Thermotropic Liquid-Crystalline Copolyester (Rodrun LC3000)/Thermoplastic Elastomer (SEBS) *In Situ* Composites: II. Mechanical Properties and Morphology of Monofilaments in Comparison with Extruded Strands

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ABSTRACT: Monofilaments of *in situ* composites were prepared from an immiscible blend of a thermotropic liquid-crystalline polymer (TLCP), Rodrun LC3000, and a thermoplastic elastomer, styrene–(ethylene butylene)–styrene (SEBS), by a melt spinning process. Mechanical properties and the morphology of the composite monofilaments were investigated and compared with those of the extruded strands previously reported. The stresses at all tensile strains of the composite monofilaments were much higher than those of the extruded strands. The tensile strengths of both extruded strands and monofilaments were comparable, but the elongation at break of monofilaments dropped considerably. The tension sets of composite monofilaments were slightly higher than those of extruded strands. All composite monofilaments with TLCP content of ≤15 wt % exhibited good elastic recovery under the applied strain up to 200%. The dynamic mechanical storage modulus at 25°C of 10 wt % TLCP composite monofilament increased fourfold compared with that of the composite extruded strand and fivefold compared with that of the neat SEBS monofilament. The dramatic enhancement in the mechanical properties of *in situ* composite monofilaments is due to the formation of finer and longer TLCP fibrils (length-to-width ratio > 100) than those formed in the extruded strands. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 90: 518–524, 2003

Key words: composites; liquid-crystalline polymers; thermoplastic elastomers; morphology; mechanical properties

INTRODUCTION

An improvement in the mechanical properties of thermoplastic elastomers can be obtained by addition of fibrous fillers.[1–4] However, incorporation of solid fibers into the molten polymer matrix normally results in an increase in the melt viscosity of the blend system. This change leads to some difficulties in processing, such as machine wear problems, poor mold filling, and increased power consumption. Increasing temperature to counterbalance viscosity may cause a thermal degradation. Also, fibers are broken down into fragments due to the high shear stress during mixing, resulting in a significant reduction in the properties of composite materials. A new possible way to circumvent these problems is the *in situ* generation of short fibers in an immiscible blend of a thermotropic liquid-crystalline polymer (TLCP) with a thermoplastic[5–7] or a thermoplastic elastomer (TPE).[8–11] TLCPs are known to possess superior physical properties, such as high strength, good thermal properties, and low melt vis-

cosity. Under appropriate processing conditions and fabrication method, the dispersed TLCP droplets can be elongated by an elongational flow field and frozen in the matrix after rapid cooling. This type of blend is therefore called an *in situ* composite.[12] The main problem to be investigated for this type of blend is to find the optimum processing conditions to obtain a fibrillar morphology for the TLCP phase. Fabrication methods for the finished products also play an important role in the formation of TLCP fibrils and the degree of molecular ordering in both the dispersed and matrix phases. It has been observed that TLCPs fabricated in the form of a fiber have a higher degree of molecular orientation than those in the form of a sheet and those formed by injection molding.[13] To enhance the mechanical properties of TPEs while maintaining their good elastic recovery, the appropriate processing conditions and fabrication techniques to generate TLCP fibers with high aspect ratio should be established. To our knowledge, investigation of the properties of TLCP/TPE *in situ* composites in the form of extruded strand in comparison with monofilament is still lacking.

The aims of our work are to establish appropriate processing conditions and especially to find the optimum amount of TLCP needed for preparation of an *in*

situ reinforced thermoplastic elastomer, styrene–(ethylene butylene)–styrene (SEBS), while maintaining the important properties of the elastomer, such as high elasticity and low tension set value. The properties of SEBS (Kraton G-1652) blended with a TLCP [Rodrun LC3000, a copolyester of *p*-hydroxy benzoic acid (HBA) and polyethylene terephthalate (PET); 60/40 mole ratio], prepared in the form of extruded strands, have been reported in Part I of this series.[14] In this second paper, we report the mechanical properties and morphology of the same composite system, but the specimens were fabricated as monofilaments by a melt spinning process. Under high extension, it is expected to create TLCP fibrils with high aspect ratio and give rise to a higher level of improvement in the mechanical properties of these filaments. The obtained results are presented in comparison with those of the extruded strands that were reported in Part I.[14]

EXPERIMENTAL

Materials

The polymer dispersed phase used in this work was Rodrun LC3000, a TLCP supplied by Unitika Company, Ltd. (Tokyo, Japan) and the matrix phase was Kraton G-1652, a TPE that was kindly provided by Shell Chemical Company (Houston, TX). Rodrun LC3000 is a copolyester of 60 mol % *p*-hydroxy benzoic acid (HBA) and 40 mol % poly(ethylene terephthalate) (PET), with a melting point of 220°C and a density of 1.4 g/cm^3. Kraton G-1652 is a triblock copolymer of SEBS, with a styrene (hard segment)/rubber (soft segment) ratio of 29:71 and a density of 0.91 g/cm^3. The materials were dried in a vacuum oven at 70°C for at least 12 h before use.

Preparation of monofilaments

In situ composites comprising SEBS and LC3000 at various compositions were prepared as extruded strands by melt blending in a twin screw extruder as described in the previous paper (Part I).[14] The monofilaments were prepared by first cutting the extruded strands into pellets and then melt spinning the pellets with a mini-extruder (screw diameter, 16 mm; length-to-diameter ratio, 24) that was equipped with a monofilament line (Randcastle, RCP-0625, Cedar Grove, NJ). The diameter of the capillary die was 1.15 mm and the screw speed was fixed at 2 rpm. The processing temperature profile was 190/220/225/230°C, representing the temperatures at the hopper zone, the two-barrel zones and the die zone, respectively. After passing through the die outlet, the monofilament was uniaxially drawn and immediately quenched in a water bath to restrict relaxation of the achieved extension of TLCP domains. A monofilament diameter of ∼500 μm was obtained. This diameter corresponds to a draw ratio of 5.3 (area ratio of the die and the filament cross sections).

Tensile tests

The tensile tester and conditions used for tensile and tension set measurements of monofilaments were the same as those described in the previous paper,[14] except that a full scale load cell of 100 N was used and the averaged value was determined from at least 10 measurements. All measurements were carried out at 25°C. The tensile properties measured were the stresses at 100 and 300% strains, tensile strength, elongation at break, and tension set.

Dynamic mechanical analysis

The dynamic mechanical analysis (DMA) was performed on a DVE-Rheospectoler (DVE-V4, Rheology, Japan) to obtain dynamic storage modulus (E'), dynamic loss modulus (E''), and the loss tangent (tan δ). The tensile strain was set at 1%, with an oscillating frequency of 10 Hz. The gauge length was set at 5 mm and the heating rate was 5°C/min. The measurements were carried out in the temperature range −100–150°C in a gentle flow of nitrogen. The dynamic moduli (E' and E'') and tan δ were recorded as a function of temperature with the detecting step of 1°C.

Morphology characterization

The fracture surfaces of all specimens were observed with a scanning electron microscope (SEM; Hitachi 2500, Ibaraki, Japan), operated at an accelerating voltage of 15 kV. Prior to examination, the specimens were dipped in liquid nitrogen for 30 min and fractured. The fracture surfaces were sputter coated with palladium for enhanced surface conductivity (Hitachi E-102 ion sputter, Ibaraki, Japan).

To inspect the actual size of TLCP fibers in the extruded strands and monofilaments more clearly, the matrix was dissolved away with toluene. The geometry of TLCP fibrils was directly observed with a polarizing optical microscope (Zeiss, model Axiolab, Carl Zeiss Jena GmbH, Jena, Germany) attached to a digital camera (Sony DSC-S85, Japan). The optical micrographs were taken at the magnifications of 100 and 500.

RESULTS AND DISCUSSION

Mechanical properties and morphology of *in situ* composite monofilaments containing 0, 10, and 20 wt % TLCP are compared with the results of extruded strands taken from the first paper[14] to illustrate the
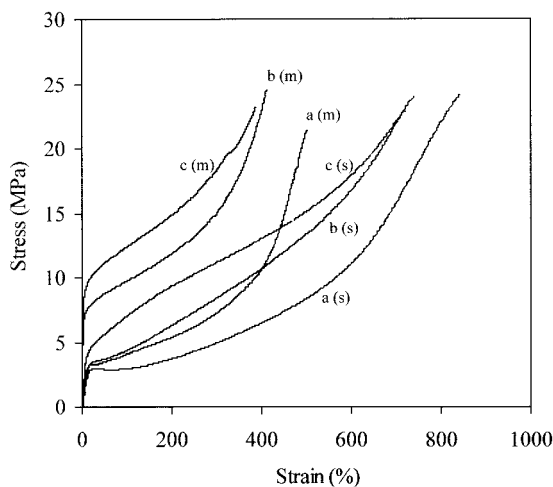
Figure 1 Stress–strain curves at 25°C of TLCP/SEBS extruded strands (s) and monofilaments (m) containing (a) 0, (b) 10, and (c) 20 wt % TLCP.

effect of fabrication methods on the properties of *in situ* composites.

Tensile properties

The stress–strain curves of extruded strands and monofilaments of neat SEBS and the composites containing 10 and 20 wt % TLCP are shown in Figure 1. All specimens exhibit similar characteristic of a typical elastomer. Comparing the curves of the neat SEBS and the composites at the same composition but fabricated by a different method, it is clearly seen that specimens fabricated in the form of monofilaments exhibit higher value of stress than do the corresponding extruded strands. For the case of neat SEBS [curves a(s) and a(m)], the difference is due to the increase of the draw ratio (from ~1 for the extruded strand to ~5 for the monofilament), which results in the deformed hard phase and the enhancement of chain orientation and

tautness of tie molecules connecting the hard styrene blocks in the drawn direction of monofilament, as suggested by Lee et al.[15] For the composites, the more pronounced difference in stress value is due to the reinforcement by TLCP fibers that have a much higher aspect ratio when formed as monofilaments. These TLCP fibers are consequently responsible for the observed increase in the tensile stress in the monofilaments compared with that in the strands. The steep increase in the stresses of all monofilaments appears in the strain region 300–500%, whereas the rapid increase in the stresses of extruded strands appears at strains >600%. Both extruded strands and monofilaments of neat SEBS exhibit a yield-like feature at a strain of ~20%. This behavior agrees well with the results of structural studies on oriented SEBS triblock copolymers reported by Lee et al.[15] and Daniel et al.[16] These authors explain that the styrene block becomes deformed as the cylindrical domains are oriented parallel to the flow direction when the polymer is extruded through a narrow capillary die. As a consequence of this deformation, the material is relatively stiff and exhibits a yield point. However, no yield point appears in all composites prepared in this work. This result may be due to the presence of TLCP fibrils, which obstructs necking of the specimens.

To compare the results in more quantitative way, the averaged values of stress at 100 and 300% strains, ultimate tensile strength, and elongation at break of extruded strands and monofilaments are listed in Table I. The tensile stress of the extruded strands increases with increasing TLCP content. Comparing stresses of the strands of composites containing 10 and 20 wt % TLCP with that of the neat matrix, the percent increases in the stress at 100% strain of the composites are ~40 and ~120%, respectively, and the percent increases in the stress at 300% strain are 56 and 113%, respectively. Compared with the neat SEBS monofilament, the monofilaments containing 10 and 20 wt %

TABLE I
Mechanical Properties of Extruded Strands and Monofilaments of neat SEBS
and TLCP/SEBS Composites Containing 10 and 20 wt % TLCP[a]

Mechanical property[b]	SEBS		10 wt % TLCP/SEBS		20 wt % TLCP/SEBS	
	Strand	Monof.	Strand	Monof.	Strand	Monof.
M100 (MPa)	3.2 (0.1)	4.2 (0.6)	4.5 (0.2)	9.7 (1.5)	7.0 (0.2)	12.4 (0.7)
M300 (MPa)	5.4 (0.2)	7.3 (1.1)	8.4 (0.2)	15.0 (1.6)	11.5 (0.4)	18.3 (1.1)
TS (MPa)	24.6 (1.9)	23.6 (3.4)	24.8 (1.4)	23.5 (3.3)	22.9 (1.2)	23.1 (4.1)
EB (%)	868 (86)	502 (55)	768 (46)	412 (62)	774 (85)	389 (30)
E' (MPa)	36.2	46.6	57.3	295	64.9	620
Set-100 (%)	5.6 (0.4)	4.0 (2.7)	6.0 (1.0)	7.4 (2.1)	6.4 (1.1)	14.8 (2.9)
Set-200 (%)	8.1 (2.6)	9.2 (1.1)	10.7 (1.1)	14.0 (2.2)	12.3 (1.2)	30.0 (5.0)
Set-300 (%)	12.2 (1.9)	16 (4.7)	15.1 (0.7)	23.6 (5.5)	26.5 (4.2)	40.2 (4.9)

[a] Properties were measured at 25°C.
[b] Tensile stress at 100% strain (M100), tensile stress at 300% strain (M300), ultimate tensile strength (TS), percent elongation at break (EB), dynamic storage modulus (E') at 25°C, and tension sets at 100% strain (set-100), 200% strain (set-200) and 300% strain (set-300).
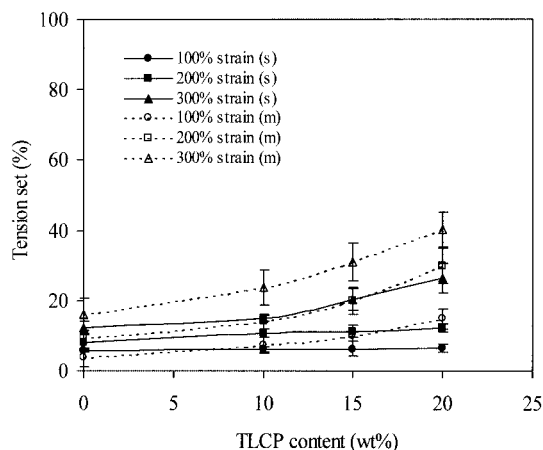
Figure 2 Tension set values of TLCP/SEBS extruded strands (s) and monofilaments (m) versus TLCP content. The measurements were performed at 100, 200, and 300% strains.

TLCP show increases in tensile stress at 100% strain of ~130 and ~200%, respectively, and at 300% strain of 105 and 150%, respectively. The higher percent increases in tensile stress of the composite monofilaments than those of the composite extruded strands are due to the more effective reinforcement by TLCP fibrils with higher aspect ratio. This reinforcement is evident in the composite morphology results that are presented in the next section. The elongation at break values of all monofilaments are reduced to one-half those of the corresponding extruded strands, but they are still >400%. In contrast, the tensile strengths of all specimens are comparable, implying that the property of the matrix phase predominates at high strain. At such a high extension, debonding at the interface and/or some fiber breakage might occur; therefore, the reinforcing elements have almost no effect on the tensile strength.

The effects of the level of applied deformation and TLCP content on the value of tension set (permanent elongation relative to the original length) of extruded strands and monofilaments are compared in Figure 2. The averaged tension set values of the specimens with 0, 10, and 20 wt % TLCP are also included in Table I. In the case of neat SEBS, the tension set values of both extruded strand and monofilament at the same degree of deformation are comparable. The tension set values of the composite monofilaments are, however, higher than those of the composite extruded strands, particularly at high degree of deformation. The higher set values of the composite monofilaments might be attributed to the debonding and/or breakage of some TLCP fibers when a high level of strain was applied. After removal of the stress, the monofilament could not completely retract to the original length due to the obstruction by some loose fibers or by fragments of broken TLCP fibers. The tension set values of both

extruded strand and monofilament increase with increasing level of TLCP concentration and with the increase degree of deformation. With addition of TLCP up to 20 wt %, the tension sets of all extruded strands under an applied deformation up to 300% strain are within the acceptable limit of the set values for thermoplastic elastomers (i.e., not exceeding 30%). The set value up to 30% under deformation of 300% elongation was reported to be typical for a triblock styrenic thermoplastic elastomer.[17] In the case of the composite monofilaments, the set values of all specimens are also under 30%, except the one with 20 wt % TLCP under 300% strain that shows a set value of 40%. This result suggests that the maximum concentration of TLCP for preparation of *in situ* composite monofilaments should not exceed 15 wt %.

Dynamic mechanical properties

DMA is a very useful method for investigation of mechanical properties, relaxation process, and the associated molecular motions and internal changes in polymeric materials as a function of temperature. In this study, the dynamic measurements were performed in the temperature range −100–150°C. Plots of dynamic storage moduli (E') and loss tangent (tan δ $= E''/E'$) versus temperature, for the neat SEBS and the composites containing 10 and 20 wt % TLCP prepared as extruded strands and monofilaments, are shown in Figures 3 and 4, respectively. The E' values at 25°C of extruded strands and monofilaments are also included in Table I. It is seen that the magnitude of E' decreases with increasing temperature, and all specimens exhibit three state regions (i.e., the glassy, rubbery, and melting states). The transition at lower
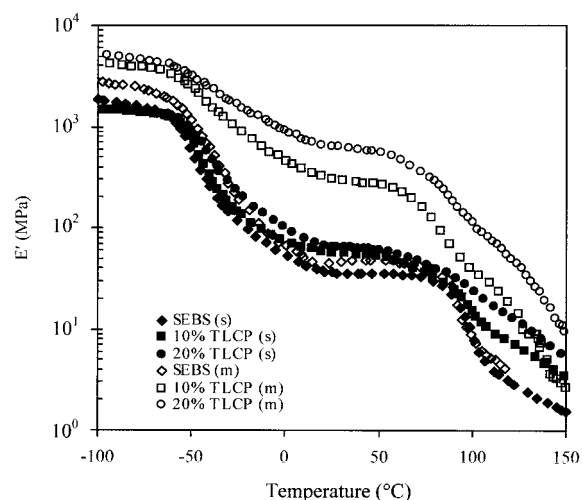

Figure 3 Dynamic storage moduli (E') as a function of temperature of the extruded strands (s) and monofilaments (m) of neat SEBS and composites containing 10 and 20 wt % TLCP.
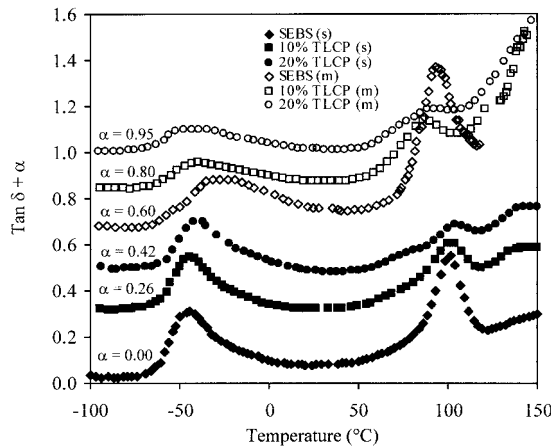
Figure 4 Tan δ as a function of temperature of the extruded strands (s) and monofilaments (m) of neat SEBS and composites containing 10 and 20 wt % TLCP (α = shift factor).

temperature is due to the glass transition (T_g) of EB block in SEBS, which is about $-45°C$, and the transition at $\sim100°C$ corresponds to the T_g of the S block. For neat SEBS, the E' curve of the monofilament is slightly higher than that of the extruded strand. The reason for the slight increase in storage modulus of the neat SEBS monofilament is the same as that described for the increase of tensile modulus. Consider E' values of all specimens in the rubbery state (-45–$100°C$); E' increases with increasing TLCP content for both extruded strand and monofilament. Interestingly, incorporation of 10 and 20 wt % TLCP into monofilaments brought about big jumps in E' values of ~5- and ~10-fold, respectively, which are higher than that of the neat SEBS monofilament. In contrast, the increase in E' for the extruded strand with addition of 20 wt % TLCP was only about twice that of the neat SEBS extruded strand. The great improvement in E' of the monofilament compared with that of the extruded strand at the same composition is attributed mainly to the reinforcement by TLCP fibrils with much higher aspect ratio. This higher aspect ratio is illustrated in the section on morphology.

The longitudinal modulus of *in situ* composite is often modeled with the Halpin–Tsai equation[18]

$$\frac{E_c}{E_m} = \frac{1 + ABX}{1 - BX} \quad (1)$$

where $A = 2(l/d)$; $B = [(E_f/E_m) - 1]/[(E_f/E_m) + A]$; and E_c, E_f, and E_m are the elastic moduli of the composite, the reinforcing fiber, and the matrix phase, respectively; and X is the volume fraction and l/d is the aspect ratio (length to width) of reinforcing fiber, which is assumed to be uniform. In addition, this equation is based on the assumption of continuity of stress and strain along the fiber/matrix interface. The value of E_c for the composite monofilament containing

10 wt % TLCP ($X = 0.07$) is calculated as 648 MPa by substituting $E_f = 59$ GPa (taken from the literature[19]), $E_m = 46.6$ MPa (obtained from DMA data at 25°C; this value is approximately equivalent to Young's modulus), and an assumed l/d value of 100, into eq. (1). This calculated value is about twice that of the measured E' value (295 MPa, at 25°C). The difference between the calculated and the measured E_c values might be due to the broad distribution of l/d values in the real system. It is interesting to note that the value of E' for 10 wt % TLCP/SEBS monofilament is about four times higher than that of the corresponding extruded strand, or comparable to that of 30 wt % TLCP/SEBS extruded strand reported in part I.[14]

In the melting state ($>100°C$), incorporation of TLCP also significantly improves E' of both extruded strand and monofilament specimens. With increasing temperature in this region, the E' values of all monofilaments drop faster than those of the extruded strands. Therefore, E' values of extruded strand and monofilament at equal composition seem to converge to the same value. For instance, the E' values of extruded strand and monofilament containing 10 wt % TLCP become equal at $\sim140°C$. The faster drop of E' in the melting state of composite monofilament might be due to the reduction of the intrinsic strength of TLCP fibers, in which the degree of molecular ordering decreased as the temperature increased, and/or due to the breakage of some TLCP fibers.

Tan δ values as a function of temperature for the extruded strands and monofilaments are shown in Figure 4. Each tan δ curve was shifted in the vertical axis by different shift factors (α) to avoid overlapping. The neat SEBS extruded strand shows two peaks around -45 and $100°C$, corresponding to the T_gs of EB block and S block, respectively. The broad peak of EB block at $-45°C$ indicates a broad distribution of relaxation times, due to the presence of the amorphous EB segments.[20] In the case of neat SEBS monofilament, the tan δ peak of EB block is even broader and shifts to higher temperature when compared with SEBS extruded strand. This result may indicate that the segmental mobility in EB block is reduced due to the increased orientation in the monofilament. The resulting EB chain alignment along the drawing direction would reduce the free volume for molecular motion and thus decreased the configurational entropy of the EB segments. Similarly, this behavior was observed and explained by Amash et al.[21] for the blend of poly(propylene) (PP) and ethylene–propylene–copolymer (EPM). The T_g of the S block in monofilament shifts slightly to a lower temperature compared with that of the S block in the extruded strand. Hence, tan δ peaks of EB and S blocks in SEBS monofilament shift towards each other, indicating an increase in the degree of miscibility of the two phases. For the composite extruded strand, the T_gs of EB block and S block
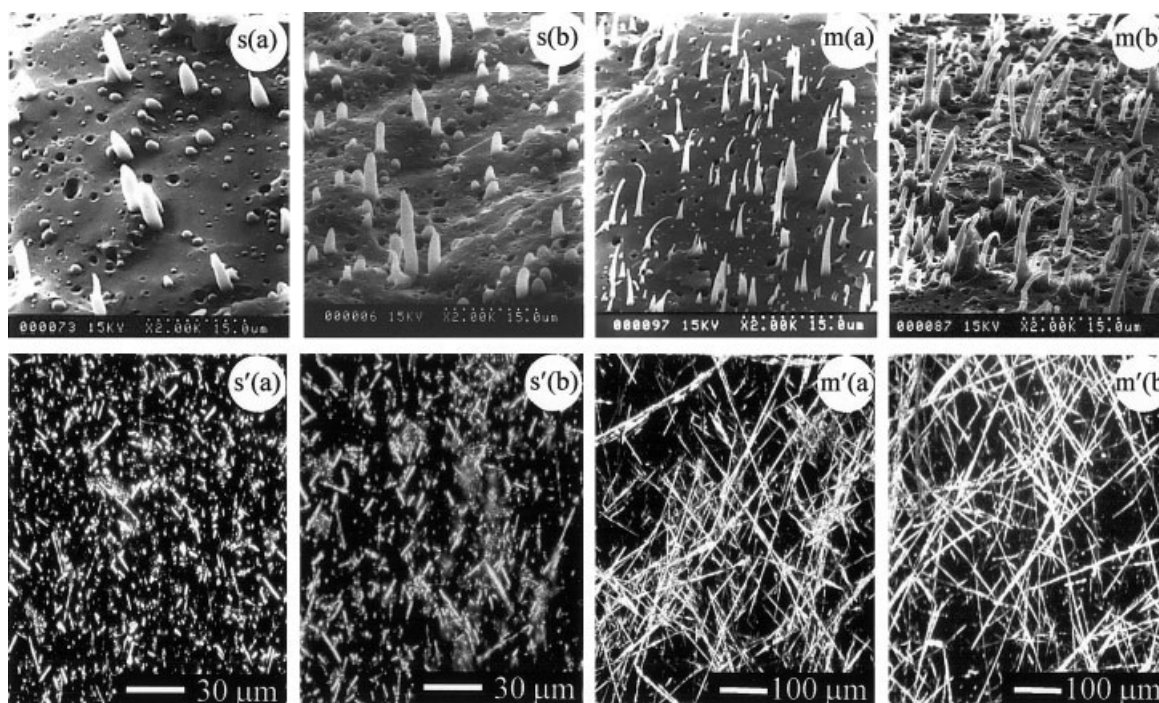
Figure 5 SEM micrographs (top row) of fracture surfaces of strands (s) and monofilaments (m); and optical micrographs (lower row) of TLCP fibrils extracted from strands (s′) and monofilaments (m′). These composites contain TLCP at (a) 10 wt % and (b) 20 wt %.

shift slightly to higher temperature with increasing TLCP content. The shift of T_g to higher temperature can be attributed to a constraint SEBS portion adsorbed on TLCP fibers. This result agrees well with that reported by Verhoogt et al.[9] for the Vectra A900/ SEBS G1650 blend system. The decrease in the tan δ peak height of EB and S blocks with increasing TLCP content was due to the dilution effect. In the case of composite monofilaments, T_gs of EB and S blocks shift slightly to lower temperature compared with the T_g of neat SEBS monofilaments. The presence of TLCP fibrils may inhibit the extension of both EB and S blocks in the matrix phase. In summary, the tan δ plots clearly demonstrate that the increase of chain orientation significantly affects the observed relaxation process, where the most affected relaxation belongs to EB rubber segment.

Morphology

The SEM micrographs of fracture surfaces at 2000× magnification (top row) and OM micrographs of extracted TLCP fibers (lower row) of TLCP/SEBS extruded strand (s) and monofilament (m) are shown in Figure 5. The composites contain (a) 10 and (b) 20 wt % TLCP. For the composite extruded strands, most of the dispersed TLCP domains appear as droplets, whereas elongated TLCP fibers with much higher aspect ratio are seen in the monofilaments. The diameters of TLCP fibers in the extruded strands and mono-

filaments are in the ranges 2–3 and 1–2 μm, respectively, whereas the length of some long fibers observed in the extruded strands and monofilaments are in the ranges 20–30 and 400–500 μm, respectively. It follows that the aspect ratios of TLCP long fibers formed in the extruded strands are in the range 10–15 and those in the monofilaments are in the range 200– 400. Therefore, a tremendous increase in fiber aspect ratio formed in the composite monofilaments is responsible for the great enhancement in the mechanical properties presented in the previous section. However, the wide distribution of TLCP fiber aspect ratio seen Figures 5 (lower row) is one of the reasons for the difference in the measured composite $E′$ value compared with the one predicted with the Halpin–Tsai equation.

CONCLUSIONS

Mechanical properties and morphology of RodrunLC3000/SEBS *in situ* composites prepared in the form of extruded strands and monofilaments were compared. The following conclusions can be drawn from this study:

1. Comparing at the same composite composition, tensile stresses of the composite monofilaments were almost twice that of the extruded strands at 100 and 300% strains. The values of tension set of the composite monofilaments were somewhat

higher than those of the extruded strands. To obtain composite monofilaments with good elastic recovery (i.e., set value < 30%), the amount of added TLCP should not exceed 15 wt %.

2. A great enhancement in dynamic storage moduli (E') was also achieved in composite monofilaments. For 10 and 20 wt % TLCP composite monofilaments, the increases in E' were by five- and 10-fold, respectively, when compared with that of the neat SEBS monofilament. For the composite extruded strand containing 20 wt % TLCP, the increase in E' was only about twice that of SEBS extruded strand.

3. The morphology of TLCP/SEBS composites strongly affected their mechanical properties. The dispersed TLCP phase appeared mostly in the form of droplets in the extruded strands, whereas long TLCP fibrils with very high aspect ratio were observed in the composite monofilaments. This difference resulted in the great enhancement in the mechanical properties of the monofilaments.

In conclusion, this work has illustrated that filaments of SEBS thermoplastic elastomer reinforced with TLCP can be produced with good elastic recovery, very high strength (4–5-fold that of the neat SEBS), and high stiffness with reinforcement loading of only 10 wt % (or ~7 vol %). More importantly, this type of composite is melt processable. In addition, ultrafine TLCP fibers with diameters of 1–2 μm could be prepared from *in situ* composite monofilaments by dissolving away the SEBS matrix with toluene. A potential application of such ultrafine fibers is the production of filters with high strength and high chemical and thermal resistance.

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