The Effect of Structural Features on Mechanical Properties of Loose Optical Fiber Poly(butylene terephthalate) Tubes

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ABSTRACT: Results of a study on the modifying mechanical properties of loose optical-fiber poly(butylene terephthalate) (PBT) tubes, produced during the standard industrial extrusion process, show that heat treatment make the structure of their material to change. The study comprised measurements of mechanical strengths properties of the tubes (tensile strength, compression strength, kinking) and determination of tube material structure [by differential thermal analysis (DTA), wide angle X-ray scattering analysis (WAXD), and scanning electron microscopy (SEM)]. Results of the study allowed observation that the annealing at 70°C for 34 h of the tubes caused the crystalline α phase to increase in the tube material from ~28.5% to ~31.5% and the structure of the existing crystallites to become more perfect. This made the values of certain mechanical properties of the tubes to increase even by as much as 30%. The tubes following such thermal treatment could be used in cables exposed to heavy-duty operation in arduous environments, where a larger margin from the standpoint of mechanical properties is required. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 86: 2130–2134, 2002

Key words: crystallization; microstructure; mechanical properties

INTRODUCTION

The loose tube–optical fibers system is the major structural and functional element of the tubular optical fiber cable. The loose tube (Fig. 1) ensures, on the one hand, an adequate excess length of optical fibers, and on the other hand, it protects the sensitive fibers against external mechanical and chemical factors.

In our previous study it was demonstrated that during the standard industrial process of extruding the PBT tubes a fine crystalline α phase forms, while its relative content there amounts to ~29% (M. Ambroziak, I. Gruin, K. Zdunek, and M. Wronikowski, unpublished observations). The value is much less than the one reported in the literature[1] for the maximal content of the crystalline phase in PBT, reported to be ~66%.

A rise in the crystalline phase in polymers is known to increase their elasticity. In this connection in the study on the ready PBT tubes produced during the standard industrial PBT tube extrusion process, the latter were heated at a temperature above the glass point (T_g) of the polymer to increase the crystalline phase content in the material. The heat treatment process was expected to bring about a significant rise in mechanical properties of loose tubes.

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EXPERIMENTAL

PBT tubes extruded in an industrial process under standard conditions were used in the study. The tubes were studied prior to (initial state) and after their special thermal treatment, which consisted of heating the tubes at 70°C for 34 h, that is, at the maximum operating temperature of the optical fiber cable. The heat treatment was carried out in a thermal chamber used for the testing of optical telecommunications cables. During the heat treatment of the tubes the optical fibers and hydrophobic gel were left inside tubes, that is, in the condition in which they commonly occur. On the other hand, prior to examining the mechanical properties and structural features of the tubes, the hydrophobic gel and optical fibers were removed from the tubes.

Methods for testing mechanical strengths of loose tubes

In the available literature no standards were found for testing mechanical properties of loose tubes, with the exception of the standard related to tube kinking property, determined in the kink test.[2] In this connection, genuine techniques of testing for tensile strength, compression strength, and kinking at room temperature were developed with the use of an Instron type 1115 testing machine. Results of the mechanical testing were worked out statistically with determination of the mean values of a given physical quantity and standard deviation of the mean. The reason for that

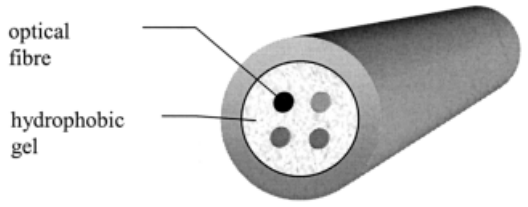

Figure 1 The loose tube–optical fiber system.

was that during the testing in point random errors prevailed, whereas systematic errors were well below the standard deviations.

Tensile test

The arrangement of tensile testing of loose tubes is shown diagrammatically in Figure 2(a).

Soft inserts were placed at the ends of the tubes under testing to prevent a precocious breaking of the tubes during their straining.

From the graphs obtained from the testing machine [diagrammatically visualized in Fig. 2(b)] the following parameters were read off: (a) F_e—value of the force at which plastic strain begins in the tube; (b) F_u—value of the force at break; (c) L_u—strain at break.

Relative plastic strain until break, ϵ_u (%), was found from formula:

$$\varepsilon_N = \frac{L_u}{L_0} \cdot 100\%$$

where L_u is the strain of sample upon break and L_0 is the sample length before tensioning (50 mm).

Tensile strength of loose tubes was evaluated by comparing mean F_e and F_u values and mean ϵ_u values.

Tensile tests were carried out with application of the following parameters: (a) total sample length of 80 mm, and (b) tensioning rate of 100 mm/min.

Compression test

Results of compression testing of loose optical fiber tubes were evaluated on the basis of the graphs from the testing machine obtained during application of a crosswise force on the tube [Fig. 3(a)]. The following parameters were found from the graphs [Fig. 3(b)]: (a) F_{elast}—value of the maximal elastic compressive force; (b) F_1—value of the compressive force at tube deflection of 0.15 mm; (c) F_2—value of the compressive force at tube deflection of 0.45 mm; (d) $F_{\text{perm}} = (F_1 + F_2)/2$—mean value of the compressive force that causes permanent strain.

To assess the compressive strength of tubes, mean values of forces F_{elast} and F_{perm} were compared.

The compression tests were carried out using the following parameters: (a) total sample length of 50 mm, and (b) compressing rate of 0.5 mm/min.

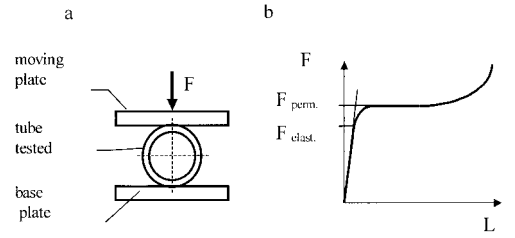

Figure 3 (a) Diagram of the setup for the compression test of loose tubes. (b) A schematic graph obtained from the testing machine, where F is the compressive force and L is the tube deflection.

Kink test

The kink test consisted in narrowing the diameter of a previously formed loop from the tube, which was secured by means of the so-called tight guide [Fig. 4(a)]. A tight guide is an element that should not affect the kink test (i.e., it should have a sufficiently small weight so as not to put much load on the tube).

From graph of Figure 4(b) the following parameters were found: (a) F_{max} = the maximal force achieved during kink test, and (b) L_{max} = displacement of the machine beam until the break of tube wall (displacement to F_{max}).

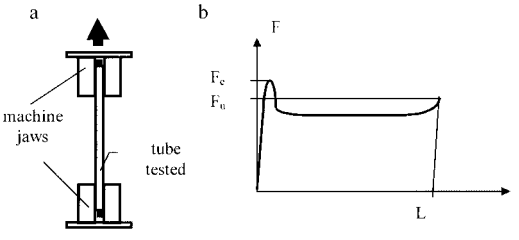

Figure 2 (a) A diagram of the setup for tensile test of loose tubes. (b) A schematic graph obtained from the mechanical testing machine, where F is the tensioning force and L is the strain of the tube.

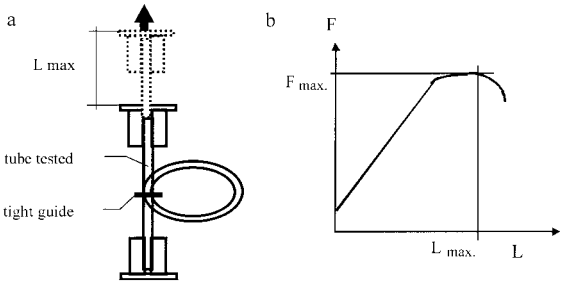

Figure 4 (a) A diagram of the setup for the kink test of loose tubes. (b) A diagrammatic graph obtained during the tube loop tightening, where F is the kinking force and L is the displacement of the testing machine beam.

TABLE I
Results of a Study of Mechanical Properties of the Tubes before and after Their Heat Treatment at 70°C for 34 h

Property		After tensile test			After compressive test		After kinking test	
Tube condition		F_e [N]	F_u [N]	E_u [%]	F_{elast} [N]	F_{perm} [N]	F_{max} [N]	L_{max} [m]
Initial	x	63.8	83.4	373	242.3	352.2	0.6847	0.0426
	S_x	±2.9	±6.9	±11	±10.8	±2.0	±0.0127	±0.0009
On heating at 70°C for 34 h	x	74.6	92.2	406	251.1	392.4	0.9977	0.0585
	S_x	±2.0	±5.9	±11	±9.0	±3.9	±0.0304	±0.0006

Notation: x—mean value of a given physical quantity, S_x—mean standard deviation, F_e—value of the force at which permanent strain in tensioning develops, F_u—value of the force at break of the tube in tensile test, E_u—relative strain at break in tensile test, F_{elast}—value of the maximum elastic force in compression test, F_{perm}—mean value of the compressive force that causes permanent strain, F_{max}—maximal value of the force achieved in kink test, L_{max}—displacement of the machine beam until tube wall break during the kink test.

The kink test was carried out at the following parameter values: (a) total sample length of 400 mm, (b) loop diameter lowering rate of 200 mm/min, and (c) initial opening of machine jaws of 50 mm.

An analysis of results of kink test consisted in comparison of the mean F_{max} and L_{max} values. Large L_{max} value corresponds to a small tube loop.

In the evaluation of structure of the loose tubes studied the DTA, WAXD, and SEM techniques were used as described by Ambroziak et al. (unpublished).

RESULTS AND DISCUSSION

In Table I are gathered values of mechanical properties obtained before and after a prolonged heating of tubes at 70°C for 34 h.

Based on the tests results obtained it can be said, that the heating of PBT tubes at 70°C for 34 h made their tensile strength rise (the F_e value increased by ~15%) and the strain to break to increase by ~10%. Also, compressive strength of tubes increased (the F_{perm} value rose by ~10%). The tube heat treatment also caused the mean L_{max} value to rise in kink test by a remarkable ~30%. This allows making tighter loops of the tube (the minimal tube bending radius is decreased) without any risk of its breaking.

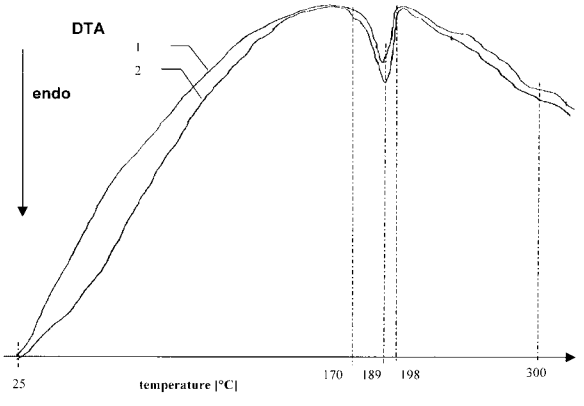

Figure 5 Examples of DTA curves of the same-weight tubes, in the initial state and on heating at 70°C for 34 h; 1—tube in the initial state; 2—tube on heating at 70°C for 34 h.

In Figure 5 are shown examples of DTA curves for the tubes of the same weight, in their initial condition and after heating at 70°C for 34 h.

The DTA study was carried out over a temperature range of 25 to 300°C. The results obtained for the tubes, both in the initial state and upon their heating at 70°C for 34 h, demonstrate the presence of one endothermic peak, which is a proof of the melting of crystalline phase in the tube material. The presence of one peak due to the melting of the crystalline phase on the DTA curve is usually evidence for the crystallites belonging to the same form of the crystalline phase that melted. The temperature range of the occurrence of the peaks is the same for the tubes studied. However, the endothermic peak area (obtained for the same-weight samples) for the heat-treated tube is larger than for the tube untreated (i.e., in the initial state). This means that the heating of tubes at 70°C for 34 h resulted in a rise in crystalline phase content from 28.45 (±0.04)% to 31.58 (±0.04)% (cf. Table II).

Shown in Figure 6 are X-ray diffraction patterns of the tubes in the initial state and on heating at 70°C for 34 h.

On the X-ray diffraction patterns obtained for the tubes, both in the initial state and upon special ther-

TABLE II
Value of the Characteristic Properties of the Tubes Obtained from the DTA Curves

Sample state tube appearance	Unit	Initial opalescent	On heating at 70°C for 34 h opalescent
$T_{peak\ start}$	°C	168 (±2)	170 (±2)
T_{peak}	°C	189 (±2)	189 (±2)
$T_{peak\ end}$	°C	198 (±2)	198 (±2)
ΔH	J/g	41.12 (±0.04)	45.63 (±0.04)
α	(%)	28.45 (±0.04)	31.58 (±0.04)

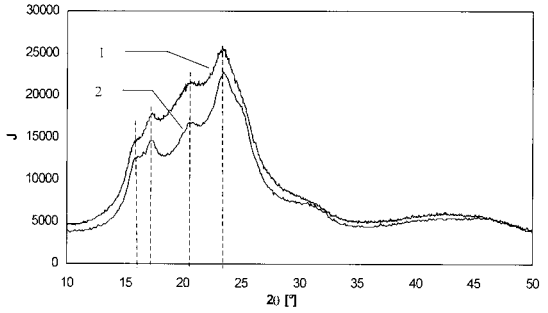

Figure 6 The X-ray diffraction patterns of the tubes in the initial state and on heating at 70°C for 34 h; 1—tube in the initial state; 2—tube on heating at 70°C for 34 h.

mal treatment, some peaks appear that are due to the crystalline phase. By comparing the X-ray diffraction patterns curves with the pertinent literature data[3] it can be concluded that the peak distribution and their corresponding intensities of the scattered X-rays are evidence for the presence in the tubes examined (both in the initial state and on heating) of the crystalline α phase only. However, on heating, the peaks become distinctly sharper, which is indicative of the fact that during the treatment a process of crystalline phase structure becomes more perfect. The X-ray diffraction pattern of the tube on heating at 70°C for 34 h lies below the pattern obtained for the tube in the initial state, which expresses less amorphous scattering. This signifies that the heating of the tubes results in rise in the crystalline phase content.

The characteristic values of parameters for the peaks were found from the X-ray diffraction patterns of Figure 6 (cf. Table III).

Presented below are examples of SEM photographs taken on brittle fractures of tubes in the initial state (Fig. 7) and on their heating at 70°C for 34 h (Figs. 8 and 9).

In Figure 7 is shown a representative structure of a brittle fracture of the tube in its initial state. Against the background of the amorphous substrate some randomly distributed spherical elements can be distinguished, which are from 10 or so to ~100 nm in size. The elements represent the crystalline phase in the

TABLE III
Value of the Parameters for the Peaks Found from X-ray Diffraction Patterns

Sample state tube appearance	Initial opalescent		Upon heating at 70°C for 34 h opalescent	
	2θ	$I_{rel.}$	2θ	I_{rel}
1	15.63	0.56	15.82	0.55
2	17.45	0.70	17.27	0.65
3	20.36	0.84	20.45	0.74
4	23.63	1.00	23.55	1.00

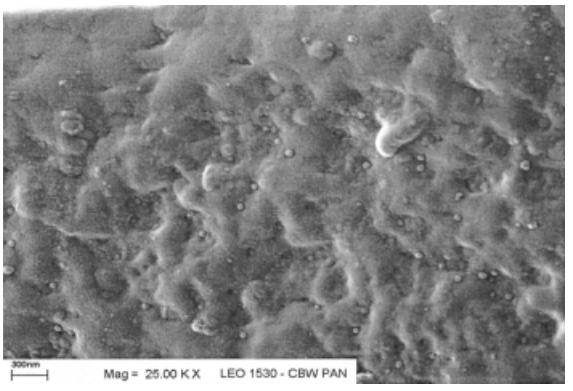

Figure 7 An example SEM photograph of a brittle fracture of the tube in its initial state.

form of spherulites, which was formed during the PBT tube fabrication process.

Figures 8 and 9 display some representative structures of brittle fractures of the PBT tubes upon their heating at 70°C for 34 h. It can be noticed on the figures that the structure on the heat treatment is nonhomogeneous concerning the size of the spherulites and their distribution density. In Figure 8 the randomly distributed spherulites of the crystalline phase, from ~50 to ~300 nm in size, can be viewed against the background of the amorphous substrate. They appear to be the original spherulites of the PBT crystalline phase, which grew in size during heating. In Figure 9 it may be observed that during heating, nucleation of new (additional) crystalline spherulites took place in the amorphous matrix. The spherulites become grouped in local clusters of similar size of ~50 nm. In the photograph some cracks in the material up to 200 nm long are also visible. These are most likely due to strong local stresses caused by contraction in volume during formation of the additional spherulites and their clusters.

It appears that postmodification of properties of PBT tubes (following their standard industrial fabri-

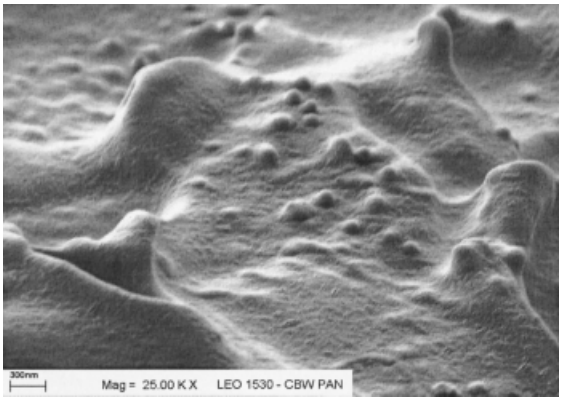

Figure 8 An example SEM photograph of a brittle fraction of the tube on its heating at 70°C for 34 h.

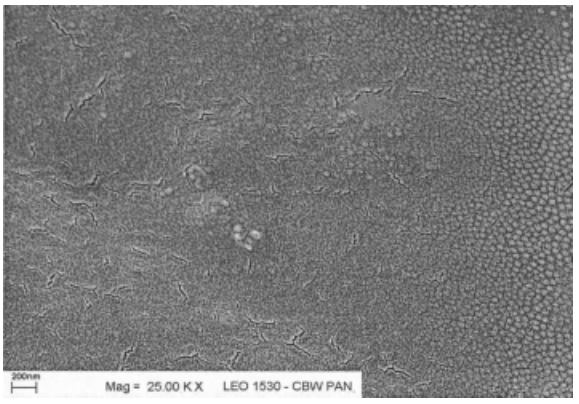

Figure 9 An example view of a brittle fraction of a PBT tube on its heating at 70°C for 34 h.

cation process) by their additional heating for prolonged time in a wound-up condition on reels at 70°C, could find use in industrial manufacturing practice, provided that the cost of energy required for such a thermal treatment of the tubes and its associated labor consumption would not be too high. The heating of the PBT tubes makes the initial excess in fiber length to become disturbed, as the tubes permanently shrink, which may result in an increased optical attenuation of the fibers. In this connection it seems that the tubes subjected to such thermal treatment should initially contain a smaller excess in fiber length, and upon heating they should be additionally rewound to another reel so as to make the fiber length excess distribution homogeneous over the entire length of the tube wound up on the reel.

The PBT tube properties could alternatively be modified by changing their standard industrial fabrication process. A reduced rate of tube cooling (e.g., by raising the temperature in the cooling vat or by increasing the speed with which the tubes are passing through the cooling vat during tube fabrication process) should result in an increased amount of crystalline phase in the tubes made and thus improve their mechanical properties.

Improved mechanical properties of the tubes appear to be desirable with reference to the cables used in heavy duty conditions, when a larger safety margin in the aspect of mechanical properties is required compared with typical cables laid underground in polyethylene pipes. The PBT tubes of increased mechanical properties could most likely find application, say, in ADSS (All-Dielectric Self-Supporting Cables) type cables. The cables are suspended on the high-voltage power transmission pylons and operate under conditions of variable temperatures, winds, icing, and electromagnetic field.

A possible use of the results of the testing in industrial manufacturing practice necessitates a detailed consideration of the options suggested above.

CONCLUSION

During standard industrial production of PBT tubes a crystalline phase is produced in the tube material in the α form, of an average content of $\sim$29%. The heating of such tubes at 70°C for 34 h brought about a rise in the crystalline phase content by $\sim$3%. This was the reason for an improvement in mechanical strength properties that rose even as much as by 30%.

The rising crystalline phase content and the perfectness of the crystallites in polymers are known to make not only the improvement of mechanical properties but likewise a betterment of other properties (including dimensional stability, resistant to temperature, resistance to chemicals).

References

1. Yasuniwa, M.; Murakami, T. Ushio, M. J Polym Sci B 1999, 37, 2420.
2. International Norm IEC 749-1-2 methods E16.
3. Li, R.; Tjong, S.; Zie, X. J Polym Sci B 2000, 38, 403.