

## Crystalline and thermal properties in miscible blends of PEEK, PPS and PEI obtained by melt compounding

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*Received 23 August, 2015*

The effects of crystal and amorphous polymers on the structure, morphology, crystalline and thermal properties of PEEK have been investigated by using FT-IR, SEM, DSC and TGA. The interaction existing in PEEK/PPS and PEEK/PEI alloys were stronger than that in PEEK/PPS/PEI alloys. The SEM results revealed that homogeneous structure exists in the alloys of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI. All of the binary and ternary alloys based PEEK exhibits single glass transition temperature ( $T_g$ ) in full composition range. When PEI was added to the PEEK/PPS alloys, the  $T_g$  of PEEK/PPS/PEI alloys increased from 146 °C to 179 °C. The crystallinity degree of PEEK increased when PPS or PEI was added in heating process, while increased firstly and then decreased with increasing the content of PPS or PEI in cooling process. The crystallization peak of PEEK/PPS/PEI alloy disappears in the ratio of 50/25/25. The thermal decomposition temperature ( $T_d$  5%) of PEEK/PPS and PEEK/PEI alloys decreased when PPS and PEI were added, the  $T_d$  of PEEK/PPS/PEI alloy in the ratio of 70/15/15 presented at 510 °C, which was lower than pure PEEK and pure PEI, higher than PPS.

**Keywords:** PEEK, PPS, PEI, plastic alloys, crystallization, thermal properties.

Влияние кристаллических и аморфных полимеров на структуру, морфологию, кристаллические и термические свойства ПЭЭК были исследованы с помощью фурье-ИК спектроскопии, сканирующей электронной микроскопии, ДСК и ТГА. Взаимодействие компонентов в сплавах ПЭЭК/ПФС и ПЭЭК/ПЭИ проявлялось сильнее, чем в ПЭЭК/ППС/ПЭИ. Сканирующая электронная микроскопия показала гомогенную структуру сплавов ПЭЭК/ПФС, ПЭЭК/ПЭИ и ПЭЭК/ПФС/ПЭИ. Все двойные и тройные сплавы на основе ПЭЭК проявляют только одну температуру перехода ( $T_g$ ) во всем интервале составов. При добавлении ПЭИ к ПЭЭК/ПФС,  $T_g$  сплавов ПЭЭК/ПФС/ПЭИ возрастала с 146 до 179°C. Степень кристалличности ПЭЭК возрастала при добавлении ПФС и ПЭИ в процессе нагревания и снижалась при добавлении ПФС и ПЭИ в процессе охлаждения. Пик кристаллизации сплава ПЭЭК/ПФС/ПЭИ исчезает в области 50/25/25. Температура термического разложения ( $T_d$ ) сплавов ПЭЭК/ПФС и ПЭЭК/ПЭИ уменьшалась при добавлении ПФС и ПЭИ.  $T_d$  сплава ПЭЭК/ПФС/ПЭИ в соотношении 70/15/15 была 510°C, что ниже, чем у чистых ПЭЭК и ПЭИ, но выше, чем у ПФС.

**Кристалічні та термічні властивості мішаних композитів ПEEK, ПФС та PEI, отримуваних змішуванням в розплаві.** Чень Цзян-бін, Го Вень-хе, Ли Чжунь-чжунь, Тянь Лі-мін

Вплив кристалічних та аморфних полімерів на структуру, морфологію, кристалічні та термічні властивості ПEEK досліджено методами Фур'є-ІЧ спектроскопії, сканувальної електронної мікроскопії, ДСК та ТГА. Взаємодія компонентів в сплавах ПEEK/ПФС та ПEEK/PEI була більш сильною, ніж в ПEEK/ППС/PEI. Сканувальна електронна мікроскопія виявила гомогенну структуру сплавів ПEEK/ПФС, ПEEK/PEI та ПEEK/ПФС/PEI. Всі подвійні та потрійні сплави на основі ПEEK дають тільки одну температуру переходу ( $T_g$ ) в усьому інтервалі складу. Додавання PEI до ПEEK/ПФС призводила до зростання  $T_g$  сплавів ПEEK/ПФС/PEI з 146 до 179°C. Ступінь кристалічності ПEEK зростала при додаванні ПФС та знижувалась при додаванні PEI. Пік кристалізації сплаву ПEEK/ПФС/PEI зникає в області 50/25/25. Температура термічного розкладу ( $T_d$ ) сплавів ПEEK/ПФС та ПEEK/PEI зменшувалась при додаванні ПФС та PEI.  $T_d$  сплаву ПEEK/ПФС/PEI при співвідношенні 70/15/15 була 510°C, що нижче, ніж у чистих ПEEK та PEI, але вище, ніж у ПФС.

## 1. Introduction

Blend alloy has been one of the most interesting subjects with respect to the polymer modification, because large improvement has been achieved in the characteristics including thermal properties, mechanical properties, crystallizability and cost effectiveness by blending appropriate polymer materials [1,2]. Poly(etheretherketone) (PEEK), as a high performance semi-crystalline engineering polymers with excellent properties such as high thermal stability, mechanical properties, chemical resistance and electrical properties, has attracted intense research attention since developed by ICI at 1978. It provides a new platform for application in aerospace, automobiles, electronics and medical devices [3-5]. However, PEEK is an expensive polymer due to the high melting point (~334 °C) and viscosity, more than four times the price of other engineering polymers, which restricts its further application.

Poly(phenylene sulfide) (PPS) possesses good thermal stability combined with good mechanical properties and chemical resistance. PPS can also act as a flow aid for plastic alloys due to its excellent melting process characterization [6-9]. The polymer poly(etherimide) (PEI) and PEEK are a class of high performance engineering polymers that are high temperature resistant and known to be miscible in the amorphous phase [10-11]. However, only two component alloys among these three plastic such as PEEK blending with PPS or PEI were reported in literatures [12-19]. Few research reports emphasized on ternary plastic alloys are based on PEEK. Recently, we investigated the effects of PEI and PES on the thermal, crystalline and tribological properties of PEEK/PEI/PES ternary alloys [20]. In this article, some

binary and ternary plastic alloys of PEEK, PPS and PEI with lower cost are prepared by melt blending. The effects of crystal polymer PPS and amorphous polymer PEI on the structure, morphology, crystalline and thermal properties of PEEK were investigated by FT-IR, SEM, DSC and TGA.

## 2. Experimental materials and sample preparation

The PEEK was supplied by Changchun Jida Engineering Plastics Research Center, with weight-average molecular weight (Mw) of 100,000. The PEI and PPS were obtained by SABIC. The materials were preliminarily dried at 140 °C for 5 hours before mixed in a high speed mixer, and then melt blended using a twin-screw extruder (model: SHJ-20, Nanjing JieYa Extrusion Equipment Co. Ltd., China).

Extrusion processing parameters: temperatures of extruder sections for 265~375 °C, screw speed 10~30 r/min, head pressure 9~11 MPa.

## 3. Characterizations

The structures of the PEEK/PPS/PEI plastic alloys were evaluated by Fourier Transform Infrared Spectroscopy (FTIR) analysis (Nicolet IS-10). The spectra were recorded from 400 to 4000  $\text{cm}^{-1}$  with a resolution of 2  $\text{cm}^{-1}$  and 32 scans. The morphologies of PEEK/PPS/PEI alloys were observed by Scanning electron microscope (HITACHI S-4800). Crystallization behaviors of the PEEK/PPS/PEI alloys were studied by differential scanning calorimetry (DSC) (TA Q-2000) on the specimens sliced from extrusion molded samples. The samples with a nitrogen atmosphere and 50~400 °C testing temperature were heated at 10 °C/min, then cooled at -10 °C/min to room temperature. Thermal stability of the alloys were examined by ther-

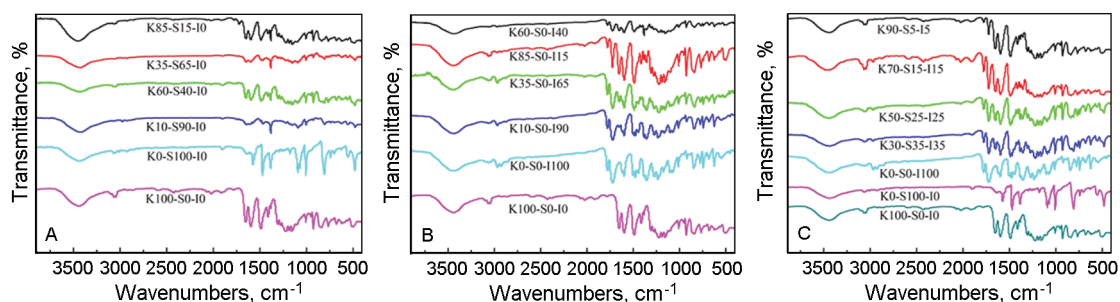


Fig. 1. FT-IR spectrum of PEEK/PPS/PEI plastic alloys: (A) PEEK/PPS alloys; (B) PEEK/PEI alloys; (C) PEEK/PPS/PEI alloys

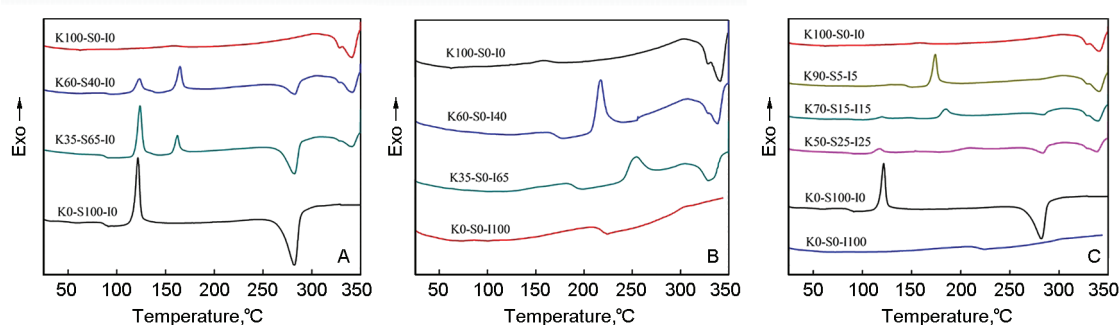


Fig. 2. DSC heating curves of binary alloys and ternary alloys: (A) PEEK/PPS alloys; (B) PEEK/PEI alloys; (C) PEEK/PPS/PEI alloys

Table 1. Detailed formulae of PEEK/PPS/PEI plastic alloys (wt %)

Materials	PEEK	PPS	PEI
K100-S0-I0	100	0	0
K85-S15-I0	85	15	0
K60-S40-I0	60	40	0
K35-S65-I0	35	65	0
K10-S90-I0	10	90	0
K0-S100-I0	0	100	0
K85-S0-I15	85	0	15
K60-S0-I40	60	0	40
K35-S0-I65	35	0	65
K10-S0-I90	10	0	90
K0-S0-I100	0	0	100
K90-S5-I5	90	5	5
K70-S15-I15	70	15	15
K50-S25-I25	50	25	25
K30-S35-I35	30	35	35

mal gravimetric analyzer (TGA) (Perkin-Elmer Pyris-1) at a heating rate of 10 °C/min in nitrogen atmosphere. The range of temperatures was from ambient temperature to 800 °C.

## 4. Results and Discussion

### 4.1 Structure and Morphology of PEEK/PPS/PEI Plastic Alloys

Figure 1 shows the FT-IR spectrum of PEEK/PPS/PEI plastic alloys with different compositions. The peak observed at 924.3  $\text{cm}^{-1}$  was due to stretching vibrations of  $\text{O}-(\text{C}=\text{O})-\text{O}$  present in PEEK. The stretching vibrations peak of  $\text{C}=\text{O}$  present at PEEK was shifted from 1652.2  $\text{cm}^{-1}$  to 1645.9  $\text{cm}^{-1}$  as PPS added, while the symmetrical stretching vibrations peak of  $\text{O}-\text{S}-\text{O}$  present at PPS was shifted from 1089.6  $\text{cm}^{-1}$  to 1083.3  $\text{cm}^{-1}$  as PEEK added. The absorbance intensities of characteristic peak for PEEK/PPS alloys decreases compared to the FT-IR spectrum of pure PPS and PEEK, as shown in Figure 1(A). The obvious interaction exists between PPS and PEEK during melt compounding can be proved.

The shifts in absorption spectra of stretching vibrations of  $\text{C}=\text{O}$ ,  $\text{C}-\text{N}$  present in PEI and stretching vibrations of  $\text{C}=\text{O}$ ,  $\text{O}-(\text{C}=\text{O})-\text{O}$  present in PEEK can not be observed, as shown in

Table 2.  $T_g$  of the alloys of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI

Materials	K100-S0-I0	K60-S40-I0	K35-S65-I0	K0-S100-I0	K60-S0-I40
$T_g / ^\circ\text{C}$	143	125	121	91	166
Materials	K35-S0-I65	K0-S0-I100	K50-S25-I25	K70-S15-I15	K90-S5-I5
$T_g / ^\circ\text{C}$	185	215	179	167	146

Figure 1(B). It was observed that the absorbent intensities of  $\text{O}-(\text{C}=\text{O})-\text{O}$  present in PEEK and C-N present in PEI have changed when PEI or PEEK was added. Compared with PEEK/PPS alloys, the change was little due to the amorphous structure of PEI polymer. The absorbent intensities of PEEK/PEI plastic alloys and interaction between PEI and PEEK increase along with the content of PEEK polymer increases.

As shown in Figure 1(C), comparing with pure PEI, PPS and PEEK, the peak position and intensities of stretching vibrations of  $\text{O}-(\text{C}=\text{O})-\text{O}$ , C=O, C-N and  $\text{O}-\text{S}-\text{O}$  present in PEEK/PPS/PEI were kept constant. The reasons why the interaction existing in PEEK/PPS or PEEK/PEI alloys was stronger than that in PEEK/PPS/PEI alloys were complicated. For PEEK/PPS and PEEK/PEI plastic alloys, the stronger interaction was due to the interaction between polar groups such as ether group and ketone group present in PEEK, PPS and PEI. For PEEK/PPS/PEI plastic alloys, the polar groups were separated into crystalline region and amorphous region. The groups were different from another group in the ternary system of PEEK/PPS/PEI plastic alloys.

Figure 2 shows the DSC heating curves of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI alloys with different compositions. The glass-transition temperature ( $T_g$ ) of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI alloys were listed in Table 2.

As shown in Fig. 2 and Table 2, only a single  $T_g$  for all the alloys during heating which have revealed that the alloys of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI with fully compatible in this temperature range. The compatibility of PEEK, PPS and PEI can be improved by increasing the uniformity of components. When the amorphous component of PEI increases, the amorphous region increases, the  $T_g$  of PEEK/PPS/PEI alloys increased from 146  $^\circ\text{C}$  to 179  $^\circ\text{C}$ .

Figure 3 shows the scanning electron microscopy (SEM) images of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI alloys. As shown in Figure 3(A) and 3(B), good dispersion of PPS particles

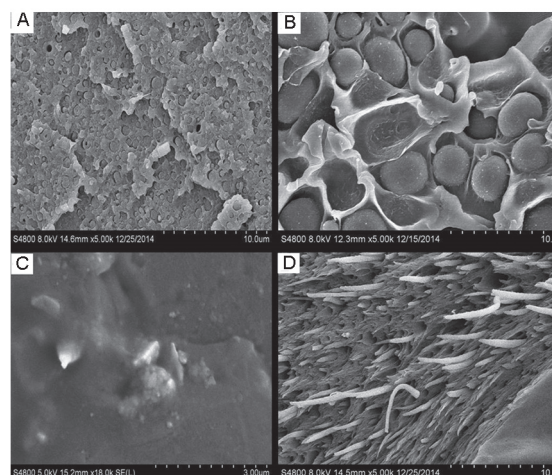


Fig. (3). SEM images of binary and ternary plastic alloys: (A) K60-S40-I0; (B) K35-S65-I0; (C) K70-S0-I30; (D) K70-S15-I15

in PEEK were observed. It was seen that the sphere-shaped dispersion of PPS increased with increasing the content of PPS. The aggregation behaviors of PPS particles were not observed. The SEM results revealed that PEEK and PPS has been completely compatible. Figure 3(C) shows that the phase interface between PEEK and PEI was blurred, there has not clear phase interface exists in PEEK/PEI alloys [21]. Figure 3(D) shows the SEM image of PEEK/PPS/PEI alloys, the existence of spinous structure and uniform distribution of PEI or PPS in the cross-section of PEEK/PPS/PEI alloys. The morphology of PEEK/PPS/PEI alloys was homogeneous. The SEM images revealed that the morphology of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI were homogeneous, which was conformed to TGA results.

#### 4.2 Crystallization behavior of *peek/pps/pei* plastic alloys

Figure 2 shows the DSC crystallization curves with heating process of binary alloys and ternary alloys. It can be seen that there was an endothermic peak but no crystallization peak in pure PEEK sample, which have proved that pure PEEK sample has completed crystallization before heating process. In the heating process of pure PPS and PEEK/PPS alloys, both endothermic peak and crystallization

Table 3.  $X_c$  and  $T_m$  of PEEK and PPS in DSC heating process

Materials	PEEK		PPS	
	$X_c$ (%)	$T_m$ (°C)	$X_c$ (%)	$T_m$ (°C)
K100-S0-I0	-	341.57	-	-
K60-S40-I0	11.20	341.89	15.86	282.76
K35-S65-I0	11.73	341.36	28.67	282.52
K0-S100-I0	-	-	25.15	282.52
K60-S0-I40	21.05	338.81	-	-
K35-S0-I65	21.95	329.67	-	-
K90-S5-I5	16.12	341.04	-	-
K70-S15-I15	7.96	339.54	12.60	284.39
K50-S25-I25	-	339.37	16.81	283.97

peak can be observed, which has revealed that the crystallinity degree ( $X_c$ ) of PEEK improved when uncompleted crystallization polymer PPS was added. Relatively, the crystallinity degree of PPS decreased with increasing the content of PEEK. Therefore, it can be proved that there was secondary crystallization during heating process in the blends in between crystallization polymers. With single glass-transition peak, the crystallization degree of compositions was obviously influenced by content.

Figure 2(B) indicated that there was only glass-transition peak in the DSC curve of pure PEI sample and there was no crystallization peak in pure PEEK sample. In the heating process of PEEK/PEI alloys, the endothermic peak and crystallization peak were observed. Therefore, it can be inferred that the crystallizability and crystallization rate of PEEK/PEI blends decreased when amorphous PEI was added. Structurally, there exists partly crystallization before heating process, because the mobility of molecular chains was limited during cooling process. During heating process, the mobility of molecular chains increased due to the total energy of blends increases, secondary crystallization can be observed.

Table 4.  $X_c$  and  $T_c$  of PEEK and PPS in DSC cooling process

Materials	PEEK		PPS	
	$T_c$ (°C)	$X_c$ (%)	$T_c$ (°C)	$X_c$ (%)
K100-S0-I0	306.16	28.82	-	-
K60-S40-I0	307.48	29.60	243.63	45.68
K35-S65-I0	305.69	24.04	243.08	51.38
K0-S100-I0	-	-	212.55	55.20
K60-S0-I40	296.60	30.65	-	-
K35-S0-I65	285.31	26.11	-	-
K90-S5-I5	305.57	32.60	-	-
K70-S15-I15	302.86	30.74	244.17	30.59
K50-S25-I25	303.34	20.05	246.01	43.44

It was shown in Figure 2(C) that the crystallization peak of PEEK decreased gradually with increasing the content of crystal polymer PPS and amorphous polymer PEI during heating process. The crystallization peak of PEEK/PPS/PEI alloy disappears in the ratio of 50/25/25. Therefore, it indicated that the crystallizability of PEEK reduced and even was eliminated when PPS and PEI were added simultaneously. It conformed to the interaction between the molecule chains as FT-IR.

The crystallinity degree ( $X_c$ ) and melting temperature ( $T_m$ ) of PEEK and PPS in heating process were presented in Table 3. During heating process, the  $T_m$  of PEEK and PPS for pure PEEK, pure PPS, PEEK/PPS alloys, PEEK/PEI alloys and PEEK/PPS/PEI alloys were almost invariable. For binary alloys, the influence of PPS or PEI content changes on secondary crystallization of PEEK was barely observed. As PPS or PEI was added, the changed of  $X_c$  for PEEK can be observed. The  $X_c$  of PEEK decreased when PPS was added and increased when PEI was added. Therefore, it can be proved that the crystallizability of PEEK inhibited by PPS was weaker than PEI dur-

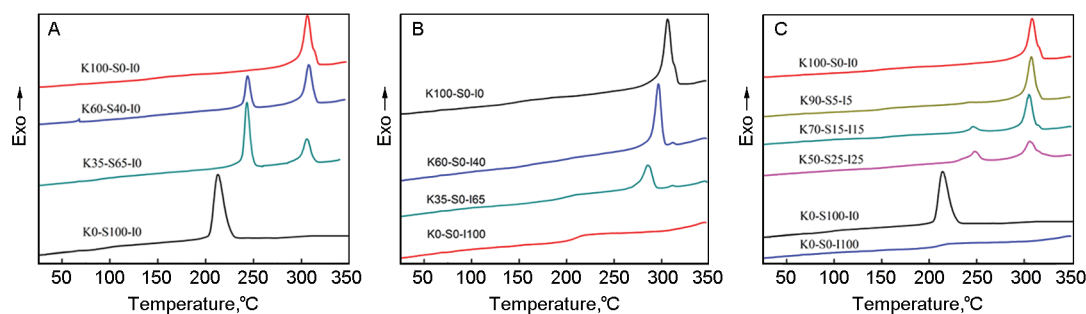


Fig. 4. DSC cooling curves of binary and ternary alloys: (A) PEEK/PPS alloys; (B) PEEK/PEI alloys; (C) PEEK/PPS/PEI alloys

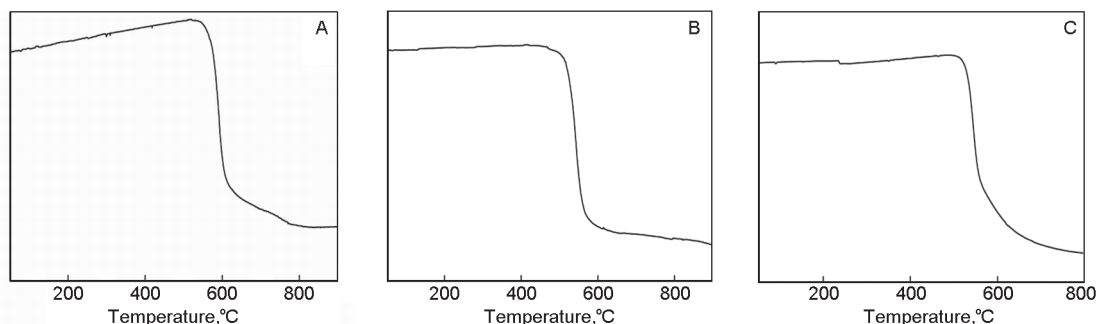


Fig. 5. TGA curves of pure PEEK, PPS and PEI: (A) pure PEEK; (B) pure PPS; (C) pure PEI

ing cooling process of blend melts. For PEEK/PPS/PEI ternary alloys, the crystallizability of PEEK changed when PPS and PEI were added simultaneously during melt blending process. The secondary crystallization of PEEK disappeared in the ratio of 50/25/25 during heating process, because PEEK has completed crystallization in this ratio during cooling process. There was no secondary crystallization of PPS by adding a small amount of PEI. With increasing the content of PEI, the secondary crystallization was observed once again.

Figure 4 shows the DSC cooling curves of binary and ternary alloys. As shown in Figure 4(A), the crystallization temperature ( $T_c$ ) of PEEK almost remains constant when PPS was added, and the  $T_c$  of PPS deviates from the lower temperature to higher one when PEEK was added. Therefore, it can be inferred that PEEK crystal was firstly formed, results in heterogeneous nucleation crystallization of PPS during cooling process and improved the  $T_c$  of PPS in PEEK/PPS alloys. With the increases of PEI, the  $T_c$  of PEEK deviated from the higher temperature to a lower one, but the variation was small, which can be observed in Figure 4(B). The crystallization temperature changes of PEEK can not be observed when PPS and PEI were added, while the crystallization temperature of PPS increased when PEEK and PEI were added simultaneously, as shown in Figure 4(C).

Table 4 shows the crystallinity degree ( $X_c$ ) and crystallization temperature ( $T_c$ ) of PEEK and PPS in cooling process. As PPS or PEI was added, the  $T_c$  of PEEK binary alloys shows a decreasing trend, the falling range of PEEK/PEI alloys was bigger than that of PEEK/PPS alloys. The  $T_c$  of PEEK/PPS/PEI alloys increases, and then decreases with PPS and PEI added simultaneously. The  $T_c$  of PPS in PEEK/PPS binary

Table 5. Td5% of alloys treated by twice heat treatment (°C)

Materials	Original	Secondary
K0-S100-I0	495	495
K100-S0-I0	550	550
K0-S0-I100	515	515
K85-S15-I0	520	535
K35-S65-I0	500	520
K60-S0-I40	515	540
K85-S0-I15	525	525
K70-S15-I15	510	545

alloys increased when PEEK was added. In Table 4, the  $X_c$  of pure PEEK and pure PPS can be measured to 28.82 % and 55.20 %. Compared with pure PEEK, the  $X_c$  of PEEK in binary alloys increased when PPS or PEI was added, but decreased when the ratio of PEEK in binary alloys was lower than that of PPS or PEI. The  $X_c$  of PPS in PEEK/PPS alloys decreased when PEEK increases. For PEEK and PPS in PEEK/PPS/PEI alloys, the changes of  $X_c$  were complicated. In the ratio of 90/5/5, the  $X_c$  of PEEK was presented to 32.60 %, while PPS was without crystal. The  $X_c$  of PEEK are 30.74 % and 20.05 % in the ratio of 70/15/15 and 50/25/25, while the  $X_c$  of PPS were 30.59 % and 43.44 %.

#### 4.3 Thermal Behavior of PEEK/PPS/PEI Plastic Alloys

In Figure 5 the thermal decomposition temperature ( $T_d$ ) of pure PEEK, pure PPS and pure PEI can be measured to 550, 495 and 515 °C. The pure PEEK with best thermal stability was mainly caused by the stronger intermolecular interaction between benzene ring structures present in PEEK molecular.

Figure 6 shows the TGA curves of binary and ternary alloys. The alloys of PEEK/PPS in the weight ratio of 85/15 and 35/65 present the

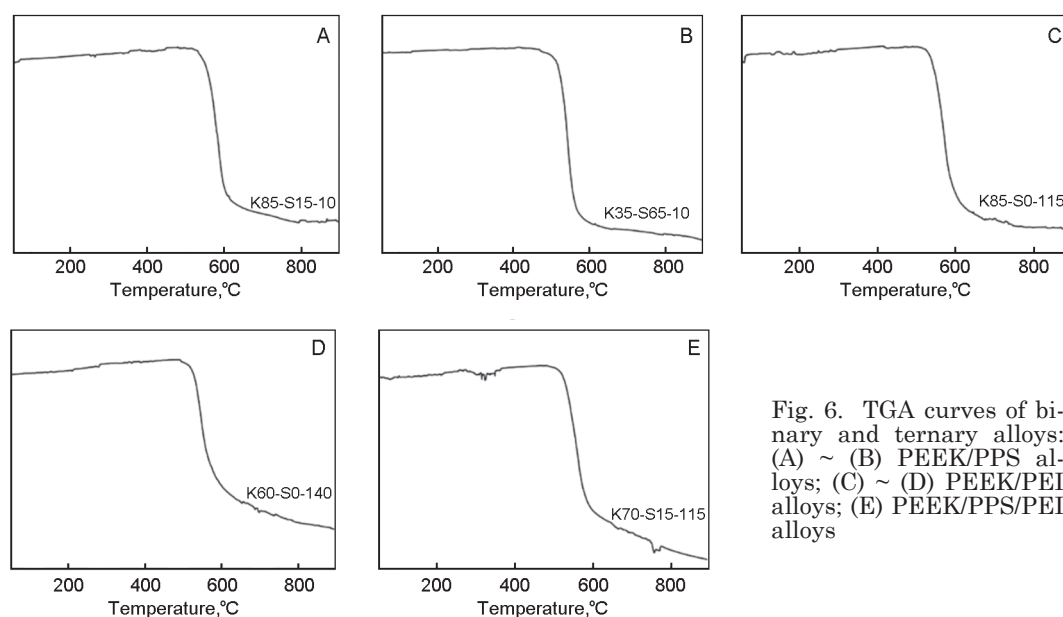


Fig. 6. TGA curves of binary and ternary alloys: (A) ~ (B) PEEK/PPS alloys; (C) ~ (D) PEEK/PEI alloys; (E) PEEK/PPS/PEI alloys

initial decomposition temperature at 520 and 500°C. Compared with pure PEEK, it can be presented in the results that the  $T_d$  of PEEK/PPS alloys decreased with increasing the content of PPS. As shown in Figure 6(C) and 6(D), the initial decomposition temperature of PEEK/PEI in the ratio of 85/15 and 60/40 presented at 525 and 515°C. Compared with pure PEEK and pure PEI, the  $T_d$  of PEEK/PEI alloys decreased when PEI was added. In Figure 6(E), the  $T_d$  of PEEK/PPS/PEI alloy in the ratio of 70/15/15 presented at 510°C, which was lower than pure PEEK and pure PEI, higher than PPS.

In order to have a better research of the effects of heat history on thermal stability of the plastic alloys, the original extrudate was treated by secondary extrusion granulation and studied by TGA. Table 5 shows the  $T_d$  5% of alloys treated by twice heat treatments. As treated by secondary extrusion granulation process, the  $T_d$  5% of pure PEEK, pure PPS and pure PEI remain content, while the  $T_d$  5% of plastic alloys is higher than original extrudate. The reason can be explained that the interaction between groups treated by secondary extrusion granulation is stronger than original extrudate.

### 5. Conclusions

The structure, morphologies, crystalline and thermal properties were measured with FTIR, SEM, DSC and TGA. The FTIR results revealed that the interaction exists in PEEK/PPS alloys was the strongest, the interaction exists in PEEK/PPS/PEI alloys was weaker

than in PEEK/PPS and PEEK/PEI alloys. The SEM images revealed that the morphology of PEEK/PPS, PEEK/PEI and PEEK/PPS/PEI alloys were homogeneous structure. The DSC results shows that all of the plastic alloys based PEEK exhibits only a single  $T_g$ , with the increases of amorphous component of PEI, the  $T_g$  of PEEK/PPS/PEI alloys increased from 146°C to 179°C. The crystallinity degree of PEEK improved when uncompleted crystal polymer PPS was added. The crystallinity degree of PPS decreased with increasing the content of PEEK. The crystallizability and crystallization rate of PEEK/PEI blends decreased when amorphous PEI was added. The crystallization peak of PEEK/PPS/PEI alloy disappeared in the ratio of 50/25/25. The TGA results shows that the  $T_d$  of PEEK/PPS and PEEK/PEI alloys decreases when PPS and PEI were added, and the  $T_d$  of PEEK/PPS/PEI alloy in the ratio of 70/15/15 present for 510°C, which was lower than in pure PEEK and pure PEI, higher than in PPS.

### Acknowledgements

This work was financially supported by the Key Projects of the Outstanding Young Talents in Colleges and Universities of Anhui Province (gxyqZD2016373) and the Department of Education of Anhui Province (KJ2013A197).

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