Instantaneous Elastic Recovery of Poly(trimethylene terephthalate) Filament

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ABSTRACT: The instantaneous elastic recoveries of poly(trimethylene terephthalate) (PTT), PET, and PBT filaments were comparatively analyzed by a tensile testing machine. The conditions of the measurement were studied and the mechanism of instantaneous elastic recovery of these three aromatic polyester filaments is discussed. The instantaneous elastic recovery of PTT filaments was significantly higher than those of PBT and PET filaments. Moreover, PTT filaments had a high instantaneous elastic recovery even at a high elongation of 20%. The outstanding instantaneous elastic recovery of PTT filaments resulted from its helical conformation in crystal lattice, which responded immediately to the applied stress and deformed as though it was a coiled spring; we suggest it was a quasispring elastic recovery mechanism. When we measured the instantaneous elastic recovery of, PTT filaments with the tensile testing machine, it was appropriate to set up the crosshead speed at 500 mm/min with an elongation of 20% and a load of 0.5 cN/tex. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 91: 1967–1975, 2004

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INTRODUCTION

Poly(trimethylene terephthalate) (PTT) is an aromatic polyester, the most common substance used in engineering thermoplastics.[1–4] More recently, PTT has drawn attention for its applications in the textile industry, especially as used in the carpet industry due to the reduction in the manufacturing cost of 1,3-propanediol, the monomer used for PTT synthesis. Nylon captures the largest market share because it has very good resiliency and bulk. However, being acid dyed, nylon suffers from poor stain resistance unless protected with stain-blocking chemicals. It is clear that PTT fiber would combine the good resiliency and bulk of nylons with the inherent stain resistance and low static generation of either PET or polypropylene (PP). Competitive pricing would make this fiber even more attractive.

The physical properties of PTT fibers have been studied in previous publications.[5–8] According to Wernys, PTT has a better recovery from bending that is superior to that of nylon 6, PET, PBT, and PP, except nylon 66.

The mechanical properties of PTT fibers were studied by Ward et al.[6] and were compared to other polyester fibers. The isothermal crystallization and melting behaviors of PTT have been studied by differential scanning calorimetry, wide-angle X-ray diffraction, and solid-state NMR.[7] The dynamic thermogravimetric kinetics of PTT were analyzed, and the effects of argon, air, and nitrogen were also investigated.[8] The crystal transition of PTT films under cyclic loading was studied by IR spectroscopy.[9] Except for these, there has been little literature addressing elastic recovery from the longitudinal stretching of these aromatic polyester fibers.

The purpose of this article is to describe a comparative study of the changes in instantaneous elastic recovery that occur when the three aromatic polyester filaments (PTT, PET, and PBT) were subjected to a mechanical tensile load. A preliminary discussion is presented of the relationship of the chain structure and the instantaneous elastic recovery of the three aromatic polyester filaments. The mechanism of recovery of the PTT filaments was deduced from the macromolecular chain conformation in the crystalline lattice as a quasispring elastic recovery mechanism. The conditions of the measurement on the instantaneous elastic recovery of PTT filaments were studied and are discussed.

EXPERIMENTAL

Materials

The materials of each polyester filament were prepared free from delustrant. PET filament FDY was

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TABLE I
Mechanical Properties of the Three Aromatic Polyester Filaments

Property	PET	PTT	PBT
Titer (dtex)	166.1	129.3	76.5
Tenacity (cN/dtex)	4.14	2.52	2.97
Breaking elongation (%)	34.6	51.1	28.8
Initial modulus (cN/dtex)	81.56	22.01	26.47
Specific breaking work (cN/dtex)	0.807	0.600	0.505

Crosshead speed = 500 mm/min; load = 0.5cN/tex.

produced by continuous spinning and drawing in a one-stage process without the as-spun yarn being taken up. A two-stage process in which the melt-spun yarn was once taken up as as-spun yarn and then drawn produced DT of PTT and PBT filaments. Some mechanical properties of the filaments are listed in Table I.

Measurements

Mechanical properties measurements

The load–elongation curves were measured with a Textechno tensile testing machine. For each material, 10 filament samples 500 mm in length were tested at a crosshead speed of 500 mm/min with a load of 0.5 cN/tex. These 10 tests were taken as the mean value. The initial modulus was calculated from the load at 0–1% elongation in the load–elongation curve. All of the measurements were taken at a temperature of 25°C and a relative humidity of 65%.

Instantaneous elastic recovery measurements

The stretch cycle experiments on aromatic polyester filaments were done with a Textechno tensile testing machine. A filament sample 500 mm in length was initially stretched into the required strain at different crosshead speeds with a load of 0.5 cN/tex. The crosshead was then immediately returned to its original position with the same speed. The instantaneous elastic recovery for the first stretch cycle was calculated according to the following formula:

$$\text{Elastic recovery} = (L_o - L) * 100\%/L_o \qquad (1)$$

where L_o is the original length of the filament, L is the irreversible length of the filament after the crosshead it was returned to its original position. These measurement procedures were repeated five times, and the instantaneous elastic recovery for the fifth stretch cycle was calculated according to eq. (1).

RESULTS AND DISCUSSION

Comparison of mechanical properties among the three aromatic polyester filaments

PET, PTT, and PBT are aromatic polyesters with similar macromolecular chain structures. The difference among them lies mainly the difference in the number of methylene groups in repeat units of the macromolecular chain. It was the difference in the number of methylene groups that resulted in the difference in physical properties among these three aromatic polyester filaments.

As shown in Table I, among the three aromatic polyester filaments, the tenacity and the initial modulus of the PET filament was the highest followed by the PBT filament, whereas those of the PTT filament were the lowest. The comparatively low modulus of PTT and PBT filaments could, therefore, be associated with the fact that the molecular conformation never corresponded to the fully extended form so that the deformation always involved bond-angle rotations and bond bending rather than bond bending and stretching. The low modulus in these cases corresponded exactly to the low crystal modulus. That means the resistance of the PTT filament to the deformation was poor and its rigidity was also poor. However, it is soft when it is used for apparel. Moreover, the breaking elongation of the PTT filament was highest among these three polyester filaments, and the scope of the deformation was fairly wide. The specific breaking work of the PTT filament was between PET and PBT, which means that the toughness of the PTT filament was higher than PBT but lower than the PET filament. Therefore, the high specific breaking work of the PTT filament was due to a high breaking elongation. It is suited for use in clothing that possesses a prominent soft handle and a high extension.

Comparison of instantaneous elastic recovery among the three aromatic polyester filaments

We measured the load–elongation curves of drawn PET, PTT, and PBT filaments in five stretch cycles by varying the elongation limits from 5 to 30% presented in Table II. Among them, the results at an elongation of 20% are depicted in Figure 1. Above this elongation, there were apparent hysteresis loops for PET, PTT, and PBT filaments. In addition to the appearance of the hysteresis loop, no sample could return to its original position because of a kind of permanent set after the load was removed and the remnant elongation increased with increasing cycles. For the PET filament, there was a prominent hysteresis loop, and the remnant elongation reached about 25% after the fifth stretch cycle even under an elongation of 5%. While under the elongation of 20%, the hysteresis loop

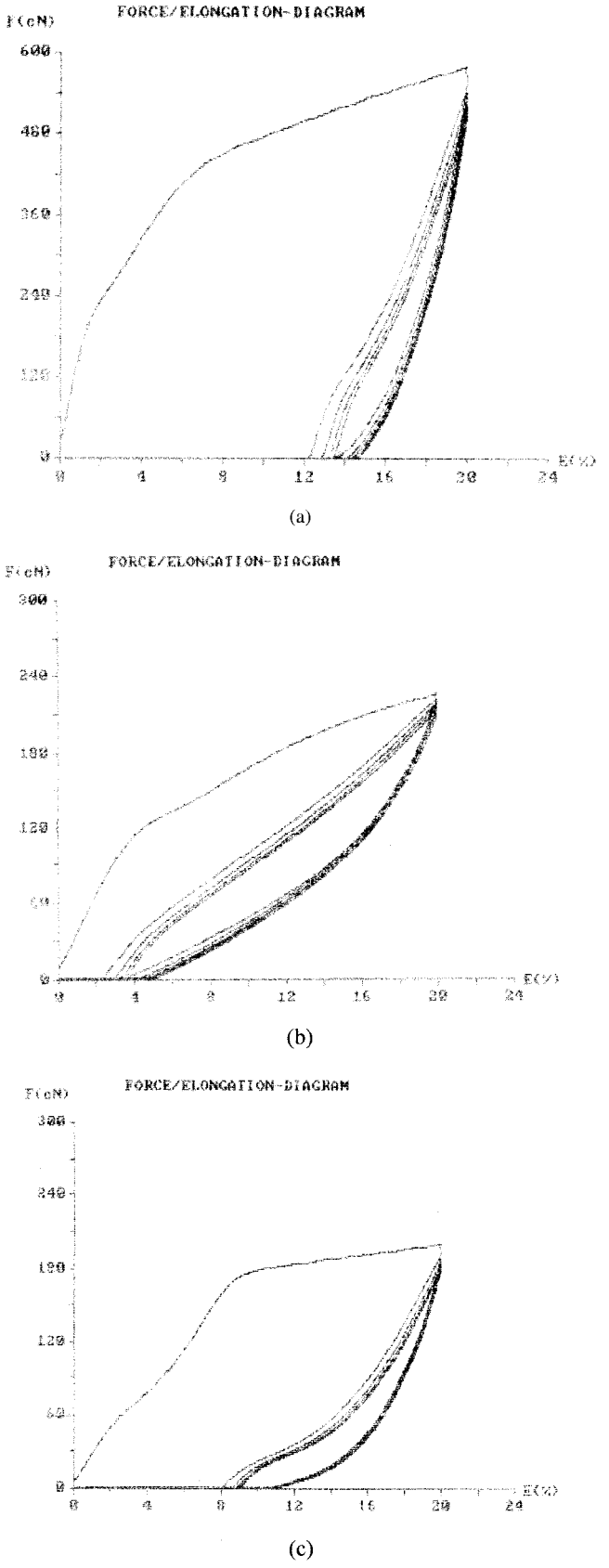

Figure 1 Instantaneous elastic recovery of the three aromatic polyester filaments during five cycles of loading–unloading with an elongation of 20%: (a) PET, (b) PIT, and (c) PBT filaments.

became very large, and the remnant elongation approached about 69% after the fifth stretch cycle shown in Figure 1. For PTT filament, there was a slight hysteresis loop, and the remnant elongation reached only about 5% after the fifth stretch cycle under an elongation of 5%. While under an elongation of 20%, the hysteresis loop became apparent, and the remnant elongation was about 20% after the fifth stretch cycle, which was weaker than that of the PET filament. For PBT filament, however, the situation of the fifth stretch cycle was between the PET and PBT filaments. The remnant elongations of the fifth stretch cycle under both 5 and 20% elongation were 7 and 45%, respectively. These results indicated that the PTT filament had a better instantaneous elastic recovery than both the PET and PBT filaments.

Table II and Figure 2 show that there were significant differences among these three aromatic polyester filaments in the instantaneous elastic recovery. The instantaneous elastic recovery of the PET filament was 20% lower than that of the PTT filament, and at a higher elongation, the difference between them was even more significant. When the elongation was 5%, the instantaneous elastic recovery for the fifth stretch cycle of the PTT filament was 25% higher than that of the PET filament under identical conditions. When the elongation was 20% the difference between them reached 49%. It dropped to 24% of the instantaneous elastic recovery for the fifth stretch cycle of the PET filament when the elongation was 30%, whereas that of PTT filament remained at a high value of 66%. It was obvious that the instantaneous elastic recovery of the PTT filament was significantly superior to that of the PET filament. Moreover, the PTT filament had a a higher instantaneous elastic recovery even at a high elongation.

From the macromolecular chain point of view, PBT and PTT have a similar chain structure; both of them should have fairly excellent elastic recovery. However, the PBT filament had a higher instantaneous elastic recovery only when the elongation was low. It can be seen from Table II and Figure I that at an elongation of 5%, the instantaneous elastic recovery for the first stretch cycle between PBT and PTT filaments was quite similar, whereas the instantaneous elastic recovery for the fifth stretch cycle between them differed from each other. The instantaneous elastic recovery of the PBT filament was lower than that of the PTT filament but significantly higher than that of PET filament. With the increase in the elongation, the instantaneous elastic recovery of the PBT filament declined significantly for the first and for the fifth stretch cycles. When the elongation was above 20%, the instantaneous elastic recovery of PBT filament declined to a large extent; the difference in the instantaneous elastic recovery between the PBT and PTT filaments was increased. For an elongation of 5%, the instantaneous

TABLE II
Instantaneous Elastic Recovery of the Three Aromatic Polyester Filaments

Elongation (%)	PET		PTT		PBT	
	First stretch cycle	Fifth stretch cycle	First stretch cycle	Fifth stretch cycle	First stretch cycle	Fifth stretch cycle
30	29.8	24.2	74.6	66.6	—	—
20	38.0	31.4	87.1	80.4	59.2	54.7
10	61.1	50.9	95.3	91.5	95.0	91.3
5	86.0	75.4	100	95.2	94.6	93.0

Crosshead speed = 500 mm/min; load = 0.5cN/tex.

elastic recovery for the fifth stretch cycle of PBT filament was about 2% lower than that of the PTT filament, but it was 23% higher than that of the PET filament. When the elongation was 20%, the difference in the instantaneous elastic recovery between the PBT and PTT filaments increased to 47%, but the difference between the PBT and PET filaments greatly increased to 74%. As the elongation increased to 30%, the PBT filament had already been broken, but the PTT filament still had a high instantaneous elastic recovery value of 66%, and the PET filament had a value of 24%.

In brief, the instantaneous elastic recovery of the PTT filament was prominently superior to that of the PET and PBT filaments. The PTT filament had not only a high instantaneous elastic recovery value at a lower elongation but also had an instantaneous elastic recovery of about 66% at the higher elongation of 30%. The PBT filament had a high instantaneous elastic recovery value only at a low elongation, but its instantaneous elastic recovery declined significantly at a higher elongation, and sometimes, it was even lower than that of the PET filament. In a comprehensive analysis and comparison of the instantaneous elastic recovery of these three aromatic polyester filaments, we concluded that that PTT filament was the best, followed by the PBT filament and the PET filament.

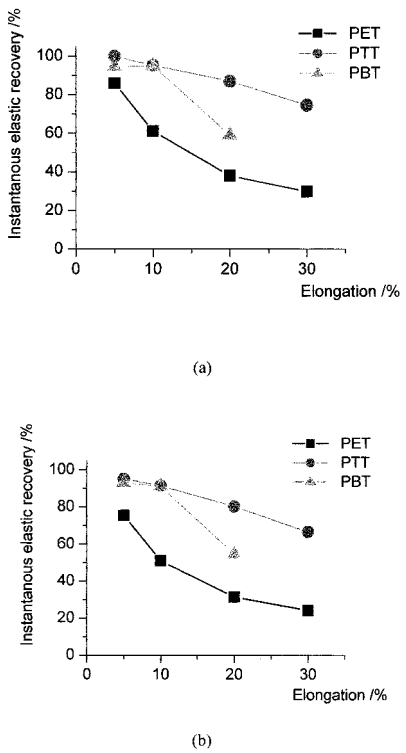

Figure 2 Instantaneous elastic recovery of the three aromatic polyester filaments for the (a) first and (b) fifth stretch cycles.

Effect of stretch cycle and crosshead speed on the instantaneous elastic recovery of the PTT filament

A multistretch cycle will affect the instantaneous elastic recovery of fibrous materials. It is shown in Table III and Figure 3 that the instantaneous elastic recovery for the fifth stretch cycle of the PTT filament was about 8% lower than that for the first stretch cycle. The higher the elongation was, the larger the difference was in the instantaneous elastic recovery between the two situations. This means that the multistretch cycle exerted a rather large influence on the instantaneous elastic recovery of the PTT filament.

Theoretically, the mechanical properties of fibrous materials are strongly time dependent, and the cross-

TABLE III
Effects of the Stretch Cycles and Crosshead Speeds on the Instantaneous Elastic Recovery of the PTT Filament

Crosshead speed (mm/min)	Instantaneous elastic recovery (%)	
	First stretch cycle	Fifth stretch cycle
20	86.7	80.4
60	87.2	80.7
100	87.0	80.6
400	87.5	80.6
500	87.1	80.4
600	87.2	80.6
1000	86.8	79.9

Elongation = 20%; load = 0.5cN/tex.

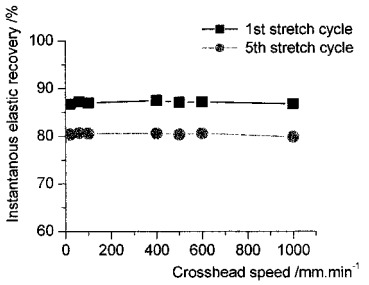

Figure 3 Effect of crosshead speed on the instantaneous elastic recovery of the PTT filament.

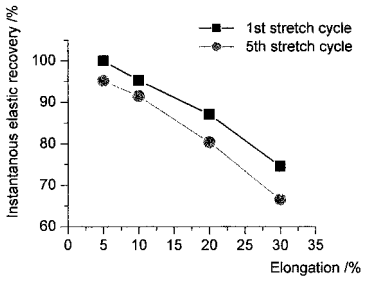

Figure 4 Effect of elongation on the instantaneous elastic recovery of the PTT filament.

head speed should exert a significant influence on the instantaneous elastic recovery of fibers. However, the crosshead speed within the range 20–1000 mm/min did not influence the instantaneous elastic recovery of the PTT filament studied here. At different crosshead speed', no matter the stretch cycle, the instantaneous elastic recovery of the PTT filament was basically unchanged. This resulted in the two straight lines shown in Figure 3. It indicated that the instantaneous deformation of the PTT macromolecular chains under the external stress easily reached equilibrium. That is why we could choose a higher crosshead speed in the measurement of the instantaneous elastic recovery of the PTT filament to increase the efficacy of the measurement. In accordance with the conditions of the measurement of the other mechanical properties, we suggest choosing a crosshead speed of 500 mm/min for the measurement of the instantaneous elastic recovery of PTT filaments.

Effect of elongation on the instantaneous elastic recovery of the PTT filament

The elongation exhibited a fairly large influence on the instantaneous elastic recovery of the PTT filament. It is shown in Table IV and Figure 4 that the instantaneous elastic recovery of the PTT filament decreased increasing the elongation. The decrease in the instantaneous elastic recovery of the PTT filament was more prominent, especially when the elongation was higher. As PTT filament was nearly completely recoverable from deformation in the time of the low elongation, its instantaneous elastic recovery was very high, and it was expressed as a typical hard spring with a reversible elastic recovery. The time dependence of the instantaneous elastic recovery after the elongation of the PTT filament increased gradually with increasing elongation. The irreversible permanent deformation resulting from creep made the instantaneous elastic recovery decrease, especially when the elongation approached the breaking elongation of the PTT filament, which increased the irreversible permanent deformation and in addition, significantly increased the time dependence of the elastic recovery after the elongation, resulting in a significant decrease in the instantaneous elastic recovery of the PTT filament.

TABLE IV
Effect of Elongation (E) on the Instantaneous Elastic Recovery of the PTT Filament

	Instantaneous elastic recovery (%)			
Stretch cycle	E = 5%	E = 10%	E = 20%	E = 30%
First stretch cycle	100	95.3	87.1	74.6
Fifth stretch cycle	95.2	91.5	80.4	66.6

Crosshead speed = 500 mm/min; load = 0.5 cN/tex.

We measured the load–elongation curves of the PTT filament in five cycles of loading–unloading by varying the elongation from 5 to 30%. It is shown in Figure 5 that there was no complete elastic recovery within the range of 5–30% elongation, and the instantaneous elastic recovery of the PTT filament decreased with increasing elongation. With 5% elongation, there was no hysteresi loop, and a complete elastic recovery existed after the first stretch cycle, whereas only a slight hysteresis loop was observed and the instantaneous elastic recovery reached 95% after the fifth stretch cycle. Above 20% elongation, the hysteresis loop began to be apparent, and the instantaneous elastic recovery dropped significantly.

The results of this study also showed that the lower elongation might have influenced the accuracy of the measurement results. At elongations of 10–30 %, the instantaneous elastic recovery of the PTT filament correlated well with the crosshead speed and the stretch cycle. The results were fairly good. At an elongation of 5%, the correlations of the instantaneous elastic recovery for the fifth stretch cycle with the crosshead speed and the multistretch cycle were poor. Even abnormal phenomena, such as the instantaneous elastic recovery of zero or negative values, could happen. The occurrence of these phenomena resulted mainly from the good instantaneous elastic recovery of the PTT filament even under a high elongation.

As clothing materials, filaments are usually used at elongations below 20%, and for the sake of compari-

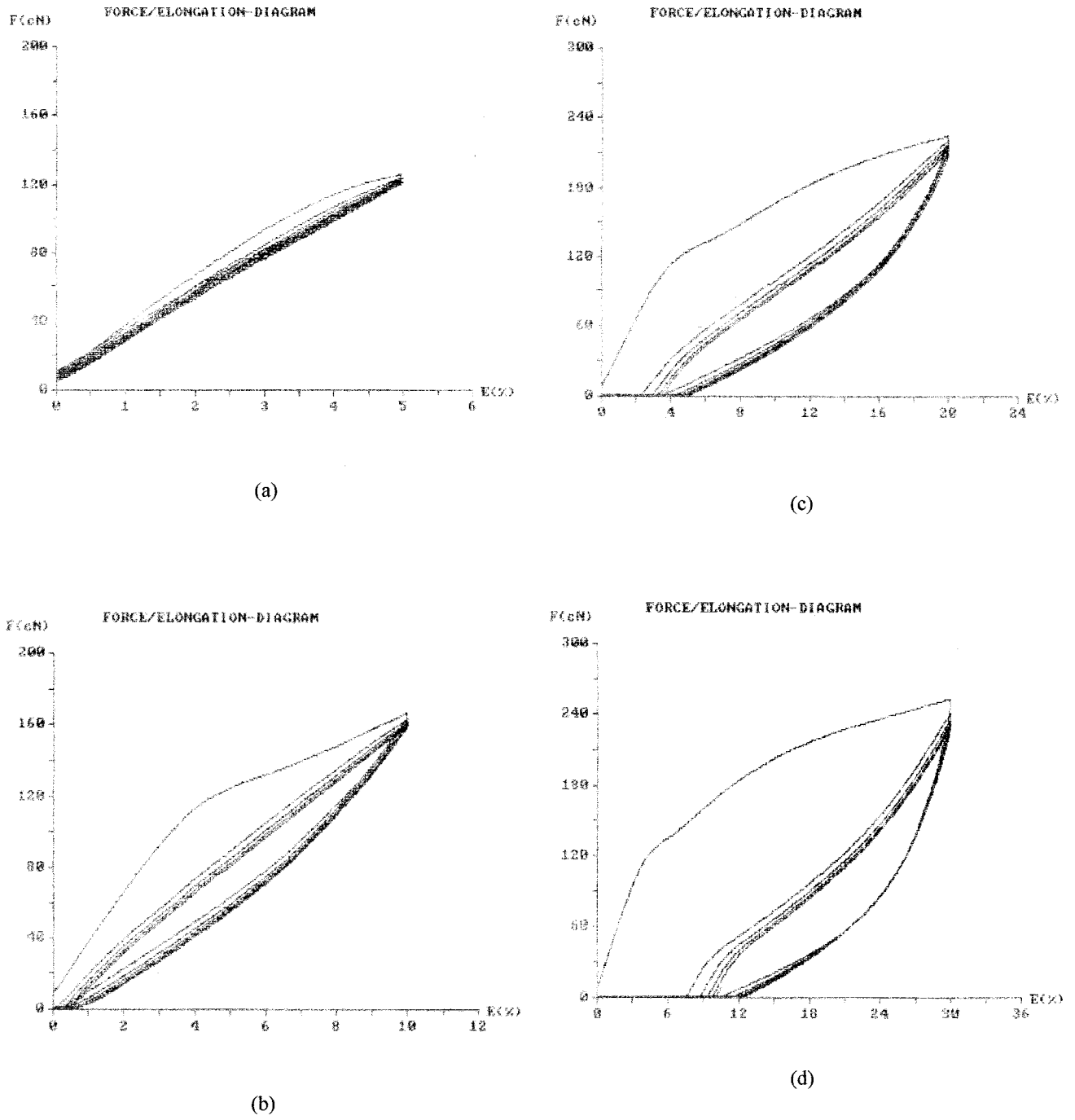

Figure 5 Instantaneous elastic recovery of PTT filament during five loading–unloading cycles: (a) 5, (b) 10, (c) 20, and (d) 30% elongation.

son with other fibrous materials, we suggest an elongation of 20% during the measurement of the instantaneous elastic recovery of PTT filaments.

Effect of load on the instantaneous elastic recovery of the PTT filament

The magnitude of load used in the measurement had some influence on the results of the instantaneous elastic recovery of fibrous materials, and setting up a load depended on the tenacity of the filaments to be measured. As the tenacity of the PET filament was high, the introduced load was 0.5 cN/tex in the measurement of its instantaneous elastic recovery. However, a lower load of 0.01 cN/tex was used in the measurement of the instantaneous elastic recovery of a polyurethane (PU) filament for its weak tenacity. Be-

TABLE V
Effect of Load on the Instantaneous Elastic Recovery of the PTT Filament at Different Elongations (Es)

Load (cN/tex)	First stretch cycle				Fifth stretch cycle			
	E = 5%	E = 10%	E = 20%	E = 30%	E = 5%	E = 10%	E = 20%	E = 30%
1.0	100	94.3	86.0	73.2	93.0	90.3	79.4	65.5
0.5	100	95.3	87.1	74.6	95.2	91.5	80.4	66.6
0.3	100	96.4	87.5	75.0	98.2	92.6	80.9	67.4
0.1	100	95.3	87.6	75.5	94.8	91.0	82.8	70.5

Crosshead speed = 500 mm/min.

cause there was some similarity in macromolecular chains between PTT and PET, the tenacity of the PTT filament was between those of PET and PU filaments but was closer to PET. It was necessary to measure the optimal magnitude of load to ensure the accuracy and precision of the measurement results. In this study, we chose a load approximate to that for the PET filament. It is shown in Table V and Figure 6 that within the load range of 0.1–1.0 cN/tex, the the measurement results changed regularly. However, the smaller the load, was the poorer the relative precision of the measurements was results and at a low elongation, the abnormal phenomena of zero or negative values of the instantaneous elastic recovery occurred. Therefore, for the sake of precision measurement and comparison with other aromatic polyester filaments, we suggest load of 0.5 cN/tex during the measurement of the instantaneous elastic recovery of PTT filaments.

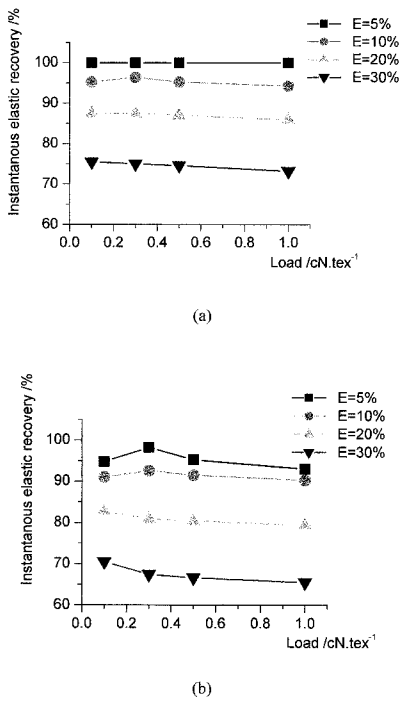

Figure 6 Effect of load on the instantaneous elastic recovery of the PTT filament at different elongations (*E*s): (a) first and (b) fifth stretch cycles.

Mechanical analysis of the instantaneous elastic recovery of the aromatic polyester filaments

It is well known that the number of methylene groups in the repeat unit influences the physical properties of many polycondensation polymers, such as polyester and polyamide; this is called the odd–even effect.[10] There are rigid benzene rings in macromolecular chains of aromatic polyesters, such as PET, PTT and PBT, which are all semicrystalline polymers. The elastic recovery behaviors of the macromolecular chains could be considered to belong to the quasispring elastic recovery mechanism. It has a prominent influence on the instantaneous elastic recovery of filaments such as structures of crystal and orientation in morphology and macromolecular chain conformation in the crystalline structure.

X-ray diffraction studies reported in a previous publication[11] showed that there are major differences between the molecular conformations in the crystalline unit cells of these three aromatic polyesters. The *c* axis dimension of PET corresponds to a molecular conformation, which is very nearly planar, with the chains almost fully extended. According to Jakeways et al.,[11] the *c* axis of the elementary lattice cell reaches about 98% of the length of the maximum drawn chain of the repeat unit in PET fibers. Only a slight stretching, about 6%, is to be expected for the fully drawn filament. Therefore, the initial modulus of the PET filament was high, which was in line with the expectation. The reversible elastic recovery of the PET filament could not reach even at a low elongation.

Although the macromolecular chains of PBT in crystalline lattice are in a planar zigzag conformation, the same as PET, the chain structure in the PBT filament is not fully extended, and this leads to major differences between PBT and PET. As there are four flexible methylene groups in the repeat unit of PBT macromolecular chains, the PBT filament has more flexibility than PET, so it has a good elastic recovery superior to that

of the PET filament. For PBT, however, two crystal forms (e.g. α and β forms) were found, and the transformation between two forms took place reversibly by mechanical deformation, changing from the α form (gauche–trans–gauche conformation) to the β form (trans–trans–trans conformation) by elongation and inversely by relaxation.[12,13] The crystal in the as-spun PBT filament belonged to the α form. The length of the elementary lattice cell in the as-spun PBT filament was about 86%. The transformation from the α form to the β form began to occur when the α form was drawn by 6% and was completed by 12%, in which the drawn β form was a length of 98% of the maximum drawn conformation of the repeat unit. It was obvious that the PBT filament could easily deform in a low elongation and return to the original reversibly when the external force was removed, so the PBT filament had a better elastic recovery than that of the PET filament and showed full recovery. For the PBT filament, however, there exists more methylene group sequences in the repeat unit than for the PET and PTT filaments. It is easy to crystallize so as to retard the deformation of macromolecular chains and influence the instantaneous elastic recovery of the PBT filament, especially at a high elongation.

For comparison with PET and PBT, there are three flexible methylene groups between terephthaloyl in the repeat unit of PTT. It is an odd number of methylene groups so there cannot be any center of symmetry in the middle of the trimethylene part of the chain. The molecular chains in the crystalline regions of PTT take a helical form, and there are probably two gauche bonds with an angle of 60° in the glycol residue, which accounts for the fact that the chains are far from fully extended. This type of molecular arrangement, in which successive terephthaloyl residues are inclined to a crystal axis by opposite inclinations, gives a *Z*-shape arrangement.[14] The fiber identity period of PTT is only 76% of the repeat distance for a fully extended chain. It has been reported that when they are subjected to external stress, the crystalline regions in PTT respond immediately as the overall strain increases.[11] It is then assumed that on longitudinal extension, some twist of the gauche toward the trans conformation should occur. The extension of macromolecular chains happens easily as there is only the transformation from the C–C bond to the C–O bond, and no transformation of the crystal forms occurs. Unlike PBT, structure deformation has not been found in PTT fibers after removal of the extension, and the conformation of PTT polymer chains is such that only departure from the gauche conformation of the trimethylene groups would result in an extension of the chain. Thus, the conformational arrangement within the trimethylene group sequences of PTT allows for a certain flexibility of the chain, which responds immediately to the applied stress and deforms as though it were a coiled spring.[15] As such, the transformation of conformation appeared to be reversible, possessing a good intrinsic elastic recovery in the macromolecular chains of PTT. It was, therefore, obvious that the elastic recovery of the PTT from the extension was very good and was considerably higher than that of PBT, particularly at a high elongation. Hence, the PTT filament had a high elasticity. This specific feature may explain the better performance in the instantaneous elastic recovery for PTT as compared to the PET and PBT filaments.[16]

CONCLUSIONS

Both the initial modulus and the tenacity of the PTT filament were lower than those of the PBT and the PET filaments, whereas the breaking elongation of the former was higher than those of the PBT and PET filaments. This is why the PTT filament possessed a softer feeling than the PBT and PET filaments.

The instantaneous elastic recovery of the PTT filament was significantly higher than those of the PBT and PET filaments. Moreover, the PTT filament had a high instantaneous elastic recovery, even at a high elongation. The PBT filament had a higher elastic recovery at low elongation, and its instantaneous elastic recovery was higher than that of the PET filament. However, the instantaneous elastic recovery of the PBT filament decreased significantly at high elongation. The results of a comprehensive comparison of instantaneous elastic recovery among these three aromatic polyester filaments showed that the PTT filament was the best, followed by the PBT filament, and then the PET filament.

When measuring the instantaneous elastic recovery of PTT filament with a Textechno tensile testing machine, we found it appropriate to set up the crosshead speed at 500 mm/min with an elongation of 20% and a load of 0.5 cN/tex.

The outstanding instantaneous elastic recovery of the PTT filament resulted from its helical conformation with the successive monomer units arranged in a crystal lattice. This conformation mades the length of the *c* axis of the PTT lattice approximately 76% that of the maximal extended conformation of repeat units and, thus, endowed the macromolecular chains of PTT with instantaneous elastic recovery after stretching. From these results, we suggest that the crystal lattice responded immediately to the applied stress and deformed as though it was a coiled spring, which is a quasispring elastic recovery mechanism. Hence, the PTT filament had a high intrinsic elasticity, resulting in a good instantaneous elastic recovery to longitudinal extension.

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