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ORIGINAL PAPER

Interfacial adhesion study on UHMWPE fiber-reinforced composites

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Abstract Ultrahigh molecular weight polyethylene (UHMWPE) fiber has many outstanding properties. However, poor interfacial adhesion of the UHMWPE fiber/polymer matrix interface limits its applications as reinforcement in high performance polymer matrix composites. Therefore, a new thermosetting resin system, named PCH, which is only composed of carbon and hydrogen elements, has been developed according to law of similar mutual solubility and the structural characteristics of UHMWPE fiber. The adhesion property was investigated by mechanical properties test, thermal performance test, and polymer solution properties test. Test results show that a strong interaction occurs between UHMWPE fiber and the PCH matrix due to the structural and polar similarity. In the case of slight difference between solubility parameters of UHMWPE fiber and cured PCH resin, it is found that the wettability of PCH resin on surface of the fiber can be improved and the difference between the coefficients of thermal expansion of the matrix and the fiber decreases with the increase of styrene added into the PCH. An optimal interfacial adhesion can be obtained as the ratio of PCH/styrene is approximately 55/45.

Keywords UHMWPE fiber · Polymer–matrix composites · Interfacial adhesion · Wettability

Introduction

Ultrahigh molecular weight polyethylene (UHMWPE) fiber has many outstanding mechanical properties, such as high strength, high modulus, low density, high

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impact resistance, and excellent abrasion tolerance. Therefore, UHMWPE fiber has been considered as reinforcing materials in high performance polymer matrix composites used in many fields where specific strength is needed such as aircraft and space structures [1]. Unfortunately, poor interfacial adhesion of the UHMWPE fiber/polymer matrix interface limits its applications. To increase the applicability of UHMWPE fibers in high performance composites, it is necessary to improve the interactions between UHMWPE fiber and matrices so that the bonding of the interfaces can be enhanced.

In order to improve the interfacial adhesion of UHMWPE fiber/polymer matrix, the mostly used techniques are to treat the fiber surface, including plasma treatment [2–5], nitrogen ion implantation [6], fast atom beams [7], EB irradiation [8], UV-initiated grafting [9], chemical treatments [10, 11], etc. These treatments on fiber surface can improve the fiber/matrix interface wetting and adhesion to some degree. These approaches effectively enhance interlaminar shear strength (ILSS) of UHMWPE fiber/polymer matrix composites, but cause damage in structural integrity of the fibers and degrade the mechanical properties of the fibers. Therefore, we can say that surface treatments for fiber have improved the interfacial adhesion of UHMWPE fiber/polymer matrix composites at the expense of the fiber's strength to some extent.

It is necessary to find a way to increase the interfacial adhesion without degrading the fiber's mechanical properties. Therefore, we have focused on modification of the matrix by developing a new thermosetting resin system, named PCH, which is only composed of carbon and hydrogen elements and structurally and polarly similar to the UHMWPE fiber according to our previous study [12]. The fiber should bond to the matrix well. Therefore, the UHMWPE fiber/PCH composites have the potential to form an unified structure and possess excellent mechanical properties. Some approaches for preparing composites with chemically compatible matrices, such as UHMWPE fiber/PE composites, where various PE (e.g., LDPE and HDPE) [13–16] have been used for the matrix, self-reinforcement composites (single polymer composites) [17–20], including a novel all-cellulose composites prepared by a surface selective dissolution method [21], and Kraton thermoplastic rubber polymers, used with UHMWPE fiber in the ballistic protection products [22], have been reported in literatures. But the resins usually used in these approaches are limited to thermoplastic polymer. Comparing with these resins, PCH resin has many advantages in process and performance. First, PCH is liquid at room temperature before cured and has a moderate curing cycle. All these can enable PCH resin to be processed as epoxy resin and molding technologies used for fiber-reinforced thermosetting resin composites such as hand lay-up (contact molding) [23, 24], autoclave molding [25], resin transfer molding (RTM) [24, 26], and filament winding process [27], which do not suit for fiber-reinforced composites with thermoplastic matrix, to be applicable for UHMWPE/PCH composites. Also appropriate preparation method can be flexibly selected according to product shape, size, and property requirements. Second, the cured PCH resin possesses nonpolar crosslinking structure which can make UHMWPE fiber/PCH composites superior to those with thermoplastic matrices in heat resistance and creep resistance.

In this study, systematical experiments on adhesion property of the UHMWPE fiber/PCH composites were conducted and UHMWPE fiber-reinforced epoxy resin composites were also prepared for comparison. The adhesion property was investigated by mechanical properties test, thermal performance test, and polymer solution properties test. Based on the above measurements, the interfacial property of the UHMWPE fiber-reinforced composites with different PCH to crosslinker ratios was characterized and the effectiveness of improving the interfacial adhesion was evaluated.

Experimental

Materials

The UHMWPE fiber filament yarn (Dyneema SK75) was obtained from the DSM high performance fiber B.V. E-51 bisphenol A liquid epoxy resin (EP) was purchased from Shanghai resin factory Co. Ltd., (China). Triethanolamine and acetone, bought from Shanghai Lingfeng Chemical Reagent Co. Ltd., (China), were used as curing agent and diluent, respectively. PCH resin is a kind of thermosetting polymer prepared by anionic polymerization of hydrocarbon monomer in the laboratory, which is liquid at room temperature before cured and yields no by-product during the curing process. Styrene, reagent grade, from Shanghai Lingfeng chemical Ltd., was used as crosslinker of PCH resin without further purification.

Preparation of resin solution and cured PCH resin

A certain proportion of styrene and peroxide initiator was added to the PCH resin, stirred uniformly and then low viscosity resin solution was prepared. According to the different ratios of the PCH resin to the styrene (70/30, 65/35, 60/40, 55/45, and 50/50), five kinds of resin solution named PCH I, PCH II, PCH III, PCH IV, and PCH V were prepared correspondingly. Subsequently, the prepared PCH resin solution was put into a well-selected metal mold and cured at 120 °C for 3 h. The cured resin was obtained after cooling the mold for later measurements.

A certain proportion of acetone and triethanolamine was added into EP resin, stirred uniformly and then low viscosity resin solution was prepared.

Preparation of unidirectional UHMWPE fiber-reinforced PCH composite and micro-composite

A winding machine was employed to manufacture the unidirectional prepregs. The resin solution was prepared well and then placed into a resin tank fixed on the winding machine. The UHMWPE fiber filament yarn was dipped in the uniformly stirred resin solution for some time, and unidirectional prepregs were prepared by the winding method. Then the prepregs were placed in the 100 °C oven to be dried for 1 h to obtain the dried prepregs. The dried prepregs were tailored according to the mold dimension, laid, and put inside stainless steel template, then were hot pressed under the processing condition (120 °C/3 h, 2 MPa) for the UHMWPE fiber/PCH prepregs, and the condition (110 °C/2 h, 2 MPa) for the UHMWPE fiber/EP prepregs. Finally, the sample was incised according to the related testing standard.

Single-fiber micro-composite was fabricated by dropping liquid method. The UHMWPE single-fiber was cut into segments in length of 5 cm which were pasted on the coordinate paper with an opening rectangular hole in the middle. A little resin drop was dripped on the fiber by a fine iron wire and cured according to temperature setting for preparation of unidirectional composite. The cured drop was a little sphere which was passed through by the fiber.

Both the unidirectional UHMWPE fiber/PCH composites containing different PCH to crosslinker ratios (70/30, 65/35, 60/40, 55/45, and 50/50) and the corresponding micro-composites are named for CP I, CP II, CP III, CP IV, and CP V, respectively.

Measurements

The ILSS measurement was carried out using an universal testing machine (CMT5105, from Shenzhen, China). The specimens for the ILSS measurement were cut using a mechanical cutting machine. The size of specimens for the ILSS measurement was 25 × 6.0 × 3.0(±0.1)mm. The ILSS was measured by three-point short beam method according to the GB3357-82 test method. The span-to-depth ratio is 5:1 and the crosshead speed for the ILSS measurement was controlled at 2 mm/min. At least ten samples were measured and the results were averaged.

Single-fiber pull-out test was conducted using a Single-Yarn Strength Tester (YG020B, from Jiangsu, China). The radius of fiber was measured with an optical microscope, and the diameter of resin drop was measured by a vernier caliper. The prepared micro-composite was clamped on the electronic single yarn strength machine and the loading rate was adjusted to 0.1 mm/min. The pull-out strength of the UHMWPE single-fiber can be calculated according to Eq. 1 [28]:

$$\tau_{\text{pull-out}} = p/(\pi d l), \tag{1}$$

where, P is the maximum pull-out load, l is the fiber diameter, and d is the resin drop diameter.

The coefficient of thermal expansion (CTE) for cured PCH resin was measured using TMA (Q400TMA, from TA Instruments, US). The CTEs for cured PCH resins containing different PCH to styrene ratios were obtained according to the GB1036-89 test method. At least three samples were measured and the results were averaged.

The JSM-6460 scanning electron microscope made in Japan was used to study the morphology of fracture surface of the laminate samples and the change of surface state of the fiber before and after pulled out. The sample surface was gold plated.

Equilibrium swelling method was applied to determine the solubility parameter (δ) of cured PCH resins. The swelling agent was prepared using heptane ($\delta = 15.14$

(J/cm^3)$^{1/2}$) and styrene ($\delta = 19.03$ (J/cm^3)$^{1/2}$), and different ratios of heptane and styrene were set to obtain different δ values according to solubility parameter formula for mixed solvent (Eq. 2) [29]:

$$\delta_{\mathrm{mix}} = \varphi_{\mathrm{h}}\delta_{\mathrm{h}} + \varphi_{\mathrm{s}}\delta_{\mathrm{s}}, \tag{2}$$

where, δ_{mix} is solubility parameter for the mixture of heptane and styrene; φ_{h} and φ_{s} are the volume fractions of heptane and styrene, respectively; and δ_{h} and δ_{s} are the solubility parameters of heptane and styrene, respectively.

Contact angle studies were performed using a contact angle meter (JC 2000, from Shanghai, China). A series of images which showed the real contact condition were recorded at different time intervals using a video camera. Each image was analyzed to calculate the contact angle using an image analysis software.

Results and discussions

Effectiveness of the PCH resin for improving interfacial adhesion

Figure 1 shows a typical swelling curve for cured PCH III, and illustrates the highest swelling ratio(Q_{max}) with the corresponding solubility parameter, δ_{s}(17.04 (J/cm^3)$^{1/2}$), which can be considered as the δ value for cured PCH III according to law of similar mutual solubility. For comparison, the δ value for UHMWPE and the δ values for cured PCH I, II, III, IV, and V are listed in Table 1. The δ values for cured PCH resins present a slightly increasing trend with the increase of styrene in the PCH resin, which can be attributed to that the styrene possesses a higher polarity than that of PCH resin.

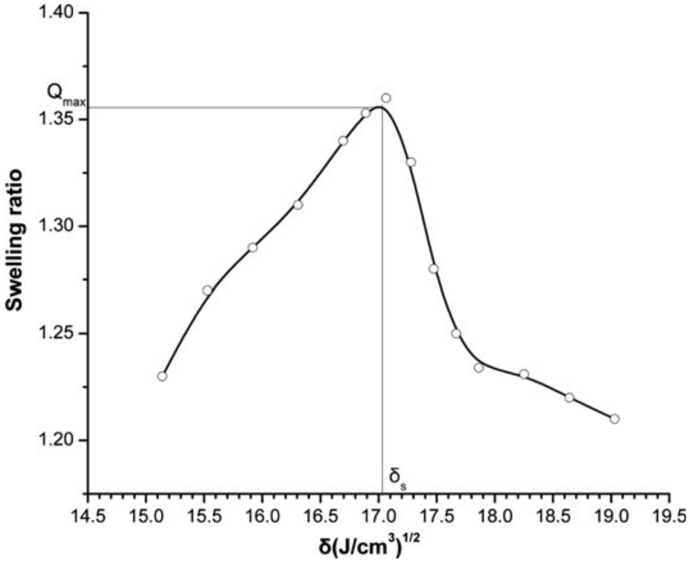

Fig. 1 Swelling curve of cured PCH III resin

Table 1 Solubility parameters and CTEs of UHMWPE fiber and different cured PCH resins

Property	UHMWPE fiber	Cured PCH resin				
		I	II	III	IV	V
Solubility parameter $(J/cm^3)^{1/2}$	16.4	16.70	16.89	17.04	17.27	17.43
CTE below T_g $(10^{-6}/K)$	−12.0	90.44	85.23	77.11	69.54	63.67

Table 1 shows a similarity of δ values of UHMWPE fiber and cured PCH resins. This similarity demonstrates that UHMWPE fiber and cured PCH resins have similar structure and polarity and consequently result in a strong interaction between UHMWPE fiber and PCH matrix, which is identified by ILSS and single-fiber pull-out strength of UHMWPE fiber/PCH composites shown in Fig. 2. Figure 2 presents excellent interfacial property for UHMWPE fiber-reinforced PCH composites, and ILSS and single-fiber pull-out strength can reach 42.6 and 21.8 MPa, respectively.

A comparison of interfacial bond strength between UHMWPE fiber/PCH composite and UHMWPE fiber/EP composites is given in Table 2 from which it can be seen that UHMWPE fiber/PCH composite has higher interfacial bond strength (ILSS and single-fiber pull-out strength) than that of UHMWPE fiber/EP composites. EP resin, traditionally used as the matrix of UHMWPE fiber-reinforced composites, has rich polar structure which differs from nonpolar PCH resin. Therefore, EP resin is unable to interact well with nonpolar UHMWPE fiber according to law of similar mutual solubility, unlike PCH resin, which has similar structure and polarity to UHMWPE fiber and can well combine with UHMWPE fiber by van der Waals force.

Figure 3 presents the microstructural changes in the single-fiber surface before (Fig. 3a) and after (Fig. 3b) pulled out. Comparing Fig. 3a with b, it is apparent that there is clearly thin resin layer adhering to the surface of fiber, which indicates a

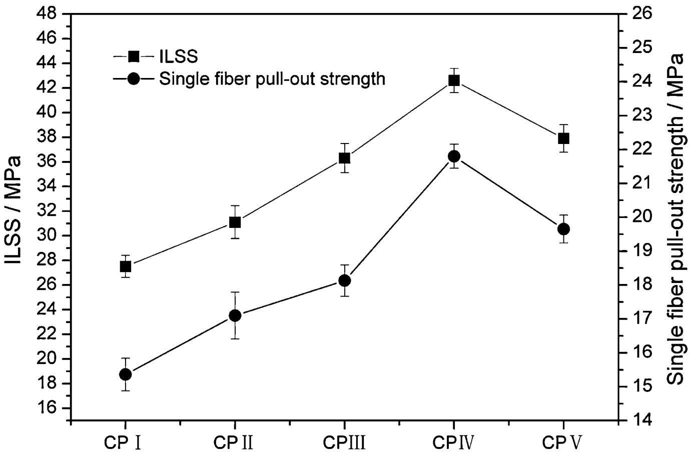

Fig. 2 The interfacial bond strength of UHMWPE fiber/PCH composites

Table 2 Comparison of interfacial bond strength for UHMWPE fiber-reinforced composites with different matrices

Sample type	ILSS (MPa)	Single-fiber pull-out strength (MPa)
UHMWPE fiber/PCH	42.6	21.8
UHMWPE fiber/EP	7.80	5.90

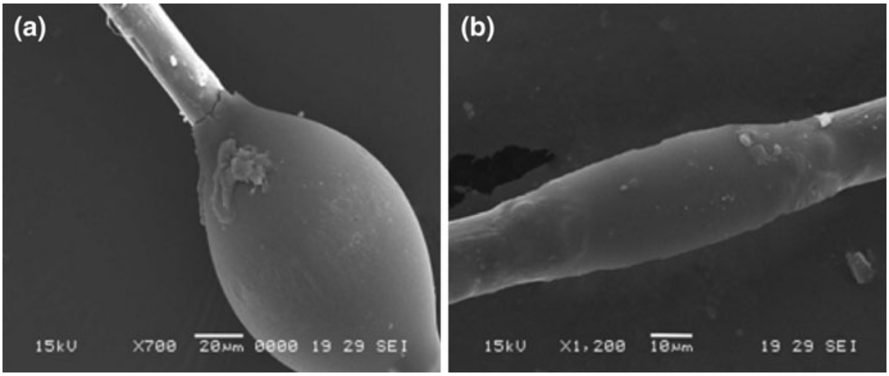

Fig. 3 SEM image of single fiber before (**a**) and after (**b**) pulled out from PCH matrix

good interfacial adhesion. Figure 4 shows a SEM image of shearing section from which it can be observed that there is detectable resin left on the surface of the UHMWPE fiber. It also proves that a good interfacial bond occurs between the UHMWPE fiber and the PCH matrix.

As shown by Mercx [10] and Peijs et al. [30], due to the highly anisotropic property of HP-PE fiber, the interlaminar shear failure mode of UHMWPE fiber-reinforced composites based on surface-treated fibers show a transition from interface debonding to fiber splitting and consequently poor ILSS. However, the PCH resin used in this study possesses similar structure and polarity with UHMWPE fiber. Without surface treatment, the nonpolar PCH solution can easily wet on the entire surface of the fiber due to their similar polarity and excellent adhesion, as shown in our previous study [12]. The PCH resin differs from the polar resins commonly used in UHMWPE fiber composites, e.g., epoxy. The difference between the polarity of epoxy and the fiber results in poor wettability of epoxy resin on surface of PE fiber and consequently clusters of nonpolar fibers in polar resin. Even if the surface-treated PE fiber was used, good wettability only occurs in the treated areas of the fibers, rather than the entire surface of the fibers. Excellent wettability of PCH resin on UHMWPE fiber can make the fiber disperse well in the resin, most probably in the form of single fiber, suggesting that the probability of fiber splitting can be greatly reduced and consequently the interlaminar shear failure is mainly dominated by interface debonding and matrix crack. The SEM of interlaminar section (Fig. 4) shows that no apparent fiber splitting can be observed and there is detectable resin left on the surface of the UHMWPE fiber, indicating

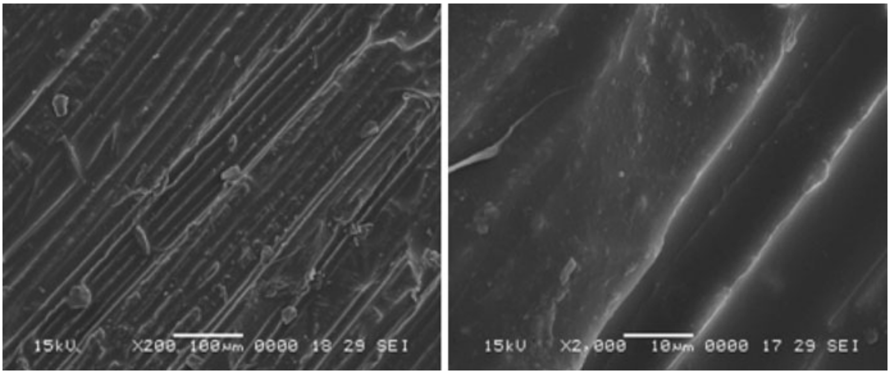

Fig. 4 SEM image of interlaminar section

that debonding and matrix crack occur in the ILSS tests. The high ILSS values shown in Fig. 2 can be most probably contributed to the excellent wettability and adhesion between UHMWPE fiber and PCH resin.

Based on the above experimental results, it is confirmed that the similar structure and polarity for UHMWPE fiber and cured PCH resin can lead to a good interfacial adhesion in UHMWPE fiber/PCH composites, but great structural and polar difference yields no strong interaction between fiber and matrix by comparison(Table 2). In theory, the closer for δ values of UHMWPE and cured PCH resin, the higher for ILSS and single-fiber pull-out strength of UHMWPE fiber/PCH composites, but experimentally an inconsistent trend is exhibited in our study (Fig. 2). With the increase of styrene added into the PCH resin, the interfacial bond strength increases, peaks as the ratio of PCH to styrene is 55/45, and then decreases. However, it is worth noting that the differences between δ values for cured PCH and UHMWPE vary from PCH I to PCH V insignificantly (Table 1), so there may be some other factors related to interfacial adhesion in the case of slight difference of δ values between UHMWPE fiber and cured PCH resin. The studies about the probable causes responsible for the above inconceivable phenomenon will be made below.

Contact angle

Wettability of resin solution on surface of the UHMWPE fiber is an important aspect for obtaining good interfacial adhesion at the stage of processing UHMWPE fiber-reinforced composite. Wettability is used to describe the extent to which resin solution spreads on fiber surface in the study of composites. It is evident that sufficient wetting can generate good interfacial adhesion and enhance the binding strength. The contact angle measurement is the commonly used method to characterize the wettability of a resin on surface of the fiber.

Figure 5 shows the actual condition of PCH resins containing different PCH to styrene ratios on surface of the UHMWPE fiber. The PCH resin possesses low contact angle on surface of the UHMWPE fiber, which indicates a good wettability

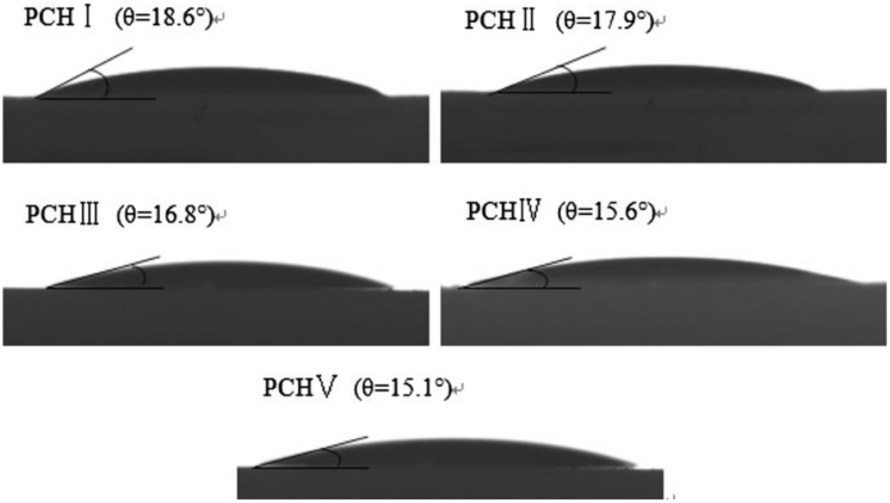

Fig. 5 Contact angles between different PCH resins and UHMWPE fiber

occurs between the PCH resin and the UHMWPE fiber. A low contact angle means a sufficient wettability, and consequently leads to a good interfacial adhesion in composites.

The curves showing contact angle as a function of time were plotted in Fig. 6. The PCH V resin (containing the most styrene) shows the lowest contact angle as compared with other test samples from Figs. 5 and 6. From the above results of contact angle, a general trend that the contact angle decreases with the increase of styrene in PCH resin and the PCH resin can wet more rapidly on surface of the fiber as the styrene increases in the PCH resin can be observed. In the curve of contact angle versus time, the initial slope (at $t = 0$) can be defined as the drop spreading rate v [31]:

$$v = \left.\frac{\mathrm{d}\theta}{\mathrm{d}t}\right|_{t=0} = \left.\frac{df(t)}{\mathrm{d}t}\right|_{t=0}, \tag{3}$$

θ is the function of time. According to Eq. 3, the relative spreading rates for different PCH resin were calculated and shown in Fig. 7. Figure 7 also gives the viscosity for different PCH resins.

From Fig. 7, it is evident that the spreading rate increases with the decrease of the viscosity of PCH resin. The PCH resin with low viscosity can spread rapidly and possesses a low contact angle on surface of the UHMWPE fiber. Therefore, it is easily speculated that the viscosity can significantly influence the wetting behavior of the PCH resin on surface of the UHMWPE fiber. The crosslinker, styrene, can perform as a diluent to lower the viscosity of the PCH resin and speed up the wetting process. With the increase of styrene in the PCH resin, the viscosity of the PCH resin becomes lower. Thereby, PCH resin with more styrene can wet more sufficiently and more rapidly on surface of the fiber, which can significantly affect the interfacial adhesion of UHMWPE fiber/PCH composites. With the increase of

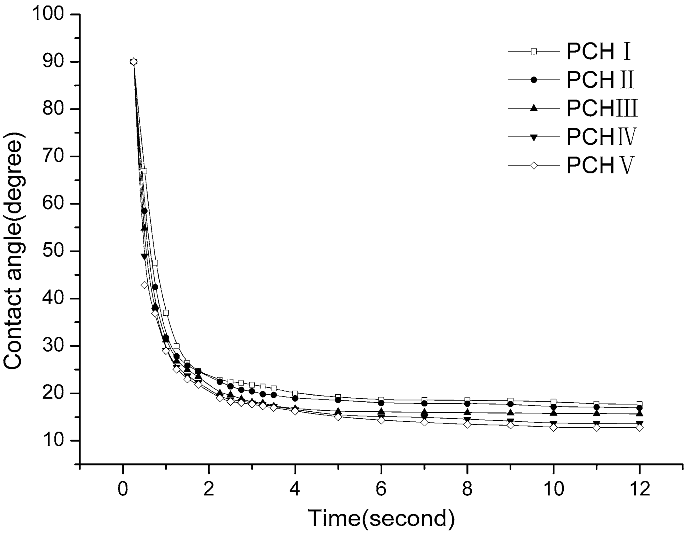

Fig. 6 Contact angle as a function of time for PCH resins containing different PCH to styrene ratios

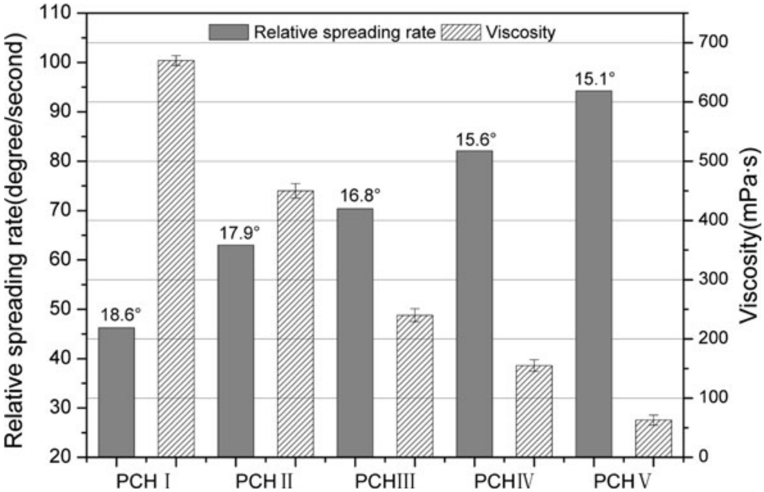

Fig. 7 Relative spreading rates and viscosity for different PCH resins

styrene added into the PCH, the interfacial adhesion of UHMWPE fiber/PCH composites can be influenced by the difference between δ values of UHMWPE and cured PCH resins and the wettability of PCH on surface of UHMWPE fiber in two different ways: (1) negative way for the former and (2) positive way for the latter. This implies that there is an optimal ratio of PCH/styrene so that an optimal interface bond can occur between fiber and matrix. This conclusion is well consistent with ILSS and single-fiber pull-out strength (Fig. 2), which show a peak value corresponding to CP IV with the PCH/styrene ratio of 55/45. This study result may explain the inconceivable phenomena depicted in Sect. 3.1 to a certain extent.

Study on thermal performance

Due to nonexistence of chemical binding within the interface of UHMWPE fiber/PCH composites, some physical properties (especially thermal performance) of fiber and PCH resins are critical in determining the interfacial adhesion of UHMWPE fiber/PCH composites. CTE is an important parameter to describe thermal expansion of materials. The fiber and PCH resin possess different CTEs (Table 1, notes that UHMWPE fiber possesses a small negative CTE, -12×10^{-6}/K). Table 1 reveals an obvious decrease in the CTE for cured PCH resins with increasing styrene content. More addition of the styrene into the PCH resin can increase the crosslinking density of cured PCH resin. A higher crosslinking density can efficiently restrain thermal expansion for cured resin by hindering molecular motion. Therefore, the CTE for cured PCH resins decreases with the increase of styrene added into the PCH resin.

The difference between CTE of fiber and that of resin matrix can significantly influence interfacial properties of fiber-reinforced composites, which can be attributed to the thermal stress due to difference between CTEs of fiber and matrix. The thermal stress can be calculated as [32]:

$$\tau_{\text{thermal}} = E_{\text{f}} \beta r_{\text{f}} (\alpha_{\text{f}} - \alpha_{\text{m}}) \left(\frac{\Delta T}{2} \right), \tag{4}$$

where, E_{f} and r_{f} are tensile modulus and the radius of the fiber, respectively; α_{f} and α_{m} are the CTEs of the fiber and the matrix, respectively; ΔT is the difference between stress-free temperature and test temperature (room temperature); and β is the shear-lag parameter.

Equation 4 demonstrates the effect of CTEs of fiber and resin matrix on the thermal stress within interface of fiber-reinforced composites. From Eq. 4, it is clear that a small difference between CTE of fiber and that of resin matrix corresponds to a small thermal stress, which implies good interfacial adhesion is achieved. The calculated results for thermal stress of composites with different PCH matrices are shown in Fig. 8 from which a decreasing trend in thermal stress can be clearly observed. The decreasing trend in thermal stress is in reasonably good agreement with the difference between CTEs for PCH matrices and UHMWPW fiber, which can be seen from Table 1.

If the difference between CTE of fiber and that of resin matrix in the composites increases, thermal stress is distributed along the fiber and micro-debonding may occur in some certain regions where the fiber and resin matrix experienced different level thermal deformation at the stage of cooling process after cured. Micro-debonding resulting from the thermal stress distributed along the UHMWPE fiber can cause degradation of interface in UHMWPE fiber-reinforced PCH composites to some extent. There is no direct evidence to illustrate the above inference, but both the macro-mechanics and the micro-mechanics experiment confirmed the effect of

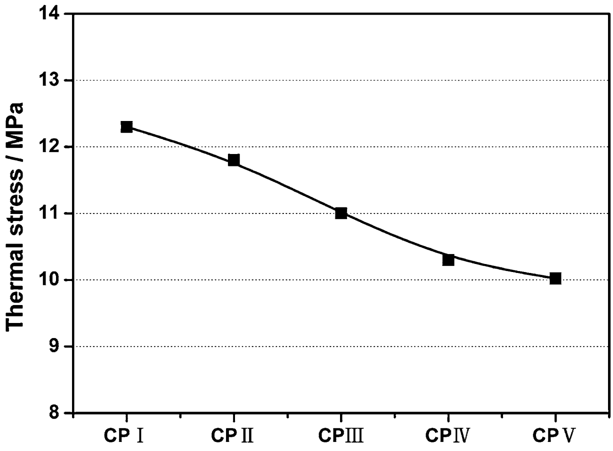

Fig. 8 Calculated results for thermal stress of composites with different PCH matrices

different thermal performance of UHMWPE fiber and cured PCH resins on the interfacial adhesion indirectly, which can be seen from the comparison between Figs. 8 and 2. As listed in Table 1, CTEs for cured PCH resins present a decreasing trend with increasing styrene added into PCH resin, which lead to a smaller difference between CTEs of fiber and matrix and consequently a decreasing trend of thermal stress within the interface of UHMWPE fiber/PCH composites (Fig. 8). This decreasing trend can cause a decrease of probability of micro-debonding, which can enhance the interfacial bond (seen from Fig. 2).

Based on the above analysis, the difference between CTE of UHMWPE fiber and that of cured PCH resin can make an impact on the interfacial adhesion of UHMWPE fiber/PCH composites whose interface does not possess chemical binding at the stage after cured (especially cooling process). So, to avoid the so-called micro-debonding, it is important to control the cooling rate after cured. This result, combined with the contact angle result, can also be regarded as a reasonable cause which is responsible for the experimental phenomenon depicted in Sect. 3.1.

Conclusion

In order to improve the interfacial adhesion of UHMWPE fiber-reinforced polymer matrix composites, a new thermosetting resin system, named PCH, which is only composed of carbon and hydrogen elements and structurally and polarly similar to UHMWPE fiber, has been developed. A strong interfacial adhesion of UHMWPE fiber/PCH composites was obtained. The test results show that ILSS and single-fiber pull-out strength can reach 42.6 and 21.8 MPa, respectively, which indicate a great potential in many engineering fields. The adhesion ability of the PCH resin to UHMWPE fiber mainly depends on its structure and polarity, but can be significantly affected by its wetting behavior and thermal performance at the stage before and after cured, respectively. Good wettability on surface of the UHMWPE fiber and small difference between CTE of cured PCH resin and that of the UHMWPE fiber conduce to obtain a good interfacial adhesion between the UHMWPE fiber and the cured PCH resins. The study results indicate that in the case of slight difference between solubility parameters of UHMWPE fiber and cured PCH resin, more styrene added into PCH resin can not only improve the wettability of resin solution on surface of UHMWPE fiber but also reduce the difference between CTEs of cured PCH resin and UHMWPE fiber, which can lead to a stronger interaction between fiber and matrix. The study reveals that an optimal interfacial adhesion can be obtained as the ratio of PCH/styrene is approximately 55/45. These study results can provide a theoretical basis for optimizing the performance of UHMWPE fiber/PCH composites and further research will be needed in future.

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