

Properties of Carbon Fiber Sized with Poly(phthalazinone ether ketone) Resin

W. B. Liu,¹ S. Zhang,¹ L. F. Hao,² W. C. Jiao,² F. Yang,² X. F. Li,¹ R. G. Wang²

¹School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China

²Center for Composite Materials and Structures, School of Astronautics, Harbin Institute of Technology, Harbin 150080, China

Correspondence to: W. B. Liu (E-mail: liuwenbo@hit.edu.cn)

ABSTRACT: We studied thermoplastic poly(phthalazinone ether ketone) (PPEK) resin as a sizing agent on carbon fiber, with emphasis on its thermal stability, surface energy, wetting performance, and interfacial shear strength (IFSS). X-ray photoelectron spectroscopy characterization was carried out to study the chemical structure of sized/unsized carbon fibers. Scanning electron microscopy and atomic force microscopy were used to characterize surface topography. TGA was used to analyze the thermal stability. Meanwhile, contact angle measurement was applied to analyze the compatibility between the carbon fibers and PPEK and the surface energy of carbon fibers. IFSS of carbon fiber/PPEK composite was examined by microbond testing. It is found that carbon fibers uniformly coated with PPEK resin had better thermal stability and compatibility with PPEK resin than the uncoated fiber. The contact angle is 57.01° for sized fibers, corresponding to a surface energy of 49.96 mJ m⁻², much smaller than that for unsized ones with contact angle value of 97.05°. The value of IFSS for sized fibers is 51.49 MPa, which is higher than the unsized fibers. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 128: 3702–3709, 2013

KEYWORDS: adsorption; molecular recognition; nanostructured polymers

Received 13 July 2012; accepted 18 September 2012; published online 8 October 2012

DOI: 10.1002/app.38605

INTRODUCTION

At present, carbon fibers have maintained their predominance as a reinforcing material for high-volume applications in aerospace, motor cars, sports goods, etc., attributing to their high-specific modulus and strength combined with their lightweight.^{1–3} However, smooth and chemically inert fiber surfaces usually result in poor matrix compatibility and weak adhesion between fibers and matrix.⁴ As a result, extensive research has been devoted to the surface treatment of carbon fibers in order to improve their interfacial adhesion, such as chemical grafting, electrochemical method, plasma treatment, and sizing method.^{5–8} Among these methods, sizing method is preferred in industry. Besides, carbon fibers are basically brittle, and so they are always sized or coated with a very thin (usually on nanometer scale) coating of a prepolymer or resin in order to prevent the fibers from damage through the process of manufacture and also to insert a polymer interlayer between carbon fiber (CF) and polymer matrix to improve the interfacial properties.^{9,10}

Sizing agents of CF, to a large extent, determine the interfacial properties of carbon fiber-reinforced plastic (CFRP) and can improve the wetting of the fiber by matrix if the main components of sizing agents are same or similar to the matrix resins.

Thus, different matrix resins usually require compatible sizing agents. Previous works in this field were mainly focused on epoxy type-sizing agents, because most matrices for CFRP are epoxy resins.^{11,12} With increasing application of thermoplastic composites, the development of thermoplastic sizing agents for CF needs immediate attention. Several researches about thermoplastic sizing agent for CF have been conducted recently.

A vinyl ester resin emulsion-type sizing agent was synthesized by phase inversion emulsification method.⁸ The emulsion sizing agent exhibits narrow and uniform distribution of particle size, which means good stability, and improves the handling characteristics of CF, while the surface of sized CF becomes smooth. Nakama et al.¹³ disclosed a high-molecular weight thermoplastic polyurethane (PU) resin as a sizing agent for treating CF, and sized CF/PU composite showed high-interlaminar shear strength. A CF-sizing agent comprised water-soluble Nylon resin and surfactant was invented by Sugiura and Maki.¹⁴ The sizing agent had good solubility in water over a wide pH range and was able to impart to carbon fibers adequate convergence for forming stable chopped carbon fibers and superior workability; thus, the sizing agent was suitable for application such as immersing said fabric in an aqueous matrix in order to impregnate the fabric with that matrix.

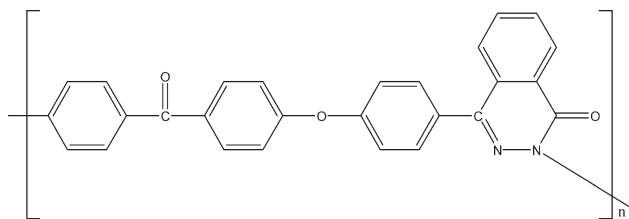


Figure 1. Chemical structure of PPEK.

To evolve demanding needs in composites with high-thermal stability and excellent mechanical properties, the application of poly(aryl ether ketone)s (PAEKs) resin such as poly(ether ether ketone) (PEEK), poly(phthalazinone ether ketone) (PPEK), and poly(phthalazone ether sulfone ketone) has been gradually increased.^{15–20} Thus, sizing agents that have same or similar chemical structure with PAEKs specialized for PAEKs resin of CF need to be explored. In this work, a thermoplastic-sizing agent for CF with PPEK resin was prepared. The sizing agent satisfies the demand of good compatibility between fiber and matrix in molding process of carbon fiber-reinforced PAEKs composites. X-ray photoelectron spectroscopy (XPS) was carried out to characterize the chemical structure of sized/unsized CF. Surface topography was determined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The effect of fibers coatings on thermal stability was investigated by thermogravimetry analysis (TGA). Surface energy of CF and wettability between CF and PPEK was characterized by dynamic contact angle measurement. Interfacial shear strength (IFSS) of CF/PPEK composite was examined by microbond method. All the results were compared between commercially available CF/PPEK composite and PPEK-sized CF/PPEK composite.

EXPERIMENTAL

Materials

PPEK resin, the chemical structure of which was shown in Figure 1, was readily synthesized by polycondensation of 4-(4-hydroxyphenyl)-2,3-phthalazin-1-one and 4,4'-difluorobenzophenone in dimethylacetamide at 190°C.²¹ The prepared PPEK had an inherent viscosity of 0.3 dL/g in 98% concentrated H₂SO₄ at 30°C and was soluble in *N*-methylpyrrolidinone (NMP) at room temperature. The glass transition temperature was 263°C, and the density was 1.33 g/cm³. Polyoxyethylene castor oil (EL) was obtained from Venture Grease Chemical Plant, China. NMP, diiodomethane, and acetone were purchased from Tianjin Chemical Regent No. 1 Plant, China. T700 SC carbon fiber, 12 K, with an average diameter of 7.15 μm, was supplied by Toray Company, Japan.

To compare the PPEK-sizing agent and the sizing agent on T700 (with the composition unknown) and get better sizing effect, the sizing agent coated on T700 CF surface was removed first. T700 CF was refluxed by boiling acetone for 48 h in a Soxhlet apparatus, then washed repeatedly with deionized water, and dried under vacuum at 110°C for 3 h (labeling as T700T, with the diameter of 7.05 μm).

Three concentrations of sizing agent, 0.1, 0.5, and 1 wt %, were used and labeled as C-1, C-2, and C-3, respectively. The PPEK resin was dissolved in NMP by constant stirring until the resin

was homogeneously dispersed under 80°C. Then the surfactant EL was added to the solution with the concentration of 0.5 wt % of the solution to improve the wettability between unsized carbon fiber and the sizing agent solution. The T700T CF was immersed in the solutions for 10 s by use of a dip tank in conjunction with automatic processing equipment for continuous running of the CF through tank. The solvent was removed, and the fiber (labeling as T700M, with the diameter of 7.15 μm, measured by SEM images) was dried under 110°C.

Characterization

Surface Topography. Carbon fibers were first coated with a Au layer of several nanometer thick and imaged with a Quanta 200 F scanning electron microscope (FEI Co., with acceleration voltage of 20 kV) to reveal the SEM images. AFM observations were carried out in noncontact mode by Solver P47 atomic force microscope made in Russia NTMDT Corp. All images were collected in air using the tapping mode with a silicon nitride probe. The scanning scope was 4 × 4 μm, and the scan rate was 1.85 Hz.

X-ray Photoelectron Spectroscopy. X-ray photoelectron spectroscopy (XPS, PHI5700) was used to evaluate the chemical structures of CF surface and to confirm the existence of PPEK coating. A Mg Kα X-ray source was used, and a binding energy range from 6 to 1350 eV was selected for the analysis. The XPS data was fitted by Gaussian–Lorentzian function. Different functional groups were assigned using reported C1s chemical shifts in various organic compounds, and the relative amounts of these groups were estimated from respective areas of assumed Gaussian–Lorentzian curves.⁹

Thermogravimetry Analysis. Thermogravimetry analysis (TGA) was used to determine the thermal stability of T700 and T700M carbon fibers. The analysis was conducted by Perkin Elmer Phris6 from room temperature to 700°C with a heating rate of 10°C min⁻¹.

Contact Angle. Contact angle measurement was applied to characterize the wettability of carbon fiber by PPEK (PPEK/NMP solution with the concentration of 15 wt %). The two liquid dynamic contact angle analyses were used to determine the dispersive (γ^d) and polar (γ^p) components of surface energy. Wilhelm plate technique was used with an insert depth of 5 mm and an insert speed of 0.008 mm/s on a DCAT 21 system (Dynamic Contact Angle Meter and Tensiometer, Germany). Water and diiodomethane were used as probe liquids, and their surface energies are listed in Table I. At least 15 specimens were tested to improve the measurement accuracy.

Interfacial Shear Strength. Interfacial shear strength (IFSS) was tested using microbond methods in Interface Strength Tester (MODEL HM410, Japan). The polymer droplet was clamped by

Table I. Surface Energies of Liquids Used for Two-Liquid Dynamic Contact Angle Test

Liquid	γ^d (mJ m ⁻²)	γ^p (mJ m ⁻²)	γ (mJ m ⁻²)
Water	22.1	50.7	72.8
Diiodomethane	44.1	6.7	50.8

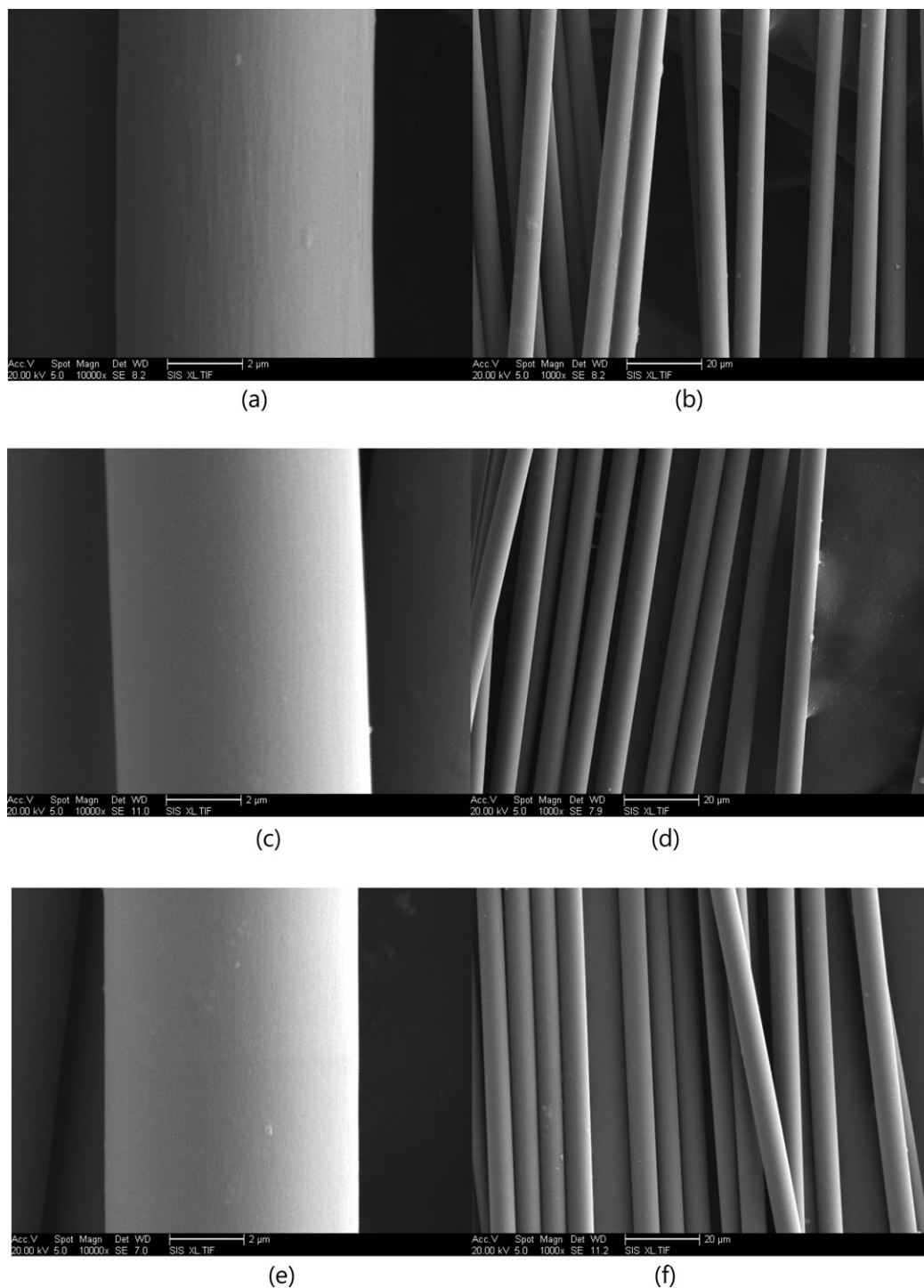


Figure 2. SEM images of carbon fibers sized with different concentrations of sizing agent (a, b) C-1, (c, d) C-2, and (e, f) C-3.

steel blades and pulled apart from fiber with a constant speed of 0.1 mm/min.

RESULTS AND DISCUSSION

Surface Topography

The SEM images of carbon fibers sized with different concentrations sizing agent are given in Figure 2. Remarkable differences in micrographs can be observed. Figure 2(a) showed that there were

a number of longitudinal streaks dispersing on the C-1-sized carbon fibers surface, which resulted from thin coating that did not cover the whole surface. The longitudinal streaks in the C-2 and C-3-sized carbon fibers almost disappeared as shown in Figure 2(c, e). A few granular substances appeared on C-3-sized carbon fibers surface due to high concentration to coat uniformly.

It can be seen from Figure 2(b, d) that there was no bundle between sized carbon fibers, and the fiber bundle had uniform

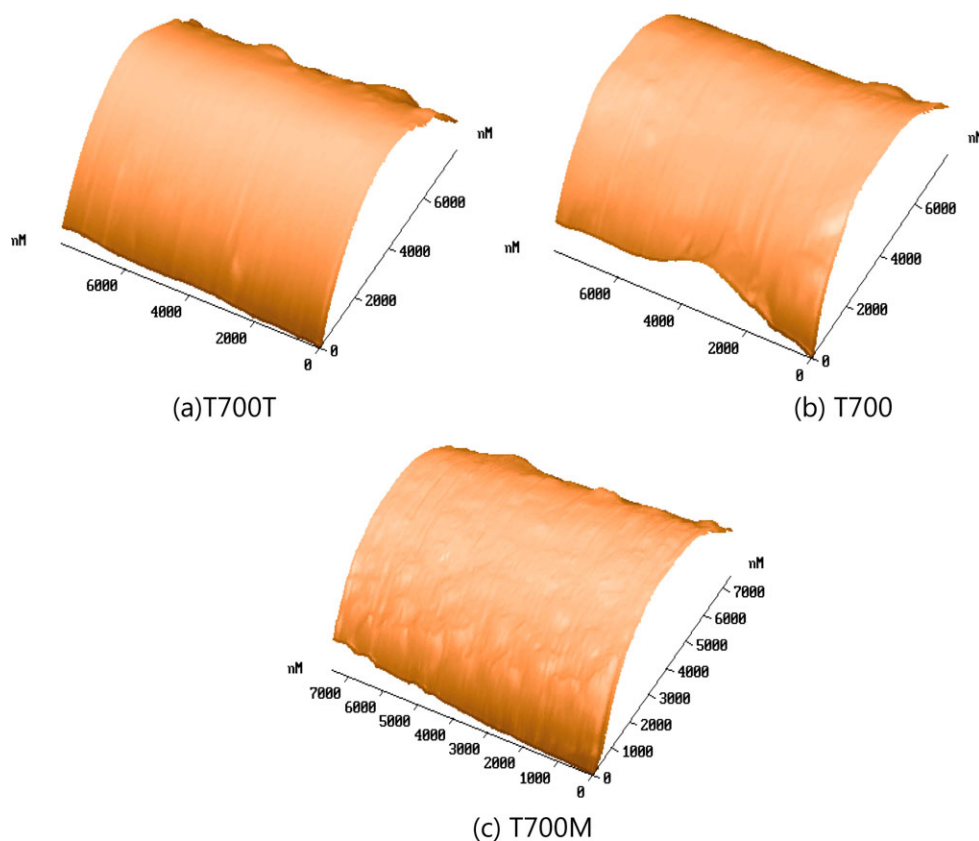


Figure 3. AFM images of carbon fibers: (a) T700T, (b) T700, and (c) T700M. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

dispersibility. However, some C-3-sized carbon fibers adhered together [as shown in Figure 2(f)], which did not satisfactory processability and other workability for further application. As a result, we chose C-2 with the concentration of 0.5 wt % as the sizing agent for the following investigation.

Figure 3 shows the AFM images of T700T, T700, and T700M carbon fibers. There was more fluctuation on T700M (as shown in Figure 3) than T700T and T700 carbon fibers. The results demonstrated that sizing improved the surface roughness of carbon fibers.

Chemical Structure

The surface composition of T700T, T700, and T700M was determined by XPS, and the results are given in Table II. Values of the atomic concentration (A.C.) are listed for each element. The surface of T700T CF was composed of carbon and oxygen elements, while the surfaces of T700 and T700M carbon fibers contained these same elements in addition to trace amounts of silicon and nitrogen, respectively. Silicon (maybe in the form of some silane-coupling reagent) and nitrogen on the surface of carbon fibers were introduced by sizing agents.

Figure 4 shows typical XPS C1s curve fit spectra for T700T, T700, and T700M carbon fibers. Values of the binding energy (B.E.) and the percent contribution (P.C.) of each curve photopeak to the total C1s photopeak²² are summarized in Table III.

The presence of C—N and C=N groups indicated that main component of sizing agent on T700M CF was PPEK.

Only C—C, C—O, and C=O peaks were detected on the T700T and T700 carbon fibers surface. The difference on these two fibers was that the ratio of C—O on T700 CF surface was higher than that of T700T CF. Sizing agent on T700 CF increases C—O group; as a result, the surface energy would be higher than T700T CF that will be discussed later. The C1s peak of T700M was decomposed into five components, which were ascribed to C—C, benzene ring, C—N, C=N, and C=O bonds, respectively. It must be noted that such a curve is quite arbitrary in view of the poor resolution of our data, and the ratio of each groups was not precise. However, it is enough to confirm the chemical structure of the sizing agent on T700M CF as we knew the main component was PPEK.

Table II. XPS Results of T700T, T700, and T700M Carbon Fibers

	C	Si	N	O
Carbon fibers	A.C. (%)	A.C. (%)	A.C. (%)	A.C. (%)
T700T	90.78	-	-	9.22
T700	76.86	1.06	-	22.08
T700M	81.97	-	1.19	16.83

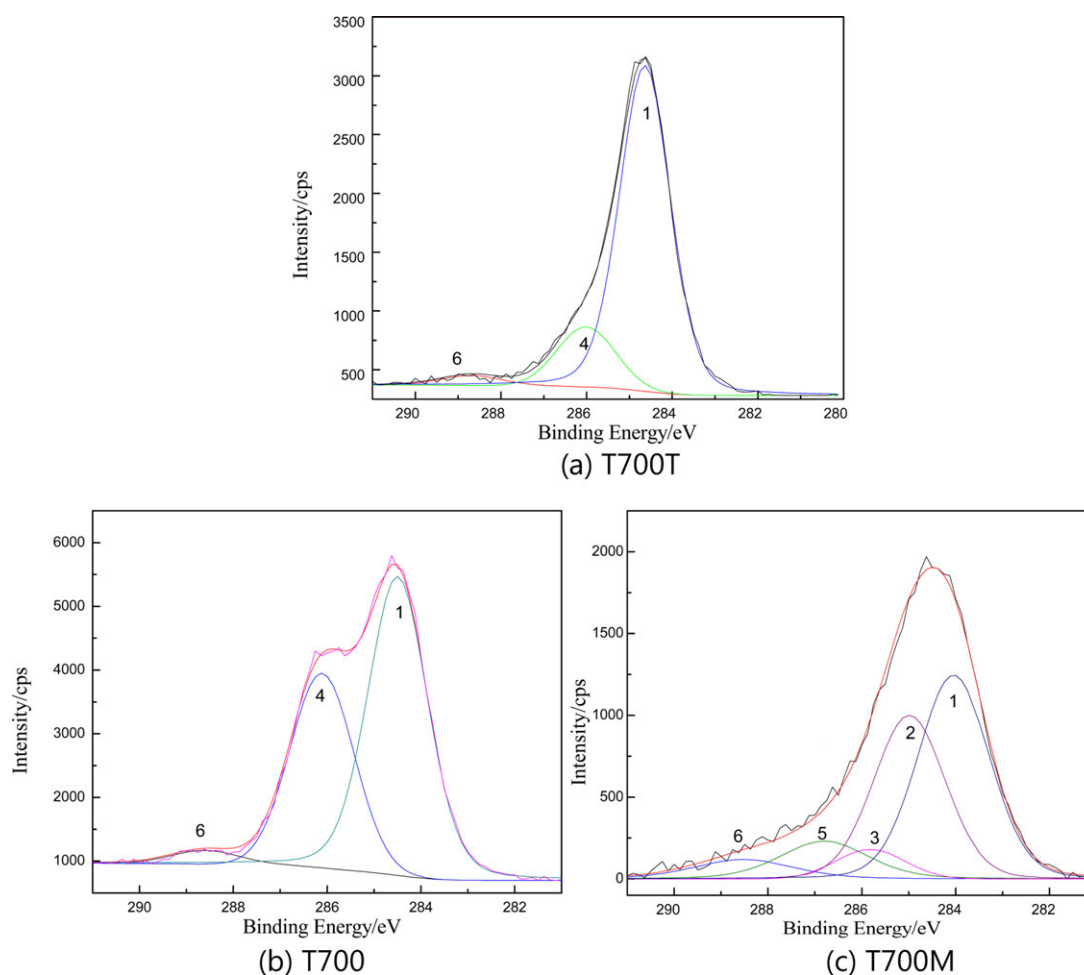


Figure 4. Curve fit of C1s photoelectron peaks: (a) T700T, (b) T700, and (c) T700M carbon fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Combined with Figure 1, it can be demonstrated that C—C, C—N, and C=N are in the form of phthalazine ring. Benzene ring, phthalazine, and C=O are much more stable than C—O; therefore, the thermal stability of T700M CF would be much higher than T700 CF. Besides, more C=O groups on T700M CF introduced by PPEK sizing agent may also improve its surface energy when compared with T700T and T700 carbon fibers.

Thermal Stability

Thermal stability of carbon fiber for many applications is necessary in determining their end use.²³ A TGA curve of T700 and

T700M carbon fibers is given in Figure 5. A sudden drop in the mass of the sample indicated the thermal degradation of the material. The materials started to lose mass at 100°C, where substantial loss in their weights was observed. According to the data, the introduction of PPEK in the sizing agent to the carbon fiber, in general, increased the mass loss temperature. At 250°C, the mass loss of T700M CF was only 0.37%, while the value of T700 CF was 0.41%; at 350°C mass loss of T700M, CF was 0.82%, while for T700 was 1.18%. Higher thermal stability of T700M CF compared to T700 CF resulted from the rigid asymmetric phenyl phthalazinone moiety into the polymer backbone,

Table III. XPS C1s Curve Fit Results of T700T, T700, and T700M Carbon Fibers

Carbon fibers	—C—C— 1		Benzene ring 2		—C—N 3		—C—O 4		—C=N 5		—C=O 6	
	B. E. (eV)	P.C. (%)	B. E. (eV)	P.C. (%)	B. E. (eV)	P.C. (%)	B. E. (eV)	P.C. (%)	B. E. (eV)	P.C. (%)	B. E. (eV)	P.C. (%)
T700T	284.14	81.27	-	-	-	-	286.10	15.71	-	-	288.69	3.02
T700	284.11	59.31	-	-	-	-	286.11	37.70	-	-	288.59	2.99
T700M	284.04	43.28	284.99	34.28	285.82	5.69	-	-	286.80	10.56	288.52	6.18

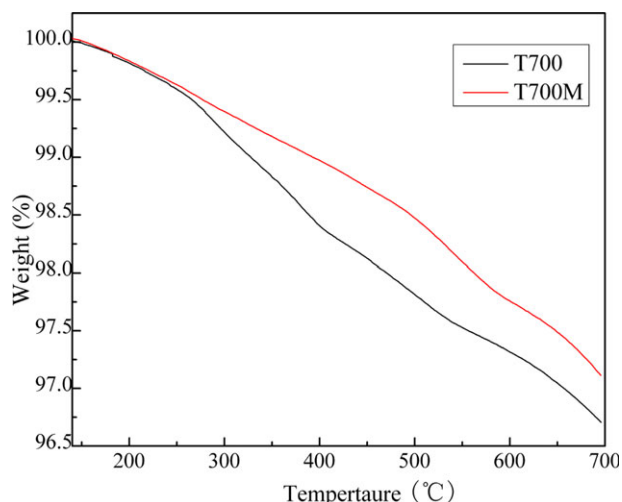


Figure 5. TGA results of T700 and T700M carbon fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

which yields the engineering polymers with high-glass transition temperatures and outstanding thermo-oxidative stability.²⁴ Higher thermal stability made T700M CF possible to be applied in high-temperature environment.

Surface Energy

Schultz et al.²⁵ proposed that the dispersive component (γ_s^d) and the polar component (γ_s^p) of the total surface energy (γ_s) of a fiber can be determined by using a two-liquid (subscripts L1 and L2) tensiometric method. With known γ_L^p and γ_L^d for equilibrium contact angle (abbreviated θ here) measurements, one can easily determine γ_s^d and γ_s^p by solving the following equations:²⁶

$$\gamma_{L1}(1 + \cos \theta_1) = 2(\gamma_s^d \gamma_{L1}^d)^{1/2} + 2(\gamma_s^p \gamma_{L1}^p)^{1/2} \quad (1)$$

$$\gamma_{L2}(1 + \cos \theta_2) = 2(\gamma_s^d \gamma_{L2}^d)^{1/2} + 2(\gamma_s^p \gamma_{L2}^p)^{1/2} \quad (2)$$

$$\gamma_s = \gamma_s^d + \gamma_s^p \quad (3)$$

In Table IV, the results of the surface free energy and its dispersive component and polar component of the carbon fibers are summarized. As a result, it can be seen that T700T CF showed the lowest surface energy due to the poor functional groups (as shown in Table III). The sizing agents of both T700 and T700M

Table IV. Surface Energies of Carbon Fibers

Carbon fibers	Contact angle (°)		γ^d (mJ m ⁻²)	γ^p (mJ m ⁻²)	γ (mJ m ⁻²)
	Water	Diiodomethane			
T700T	71.78	74.68	8.93	22.44	31.37
T700	64.23	53.91	21.11	18.50	39.61
T700M	49.40	44.77	19.14	30.82	49.96

Table V. Contact Angle Results of Carbon Fibers/PPEK Solution

Carbon fibers	Contact angle (°)
T700T	97.05
T700	73.11
T700M	57.01

carbon fibers improved the surface energy. The increase in the surface-free energy of T700M was mainly influenced by its polar component. The polar component that is correlated with the amount of functional groups (such as C=O) was 30.82 mJ m⁻², much higher than the values of T700 and T700T carbon fibers (18.50 and 22.44 mJ m⁻², respectively). For T700 CF, the content of C=O group was lower than T700T CF (3.02% for T700 and 2.99% for T700T) as shown in Table III, and so its polar component was lower than T700T CF. More active group resulted in higher surface energy with the value of 49.96 mJ m⁻², improving 59.26% compared to T700T CF (with the value of 31.37 mJ m⁻²). These results indicated that the sizing agent, especially sizing agent with PPEK, can increase the surface energy of CF.

Wettability

It has been reported that the presence of sizing agent may improve the wetting performance of the fiber by the matrix resin.²⁷ The wetting of carbon fiber by PPEK/NMP solution (with the surface tension of 42.06 mNm⁻¹, which was determined by DCAT21) was measured, and the results are given in Table V. The wetting ability of PPEK solution on the T700T CF was poor, and the contact angle between PPEK solution and T700T CF was 97.05°. The wettability of T700 and T700M carbon fibers is better than that of T700T CF. Comparison of contact angle data based on the different carbon fibers is shown in Figure 6. T700M CF has the best wettability with PPEK/NMP solution with the contact angle of 57.01°, and the value of T700 CF is 73.11°.

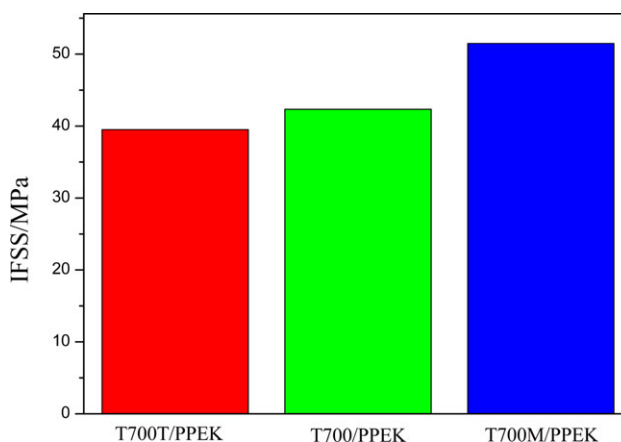


Figure 6. IFSS results of different carbon fiber-reinforced PPEK composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

According to “similar dissolve mutually theory,” good compatibility leads to good wettability.²⁸ On the other hand, higher surface energy on CF surface makes matrix wet fiber easily. The content of C=O bond, which is the same structure with PPEK, is higher on T700 CF surface than T700T CF, resulting in better wettability on T700 CF. T700M CF shows best wettability with PPEK resin not only due to the same chemical structure on its surface but also its higher surface energy. Sizing agent with thermoplastic PPEK resin improved the wettability of CF by PPEK obviously.

Interfacial Shear Strength

It has been studied that better wetting can enhance the interfacial shear strength (IFSS) by improving the work of adhesion; high-surface energy indicates that fiber contains more polar groups on the surface, and interfacial adhesion can be improved by the strong interaction between resin and the polar groups.^{15,29,30}

The effect of sizing agent on the IFSS of CF/PPEK composites has been studied, and the results are listed in Figure 6. The IFSS of T700M carbon fibers composite was 51.49 MPa, which was higher than those of 700T and T700 carbon fibers composites with the value of 39.51 and 42.33 MPa, respectively. After sizing with PPEK, IFSS of 700M CF/PPEK composite was improved 30.32% compared to 700T CF/PPEK composite. Better wetting between T700M CF and PPEK/NMP solution, more rough surface and higher surface free energy of T700M CF resulted in higher IFSS. For carbon fiber-reinforced thermoplastic composites, there was no or little chemical bond at the interface. However, interfacial adhesion can also be improved by changing surface energy to increase the wettability thus providing intimate molecular contact and promoting secondary forces.⁹ Increasing surface roughness of carbon fiber improved the mechanical interlocking between CF and PPEK resin. As a result, sizing with PPEK resin on carbon fiber improved the IFSS of CF/PPEK composite. Carbon fibers sizing with PPEK can also be used to reinforce other PAEKs resin, and it needs to be further investigated.

CONCLUSIONS

The results of this study revealed that sizing agent with thermoplastic PPEK resin improved carbon fibers surface and interfacial properties of carbon fiber composites. XPS results confirmed that the main component of sizing agent on T700M CF was PPEK resin. T700M CF performed better thermal stability than that of T700 CF. Because of more functional groups on surface, T700M CF showed higher polar component and surface energy than T700 and T700T carbon fibers. Surface energy of T700M CF was 49.96 mJ m⁻², improving 59.26% compared to T700T CF. The wetting performance of T700M was best with the contact angle of 57.01°, and the values were 73.11° and 97.05° for T700 and T700T carbon fibers, respectively. IFSS of T700M CF/PPEK composite was 51.49 MPa and improved 30.32% compared to 700T CF/PPEK composite. Carbon fiber sizing with PPEK can distinctly improve their thermal stability, surface energy, wetting performance, and IFSS.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support from the National Basic Research Program of China (973 Program, No. 2011CB605605) and the National Natural Science Foundation of China (No. 90916008).

REFERENCES

- Li, D. F.; Wang, H. J.; Wang, X. K. *J. Mater. Sci.* **2007**, *42*, 4642.
- Meyer, D. R. In Proceedings of the 50th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics, and Materials Conference, 17th, Palm Springs, California, America, May 4–7, 2009; Deft University of Technology Ed.; the American Institute of Aeronautics and Astronautics, Inc.: Delft, **2009**.
- Zhang, D. H.; Ye, L.; Deng, S. Q.; Zhang, J. N.; Tang, Y. H.; Chen, Y. F. *Compos. Sci. Technol.* **2012**, *72*, 412.
- Montes-Moran, M. A.; Young, R. J. *Carbon* **2002**, *40*, 845.
- Cao, H. L.; Huang, Y. D.; Zhang, Z. Q.; Sun, J. T. *Compos. Sci. Technol.* **2005**, *65*, 1655.
- Zhao, F.; Huang, Y.; Liu, L.; Bai, Y.; Xu, L. *Carbon* **2011**, *49*, 2624.
- Zhao, F.; Huang, Y. D. *Mater. Lett.* **2011**, *65*, 3351.
- Zhang, R. L.; Huang, Y. D.; Li, N.; Liu, L.; Su, D. *J. Appl. Polym. Sci.* **2012**, *125*, 425.
- Dilsiz, N.; Wightman, J. P. *Carbon* **1999**, *37*, 1105.
- Xu, Z. W.; Huang, Y. D.; Zhang, C. H.; Liu, L.; Zhang, Y. H.; Wang, L. *Compos. Sci. Technol.* **2007**, *67*, 3261.
- Sugiura, N.; Taguchi, M.; Saito, T.; Okuda, T. Eur. Pat. 1,413,670,B1 (**2004**).
- Sumida, A.; Toyokazu, M.; Motoi, I.; Itsuki, T. K.; Hiroshi, S. K.; Manabu, S. K. U.S. Pat. 5,298,576 (**1994**).
- Nakama, K.; Yamashita, H.; Kuroda, H.; Kashimoto, M. U.S. Pat. 4,474,906 (**1984**).
- Sugiura, N.; Maki, N. U.S. Pat. 7,135,516,B2 (**2006**).
- Chen, P.; Lu, C.; Yu, Q.; Gao, Y.; Li, J.; Li, X. *J. Appl. Polym. Sci.* **2006**, *102*, 2544.
- Fracasso, R.; Rink, M.; Pavan, A.; Frassine, R. *Compos. Sci. Technol.* **2001**, *61*, 57.
- Jian, X. G.; Chen, P.; Liao, G. X.; Zhu, X. L.; Zhang, S. H.; Wang, J. Y. *Acta Polym. Sin.* **2003**, *4*, 469.
- Liu, W. B.; Wang, R. G.; Zhang, H. T.; Jia, J.; Jiao, W. C.; Xie, H. Q. *Compos. Interf.* **2004**, *11*, 441.
- Zhang, Y. H.; Sun, X. B.; Xu, R.; Niu, Y. M.; Wang, G. B.; Jiang, Z. H. *Mater. Chem. Phys.* **2006**, *99*, 465.
- Gao, Y.; Robertson, G. P.; Guiver, M. D.; Jian, X. G. *J. Polym. Sci., Part A: Polym. Chem.* **2003**, *41*, 497.
- Berard, N.; Paventi, M.; Chan, K. P.; Hay, A. S. *Macromol. Symp.* **1994**, *77*, 379.
- Mercier, D.; Rouchaud, J. C.; Barthes-Labrousse, M. G. *Appl. Surf. Sci.* **2008**, *254*, 6495.

23. Rezaei, F.; Yunus, R.; Ibrahim, N. A. *Mater. Des.* **2009**, *30*, 260.
24. Pan, J. L.; Li, K.; Chuayprakong, S.; Hsu, T.; Wang, Q. *ACS Appl. Mater. Int.* **2010**, *2*, 1286.
25. Schultz, J.; Cazeneuve, C.; Shanahan, M. E. R.; Donnet, J. B. *J. Adhes.* **1981**, *12*, 221.
26. Park, S. J.; Kim, M. H.; Lee, J. R.; Choi, S. *J. Colloid Interf. Sci.* **2000**, *228*, 287.
27. Huttinger, K. J.; Krekel, G. *Carbon* **1991**, *29*, 1065.
28. Liu, J.; Ge, H.; Chen, J.; Wang, D.; Liu, H. *J. Appl. Polym. Sci.* **2012**, *124*, 864.
29. Park, J. M.; Kim, D. S.; Kong, J. W.; Kim, M.; Kim, W.; Park, I. S. *J. Colloid Interf. Sci.* **2002**, *249*, 62.
30. Nunes, J. P.; Hattum, F. W. J. V.; Bernardo, C. A. J. *Thermoplast. Compos. Mater.* **2004**, *17*, 523.