared with the results of the sample exposed to microwave for 60 seconds using the same lossy are given in *Table IV.*

Table IV. Order Factor, Orientation Angle and Crystal Size calculated from X-ray diffraction for PET-POY control samples exposed to microwave and heat-set in same lossy (Glycerol) by conventional heating for 60 seconds.

Sr. No.	Time of Treatment (seconds)	X-ray data		
		Order Factor	Orientation Angle (deg.)	Crystal Size (A°)
1.	Exposed to Microwaves	0.64	7.9	40.74
2.	Conventional heating	0.51	12.0	31.34

It is observed from the *Table IV* that the order factor, crystalline orientation and crystal size obtained by the sample exposed to microwaves are very high compared to the sample when heat-set in the same lossy by conventional heating for the same time interval. This shows that the microwave radiations could generate the desired higher crystalline orientation, crystallinity level and crystal size. It therefore appears that microwave radiation in conjuction with lossy material has accelerated action on orienting the molecular chains of polyester. Thus, the potential of microwave radiation in improving the fine structure parameter is clearly observed in this investigation. However, more study is needed before this treatment can be recommended for industrial use.

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