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Crystallization and Mechanical Properties of Continuous Carbon Fiber Reinforced Polyether-ether-ketone Composites

Kaili Zhu, Hongsheng Tan*, Yajie Wang, Changheng Liu, Xiaomin Ma, Jie Wang, and Haini Xing

School of Materials Science and Engineering, Shandong University of Technology, Zibo 255000, China

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Abstract: The effect of two different continuous carbon fibers (CCF1 and CCF2) with different material properties on structure-properties of continuous carbon fibers reinforced polyether-ether-ketone prepreg tapes (CCF/PEEKPT) prepared by a wet powder impregnation process were investigated. The effects of fiber content on the tensile properties, dynamic mechanical behavior, crystallization melting behavior and fracture morphology of prepared prepreg tapes were performed by the dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC) and scanning electron microscope (SEM) etc. The results showed that, the tensile strength of CCF1/PEEKPT increased with increasing of the carbon fiber content within 50-75 wt%. Meanwhile, the tensile strength of 50-65 wt% CCF2/PEEKPT had the same change trend, but then decreased when the fiber content of composites was higher than 65 wt%. The storage modulus (E') of composites increased with adding content of carbon fiber. The composites still maintained high deformation resistance when the temperature rose to 290 ^{o}C. Compared with pure PEEK, the crystallinity and crystallization rate of the composites both increased with the occurrence of fiber inducing PEEK crystallization, but the crystallization onset temperature, crystallization temperature and melting temperature of CCF/PEEKPT moved to low temperatures with adding of fiber content. In general, these observations suggested that CCF hindered the movement of the polymer chain segment and constrained the spherulites growth of PEEK for CCF/PEEKPT.

Keywords: Unidirectional prepreg tapes, Continuous carbon fiber reinforced, PEEK, Mechanical properties, Crystallization melting behavior

Introduction

High performance and lightweight were important research directions in the field of composite materials. Benefiting from convenient processing, good heat resistance, excellent impact resistance and recoverable properties, thermoplastic composites were applied to various high-performance structural materials, which partly replaced the conventional thermoset composites [1,2]. In recent years, carbon fiber (CF) was favored by researchers for its excellent properties, such as light weight, high strength, high modulus and high temperature resistance. However, carbon fiber was difficult to be applied directly because it was a linear material. Hence, selecting suitable resin matrix and preparing CF reinforced resin composites were one of the approaches to solve the problem of restricting the CF application. Polyether-ether-ketone (PEEK) was a kind of fully aromatic semicrystalline thermoplastic engineering plastics, and had excellent mechanical properties [3], corrosion resistance [4], thermal stability [5] and biocompatibility [6], which could be applied to aerospace, automotive, high temperature applications, biomedicine and other fields. It was considered the most promising thermoplastics.

At present, many studies were carried out on CF reinforced PEEK (CF/PEEK) composites [7-11]. Short and long fibers reinforced with simple processing method were widely used in CF/PEEK composites, and its mechanical properties were obviously lower than that of continuous carbon fiber reinforced PEEK (CCF/PEEK) composites. Garcia-Gonzalez *et al.* [12] studied the effect of short CF on the mechanical impact behavior of CF/PEEK composites fabricated by injection molding technology. The results showed that short CF reinforced PEEK composites exhibited the low absorption energy capability and a brittle failure comparing with unfilled PEEK. Hence, the development and application of CCF/PEEK composites received more attention. Fujihara *et al.* [13] studied the effect of nine different fabrication conditions on bending properties of the CCF/PEEK composites based on a micro-braiding fabrication method. It could be found that the 380 ^{o}C and the 20 min holding time were optimal experimental conditions. Under this condition, the bending modulus and bending strength of CCF/PEEK composites were 95 GPa and 1300 MPa, respectively. Xu *et al.* [14] investigated the effect of the molding temperature and molding time on the mechanical properties of unidirectional CCF/PEEK composites via the wrapped yarn method, according to thermal and fracture morphology characterizations. The tensile strength was 1500 MPa, which was lower than the most representative product APC-2 (CCF/PEEK). Typically, for unidirectional AS4/PEEK (60 % by volume CF), its tensile strength was about 2100 MPa. Zhang *et al.* [15] reported the novel CCF/PEEK composites fabricated by a solution impregnation process, ascribed to introduction of crosslinkable phenylethynyl pendant to PEEK. The longitudinal tensile strength of the CCF/PEP-PEEK composites was increased to 1610 MPa. Lee *et al.* [16] evaluated the effect of preheating time on the tensile strength and crystallization of PEEK and CCF/PEEK

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*Corresponding author: hshengtan@163.com

composites prepared by compression molding. The results indicated that the tensile strength and crystallization of CCF/PEEK composites were improved with increasing preheating time. The preparation process of CCF/PEEK composites above were complex as well as had high production cost, which limited their application in industrial production. However, a wet powder impregnation process had the advantages of low concentration of surfactant additive, no health hazard and easy handling of additives, simple processing method and low cost. In recent decades, researchers prepared natural and glass fiber reinforced polypropylene composites [17,18], continuous carbon fiber reinforced polyphenylenesulfide composites [19,20] by powder impregnation process. At present, there were few studies on the preparation of CCF/PEEK composites by wet powder impregnation process, especially the CCF/PEEK prepreg tapes.

In order to further improve the preparation efficiency and reduce the cost of CCF/PEEK composites, in this work, the continuous carbon fibers reinforced polyether-ether-ketone prepreg tapes (CCF/PEEKPT) were prepared by a wet powder impregnation process. And the effects of two different CCFs and fiber content on the tensile properties, dynamic mechanical behavior, crystallization melting behavior and fracture morphology were evaluated. The mechanical properties of composites with different fiber contents were compared to reveal the reinforcing mechanism of CCF/PEEKPT. The use of a non-isothermal method was preferred to analyze the crystallization behavior, since PEEK and PEEK composites processing were usually performed under dynamic non-isothermal conditions. Therefore, it was meaningful to provide information on the effect of the fiber content on the crystallization behavior of the resin during the processing. These results would help to broaden the application of PEEK composites in aerospace, automobile and other fields.

Experimental

Materials

PEEK powders were supplied by Jiangsu Huajun New Material Co., Ltd., China. Carbon fibers (T700SC-12K) were purchased from Japan Toray Co. with tensile strength of 4900 MPa, modulus of 230 GPa, density of 1.8 g/cm^3, and diameter of 7 μm, labeled as CCF1. Carbon fibers (TC35R-24K) were produced by Taiwan Taili with tensile strength of 2424 MPa, modulus of 240 GPa, density of 1.8 g/cm^3, and diameter of 7 μm, labeled as CCF2. Deionized water was homemade in the laboratory. Solvents and other additives were domestic industrial products. It provided the two different CCFs with different tensile strength and modules. In previous literatures, CF/PEEK composites had given different material properties in terms of mechanical strength, modules and crystallization melting behavior, in the presence of fibers with different properties [21,22].

Preparation of CCF/PEEK Prepreg Tapes

The preparation process of CCF/PEEKPT included the following steps: firstly, the PEEK powder was uniformly dispersed in the dispersion; secondly, the CCF were spread evenly in the sizing tank by adjusting the tension, which made the PEEK attach to the surface of the CCF; thirdly, the fiber bundles were pre-prepared the self-designed preheated melting mold, and the CCF/PEEK prepreg tapes were prepared by extrusion and finally coiled. The prepreg tapes were prepared by the wet powder impregnation process with low cost and less time than micro-braiding fabrication, the wrapped yarn and solution impregnation [13-15].

Measurements

Tensile Property

Tensile properties were measured using a Model 5969 universal material testing machine manufactured by INSTRON, Inc. Tensile specimens of prepreg tapes were prepared according to ASTM D3039/D3039M-14, and the stretching speed was 10 mm/min. Figure 1 showed the dimensions of the specimen for tensile test. The length of gauge was138 mm and the aluminum tabs were 56 mm. The tabs with thickness of 0.6 mm were attached to the specimens to avoid possible damage when the specimens were gripped.

Dynamic Mechanical Analysis

Dynamic Mechanical Analysis (DMA) test were carried out using a Q800-DMA instrument manufactured by TA, USA, operating in tensile mode and a frequency of 1 Hz, running from 0 to 320 ^{o}C at a heating rate of 5 ^{o}C/min under nitrogen atmosphere. The specimen dimensions were 40×1×0.1 mm.

Differential Scanning Calorimeter

The DSC test was conducted on a Q2000-DSC differential scanning calorimeter manufactured by TA Instruments of the United States. These samples were heated at 20 ^{o}C/min from room temperature to 400 ^{o}C under nitrogen atmosphere,

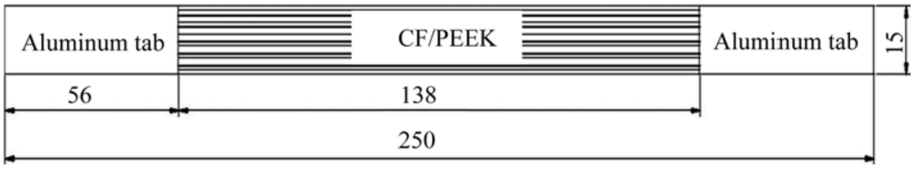

Figure 1. Dimension of the tensile specimen (mm).

and held for 2 min to remove the previous thermal history, ensuring that the sample preparation process has no effect on the crystallization behavior during the study, then cooled it down to 0 °C at a rate of 10 °C/min, after then the samples were reheated to 400 °C at a rate of 10 °C/min. The DSC curve of the sample was obtained.

The degree of crystallinity was calculated using the following formula (1):

$$X_c(\%) = \frac{\Delta H_c}{(1-W_f)\Delta H_f^0} \times 100\% \tag{1}$$

where ΔH_c was the melting enthalpy of composites, ΔH_f^0 was the theoretical melting enthalpy of PEEK with the value of 130 J/g, W_f was mass fraction of CCF.

The relative crystallinity ($X(t)$) was calculated using the following formula (2):

$$X(t) = \frac{\int_{t_0}^{t}\left(\frac{dH}{dt}\right)dt}{\int_{t_0}^{t_e}\left(\frac{dH}{dt}\right)dt} \times 100\% \tag{2}$$

where H was heat flow, t was crystallization time, t_0 was the onset crystallization time, t_e was the end crystallization time.

The crystallization time (t_c) was calculated using the following formula (3):

$$t_c = \frac{|T-T_0|}{a} \tag{3}$$

where a represents the cooling rate and T_0 was the crystallization onset temperature.

Scanning Electron Microscope

Fracture surfaces of tensile specimens were observed by a scanning electron microscope (Quanta 250 FEI Inc.) after coating gold-palladium.

Results and Discussion

Mechanical Properties

Tensile

Figure 2 presented the effect of two different grades of CCF on the tensile properties of CCF/PEEKPT. As shown, the tensile strength of CCF1/PEEKPT increased with increasing the carbon fiber content. The sample with 75 wt% CCF1/PEEKPT had the highest tensile strength of reaching 1860 MPa, which increased by 56.04 % in comparison to the sample with 50 wt% CCF1/PEEKPT and rose a 18 times comparing with pure PEEK. Meanwhile, the tensile strength of CCF2/PEEKPT increased and then decreased with the carbon fiber content increasing. The highest tensile strength was 1100 MPa for the samples with 65 wt% CCF2/PEEKPT, which had increased by 59.88 % comparing with 50 wt% CCF2/PEEKPT and had a 18 times increase comparing with pure PEEK.

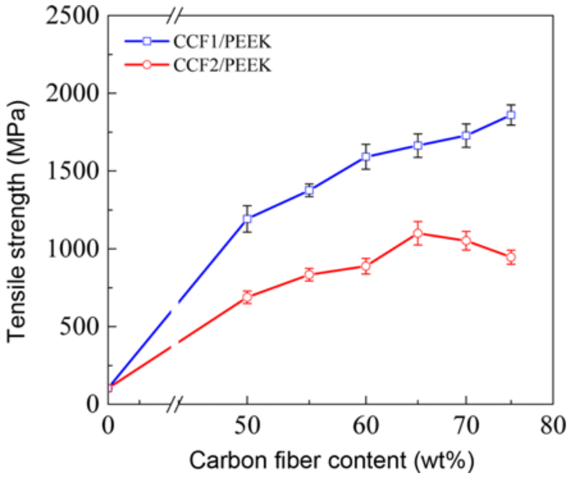

Figure 2. Tensile strength of CCF/PEEKPT against content of CCF.

The tensile strength of composites was determined by the matrix strength, fiber strength, and interfacial adhesion. The composite material was subjected to an external load, which could be effectively transmitted to the fiber through the resin matrix, thus carbon fiber played a critical enhancement role in composite. There was a huge difference in the reinforcement effect of the fiber with difference in tensile properties. The tensile strength of CCF1 was up to 4900 MPa, which had strong effect on matrix reinforcement, so that the tensile strength of the composite was higher. The tensile strength of CCF2 was 2424 MPa, which was lower than CCF1. The content of resin was low when the fiber content was higher than 65 wt%. Owing to the greater frictional force between the prepreg and the mould in the process of the prepreg tapes prepared, the fiber appeared the phenomenon of broken wire because of the low strength. Therefore, the effective fiber content bearing the load could be reduced during the tensile test. The tensile strength of the prepreg tapes became lower due to the fiber reinforcement decreased.

Figure 3 displayed the stress-strain curves of CCF/PEEKPT. It revealed the damage mechanism of the prepreg tapes which occurred the brittle fracture during tensile testing. This process was divided into two stages. In the first stage (see number 1 arrow in Figure 3), the stress-strain curves showed a linear relationship. The stress linearly increased with the increasing of displacement no matter which content of CCF. Under the action of external forces, the longitudinal parallel arranged fibers were carrying load together, which leaded to the load on the prepreg tapes gradually increasing. In the second stage (see number 2 arrow in Figure 3), the prepreg tapes appeared the phenomenon of fracture gradually when it continued applying load. Because the thickness of the prepreg tapes was thinner, the edge part broke first under the effect of external force, and the remaining effective loading external force of fiber tow became smaller and the prepreg tapes was gradually broken

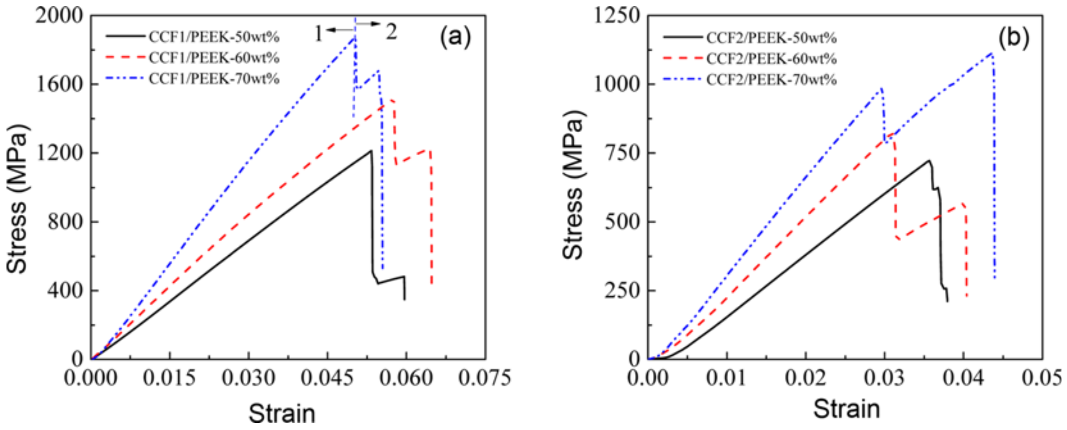

Figure 3. Strain-stress curves of CCF/PEEKPT; (a) CCF1/PEEK and (b) CCF2/PEEK.

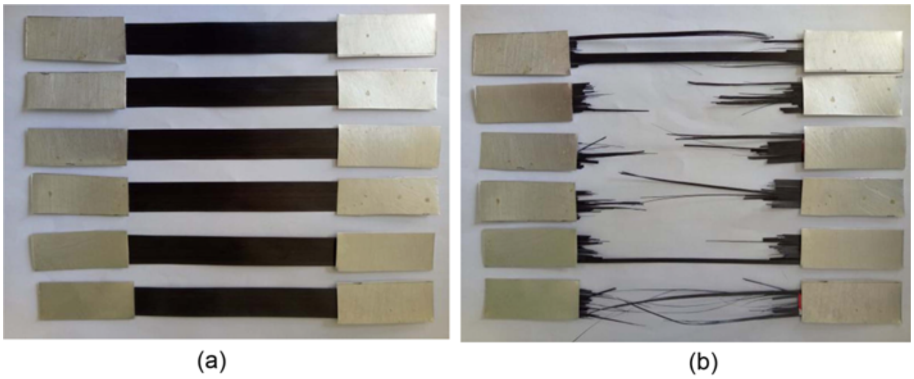

Figure 4. Tensile specimens photos before and after tensile testing; (a) before and (b) after.

when it continued to apply load. Figure 4 depicted tensile specimen photos before and after tensile testing. It was concluded that the fracture of the prepreg tapes was uneven which corresponded to the fluctuation of the stress-strain curves in the second stage as shown in Figure 3.

Fracture Morphology of Tensile Specimens

Figure 5 displayed the fracture morphology for CCF/PEEKPT. As shown in Figure 5(a) and Figure 5(b), fibers uniformly dispersed and wrapped with substantial PEEK matrix with less voids, which illustrated the better interfacial adhesion between fibers and PEEK matrix. This was an important reason for the high tensile strength of CCF1/PEEKPT in Figure 2. As shown in Figure 5(c) and Figure 5(d), the fiber appeared the phenomenon of multiple breakpoints in the cross sections of CCF2/PEEKPT. Although there was little residual PEEK adhering on surfaces of CCFs and fiber-matrix debonding accompanied by pull-out of CCFs. It indicated the weak interfacial adhesion between fibers and PEEK matrix. Correspondingly, this was one of the reasons for the low tensile strength of CCF2/PEEKPT in Figure 2.

Dynamic Mechanical Analysis

The influence of temperature on storage modulus (E') of the CCF/PEEKPT had been investigated by the DMA. E' characterized the ability to resist deformation of materials, which was closely related to the mobility of polymer molecular chain segments and side groups [23]. Figure 7 showed the evolution of the storage modulus as a function of temperature. The E' of the prepreg tapes were proportional to the fiber content, which were increased with increasing content of fiber. The E' of CCF1/PEEK-50 wt%, CCF1/PEEK-70 wt%, CCF2/PEEK-50 wt% and CCF2/PEEK-70 wt% at the initial temperature were 69.69, 100.83, 81.06 and 106.74 GPa, respectively.

The elastic modulus of different grades of fiber was different. The E' of the composite was closely related to the elasticity modulus of the fiber. The elastic modulus of the fiber was high, and the E' of the corresponding composite was high. The prepreg tapes were subjected to alternating tensile stress which was effectively transferred to the CF through the matrix. Therefore, the stress of the prepreg tapes was mainly borne by CF. The higher the elastic modulus of the fiber was, the higher the E' of the composite material was, and the stronger the deformation resistibility ability was. PEEK was in the glassy state region which was mainly confined to vibration and short range rotation when the

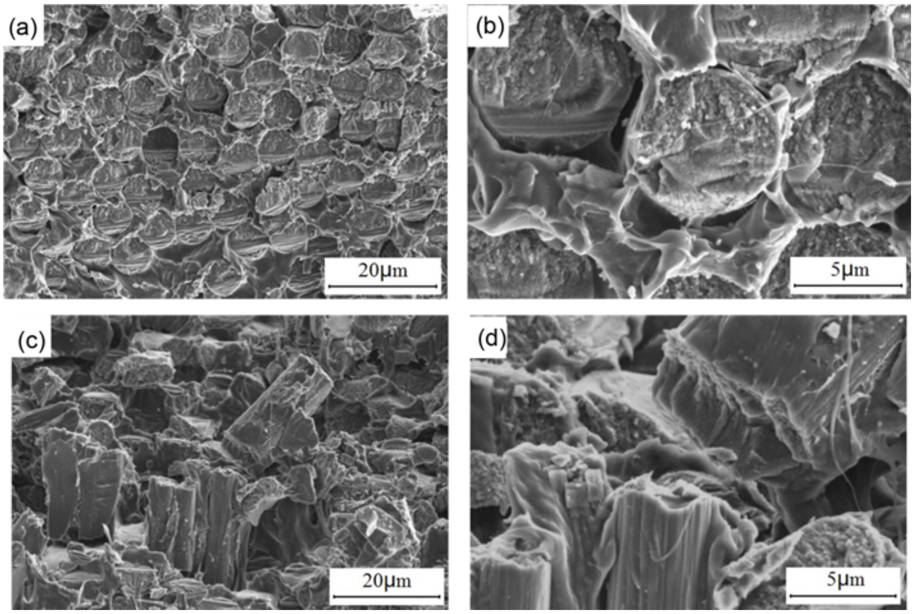

Figure 5. SEM images of fracture morphology for CCF/PEEKPT with two different fibers; (a) CCF1/PEEK×5000, (b) CCF1/PEEK ×20000, (c) CCF2/PEEK×5000, and (d) CCF2/PEEK×20000.

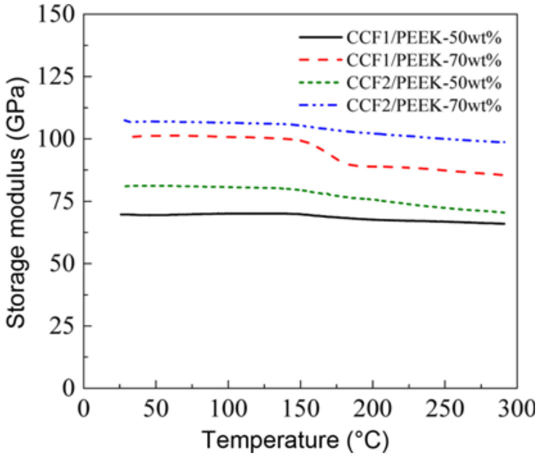

Figure 6. Storage modulus versus temperature of CCF/PEEKPT.

temperature was below 150 °C *E′* remained relatively stable. The segment had enough energy to move in a cooperative way, where PEEK began to change from glassy state to high elastic state when the temperature rose to 200 °C from 150 °C. *E′* had a declined trend where the temperature of *E′* decreased significantly was T_g at about 180 °C. PEEK was in a high elastic region when the temperature was above 150 °C, where the parallel arrangement of CCF limited the movement of the molecular chain because of the physical entanglement in the molecules. Hence, *E′* decreased slowly. When the temperature rose to 290 °C, the remaining *E′* of CCF1/PEEK-50 wt%, CCF1/PEEK-70 wt%, CCF2/PEEK-50 wt% and CCF2/PEEK-70 wt% were 65.99, 85.45, 70.48 and 98.69 GPa, respectively. The decline rate of *E′* was only 5-15 %. It was concluded that CCF/PEEK composites still maintained high deformation resistance at high temperatures.

Crystallization and Melting Behavior

Non-isothermal Crystallization

In order to investigate the influence of the two different CCFs on the properties of the prepreg tapes, it was necessary to compare crystallization melting behavior of the prepreg tapes. There were two major mechanisms affecting the crystallization of PEEK in the crystallization process of fiber reinforced polymer composites: on the one hand fiber promoted heterogeneous nucleation, on the other hand fiber hindered the movement of polymer chain segments. The crystallization behavior of the pure PEEK compared with CCF/PEEKPT. If heterogeneous nucleation was dominating, it would accelerate the orderly arrangement of the polymer molecules on the CF and produce the crystallization, which would cause T_c to move towards high temperature. Conversely, if hindering the movement of polymer chain segments was dominating, it played a negative role of the formation of complete microcrystals, which would cause T_c to move towards low temperature [24].

Figure 7 displayed crystallization exotherms of pure PEEK and CCF/PEEKPT with different fiber content. As shown in Figure 7 and Table 1, T_0 and T_c moved to low temperature with adding carbon fiber comparing with pure PEEK. The X_c of pure PEEK, CCF1/PEEK-50 wt%, CCF1/PEEK-70 wt%, CCF2/PEEK-50 wt% and CCF2/PEEK-70 wt% were 34.04, 35.62, 37.46, 36.29, 37.26 %, respectively. It could be concluded that the crystallinity of the prepreg tapes increased gradually with increasing fiber content. Due

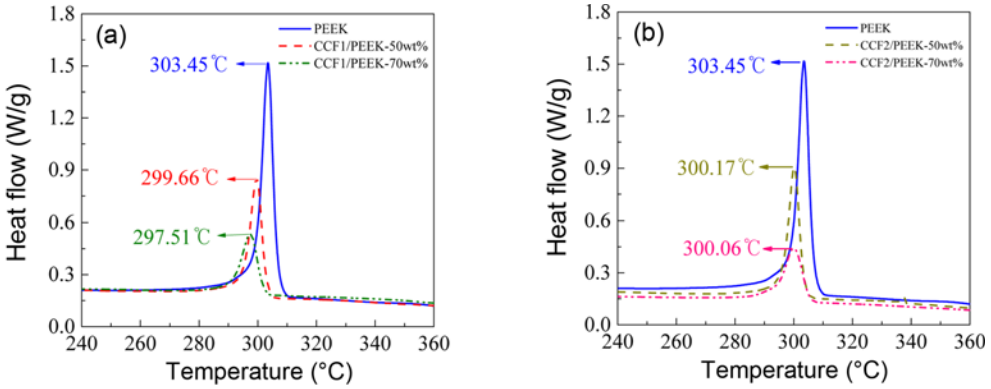

Figure 7. Crystallization exotherms at different fiber content for pure PEEK and CCF/PEEKPT; (a) CCF1/PEEK and (b) CCF2/PEEK.

Table 1. Values of T_0 (crystallization onset temperature), T_c (end crystallization temperature), ΔH_c (the crystallization enthalpies) and X_c (absolute crystallinity), obtained from equation (1) for all five samples with different fiber content

Materials	T_0 (°C)	T_c (°C)	ΔH_c (J/g)	X_c (%)
PEEK	312.41	276.02	44.24	34.04
CCF1/PEEK-50 wt%	306.34	280.56	23.15	35.62
CCF1/PEEK-70 wt%	304.45	285.87	14.61	37.46
CCF2/PEEK-50 wt%	309.38	286.25	23.59	36.29
CCF2/PEEK-70 wt%	307.10	288.15	14.53	37.26

to the interaction force between PEEK and CF and the induction of graphite microcrystals in CF, the PEEK chain tended to overcome the position resistance caused by partial overlap between the adjacent C-H bonds of the benzene ring with the confront conformation, reaching the conformation close to the graphite grain boundary, thus forming a transverse crystal by forming a conjugate structure with the graphite microcrystal [25]. Nucleation of the PEEK matrix mainly occurred on the surface of the fiber for the CCF/PEEKPT. The factor determining the size of the crystal was the temperature and the distance of two adjacent fibers. However, for CCF/PEEKPT with higher fiber content, the distance of two adjacent fibers was a key factor in determining the size of the crystal [26]. If the fiber content of the CCF/PEEKPT was higher than 50 wt%, the distance between two adjacent fibers was smaller relative to low fiber content composites, which hindered the movement of the polymer chain segment and constrained the spherulites growth [27]. It could be concluded T_c moved to low temperature. T_0 moved to low temperature, which indicated that the increase of fiber content would lead to crystallization of the prepreg tapes at lower temperature [28]. The influence of different types of fibers on the crystallization of PEEK needed to take into account the density and diameter of fibers. Since CCF1 and CCF2 had the same density and diameter, the hindrance to the movement of their segments was similar. Hence, the polymer achieving the same weight percentage had similar crystallinity.

$X(t)$ vs. Time was presented in Figure 8. The data of graph could be directly calculated by equation (2) and equation (3). As shown in Figure 8, all samples had the same shape S curve. The crystallization time of the composites decreased with increasing of fiber content comparing with pure PEEK, indicating that the fiber played a heterogeneous nucleation effect on PEEK and promoted the crystallization of the resin. The curved shape of the lines at the end of crystallization

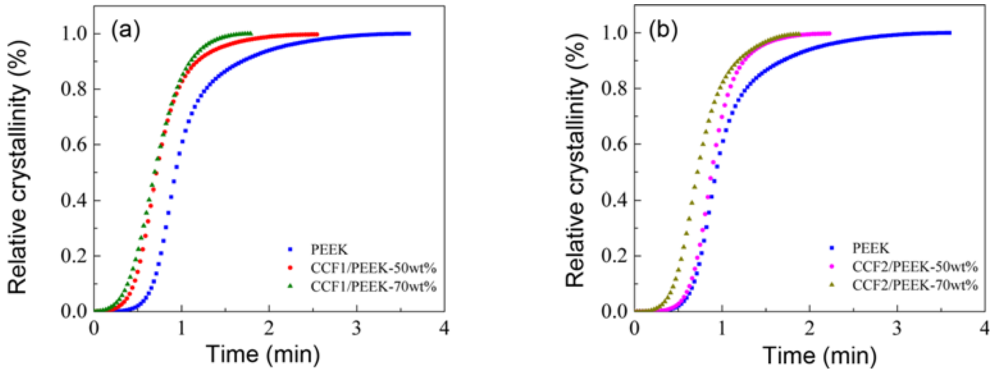

Figure 8. $X(t)$ (Relative crystallinity percentage with respect to time) versus time for CCF/PEEKPT with different fiber content; (a) CCF1/PEEK and (b) CCF2/PEEK.

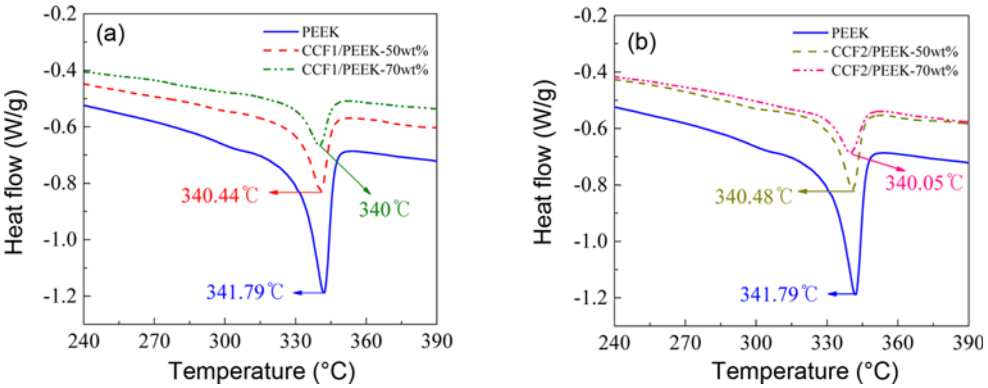

Figure 9. Heating curves of pure PEEK and CCF/PEEKPT after the non-isothermal crystallization study; (a) CCF1/PEEK and (b) CCF2/PEEK.

period could be attributed to spherulite impingement [28].

Melting Behavior

Figure 9 displayed the melting curves of pure PEEK and CCF/PEEKPT with different fiber content after non-isothermal crystallization. It showed that the T_m of CCF/PEEKPT was found to be lower than that of the pure PEEK, probably due to the addition of fibers constraining the spherulites growth. Spherulites of composites formed at a lower temperature tend to be smaller and produce more defects with increasing adding fiber, leading to a lower T_m in subsequent heating [24]. By comparing the curves of the CCF/PEEKPT with the same fiber content in Figure 9, it could be seen that the fluctuation range of the T_m of the resin matrix was within the error range. It concluded that adding the same density and diameter of CCF had less influence on the melting behavior of the PEEK matrix.

Conclusion

In this study, the CCF/PEEKPT was prepared by wet powder impregnation process. The experimental results showed remarkable enhancements in mechanical properties of the prepreg tapes as increasing two different CCFs with tensile strength and modules. However, it had less influence on the crystallization melting behavior of the prepreg tapes owing to the two different CCFs with the same density and diameter. The results of mechanical tests showed that mechanical performances of CCF/PEEKPT were improved obviously with increasing of CCF. The tensile strength of 75 wt% CCF1/PEEKPT was 1860 MPa, which was increased by 56.04 % comparing with 50 wt% CCF1/PEEKPT. And the tensile strength of 65 wt% CCF2/PEEKPT was 1100 MPa, which was improved by 59.88 % comparing with 50 wt% CCF2/PEEKPT. This could be further confirmed by SEM observations on fracture morphology of prepreg tapes, where the dominant failure mechanisms existed great variations. The E' of prepreg tapes increased with adding content of carbon fiber. The decline rate of E' was only 5-15 %, when the temperature was 290 ^{o}C. It indicated that the CCF/PEEKPT still maintained high deformation resistance at high temperature. The crystallinity and crystallization rate of the composites both increased with the occurrence of fiber, which indicated that the fiber induced the crystallization of the PEEK matrix. However, high weight of fiber content CCF/PEEKPT constrained the spherulites growth by an impingement mechanism causing T_0 and T_c to move towards low temperatures. With the increasing of fiber content, the T_m of composites moved towards low temperature. Spherulites of CCF/PEEKPT formed at a lower temperature tend to be smaller and produce more defects with increasing adding fiber, leading to a lower T_m in subsequent heating.

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