

Effect of Thermotropic Liquid Crystalline Poly(ether ketone)arylates on the Processibility and Properties of Poly(ether ether ketone)s Fibers

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ABSTRACT: A series of composite fibers based on poly(ether ether ketone)s (PEEK) and a thermotropic liquid crystalline poly(ether ketone)arylates (PEKAR) have been prepared by melt spinning. The structure, compatibility, and properties of these composite fibers were investigated in detail by rheological measurements, differential scanning calorimetry, thermogravimetric analysis, wide-angle X-ray diffraction, scanning electron microscopy, orientation degree test, and mechanical properties test. The results showed that the addition of PEKAR could reduce the apparent melt viscosity of the blends obviously, which is beneficial in improving the processibility of PEEK at a relatively low temperature. After adding 1 wt % PEKAR to PEEK, the tensile strength of the post-treatment fiber improved by 8.8%, whereas the crystallinity of the as-spun fiber increased from 21.76% to 31.51%, and the orientation degree also increased with the addition of PEKAR. The result of morphology research suggested that PEKAR had a good compatibility with PEEK resin. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40595.

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INTRODUCTION

Poly(ether ether ketone) (PEEK) is a kind of wholly aromatic thermoplastic (TP), and its macromolecular backbones consist of rigid benzene ring, flexible ether, and aryl ketone groups. These groups impart their excellent mechanical properties, high heat resistance and thermal stability, outstanding abrasion performance, and other excellent properties.^{1–3} They also give PEEK a very high melting point (around 334°C) and a quite high melting viscosity. Therefore, the processing temperature of PEEK is above 400°C, which means huge energy consumption and high requirements on equipment.^{4,5} Thus, it is meaningful to improve the processibility of PEEK at lower temperature.

Two methods were generally used to improve processibility of PEEK in previous works. One is chemical modification, in which some new types of PEEKs were synthesized by introducing large side groups to prepare hyperbranched PEEKs to make it processible in solvent. But they found that it was costly and would reduce the thermal endurance of PEEK.^{6–10} Another way is to blend PEEK with other polymers, such as polyphenylene sulfide, polyether sulfone, and so on. It was proven to be effective in decreasing the melting viscosity of PEEK, but the thermal properties and mechanical properties declined with the addition of these polymers.^{11,12}

Thermotropic liquid crystalline polymer (TLCP) is considered to be an ideal processing aid agent. It was found that adding a small amount of liquid crystalline polymer could reduce the viscosity of the TP matrix significantly. Several opinions were proposed to explain this effect: (1) low-viscosity liquid crystalline polymer melt migrates toward the wall; (2) the oriented TLCP microfibril slides past one another; and (3) the interfacial slip-page between the two melts.¹³ Moreover, TP matrix will be reinforced by the formed microfibril. These polymer composites are commonly known as *in situ* composites.¹⁴ Certainly, TLCPs were also used to ameliorate the processibility of PEEK in previous works. The rheological behavior, dynamic mechanical behavior, and other properties of PEEK/Vectra 950A blends were studied by Isayev and co-workers.^{15–17} It was found that the addition of TLCP significantly reduced the melting viscosity of PEEK, especially at a shear rate of 118 s⁻¹; the mechanical properties were improved when the content of TLCP exceeded 10 wt %. In previous works of our group, we synthesized a thermotropic liquid crystalline polyarylate (PEAR) and applied it in facilitating the processibility of PEEK and polyphenylene sulfide.^{18–20} However, both Vectra 950A and PEAR were incompatible or semicompatible with PEEK; as a result, the composites could not achieve the best mechanical properties as we

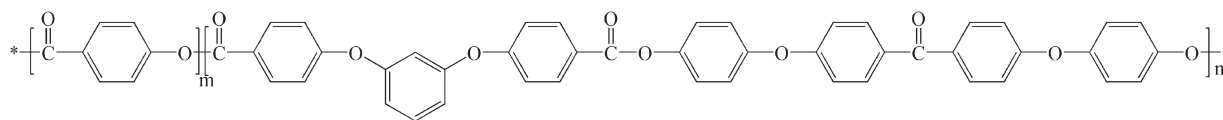


Figure 1. The structure of PEKAR.

expected. It is worthwhile to find a kind of TLCP that is compatible with PEEK to improve the processibility and mechanical properties of PEEK simultaneously.

In the present work, a new TLCP, namely poly(ether ketone)arylates (PEKAR), was used to improve the processibility of PEEK at a relatively low temperature. The main chain of PEKAR was linked by aryl-ether, aryl-ketone, and ester groups, which is more similar to PEEK's backbone compared with Vectra and PEAR used in our another work.²⁰ It was expected to be more compatible with PEEK, which means a stronger interaction force between PEEK and PEKAR. Thus, it should work better in inducing orientation arrangement of PEEK chain and reinforcing PEEK. Meanwhile, PEAR was used as a control experiment to investigate the effects of compatibility on the processibility and properties of TLCP/PEEK composite. Rheological behavior of PEEK/PEKAR blends was investigated by high-pressure capillary rheometer in detail, and the processibility of the blends was studied by melt spinning. Also, the thermal properties, crystallization, mechanical properties, and morphology of the composite fibers were investigated in detail by differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), wide-angle X-ray diffraction (WAXD), and scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

The Victrex 2000P PEEK with a melting temperature of 334°C was purchased from Degussa. The chemical structure of PEKAR used in this study is shown in Figure 1, which was synthesized from *p*-acetoxy-benzoic acid, 1,3-bis(4'-carboxyphenoxy)benzene, and 4,4'-bis(3-hydroxy phenoxy)-benzophenone with a molar ratio of 70 : 15 : 15. The synthesis procedure was reported in our previous work.²¹ The intrinsic viscosity of PEKAR is 0.72 dL/g, and the melting temperature is 319.2°C, but the clear point is not detected below 400°C. The initial decomposition temperature of PEKAR tested by TGA is 406.2°C (in N₂).

Blends Preparation

PEKAR and PEEK powders were blended homogeneously by DFT-100 high-speed versatile disintegrator (DFT-100; Jinpeng Test Mart, China; spindle speed is 24,000 rpm), and PEKAR weight ratios were set as 0.5, 1.0, and 2.0 wt %, respectively. The PEEK/PEKAR blends were marked as PEEK-K-wt %, where wt % is the weight percentage of PEKAR in the blends and "K" represents PEKAR. For a comparative study, the blends of PEEK/PEAR described in our previous work²⁰ were used as control samples, which were marked as PEEK-E-wt %. To remove the water and residual solvents, the blends were dried for at least 2 h at 200°C under vacuum.

Fibers Preparation

The as-spun fibers of pure PEEK and PEEK/PEKAR were prepared by melt spinning machine with a single orifice ($D = 0.8$ mm, $L/D = 8/1$). The spinning temperature was set as 365°C, 370°C, and 380°C, respectively. PEEK and PEKAR powder were blended by high-speed versatile disintegrator (24,000 rpm). Then the mixture was added into feed hopper of the melt spinning machine (shown in Figure 2), the extrusion speed at spinneret was set at 5.85 m/min, which was controlled by the metering pump, and the take-up speed was 340 m/min. Then the as-spun fibers were subjected to a hot tensile process and a thermosetting process (shown in Figure 3). The heat drawing was carried out at 210°C (the draw ratio was 3.4) and heat setting at 200°C (the heat setting time was 51 s).

Testing and Analysis

The rheological measurement was carried out by a high-pressure capillary rheometer (Rosand RH7D, Malvern, England) with diameter of 1 mm and a capillary length-to-diameter (L/D) of 16 mm. The measurements were performed at a shear rate range from 20 to 1000 s⁻¹ at the temperatures of 370°C, 380°C, and 390°C, respectively.

DSC (Netzsch, Germany) curves were measured in the temperature range of 50°C–380°C at a heating rate of 10°C/min under flowing nitrogen (20 cm³/min). The temperature and heat enthalpy were calibrated with indium standard before the experiment running. The crystallinity ($X_c\%$) was calculated according to the following equation:

$$X_c\% = [(\Delta H_m + \Delta H_{rc}) / (w \times \Delta H^0)] \times 100\%$$

where ΔH^0 represents the melting enthalpy of the 100% crystalline PEEK, ΔH_m is the melting enthalpy of the sample, ΔH_{rc} is the recrystallization enthalpy, and w represents the weight percentage of PEEK component in the composite fibers.

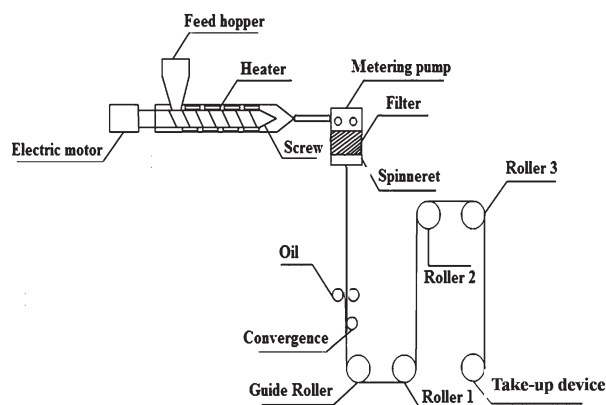


Figure 2. The equipment diagram of melt spinning machine.

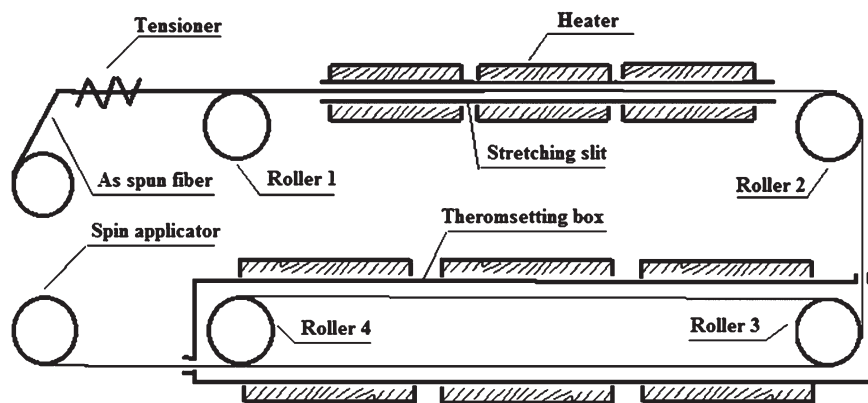


Figure 3. The equipment diagram of hot drawing machine and hot setting machine.

TGA test was taken by a thermogravimetric analyzer (SDTQ600) under N_2 atmosphere at a heating rate of $20^\circ\text{C}/\text{min}$ and a N_2 flow rate of $100\text{ cm}^3/\text{min}$, respectively. The temperature range was evaluated from room temperature to 800°C .

The orientation degree of the molecular chain was determined by measuring the transit time of the ultrasonic wave between two transducers coupled to the specimens. This measurement was carried out by an SCY-III model fiber sonic velocity meter (Donghua University, China). The variable “ f ” represents the chain orientation factor of the fiber, which can be calculated according to the equation $f = 1 - \left(\frac{C}{C_\mu}\right)^2$, where, C and C_μ represent the sonic velocity of the above samples and unoriented PEEK sample, respectively. The value of C_μ is 1.487 km/s and it was tested by using free-run PEEK samples instead of unoriented fibers.

X-ray diffraction patterns of the fibers were measured using a X'Pert Pro(Philips) X-ray diffractometer operating in the 2θ range of 5° – 40° at a scanning rate of 12° min^{-1} with nickel-filtered $\text{Cu K}\alpha$ radiation ($L = 1.54\text{ \AA}$) at room temperature. The morphology of the fibers was examined by field-emission scanning electron microscope (FEI Sirion-200, USA) at ambient condition. SEM photographs of the cross-section of the as-spun fibers were used to study the two-phase compatibility, whereas the surface photographs of the as-spun fibers were used to study the impact of PEKAR on the processibility of PEEK.

Tensile testing was performed on a single fiber tensile strength tester (YG061, China) at room temperature. The initial length was 20 mm , and tensile speed was $20\text{ mm}/\text{min}$. An average of 10 replicas was used.

RESULTS AND DISCUSSION

Processibility

Figure 4 reveals the effect of the PEKAR content on the rheological behavior of the blends at different temperatures, and the details of shear range from 20 to 100 s^{-1} are presented in the inserts. Different from the classical non-Newtonian fluid, the rheological behavior of PEEK and PEEK/PEKAR at high temperature or in high PEKAR proportion can be divided into three regions. Region 1 is the first shear thinning region whose apparent melt viscosity sharply decreases with the increase in

shear rate. It is not apparent at low temperature especially when the content of PEKAR is low. With increasing the test temperature or the content of PEKAR, Region 1 will get more apparent. It is because the disentanglement and the orientation of the molecular chain under shear stress could induce the decrease of the apparent melt viscosity. The addition of PEKAR or the elevation of test temperature facilitates the orientation or the motion of molecular chain of the blends; as a result, Region 1 gets more apparent and wider. Region 2 is the shear thickening region. With further increase in the shear rate, there is a mutation area where the apparent melt viscosity increased with the elevation of shear rate. This is due to the slight physical crosslink structure, which may result from the enhanced intermolecular force of the ordered area or the tangles between main chain and branched chain.^{19,20} Region 3 is the second shear thinning region where the apparent melt viscosity decreased with the increase in the shear rate. Meanwhile, the apparent melt viscosity drops more gently after adding PEKAR, which suggests that PEKAR lowers the sensitivity of the viscosity to the shear rate; as a result, the processibility tends to be more stable.

It can also be seen that the apparent viscosity of samples decreases obviously with the addition of PEKAR at 365°C and 370°C . The reason can be explained by the morphological changes of PEKAR and PEEK chains in capillary die. The PEKAR chains can easily orientate under shear stress, which can induce the orientation of PEEK chains and lead to the decrease in the viscosity of the blends. However, the apparent viscosity of PEEK/PEKAR tends to be consistent with neat PEEK at 390°C with the increase in shear rate, because PEEK can be plasticized completely on the condition of high shear rate and temperature. The results suggest that PEKAR can act as a processing aid by reducing the viscosity of PEEK melt during the process at relative low temperature.

Figure 5 unfolds a clear comparison between PEAR/PEEK blends and PEEK/PEKAR blends on the viscosity at 365°C . It can be seen that PEKAR works better than PEAR in reducing viscosity of PEEK at all shear rate ranges. This is associated with the similar structure of PEKAR and PEEK, which means that there is a bigger interfacial force between PEKAR and PEEK than that of PEAR. As a result, PEKAR works more outstanding in reducing the melt viscosity of PEEK.

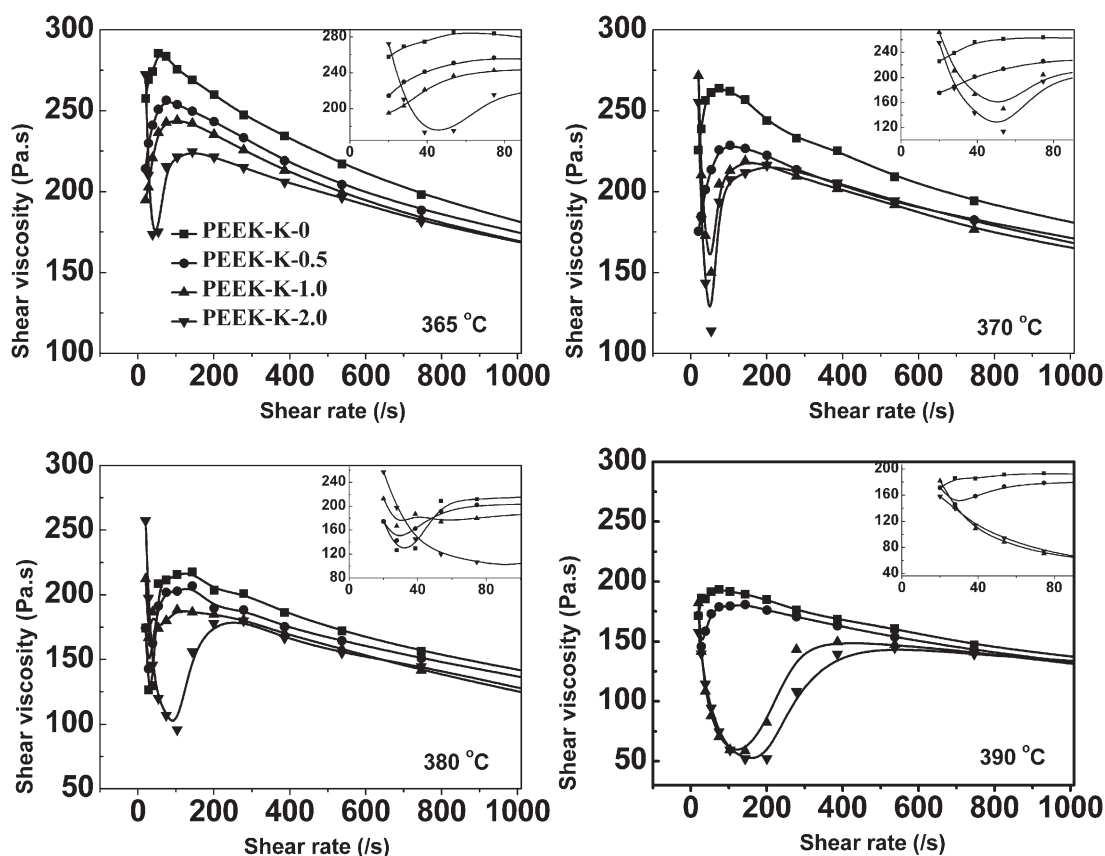


Figure 4. Plots of shear viscosity as a function of shear rate for the blends at different temperatures.

The rheological research shows that PEKAR is effective in reducing the viscosity of PEEK melt, which means that PEEK/PEKAR blends can be processed under lower temperature. To confirm this conclusion, the processibility of the blends was tested at different temperatures by melt spinning. The key parameters are shown in Table I. It can be seen that the processibility of neat PEEK is poor at 370°C and 365°C because PEEK cannot plasticize completely below 380°C, which results in high pack pressure and frequent filament breakage. After adding

0.5 wt % of PEKAR, the processibility of PEEK at 370°C improved significantly. When the content of PEKAR reaches 1 wt %, the pack pressure decreases about 50% and the as-spun fibers become smooth with no breakage at 365°C. Therefore, we can say that the processibility of PEEK is significantly improved after adding a small amount of PEKAR.

Table I. The Processibility of Neat PEEK and PEEK/PEKAR Blends

Samples	Processing temperature (°C)	Spinning pack pressure (MPa)	Texture of as-spun fibers	Filament breakage severity
PEEK-K-0	365	1.55	Rough	Frequent
	370	1.3	Rough	Occasional
	380	0.55	Smooth	None
PEEK-K-0.5	365	1.05	Rough	None
	370	0.7	Smooth	None
	380	0.3	Smooth	None
PEEK-K-1.0	365	0.75	Smooth	None
	370	0.3	Smooth	None
	380	0.2	Smooth	None
PEEK-K-2.0	365	0.7	Smooth	None
	370	0.25	Smooth	None
	380	0.2	Smooth	None

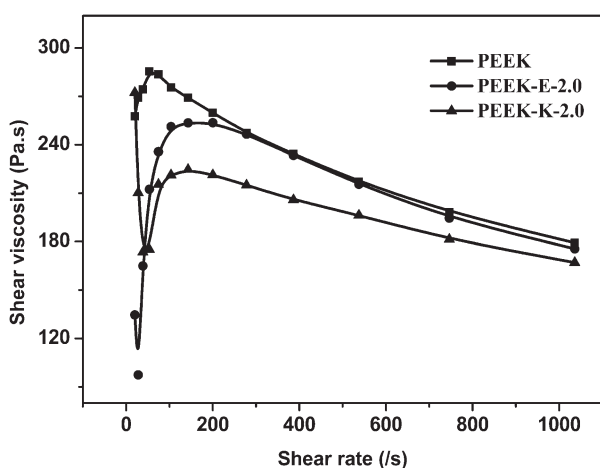


Figure 5. Plots of shear viscosity as a function of shear rate for different TLCP.

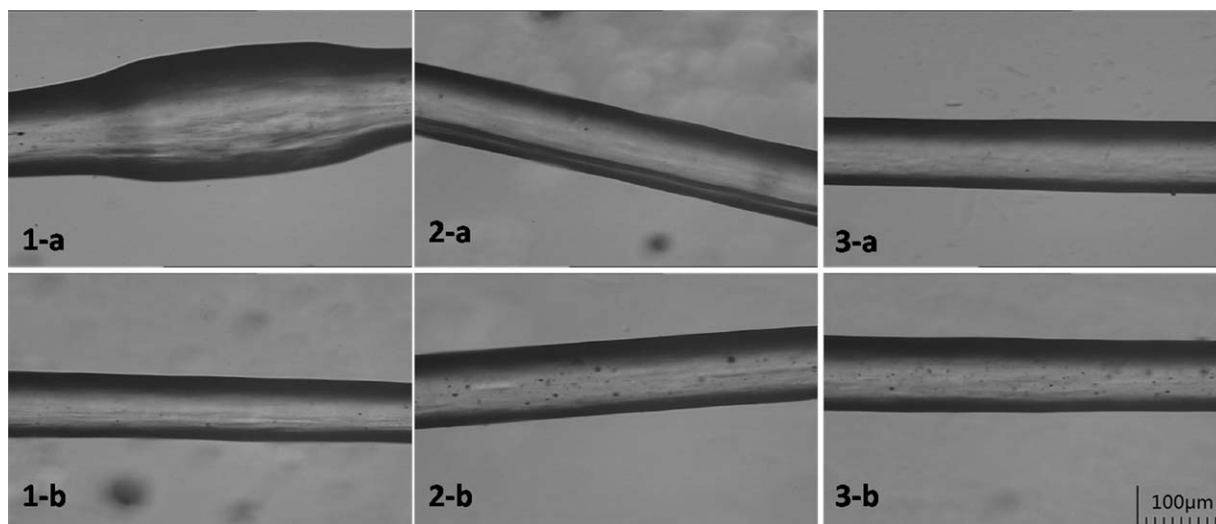


Figure 6. The optical micrographs of as-spun fibers. 1-a: PEEK-K-0 (365°C, $\times 640$); 2-a: PEEK-K-0 (370°C, $\times 640$); 3-a: PEEK-K-0 (380°C, $\times 400$); 1-b: PEEK-K-0.5 (365°C, $\times 640$); 2-b: PEEK-K-1.0 (365°C, $\times 640$); and 3-b: PEEK-K-2.0 (365°C, $\times 640$).

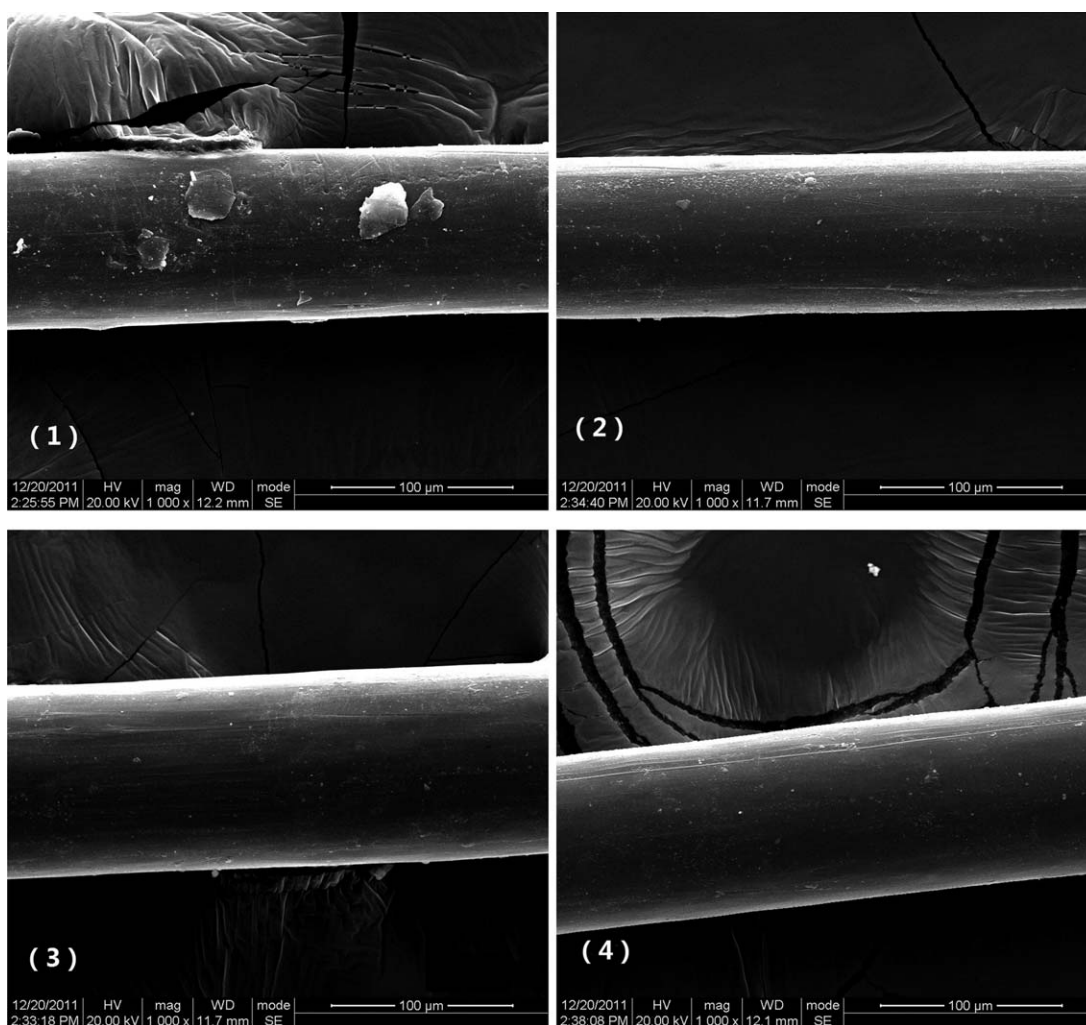


Figure 7. Scanning electron micrographs of the as-spun fibers prepared at 365°C. (1) PEEK-K-0 ($\times 1000$); (2) PEEK-K-0.5 ($\times 1000$); (3) PEEK-K-1.0 ($\times 1000$); and (4) PEEK-K-2.0 ($\times 1000$).

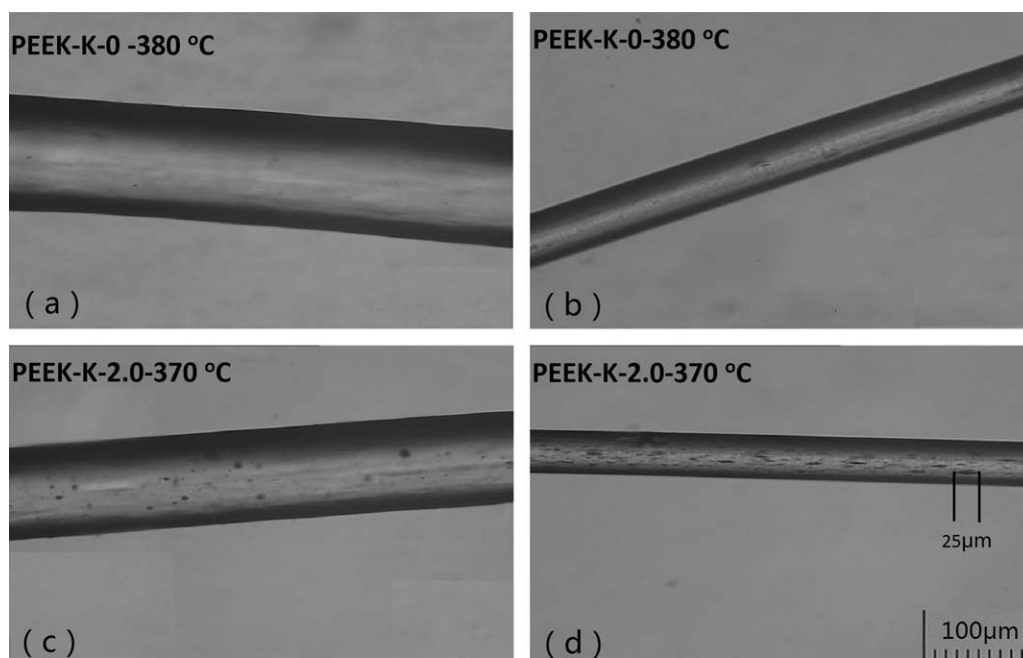


Figure 8. The optical micrographs of as-spun fibers (a and c) and post-treatment fibers (b and d).

Morphology

The morphological research was conducted by optical microscope and field-emission scanning electron microscope, and the results are shown in Figures 6 and 7, respectively. From Figure 6(1-a), it can be seen that the as-spun fibers of neat PEEK prepared at 365°C display rough skin and uneven thickness, and the smooth and even fibers of neat PEEK could only be prepared at 380°C or higher temperature as is shown in Figure 6(3-a). However, after adding 1 wt % of PEKAR, even thickness fibers without defects can be got at 365°C [shown in Figure 6(2-b)]. On comparing the SEM images shown in Figure 7, it can be found that the as-spun fibers of neat PEEK prepared at 365°C exhibit a rough skin, but after the addition of PEKAR, the defects on as-spun fibers decreased significantly when the proportion of PEKAR is 1 or 2 wt %.

From the rheological and morphology researches it can be concluded that PEKAR can act as an effective process aid. When

the content of PEKAR is 1 or 2 wt %, we can get even thickness and smooth composite fiber at 365°C, whereas the neat PEEK cannot continuously spin at this temperature.

Figure 8 displays the optical microscope images of the as-spun fibers and the post-treatment fibers. It can be found that PEKAR domains exhibit spherical morphology in as-spun fiber, and then the spherical domains were deformed into fibril shape by tensile stress after the post-treatment process. The cross-section morphology of the as-spun fibers can be seen in Figure 9, and PEKAR domain can be found with a diameter around 1 μm. Compared with the PEAR/PEEK composite fibers, PEKAR domains in PEEK/PEKAR composite fibers are much smaller than PEAR domains, and there is no void space between PEKAR phase and PEEK phase; in contrast, the PEAR domains in PEAR/PEEK composite is bigger and void spaces can be found between the PEAR domain and PEEK matrix. This is because the different melt viscosity ratio and lower interfacial

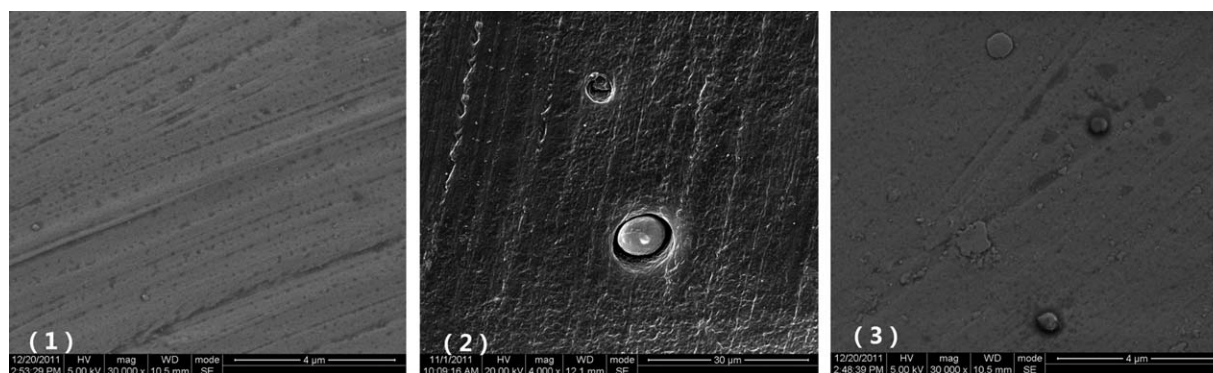


Figure 9. SEM images of the cross-sections of composite fibers. (1) Neat PEEK (×30,000); (2) PEEK-E-2.0 (×4000); and (3) PEEK-K-2.0 (×30,000).

Table II. DSC Data of PEEK and PEEK/PEKAR Blends

Sample	T_g (°C)	T_{rc} (°C)	ΔH_{rc} (J g ⁻¹)	T_m (°C)	ΔH_m (J g ⁻¹)	T_c (°C)	X_c (%)
PEEK-K-0	137	169	-21.28	344	49.57	301	21.76
PEEK-K-0.5	135	168	-17.39	344	49.44	304	24.78
PEEK-K-1.0	134	165	-14.14	343	63.11	305	38.05
PEEK-K-2.0	135	167	-17.96	343	58.11	305	31.51

T_g , glass-transition temperature; T_{rc} , recrystallization temperature (cold crystallization temperature); ΔH_{rc} , recrystallization enthalpy; T_m , melt temperature; ΔH_m , melting enthalpy; T_c , crystallization temperature; X_c , crystallinity.

tension result in different capillary index, which will influence the domain size.²² It can be concluded that PEKAR has a better compatibility with PEEK than PEAR has, as we expected.

Thermal Properties and Crystallization Behaviors

Thermal properties of the as-spun fibers were studied by DSC, and the results are summarized in Table II. It can be seen that T_g values of the blends move to lower value with the addition of PEKAR, and the same trend can be seen in DMA (data shown in Figure 10). Generally speaking, the lower value of the glass-transition temperature is always caused by the amplification of free volume after adding the process aids.

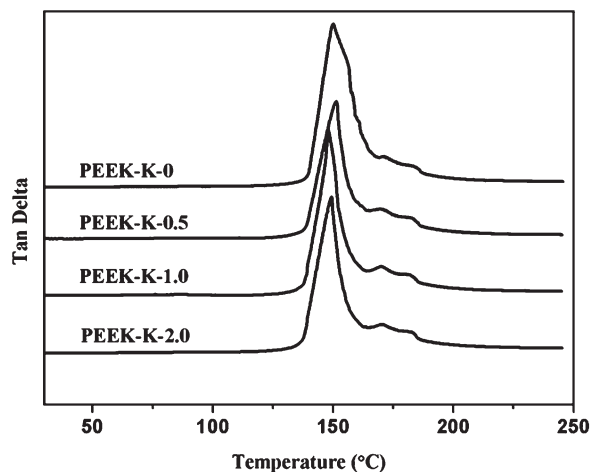


Figure 10. The DMA data of PEEK and PEEK/PEKAR blends.

The recrystallization peak (T_{rc}) of the blends decreased when the percentage of PEKAR increase from 0 to 1 wt %, and then increased when the proportion of PEKAR increase from 1 to 2 wt %, and the enthalpy value of T_{rc} peaks have the same trend. On the contrary, the crystallinity of the as-spun fiber increased first and then decreased with the increase in PEKAR's proportion. The reason can be explained by that PEKAR is easy to achieve orientational arrangement under shear conditions, which can induce the disentanglement and the orientational arrange of PEEK chain. It became easier for these relatively ordered molecular chains to enter the crystal lattice. Therefore, the crystallinity of the composite fibers increased with elevating PEKAR contents. However, too much of PEKAR will hinder the growth of PEEK crystal. As a result, the crystallinity decreased with further increasing the PEKAR content.

Moreover, the crystallization temperature determined by DSC shifted to higher value after the addition of PEKAR. As we know, PEKAR is in liquid crystalline state at 300°C or higher temperature, which means that the TLCP can act as a nucleating agent in a higher temperature to facilitate the crystallization of PEEK.

The crystallization of the as-spun fibers and post-treatment fibers was studied by WAXD (presented in Figure 11). Figure 11(a) shows that there is no any sharp diffraction peak, which means that the as-spun fibers of both neat PEEK and PEEK/PEKAR (2 wt % of PEKAR) blends have very low crystallinity. After the post-treatment, the WAXD patterns of those two samples presented in Figure 11(b) show strong reflections with four

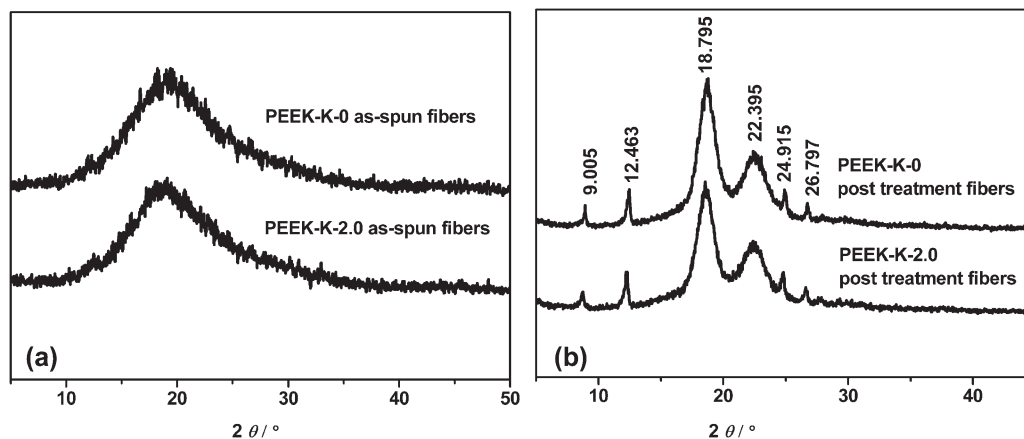


Figure 11. WAXD patterns of (a) neat PEEK and (b) 2 wt % PEEK/PEKAR fibers.

Table III. Mechanical Properties of Post-Treatment Fibers Prepared at 380°C

Sample	Diameter (μm)	Tensile strengths			Elongation at break	
		cN/dtex	MPa	Standard deviation (%)	Percentage	Standard deviation (%)
PEEK-K-0	50.55	6.58	838.3	3.97	15.25	4.92
PEEK-K-0.5	50.55	6.90	879.0	6.87	15.45	7.71
PEEK-K-1.0	50.55	7.16	912.2	4.22	16.84	4.89

peaks $2\theta = 18.80^\circ$, 22.40° , 24.91° , and 26.9° , which are assigned as the lattice planes of (110), (111), (200), and (211), respectively. There is little difference in the crystal form between the neat PEEK fibers and PEEK-K-2.0 composite fibers.

Mechanical Properties

The mechanical properties of post-treated fibers are listed in Table III. A steady increase in strength can be observed with the increasing proportion of PEKAR, and this rising trend slows down when the proportion of PEKAR reaches 1 wt %. Meanwhile, the elongation at break remains around 15%, which indicates that the toughness of the composite fibers was not weakened by the addition of PEKAR.

Thermal Stability

Thermal stability of the blends was evaluated by TGA, and the results are listed in Table IV. It can be seen that the introduction of PEKAR can result in a negative contribution to the thermal stability. Both the initial degradation temperatures (T_{id}) and maximal degradation temperature (T_{md}) decrease with the addition of PEKAR due to the relatively poor thermal stability of PEKAR. However, there is little change in the char yields at 800°C for all the blended samples and the neat PEEK sample.

CONCLUSIONS

In the present work, PEEK/PEKAR composite fibers were prepared by melt spinning, and the effects of PEKAR on the processibility and other properties of PEEK fibers were investigated. The following conclusions are made in summary:

1. PEKAR can function as a processing aid by reducing the viscosity of PEEK matrix so that the composite can be processed at 365°C, whereas the neat PEEK can only be processed at 380°C or higher temperature.
2. The mechanical properties of post-treatment fibers produced at 380°C were increased by 8.8% after adding 1 wt % PEKAR into PEEK matrix.
3. The crystallinity of as-spun fibers increased from 21.76% to 31.51%, and glass-transition temperature decreased, whereas the crystallization temperature increased with the addition of PEKAR. Both of the initial degradation temperatures and the maximal degradation temperatures decrease with the increase in the PEKAR content.
4. PEEK/PEKAR has better compatibility than PEAR/PEEK blends. In general, PEEK blending with 2 wt % of PEKAR can exhibit good processing properties, and the composite fibers have good mechanical properties and excellent thermal properties.

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Table IV. TGA Data of PEEK and PEEK/PEKAR As-Spun Fibers

Samples	T_{id} (°C)	T_{md} (°C)	Char yield (%)
PEEK-K-0	573	595	51.47
PEEK-K-0.5	569	593	52.05
PEEK-K-1.0	568	590	51.09
PEEK-K-2.0	566	588	51.40

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