Effect of Structural Features of Poly(butylene terephthalate) Tubes on the Useful Properties of the Loose Tube/Optical Fibers System in the Tubular Optical Fiber Cables

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ABSTRACT: To verify a hypothesis that a relationship exists between the useful properties of the loose tube/optical fibers system and the structural features of poly(butylene terephthalate) (PBT) tubes, optical attenuation of optical fibers was studied both in the tubular optical fiber cable and in the tube proper. Examinations of the tube material structure was also performed by the DTA, SEM, and WAXD techniques. The results obtained allowed conclusion that during the standard industrial PBT tube extrusion process a finely dispersed crystalline α phase is formed in a quantity of ~29%. The value is much less compared with the literature value of 66% (Yasuniwa et al., J Polym Sci B 1999, 37, 2420[1]) reported for a possible maximal content of the crystalline phase in PBT. Accordingly, it was assumed that the quantity of the crystalline phase in the tubes can be made to increase by their heating at a temperature above the glass transition temperature (T_g) of PBT. Results of the study corroborate the soundness of the assumption that during the optical cable conditioning process over its working temperature range, i.e., from −40 to 70°C, crystallisation of PBT tubes is likely to occur to result in their shrinkage which, as proven, affects the optical fibers attenuation, which is a major useful property of optical cables. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 86: 2124–2129, 2002

Key words: crystallization; microstructure; plastics

INTRODUCTION

Optical fibers incorporated into an optical communication cable are used to transmit information in the form of optical pulses of a specific wavelength. The major structural and functional element of the tubular optical fiber cable is the loose tube/optical fibers system. Useful properties of such a system include, first, its capability for undisturbed transmission of an optical signal, estimated, for example, in terms of optical attenuation. The loose tube performs as a protection of the sensitive optical fibers against external mechanical and chemical factors. A real design of this kind the length of the optical fibers used is somewhat greater in relation to the tube length (Fig. 1).

The excess fiber length in the tube (on the level of 0.05%) is selected during an industrial loose tube extrusion process schematically shown in Figure 2. Generally the tube extrusion line consist of these given devices: optical fibers feeder, pump supplying a hy-drophobic jelly, extruder with a head shaping a tube, tube drawing wheel, two water cooling baths, and a tube take-up reel.

The excess length of the fibers in the tube is devised to make up for the changes in cable dimensions as the result of mechanical forces and external environment acting on it. The excess fiber length should be so matched, on the one hand, that the tensile stresses (developed, for instance, during cable laying out) should not be transferred to the mechanically vulnerable, fragile fibers, and on the other hand, that the compressive stresses (set up, for instance, as the result of contraction in volume) should not bring about increased attenuation of the fibers due to loss in optical power of light at the micro- and macrobends in the fibers. Therefore, the major requirements for the loose tube/optical fibers system is such of a dimensional stability of the tube over the service temperature range of the optical fiber cable (i.e., from −40 to +70°C), which does not affect the initial excess length of the fibers established during the cable manufacturing process.

From the standpoint of the materials selection, while the material properties of the optical fiber are unchangeable in practice, the material properties of the tube is a trade-off between the complexity of the material requirements, often difficult to be reconciled.
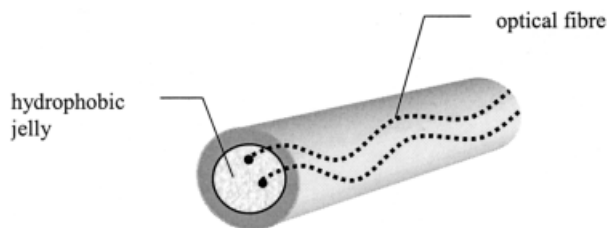
Figure 1 The excess of optical fiber length in the loose tube system.

A major material used today in the loose tube manufacture is poly(butylene terephthalate) (PBT).[2–4] The polymer features a rather good combination of mechanical, chemical, and process properties.

PBT ranks among the thermoplastic polyesters. Its T_g is reported to range from 22 to 65°C.[5–10] The material is, in general, partly crystalline.[11–25]

In PBT two crystalline phases, viz.: α and β are known to occur. Typically, the α form is present in the relaxed material, whereas the β form is found in the material under elastic stress that induces a relative strain up to ~10–14%. Examination of the morphology of these two forms mentioned showed the difference to be explainable in terms of diverse conformations of the aliphatic fragments of the macromolecule chain, i.e., ($-CH_2-$)$_4$. The α form is due to the conformation of the chain fragments mentioned, referred to as *gauche-trans-gauche*, whereas the β form is associated with the *trans- trans- trans* conformation. The $\alpha \leftrightarrow \beta$ transformation is reversible during elastic deformation upon removal of the load from sample.[26–36] The β form may also be stable, yet it may be expected in samples earlier subjected to strains greater than elastic strains.[37–38]

The aim of this study was to verify the hypothesis that the useful properties of the loose tube/optical fibers system are significantly affected by the structural features of the PBT fabricated under standard industrial conditions into loose optical cable tubes.

Elucidation of the effect of the tube structure on the useful properties of the optical fiber systems under discussion may be utilized, among others, in the industrial optical cable tube production process to maintain high-quality products, as well as in a possible modification of the process.

EXPERIMENTAL

The effect of temperature on attenuation of optical fibers

In a Heraeus temperature chamber model HZT 6050, the optical fiber cable containing six PBT tubes stranded around a strengthening core element (each tube contained four SMF 1528 type single-mode fibers) and a separate PBT tube (which contained four SMF

1528 type single-mode fibers) were placed. Both the cable and tube were wound up on separate reels. The fibers of the cable and of the tube were fused in series to make up two separate measuring systems. Optical power vs. temperature was measured using a Hewlett Packard power meter model 8153A at a wavelength of 1550 nm with an accuracy of 0.005 dB.

Structural investigations

Samples for the study were made from representative lengths of PBT loose tubes. Tubes were fabricated under the typical industrial conditions extrusion process. It is called standard conditions.

Prior to the experiments the hydrophobic gel and the optical fibers were removed from the inside of the tubes using compressed air. In the study of the structure of loose tubes the following techniques were used: (a) differential thermal analysis (DTA), (b) wideangle X-ray scattering analysis (WAXD), and (c) scanning electron microscopy (SEM).

DTA

Tube samples, carefully cleaned of the gel with petroleum spirits, were placed in platinum crucibles. The study was carried out using a derivatograph[39] under the following conditions: heating rate—2.5°C/min, temperature range—from the room temperature to 500°C, load sensitivity—200 mG, DTA sensitivity—1/1, and recorder chart speed—$V = 2$ mm/min.

To determine the relative crystalline phase content in the tube under examination, the heat of fusion of the crystalline phase, ΔH_m, was calculated using formula:[40]

$$\Delta H_m = K \cdot \frac{F}{M \cdot V} \tag{1}$$

where ΔH_m is the the heat of fusion of the crystalline phase (J/G); K is the calibration coefficient for a given apparatus (J/ms); $K = 0.4114$ (J/ms); F is the surface
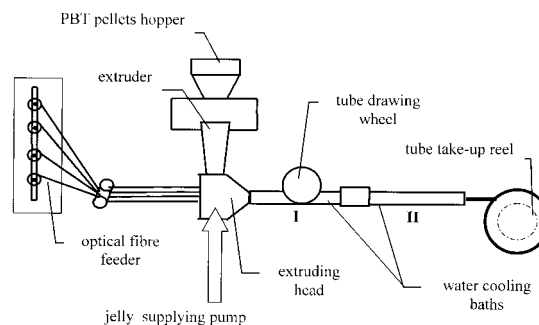
Figure 2 A scheme of the industrial loose tube extruding line.
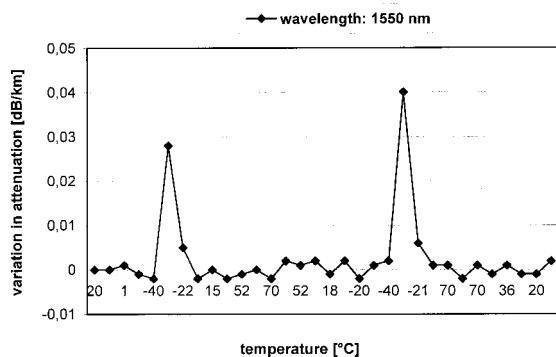
Figure 3 The effect of temperature (over a range from −40 to 70°C) on variation in attenuation of single-mode optical fibers in a cable composed of six tubes with four fibers each.

area under the peak formed during fusion of the crystalline phase (m²); M is the mass of sample (G); and V is the recorder chart speed (m/s).

The relative crystalline phase content in tubes, α (%), was found from the formula:

$$\alpha = \frac{\Delta H_m}{\Delta H_{PBT}} \cdot 100\% \qquad (2)$$

where $\Delta H_{PBT} = 144.5$ J/g,[41] and is the heat of fusion of the 100% PBT crystalline phase.

SEM

Brittle fractions of the tubes were obtained by breaking them by freezing in liquid nitrogen. The sample surface was coated by sputtering with a gold film ~5 nm thick. Observations and the SEM pictures were made using a LEO model 1530 microscope. The microscope, with the so-called cold cathode, enables observations of highly magnified structure.

WAXD

Samples used in the study were made in the form of flat tubes placed one close to another on a glass plate on which an adhesive tape was previously spread out. This arrangement was devised to simulate a solid PBT sample.

The examinations were carried out using an X-ray diffractometer equipped with an X-pert goniometer. The wavelength of the X-rays used was 0.154 nm (CuK$_\alpha$ with a Ni filter).

RESULTS AND DISCUSSION

Shown in Figure 3 are some of the results of the study on the variation of attenuation of optical fibers in a tubular cable during two temperature cycles (from

−40 to 70°C). The cable was withheld at −40 and 70°C for 17 h.

The study was started at 20°C. Subsequently, the temperature was lowered to −40°C, which made the optical attenuation of the fibers to increase (the first peak) compared to the initial condition. As the linear expansion coefficient of PBT is by about three orders of magnitude higher than that of SiO₂, which is the major material of the optical fiber, then, as the result of a temporary shrinkage of the tubes at low temperatures, the fiber excess length in the tubes increased. The effect made the attenuation of the fibers to increase as the result of a loss in optical power at microbends. Subsequently, the temperature was raised to 70°C, which had no effect on the attenuation of the fibers. Surprisingly, a renewed decrease of temperature to −40°C brought about another change in the attenuation of the fibers (the second peak) and so to a much more pronounced degree than during the first low-temperature conditioning. The result of the study is not fortuitous, as it was observed repeatedly. A possible explanation of this fact, as well as of the fact of a greater intensity of the second peak at lower temperatures (upon previous heating at 70°C for a long period of time) compared with the first peak, is that the tubes during the prolonged heating at temperatures above T_g underwent crystallization and a permanent shrinkage due to a previous crystallization, which was superimposed on the temporary shrinkage at low temperature (i.e., at −40°C). This resulted in an increased excess length of the fibers in the tubes and, as a result, an increased optical attenuation of the fibers.

This kind of test is commonly performed for optical fiber cables. According to the standard,[42] the permissible change in attenuation of the single-mode fibers during the two temperature cycles (from −40 to 70°C) should not be greater than 0.1 dB/km and upon completion of the test—not greater than 0.05 dB/km (for a wavelength of 1550 nm).

To demonstrate that it is the permanent tube shrinkage caused undoubtedly by the tube polymer crystallization that determines the useful properties of the optical fiber cable, a nonstandard investigation of the temperature variations of attenuation of the fibers in the tube was carried out (Fig. 4). During that test the tube was being successively withheld at −20 and 70°C for 17 h.

As seen from Figure 4, the optical fibers in the PBT tube over a temperature range from ~1 to −20°C show variations in attenuation above 0.1 dB/km (for a wavelength of 1550 nm). This is due to the fact that on the permanent shrinkage, which is the results of crystallization of the tube PBT polymer at the temperature above its T_g, the temporary shrinkage is superimposed, which developed at low temperature. This effected too considerable excess of the fiber length in the
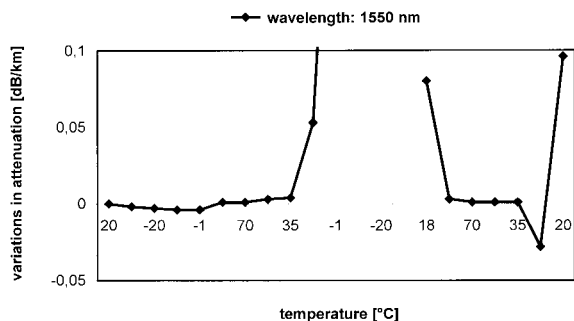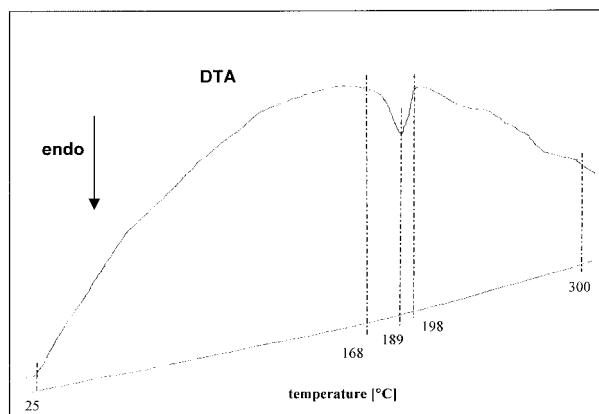
Figure 4 The effect of temperature on variations in attenuation of single-mode fibers in a BPT tube in two temperature cycles (−20 and 70°C).

Figure 6 A view of the surface of PBT tube brittle fracture for the tube fabricated under standard industrial conditions on a microphotograph.

tube and an undesirable rise in attenuation of the fibers. The shrinkage is so great that even upon completion of the thermal test, the fiber attenuation at room temperature is above 0.05 dB/km. The thermal test caused a permanent and irreversible, under given conditions, change in tube length, which affected the excess fiber length established during the extrusion operation. For fibers in the tubes tests of such a kind are not commonly performed.
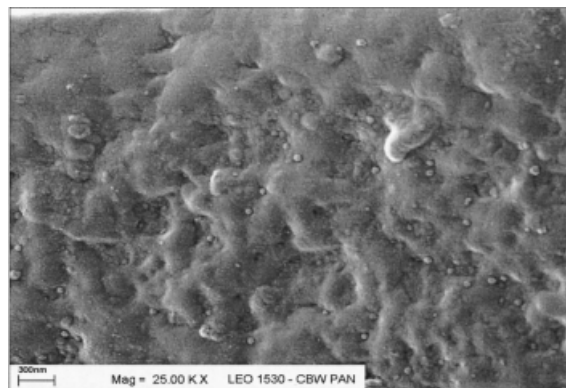
To substantiate the concept of a relationship between the useful properties of the loose tube/optical fibers system and the physical structure of the PBT tubes, a number of preliminary structural studies were carried out.

It was unambiguously found that in the PBT tubes fabricated under standard industrial conditions a crystalline phase exists. This can be inferred from the run of the DTA curves measured for such tubes, some of which are shown for illustration in Figure 5.

Heating of the tubes during the DTA investigation at a rate of 2.5°C/min over a temperature range from ca. 25°C to ~300°C allowed finding a single endothermic peak on the curve, which is indicative of the melting of the crystalline phase. The presence of one melting peak only suggests that in the tubes examined

only one crystalline phase form exists. This finding, however, should not be extended over PBT polymer of just any thermal history. It should be realized that the heating rate may induce structural changes in PBT.[43–45]

The characteristic temperatures of the melting peak of the PBT tube crystalline phase are: (a) melting start, 168°C; (b) melting end, 189°C; (c) curve return end upon melting, 198°C.

The value of the heat of melting of the crystalline phase found allowed to determine the percentage of the crystalline phase present in the tubes sampled from representative fabricated sections. The percentage of the crystalline phase in the tubes examined was found to be 28.8%. Elsewhere,[1] upon application of special heat treatment conditions and at an increased pressure, the crystalline phase in PBT polymer (but not in PBT tubes) was made to rise to a level of 66%. The value appears to be the highest attainable for this phase.

To substantiate the presence of the crystalline phase in the tubes, SEM microscopic observations were carried out on tube brittle fractures. An SEM photograph is shown for illustration in Figure 6.

The tiny spherical precipitates seen are most likely the spherulitic crystalline phase in PBT. Similar images of finely dispersed precipitates of the PBT crystalline phase were reported earlier.[46]

In a search for other forms of crystalline phase in the tubes under study, not detected by the DTA technique, structural X-ray analysis was carried out on the same tubes. Shown in Figure 7 is an example of an X-ray diffraction pattern of the PBT tubes fabricated under standard industrial conditions.

The study was made over an angle range (of the goniometer) of 10–50°. The four peaks on the figure are evidence of the fact that the tube contains a crystalline phase in its structure.

In comparing the results of Table I obtained with the literature data[47] (Table II), it can be inferred that the

Figure 5 Example of the DTA plot for the PBT tube fabricated under standard industrial conditions.
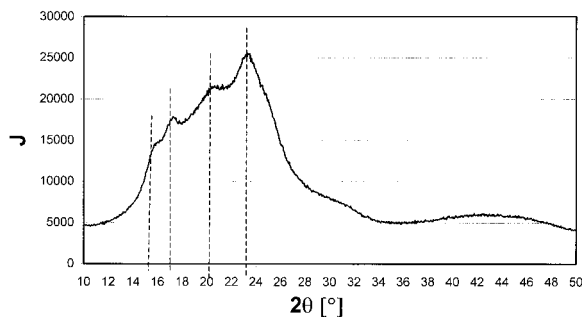
Figure 7 An illustrative X-ray diffraction pattern of a PBT tube fabriated under standard industrial conditions.

TABLE II
Characteristic Peaks from the PBT α-Form Reported in the Literature[47]

Peak no.	Angle of X-rays scattering 2θ (°)	Relative intensity of the scattered X-rays J/J_0 (%)
1	15.81	24.7
2	17.15	61.8
3	20.44	19.6
4	23.18	100.0
5	25.07	66.6

peak distribution and their corresponding intensities of the scattered X rays provide evidence for the occurrence of the α form of the crystalline phase in the tube examined.

SUMMARY OF RESULTS

Results of the study have shown that during the standard industrial PBT tube extrusion process a finely disperse crystalline phase is formed in the plastic material. The phase is of the α form present in an amount of ~29%. The value is much lower than the maximal content of the crystalline phase liable to develop in PBT estimated at 66%. In this connection, the crystalline phase may be increased in the tubes by their prolonged heating at a temperature above T_g of the tube material. Results of the study demonstrated that during optical fiber cable conditioning over its operating temperature range, i.e., from −40 to 70°C, the crystallization process is likely to occur in the PTB tubes, which results in tube shrinking. This effect was shown to increase attenuation of light in optical fibers. It was unambiguously found from the study that there is a clear correlation between the useful properties of the loose tube/optical fibers system and the physical structure of the PBT tubes.

CONCLUSION

The occurrence of a crystalline phase in PBT tubes fabricated under standard industrial conditions signifies that the crystallographic form and the crystalline

phase content in the tubes can be controlled. This can be achieved by modification of conditions of tube extrusion, treatment, and storage of the tubes produced.

The control over the quantity and kind of the crystalline phase in the tubes may be critical in the aspect of mechanical properties of the tubes. These properties can affect the useful properties of the tube/fiber system and, in consequence, have an effect on transmission properties of the fibers in optical fiber cables.

The results available in the literature prove that the mechanical properties of PBT depend on the percentage of the α and β forms present in the structure of the tube material, and that more advantageous mechanical properties are achieved when the β form prevails in the structure. This is due to the fact that the β form ensures a higher Young's modulus and a higher value of the stress limit of the forced elasticity.[37] Considering the foregoing, it might seem desirable that in the structure of the tube material that also the β form of the crystalline phase would be present.

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TABLE I
Peak Parameters Read Off the Plot

Peak no.	Angle of X-rays scattering 2θ (°)	Relative intensity of the scattered X-rays J/J_0 (%)
1	15.63	56
2	17.45	70
3	20.36	84
4	23.63	100

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