# Novel CO2 Laser Drawing of Thermotropic Liquid Crystal Polymer and Poly(ethylene 2,6-naphthalate) Blend Fibers

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**ABSTRACT:** Thermotropic liquid crystal polymer (TLCP)/poly(ethylene 2,6-naphthalate) (PEN) were prepared by a melt blending, and were melt spun by a spin-draw process. In this study, we suggest novel drawing technology using the $CO_2$ laser that can directly and uniformly heat up fiber inside to prevent the formation of ununiform structures in conventional heat drawing process. The properties of the heat/laser drawn TLCP/PEN blend fibers were superior to those of any other handled fibers, and were rather more excellent than those of TLCP/PEN blend fibers annealed at 135°C for 10 min. It was confirmed that the $CO_2$ laser drawing made it possible to achieve the optimal drawing effect by draw ratio. The combined heating and $CO_2$ laser-drawing method has a great potential for industrial applications as a novel fiber-drawing process, and it can also be applied continuously to conventional spin-draw system. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 205–211, 2007

**Key words:** drawing; fiber; liquid crystalline polymers; polyesters

## INTRODUCTION

The melt blending of thermotropic liquid crystal polymer (TLCP), with relatively low melt viscosity, and thermoplastic polymer has attracted great attention because of their high mechanical properties, excellent thermal stability, and good processability.[1–12] TLCPs can be oriented to form fibrils in melt processing, and the oriented fibrous structures are developed in the extruded TLCPs. The development of highly ordered structures results in self-reinforcing characteristics.[13,14] Because of this reason, the melt blend fibers consisting of TLCP and thermoplastic polymer that prepared by conventional melt blending and spinning processes can be used for high performance fibers in various industrial fields.[8–10]

The structure and properties of a fiber are significantly affected by not only spinning process but also handing process after spinning, including drawing and annealing. In general, the drawing is a process that stretches the molecular chains of fibers along the direction of fiber axis in temperature range between glass transition temperature and melting temperature of a fiber. The drawing of a fiber results in the increase in the crystallinity and the orientation of amorphous region in fibers. As a consequence, the drawing process has a significant effect on the final properties of fiber. The heat-drawing processes, such as a roll drawing and a zone drawing, are in common use to perform the drawing process of conventional polyester fibers. In the roll-drawing process, surface of fibers is preferentially heated-up by heated roller, and that of the polyester fibers is also preferentially heated-up by high temperature air in the zone-drawing process.[15–17] However, because of the low thermal conductivity of polymers, a large temperature distribution is generated in fibers with distance from heat-source during drawing process. Therefore, the conventional heat-drawing process cannot heat up the fiber uniformly and continuously along the cross-sectional direction of the fibers, leading to the formation of ununiform or skin/core structures in fibers. Because of these drawbacks, it is limited to manufacture high performance fibers by conventional heat-drawing processes.

To improve uniformity of orientation of molecular chains, the drawing technology using $CO_2$ laser that can directly and uniformly heat up inside of fiber has been suggested in recent years.[17–23] The $CO_2$ laser that emit monochromatic light exhibits excellent coherence, good directionality, and high brightness, which make it possible to heat up directly and uniformly the inner part of the fiber.[24–26] However,

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$CO_2$ laser-drawing methods reported in the literature have serious disadvantage in that the processes were performed at very low drawing speed, and the low-drawing process is not possible to be applied in commercial scale process. In addition, no study on comparing the properties of laser drawn fibers with those of other handled fibers has been published to date.

In this study, poly(ethylene 2,6-naphthalate) (PEN) fibers reinforced with thermotropic liquid crystal polymer (TLCP) were manufactured by melt blending and spinning processes, and the $CO_2$ laser-drawing process was performed with the heat/$CO_2$ laser-drawing machine designed and constructed by our laboratory. Effects of $CO_2$ laser drawing on the mechanical properties of the blend fibers are discussed in comparison with heat drawing, cold drawing, and annealing processes in this article. Structure and properties of the blend fibers were investigated in terms of birefringence measurement, wide-angle X-ray diffraction (WAXD) analysis, differential scanning calorimetry (DSC), and tensile testing. In this study, we newly intend to suggest that the combined heating and $CO_2$ laser-drawing method have a great potential for industrial applications as a novel fiber-drawing process, making it possible to apply continuously to conventional spin-draw system.

## EXPERIMENTAL

### Materials and preparation of TLCP/PEN blend fibers

The conventional thermoplastic polymers used were the poly(ethylene 2,6-naphthalate) (PEN) with an intrinsic viscosity of 0.93 dL/g containing 0.1 wt % of dimethyl terephthalate (DMT) to enhance the processability of the blends, supplied by Hyo Sung, Korea. The thermotropic liquid crystal polymer (TLCP) used was flexible thermotropic liquid crystal copolyester synthesized from poly (*p*-hydroxy benzoate) (PHB) and poly (ethylene terephthalate) (PET) with a molar ratio of 80 : 20, purchased form Unitika, Japan. All the materials were dried at 120°C *in vacuo* for 12 h before being used to minimize the effect of moisture. The TLCP/PEN blends were prepared by a melt blending in Haake rheometer (Haake GmbH, Germany) equipped with a twin-screw. The predetermined TLCP/PEN blend compositions were 1 : 99 and 5 : 95 (w/w), respectively. The temperatures of the heating zones from the hopper to the die were set to 290, 300, 310, and 300°C, and the screw speed was fixed at 15 rpm. Melt-blended polymer chips were melt spun in the twin extruder that had a four hole-spinneret with a diameter of 1 mm, and the output rate was controlled at 5 g/min per hole. Then, the TLCP/PEN blended fibers were spun by spin-draw spinning method. During spin-draw process, the speeds of feed and take-up rollers in TLCP1/PEN blend fibers were 380 and 2550 m/min, respectively, and those in TLCP5/PEN blend fibers were 430 and 2550 m/min, respectively. In this study, the numerals 1 and 5 donate the weight percentage (wt %) of TLCP added to PEN.

### Drawing process on TLCP/PEN blend fibers

To investigate the effect of heat in drawing process on properties of the as-spun fibers, annealing process was performed at various temperature and for different time periods. The as-spun fibers were drawn in various conditions by using the heat/$CO_2$ laser-drawing machine self-constructed in our laboratory, as shown in Figure 1. Cold-drawing and heat-drawing processes were performed to characterize the effects of drawing on properties of the blend fiber, in comparison with that of $CO_2$ laser drawing. In the $CO_2$ laser-drawing process, $CO_2$ laser system was installed between feed roller and take-up roller, and $CO_2$ laser beam was irradiated continuously to the blend fibers in perpendicular to the direction of fiber drawing. Drawing conditions of TLCP/PEN blend fibers are shown in Table I. The draw ratio was calculated by the ratio of the speed of feed roller to that of take-up roller, and the diameter of feed and take-up rollers was equally 10 cm. The wavelength and applied generating power of $CO_2$ laser system (J48-2 model, Synard) were 10.6 μm and 37.5 W, respectively. The beam diameter and the beam divergence angle of $CO_2$ laser were 5.0 mm and 0.1 mrad, respectively. The real generating power of $CO_2$ laser that irradiated fiber was controlled by the laser power meter (POWER WIZARD™ 250 model, Synrad). From an industrial perspective, $CO_2$ laser-drawing methods reported in the literature are not appropriate for applying a practical-drawing process in industrial fields, because the $CO_2$ laser-drawing process was performed at very low drawing speed. Therefore, in this study, faster drawing speed should be applied to achieve the

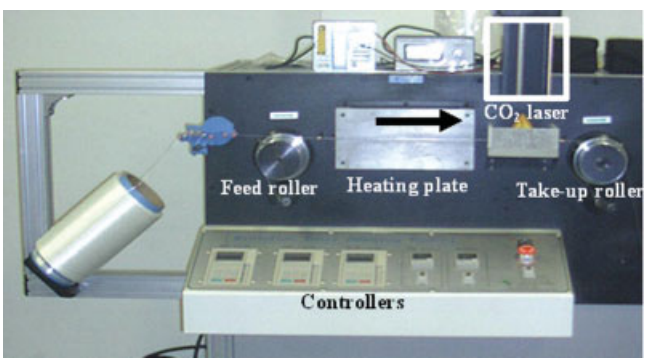


**Figure 1** Heat/$CO_2$ laser-drawing machine. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

**TABLE I**
**Drawing Conditions of TLCP/PEN Blend Fibers**

| Sample | Feed roller speed (rpm) | Heating plate temperature (°C) | $CO_2$ laser power (W) | Take-up roller speed (rpm) | Draw ratio |
|---|---|---|---|---|---|
| Cold drawn TLCP1/PEN fiber | 200 | – | – | 400 | 2 |
| Laser drawn TLCP1/PEN fiber | 200 | – | 35.5 | 600 | 3 |
| Heat drawn TLCP1/PEN fiber | 200 | 220 | – | 700 | 3.5 |
| Heat/laser drawn TLCP1/PEN fiber | 200 | 220 | 35.5 | 750 | 3.8 |
| Cold drawn TLCP5/PEN fiber | 200 | – | – | 400 | 2 |
| Laser drawn TLCP5/PEN fiber | 200 | – | 35.5 | 600 | 3 |
| Heat drawn TLCP5/PEN fiber | 200 | 220 | – | 700 | 3.5 |
| Heat/laser drawn TLCP5/PEN fiber | 200 | 220 | 35.5 | 750 | 3.8 |

requisite for commercial procedures when compared with the other reports.

### Characterization

The birefringence of the TLCP/PEN blend fibers was measured with Olympus polarizing microscope equipped with a Break compensator. The mechanical properties of TLCP/PEN blend fibers were measured at room temperature with an Instron 4465 tensile testing machine equipped with standard fiber grips, according to the procedures in the ASTM D 638 standard. The gauge length and crosshead speed were set to 20 and 5 mm/min, respectively. WAXD analysis was performed with a Rigaku Denki X-ray diffractometer with Ni-filtered Cu Kα X-rays ($\lambda = 0.1542$ nm). The apparent crystallite size ($L_{hkl}$) in the blend fibers was calculated with Scherrer's equation:[27]

$$L_{hkl} = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

where β is the half-width of the reflection peak, θ is the Bragg angle, $K$ is a correction factor ($K = 0.9$), and λ is the wavelength of the X-ray beam used. The interplanar spacing ($d$) and the number of repeat units per crystal in the blend fibers were calculated with the Bragg equation, $n\lambda = 2d \sin\theta$, and $L_{hkl}/d$, respectively. The thermal behaviors of blend fibers were investigated with DSC (TA instrument 2010) under nitrogen over the temperature range of 40 to 300°C employing a scanning rate of 10°C/min. The crystallinity of the TLCP/PEN blend fibers was calculated with the following equation:

$$X_{dsc}(\%) = \frac{\Delta H_m}{(1-b) \times \Delta H_m^o} \quad (2)$$

where $b$ is the content of TLCP in fiber, $\Delta H_m$ is the heat of fusion of sample, and $\Delta H_m^o$ is the heat of fusion of perfect crystal (103.2 J/g).

## RESULTS AND DISCUSSION

The structural behaviors of annealed polymers are altered by the accumulation of various structural processes, such as the disorientation, the recrystallization by nucleation and growth, the shrinkage, and the crystal decomposition; these changes are natural responses to the temperature and time during annealing.[8–10,28,29] The dependence of tensile strength and modulus on annealing temperature and time is shown in Figures 2 and 3. When the annealing temperature was 135°C, TLCP/PEN blend fibers exhib-

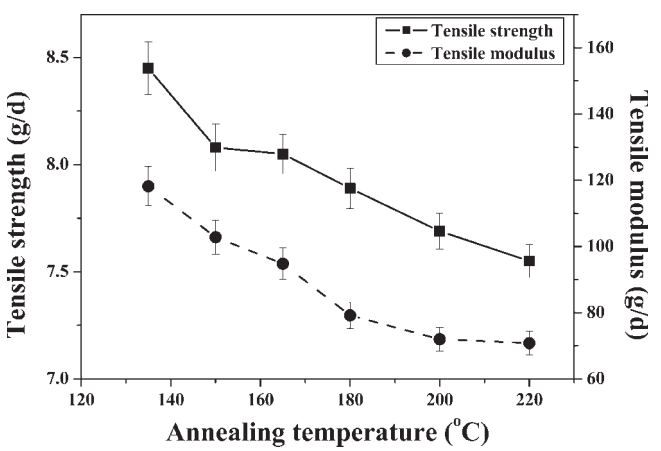


**Figure 2** Effect of annealing temperature on mechanical properties of TLCP1/PEN blend fibers annealed for 2 h.

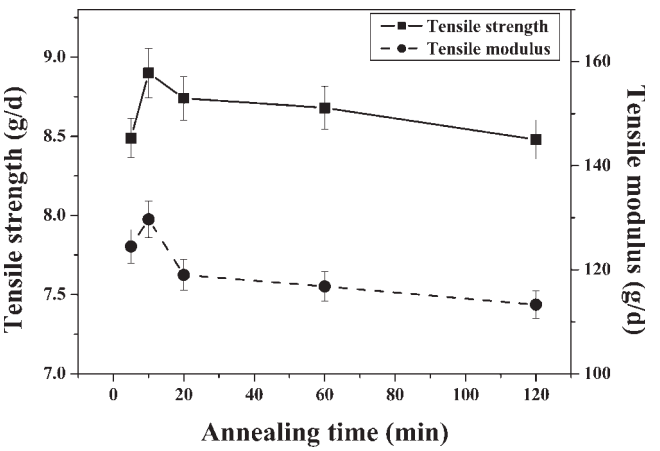


**Figure 3** Effect of annealing time on mechanical properties of TLCP1/PEN blend fibers annealed at 135°C.

**TABLE II**
**Tensile Strength and Modulus of TLCP/PEN Blend Fibers Before and After Annealing**

| | Before annealing | | | After annealing | | |
|---|---|---|---|---|---|---|
| Sample | Birefringence | Tensile strength (g/d) | Tensile modulus (g/d) | Birefringence | Tensile strength (g/d) | Tensile modulus (g/d) |
| TLCP1/PEN fiber | 0.416 | 7.22 | 103.9 | 0.436 | 8.90 | 129.70 |
| TLCP5/PEN fiber | 0.395 | 7.20 | 106.81 | 0.424 | 7.99 | 125.55 |

ited the maximum values of tensile strength and modulus. In addition, the tensile strength and modulus of the annealed blend fibers increased with increasing annealing time up to 10 min, and thereafter decreased. Thus, for TLCP/PEN blend fibers, the optimal annealing time was 10 min when annealing temperature was 135°C. The birefringence ($\Delta n$), tensile strength, and modulus of unannealed and annealed blend fibers are shown in Table II. The mechanical properties of TLCP/PEN blend fibers improved by annealing process, and the increase of birefringence may cause the improvement of mechanical properties of the annealed fibers.

The variation of birefringence of the TLCP/PEN blend fibers drawn at various conditions are shown in Table III. The relatively high birefringence of the blend fibers may be explained by the effect of spin-draw and TLCP phase. When TLCP is incorporated as a minor component into the blend fibers, it can form highly elongated domains parallel to the flow direction and improve the orientation, resulting in higher birefringence of the blend fibers.[8–10,30] Van Eijindhoven-Rivera et al.[31] reported that the degree of molecular orientation depended on the TLCP content in the blends and on the stress-transfer in TLCP-based polymer blends, and they proposed that the formation of uniform and continuous fibrils was accompanied by a high degree of orientation of TLCP phase. In general, the increase of birefringence means the development of molecular orientation. However, TLCP1/PEN blend fibers exhibited higher birefringence than that of TLCP5/PEN blend fibers. This result may be explained by lower speed of feed roller in the TLCP1/PEN blend fiber during the spin-draw process when compared with the TLCP5/PEN blend fiber. It can be deduced that, in this spin-draw process, the applied shear stress has more significant effect on molecular orientation of the blend fibers than TLCP content. The change in diameter of the TLCP/PEN blend fiber drawn at various conditions is shown in Table III. The diameters of the TLCP/PEN blend fibers decreased with increasing birefringence, indicating that the smaller diameter of the blend fibers was obtained at higher orientation.[9] The diameter of the TLCP1/PEN blend fibers was smaller than that of the TLCP5/PEN blend fibers, and this attributed to higher birefringence of TLCP1/PEN blend fibers.

The effect of drawing condition on the tensile strength and modulus of TLCP/PEN blend fibers is also shown in Table III. If $CO_2$ laser drawing is uniformly and continuously applied to the blend fibers, it is expected that the mechanical properties of laser drawn fibers were superior to those of heat drawn fibers. However, the mechanical properties of the laser drawn fibers were less excellent than those of heat drawn fibers, which may be explained by the fact that $CO_2$ laser was not able to supply enough energy to fully heat up the TLCP/PEN blend fibers in this experimental condition. Therefore, we suggest

**TABLE III**
**Mechanical Properties of the TLCP/PEN Blend Fibers**

| Sample | Birefringence | Diameter (μm) | Tensile strength (g/d) | Tensile modulus (g/d) |
|---|---|---|---|---|
| Undrawn TLCP1/PEN fiber | 0.416 | 29.5 | 7.22 | 103.9 |
| Cold-drawn TLCP1/PEN fiber | 0.417 | 29.1 | 7.61 | 115.3 |
| Laser-drawn TLCP1/PEN fiber | 0.430 | 28.7 | 7.98 | 119.4 |
| Heat-drawn TLCP1/PEN fiber | 0.433 | 28.4 | 8.66 | 126.1 |
| Heat/laser-drawn TLCP1/PEN fiber | 0.441 | 27.6 | 8.97 | 149.6 |
| Annealed TLCP1/PEN fiber | 0.436 | 28.5 | 8.90 | 129.70 |
| Undrawn TLCP5/PEN fiber | 0.395 | 30.0 | 7.20 | 106.81 |
| Cold-drawn TLCP5/PEN fiber | 0.409 | 29.5 | 7.30 | 112.83 |
| Laser-drawn TLCP5/PEN fiber | 0.411 | 29.5 | 7.73 | 115.78 |
| Heat-drawn TLCP5/PEN fiber | 0.420 | 29.3 | 7.81 | 123.74 |
| Heat/laser-drawn TLCP5/PEN fiber | 0.427 | 27.7 | 7.94 | 131.15 |
| Annealed TLCP5/PEN fiber | 0.424 | 28.4 | 7.99 | 125.55 |

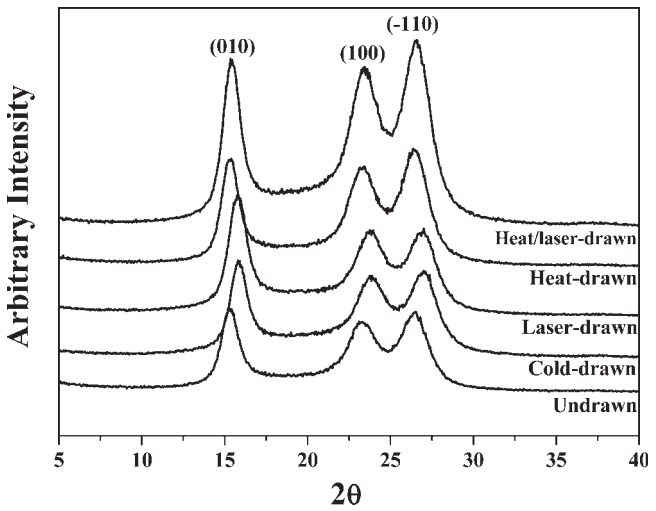


**Figure 4** WAXD patterns of TLCP1/PEN blend fibers.

novel $CO_2$ laser-drawing process combined with preheating method to overcome this problem. The major advantage of novel $CO_2$ laser-drawing process is as follows: $CO_2$ laser with sufficient heat energy that supplied for fully heating up the blend fibers by preheating can considerably reduce temperature distribution with distance from heat-source to obtain the optimal drawing effect. Therefore, the properties of the heat/laser drawn TLCP/PEN blend fibers could be expected to be more excellent than those of other handled fibers, which were confirmed by the results summarized in Table III. The tensile strength of heat/laser drawn TLCP/PEN blend fibers was similar to that of TLCP/PEN blend fibers annealed at optimal annealing condition. In addition, the tensile modulus of heat/laser drawn TLCP/PEN blend fibers was higher than that of TLCP/PEN blend fibers annealed at 135°C for 10 min. These results suggest that the $CO_2$ laser method that was suggested by our laboratory was much more effective fiber handling process than other methods.

The WAXD profiles of the TLCP/PEN blend fibers are shown in Figure 4. Three characteristic crystalline peaks were observed at 15.6°, 23.4°, and 26.8°, which were attributed to the (010), (100), and (−110) reflections, indicating the α-form crystalline structures of PEN. The drawing or annealing processes used in this study has little effect on the position of characteristic crystalline peaks of the TLCP/PEN blend fibers. The diffracting intensity of the heat/laser drawn fibers was higher than that of any other fibers, which was attributed to the development of more ordered crystallites and the increase in the relatively crystallinity when compared with the other treated fibers. Thus, it can be deduced that the $CO_2$ laser drawing combined with preheating is more effective than other methods of improving fiber properties. Structural parameters of the TLCP/PEN blend fibers are shown in Table IV. According to Scherrer's equation, the narrow diffraction peaks in the WAXD patterns are related to crystals that are large in the direction perpendicular to the observed crystal plane.[24] Structural parameters of the TLCP/PEN blend fibers were varied with the applied drawing method, as shown in Table IV. It can be seen that the crystalline structures of TLCP/PEN fibers were varied with the applied drawing method, and they were more sensitive to drawing condition than drawing ratio. Thermal behaviors of the TLCP/PEN blend fibers are shown in Table V. The drawing processes have little effect on the glass transition and melting temperatures. However, the drawn blend fibers exhibited higher crystallinity than undrawn blend fibers.

The changes of birefringence for TLCP/PEN blend fibers with draw ratio are shown in Figure 5. Birefringence of the blend fibers increased with increasing draw ratio, indicating that the molecular orientation of the blend fibers was promoted along the fiber axis with increasing draw ratio. As shown in Figure 6, the linear relationship between the degree of crystallinity and draw ratio can be observed. In general, the increase of crystallinity and draw ratio improves the mechanical properties of fiber. The tensile strength and modulus of TLCP/PEN blend fibers with draw ratio are shown in Figure 7. Both tensile strength and modulus of the blend fibers increased with increasing draw ratio, which was attributed to the improvement of molecular orientation, the development of more ordered crystalline

**TABLE IV**
**Structural Parameters of the TLCP1/PEN Blend Fibers**

| Samples | $d$ (Å) | | | $L_{hkl}$ (Å) | | | Number of repeating units per crystal | | |
|---|---|---|---|---|---|---|---|---|---|
| | (010) | (100) | (−110) | (010) | (100) | (−110) | (010) | (100) | (−110) |
| Undrawn TLCP1/PEN fiber | 5.79 | 3.83 | 3.36 | 79.7 | 59.0 | 55.1 | 13.8 | 15.4 | 16.4 |
| Cold drawn TLCP1/PENfiber | 5.59 | 3.74 | 3.29 | 84.6 | 60.9 | 61.8 | 15.1 | 16.3 | 18.8 |
| Laser drawn TLCP1/PEN fiber | 5.60 | 3.74 | 3.31 | 84.2 | 61.4 | 57.2 | 15.0 | 16.4 | 17.3 |
| Heat drawn TLCP1/PEN fiber | 5.79 | 3.81 | 3.31 | 79.7 | 60.9 | 57.2 | 13.8 | 16.0 | 17.0 |
| Heat/laser drawn TLCP1/Pen fiber | 5.75 | 3.80 | 3.36 | 79.7 | 61.3 | 55.1 | 13.9 | 16.1 | 16.4 |

**TABLE V**
**Thermal Behavior of TLCP1/PEN Blend Fibers**

| Samples | $T_g$ (°C) | $T_m$ (°C) | $\Delta H_m$ (J/g) | $X_{dsc}$ (%) |
|---|---|---|---|---|
| Undrawn TLCP1/PEN fiber | 122.3 | 273.3 | 52.9 | 52.8 |
| Cold-drawn TLCP1/PEN fiber | 122.5 | 273.3 | 56.2 | 55.0 |
| Laser-drawn TLCP1/PEN fiber | 122.6 | 273.8 | 58.2 | 57.0 |
| Heat-drawn TLCP1/PEN fiber | 122.5 | 274.1 | 59.1 | 57.8 |
| Heat-laser drawn TLCP1/PEN fiber | 122.7 | 274.0 | 60.0 | 58.7 |

structure, and the increase in the crystallinity. From the above-mentioned results, it can be deduced that draw ratio is a more important factor than drawing method in determining the mechanical properties of the TLCP/PEN blend fibers.

## CONCLUSIONS

PEN reinforced with the TLCP blend fibers were prepared by the melting blending and spinning processes. The laser-drawn blend fibers exhibited lower fiber properties than that of heat-drawn blend fibers because $CO_2$ laser was not enough to fully heat up the blend fibers. Tensile strength and modulus of TLCP/PEN blend fibers increased with increasing draw ratio, which was attributed to the improvement of molecular orientation, the development of more ordered crystalline structure, and the increase in the crystallinity. Draw ratio was more important factor than drawing method, in determining mechanical properties of the TLCP/PEN blend fibers. There are major advantages of the heat/laser drawing process: enough heat energy can be supplied for the blend fibers by preheating, and then temperature distribution with distance from heat-source can be considerably reduced by applying $CO_2$ laser. Therefore, the properties of the heat/laser drawn TLCP/PEN blend fibers were more excellent than those of any other handled fibers, and the draw

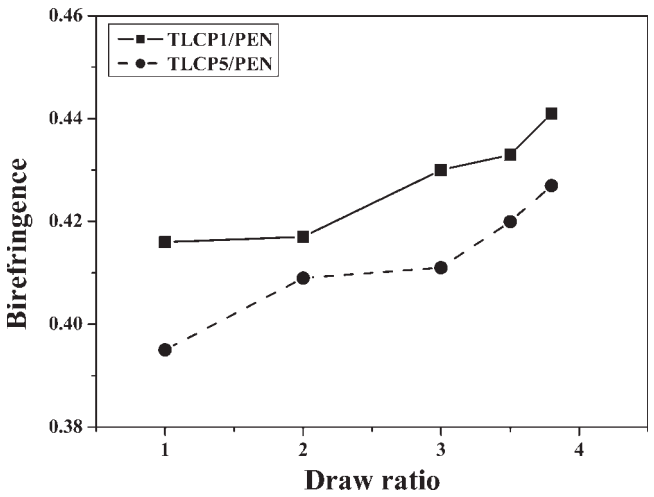


**Figure 5** Relationship between draw ratio and birefringence of TLCP/PEN blend fibers.

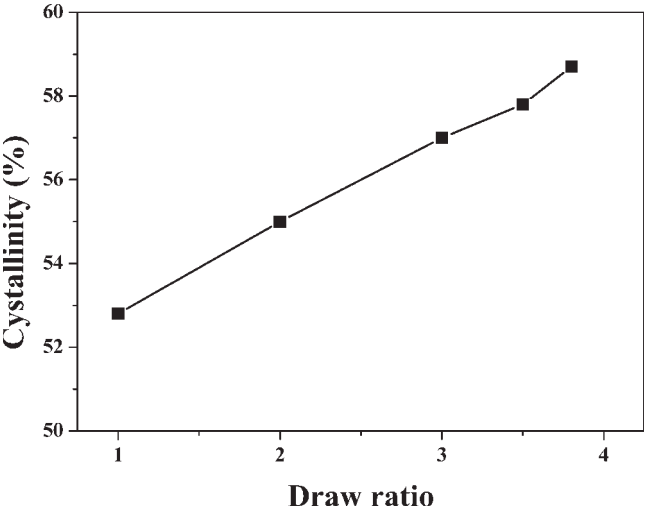


**Figure 6** Relationship between draw ratio and crystallinity of TLCP1/PEN blend fibers.

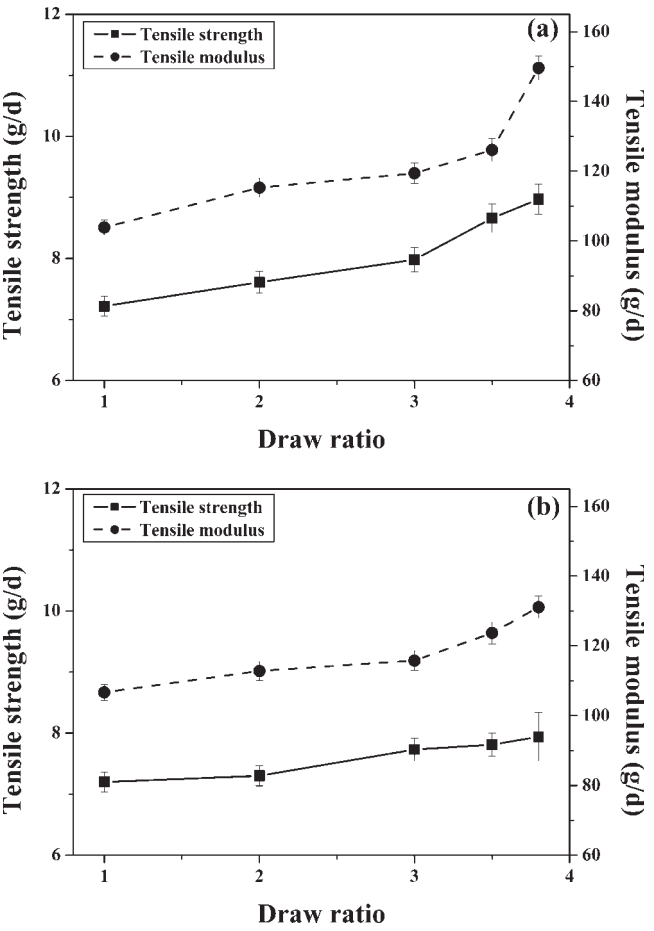


**Figure 7** Relationship between draw ratio and tensile properties of (a) TLCP1/PEN and (b) TLCP5/PEN blend fibers.

ratios of them were the largest. These results suggest great potential for the application of $CO_2$ laser as a novel fiber drawing process. In particular, the combined preheating and $CO_2$ laser drawing process may be well appropriated for fiber drawing process because the improvement effect by this system is more significant than other drawing methods.

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