Friction and Wear Behavior of Glass Fabric/Phenolic Resin Composites with Surface-Modified Glass Fabric

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ABSTRACT: Untreated, air-plasma-bombarded, and β-aminoethyltrimethoxylsilane-silanized glass fabric (GF) was used to prepare GF/phenolic composites by dip coating in a phenolic adhesive resin and successive curing. The tribo-performances of these GF/phenolic composites sliding against AISI-1045 steel were evaluated with a pin-on-disc wear tester. The chemical composition of the untreated and surface-treated GF was analyzed with Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy. The interfacial regions between the phenolic resin and GF and the worn surfaces of the composites were analyzed with scanning electron microscopy. The results show that the GF/phenolic composite with β-aminoethyltrimethoxylsilane-silanized GF had the highest load-carrying capacity and best tribo-performance, and it was followed by the composite with plasma-treated GF. The improved tribo-performance of the GF/phenolic composite made of surface-treated GF was attributed to the strengthened interfacial bonding between the treated GF and the phenolic adhesive resin. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 594–601, 2009

Key words: composites; fibers; structure

INTRODUCTION

Polymers and their composites are being increasingly employed because of their good strength and low densities. Besides, a wider choice of materials and the ease of manufacturing make them ideal for engineering applications.[1–3] On account of their good combination of properties, fabric composites have been considered good candidates for tribological applications such as aircraft brake disks and bearing linings.[4–8] There are many characteristics that make the materials attractive in comparison with conventional metallic systems and coating composites. They display good self-lubricity, antiwear ability, and a large load-carrying capacity as well as a low density. Unfortunately, little has been reported on the friction and wear behaviors of fabric composites[9,10] and especially on the influence of the surface treatment of glass fabric (GF) on the tribo-performance of GF/phenolic composites.

Fabric composites are composed of fiber fabric and adhesive resin, and they can adhere to a metal surface in the presence of an adhesive resin. The binder is an important constituent in fabric composites: it connects fibers to form structural composites. Among binders, phenolic resins are widely employed because of their very good mechanical properties, active chemical reactivity, and good adhesive strength.[11,12] Fabric is the principal constituent in fabric composites. It occupies the largest volume fraction of fabric composites and shares the major portion of the load acting on a composite structure. Among fabric composites, GF is widely employed and costs very little in comparison with other advanced fabrics such as carbon fabric and Kevlar fabric. Moreover, glass fiber/fabric, exhibiting high tensile strength and tensile modulus as well as good thermal conductivity, has been widely used to promote the wear resistance and mechanical properties of various polymer–matrix composites.[13–15] For example, glass fiber has been attracting much attention in the modification and reinforcement of polytetrafluoroethylene because of its good thermal stability and relatively low cost.[14,15]

We have demonstrated that the properties of the interfacial region between the fiber and adhesive resin make a critical contribution to the quality of fabric composites.[9,10] In addition, the physical–chemical interactions between the fiber and resin at the

fiber–resin interface play an important role in the mechanical and tribological properties of fabric composites. There are some defects in glass fiber, such as poor compatibility with adhesive resins and a large friction coefficient.[14–16] Many surface modification methods for glass fiber, such as the use of coupling agents and plasma treatment, can improve interfacial adhesion between the glass fiber and polymer matrix. The improved interfacial adhesion can enhance the mechanical or tribological properties of surface-modified glass-fiber-reinforced polymer–matrix composites.[14,15,17–20] Therefore, we treated GF with plasma bombardment and β-aminoethyltrimethoxysilane (OB551) to endow a GF/phenolic composite with good tribological properties. In this study, the tribo-performance of GF/phenolic composites composed of untreated GF (pure GF) and surface-treated GF was comparatively investigated. To the best of our knowledge, this is the first report about the influence of the surface treatment of GF on the tribo-performance of GF/phenolic composites.

EXPERIMENTAL

Materials

Plain-weave GF was woven with E-glass fiber provided by the Nanjing Academy of Glass Fibers (Nanjing, China). The phenolic adhesive resin (no. 204) was provided by Shanghai Xingguang Chemical Plant (Shanghai, China). The OB551 coupling agent was purchased from Diamond New Material of Chemical, Inc. (Hubei, China). Ethanol, an analysis-grade reagent, was purchased from Tianjin No. 1 Chemical Reagent Plant (Tianjin, China).

Surface modification of GF

GF was desized via dipping in acetone for 24 h and boiling in distilled water for 10 min; this was followed by cleaning with acetone in an ultrasonic bath. The cleaned GF was bombarded with air plasma and silanized with OB551. Silanization of the cleaned GF was performed with 2, 5, and 10% (v/v) OB551 ethanol aqueous solutions at 50°C for 3 h. Then, the silanized GF was cleaned with deionized water and dried at 100°C for 2 h in an oven. The cleaned GF was bombarded with air plasma at 20 W for different times under a low vacuum of 0.5 Pa on a PICO (Germany) plasma apparatus. The chemical composition of the pure GF, air-plasma-bombarded GF (plasma GF), and 5% OB551-silanized GF (OB551 GF) was analyzed with a Bruker IFS/66v Fourier transform infrared (FTIR) spectroscope and a PHI-5702 multifunctional X-ray photoelectron spectroscope.
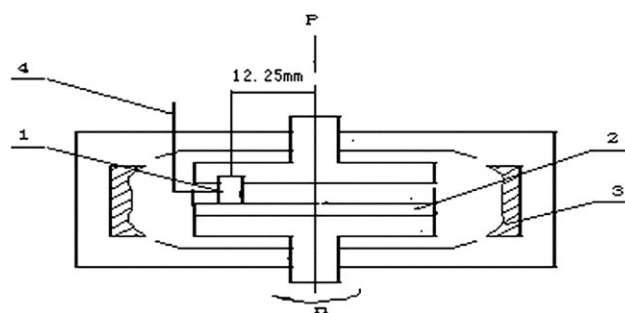

Figure 1 Schematic diagram of the pin-on-disc (pin φ (diameter) = 2.0 mm and disc φ (diameter) = 44.0 mm) friction and wear tester: P = applied load, 1 = counterpart pin, 2 = fabric-composite-coated AISI-1045 steel disc, 3 = electric furnace, and 4 = thermocouple.

Preparation of the composite

The pure GF, silanized GF, and plasma GF were immersed in the phenolic adhesive resin to allow coating by the adhesive resin. The immersion of the GF in the adhesive resin and the successive drying of the coated GF around 60°C were repeated, and GF/phenolic composites about 250–300 μm thick were obtained as the phenolic adhesive resin was used. Finally, the composite coating was adhered to an AISI-1045 steel substrate with a surface roughness of 0.45–0.60 μm with the phenolic adhesive resin and then heated at about 60°C for 24 h in air at atmospheric pressure. The final target of the GF/phenolic composite was obtained by the curing of the composite coating on the steel substrate at 180°C and 0.15–0.20 MPa for 2 h. The resulting GF/phenolic composites of pure GF, plasma GF, and OB551 GF are also called composite-U, composite-P, and composite-S, respectively. These composites were developed with the GF concentration kept at 60–65 wt %. The interfacial region between the GF and phenolic adhesive resin of various composites was observed with a JEOL JSM-5600LV scanning electron microscope.

Friction and wear test

The friction and wear behaviors of the GF/phenolic composite supported on an AISI-1045 steel disc sliding against an AISI-1045 steel pin with a diameter of 2 mm were evaluated on a Xuanwu-III test rig (produced by Lanzhou Institute of Chemical Physics, China) as shown in Figure 1. Before the tests, the pin was successively mechanically polished with 350-, 700-, and 900-grade waterproof abrasive paper to a surface roughness of 0.15–0.30 μm and then cleaned with acetone. The sliding was performed under ambient conditions at a sliding speed of 0.26 m/s with a normal load within 78.4–219.6 N at temperatures ranging from room temperature to 230°C over a period of 2 h, except when otherwise

TABLE I
Effect of the Air-Plasma Treatment Time at 20.0 W of the GF on the Friction Coefficient and Wear Rate of the GF/ Phenolic Composites at 148.9 N and Room Temperature

Treatment time (min)	Friction coefficient	Wear rate $[10^{-14} \text{ m}^3 \text{ (N m)}^{-1}]$
0 (pure GF)	0.253	8.91
5	0.246	7.69
10	0.234	6.87
15	0.236	7.06
20	0.239	7.56

indicated. At the end of each test, the composite coating on the steel was cleaned and dried, and then its wear volume loss was obtained by the measurement of the wear scar and wear depth on a micrometer (±0.001 mm). The wear rate represents the wear volume per unit of applied load and sliding distance, and it was calculated with the same formula even if the tested composite sample was unable to endure sliding for 2 h. During the test, the friction force was measured with a sensor and fed into a computer continuously. The friction coefficient was the result of the ratio of the measured force to the applied load. The environmental temperature of the friction condition was controlled with an electric furnace and was monitored with a thermocouple in the furnace. Each experiment was carried out three times, and the average value was considered (the measurement errors of the friction coefficient and wear rate are ±5 and ±10%, respectively).The morphologies of the worn surfaces of the composites were analyzed on a JEOL JSM-5600LV scanning electron microscope.

RESULTS

Friction and wear

The influence of plasma GF at 20.0 W on the friction coefficient and wear rate of the GF/phenolic composites at 148.9 N and room temperature is shown in Table I. The air-plasma treatment of GF led to a small decrease in the friction coefficient and a more significant decrease in the wear rate of the GF/phenolic composites. The GF/phenolic composite with plasma GF for 10 min at 20.0 W showed the lowest

TABLE II
Effect of the OB551 Concentration on the Friction and Wear Properties of GF/Phenolic Composites with OB551 GF at 148.9 N and Room Temperature

OB551 concentration (%)	Friction coefficient	Wear rate $[10^{-14} \text{ m}^3 \text{ (N m)}^{-1}]$
0 (pure GF)	0.253	8.91
2	0.236	6.42
5	0.231	5.81
10	0.232	6.03

wear rate and friction coefficient. However, the wear rate and friction coefficient of the GF/phenolic composite increased with a further increase in the air-plasma treatment time, and this might be attributed to the harm to the GF's mechanical properties from the long-playing bombardment of air plasma.[8]

Effects of the OB551 concentration in an aqueous ethanol solution on the friction coefficient and wear rate of the GF/phenolic composites with OB551 GF at 148.9 N and room temperature are shown in Table II. The GF/phenolic composite composed of 5% OB551 silanized GF (OB551 GF) exhibited the best tribo-performance. An excessive concentration of OB551 may have eroded the GF and then harmed the mechanical properties of GF, and this could have led to the degradation of the tribo-performance of the GF/phenolic composite.[19]

Typical variations of the friction coefficient of GF/ phenolic composites with pure GF (composite-U), 10-min plasma GF (composite-P), and OB551 GF (composite-S) with the testing time at 148.9 N and room temperature are shown in Figure 2. The friction coefficients of various GF/phenolic composites initially increased with an increase in the testing time up to 20 min. Then, the friction coefficients decreased with a further increase in the testing time because the worn surfaces became smoother from the repeated sliding between the composites and the counterpart pin. At the same time, the surface treatment of GF with air plasma or a silane coupling agent led to a small decrease in the friction coefficient in comparison with that of the pure GF/phenolic composite.

A series of friction and wear tests at a fixed velocity and various loads were conducted under dry sliding for a test duration up to 120 min, and the largest normal load that the composite could endure was cited as the maximal load-carrying capacity.
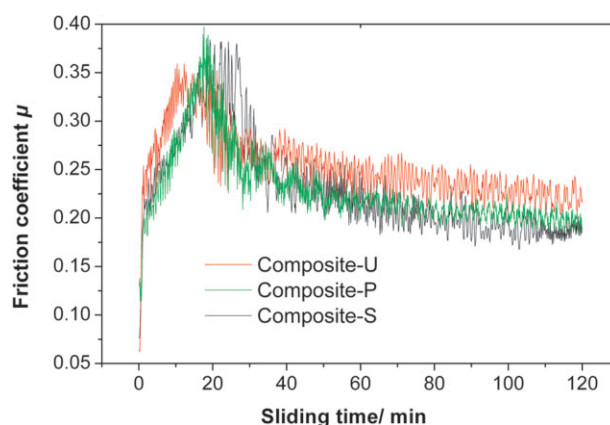

Figure 2 Typical friction coefficients of the composites made of pure and surface-treated GFs at 148.9 N. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
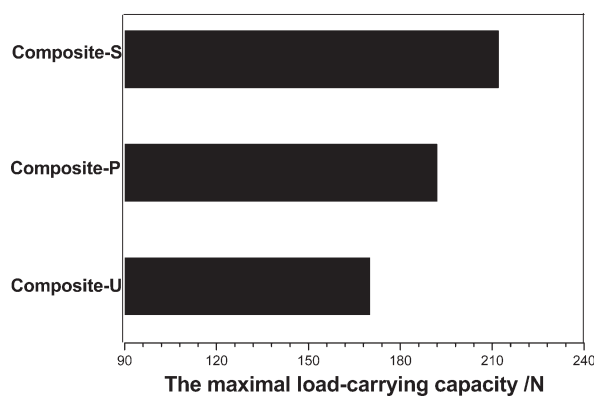
Figure 3 Comparisons of the maximal load-carrying capacities of the GF/phenolic composites with the pure and surface-treated GFs at 0.26 m/s.

Figure 3 compares the maximal load-carrying capacities of composite-U, composite-P, and composite-S. The GF/phenolic composite composed of OB551 GF and plasma GF (for 10 min) showed much better load-carrying capacity than the composite composed of pure GF. Moreover, the composite made of OB551 GF (composite-S) exhibited the largest load-carrying capacities among the various composites; this capacity was 27% larger than that of the composite made of pure GF.

Figure 4 illustrates that the GF/phenolic composite made of OB551 GF and plasma GF had a slightly smaller friction coefficient than the composite made of pure GF; moreover, the former had better wear resistance than the latter as well, regardless of the applied load. The GF/phenolic composite with the pure GF and plasma GF could not endure sliding for 120 min at the loads of 172.5 and 196.0 N, respectively. However, the GF/phenolic composite composed with the OB551 GF could endure sliding for more than 120 min at the load of 196.0 N under the same sliding condition. As a result, the GF/phenolic composite made of OB551 GF had the best comprehensive tribo-performance under different loads in comparison with the composites made of pure GF and plasma GF. At the same time, the friction coefficients of the various composites decreased with increasing applied load, but their wear rates increased with an increase in the applied load.

Figure 5 presents the influence of the environmental temperature on the friction and wear behavior of the composite at 148.9 N. The test was carried out because there was a concern that the environmental temperature could influence the friction and wear performance of the fabric composite and therefore affect its applied life. The results showed that the friction coefficient of the composite composed of OB551 GF decreased with increasing environmental temperature, whereas the wear rate initially decreased with the environmental temperature
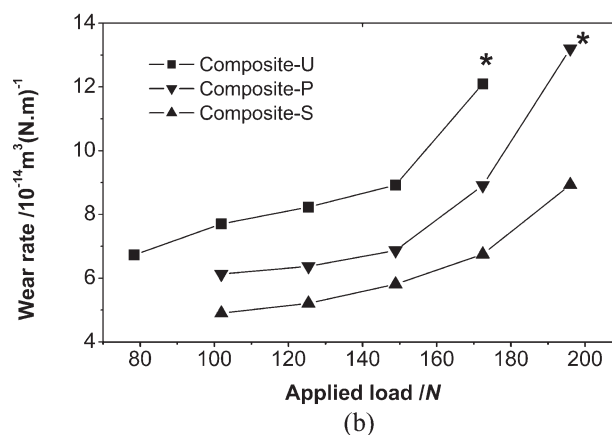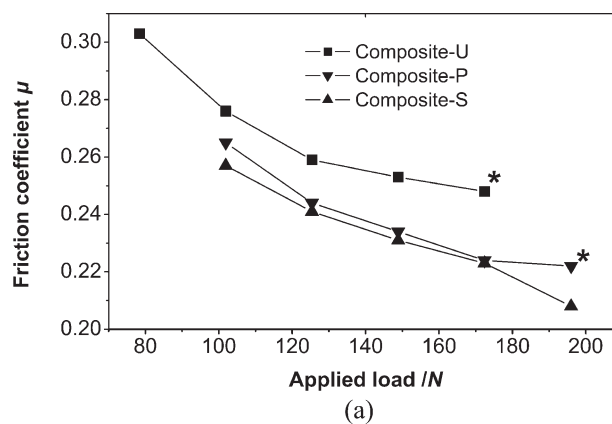

Figure 4 Comparisons of the friction coefficients and wear rates of the GF/phenolic composites with pure and surface-treated GFs with increasing applied load (the asterisk indicates that the composite could not endure sliding for 120 min under these conditions): (a) the friction coefficient versus the load and (b) the wear rate versus the load.

increasing up to 170°C and then rapidly increased as the environmental temperature increased from 170 to 230°C. It is known that a worn surface can be polished to a certain extent because of softening of the
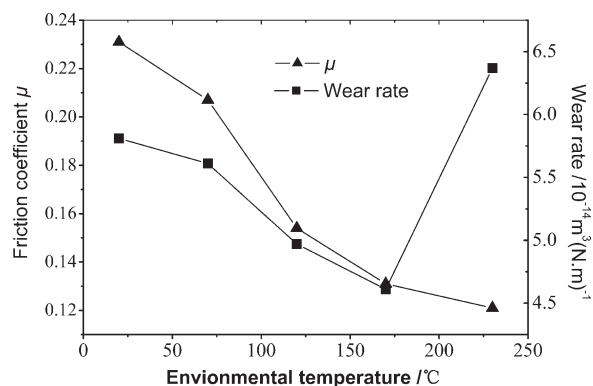

Figure 5 Effect of the environmental temperature on the friction and wear properties of the GF/phenolic composite with OB551 GF at 148.9 N.
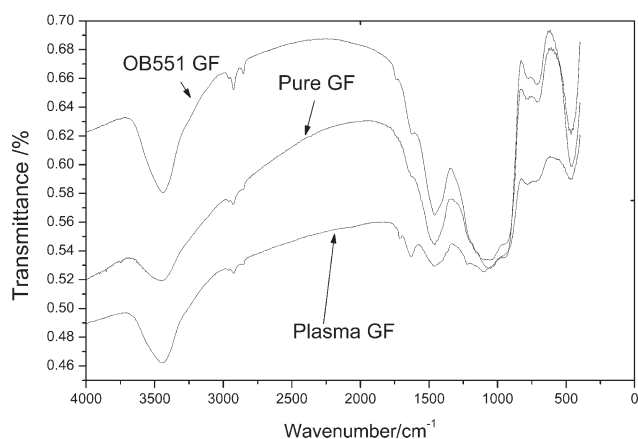
Figure 6 IR spectra of the pure, plasma, and OB551 GFs.

adhesive resin with an increase in the environmental temperature, and this results in a reduced friction coefficient of the composite.[8] On the other hand, a high contact temperature at the friction surface can induce accelerated breakage of the matrix, especially in the interfacial region.[10] As a result, the wear rate of the GF/phenolic composite made of OB551 GF rapidly increased as the environmental temperature increased from 170 to 230°C.

FTIR spectroscopy of GF

To investigate possible changes in the chemical composition of GF bombarded with air plasma or silanized with OB551, FTIR spectroscopy measurements in the mid-infrared region (4000–400 cm^{-1}) were performed. FTIR spectra were recorded with powder samples, which were obtained from cut glass fibers and dispersed in dry KBr with a Bruker IFS/66v apparatus. As shown in Figure 6, some new peaks appeared and some peaks broadened in the spectra of the glass fiber in OB551 GF. The characteristic stretching bands from methylene and methyl groups can be observed at 2850 and 2925 cm^{-1}, respectively, for the OB551 GF. The band centered at 1000–1100 cm^{-1} broadened for the OB551 GF in comparison with the pure GF, and this resulted from the contributions from Si—OH and Si—O—Si stretching bands. The stretching bands from the amidogen at 3500 cm^{-1} were significantly strengthened in the OB551 GF. As a result, it could be inferred that chemical reactions were achieved between the GF and the silane coupling agent, and this indicated that the GF was successfully coated with OB551. Moreover, some new spectra appeared for the glass fiber in the air-plasma-bombarded fabric in comparison with the pure GF. The peaks at 1750 and 1230 cm^{-1} were assigned to the stretching vibration of C=O and C—O in carboxyl, respectively, and the peak at 1650 cm^{-1} was ascribed to the stretching of

C=O. The stretching vibration of O—H at 3450 cm^{-1} was more obvious in the plasma-treated glass fiber than that in the pure GF. Therefore, some active groups, such as carboxyl and carbonyl groups, had been introduced onto the surface of the GF when the GF was bombarded with air plasma.
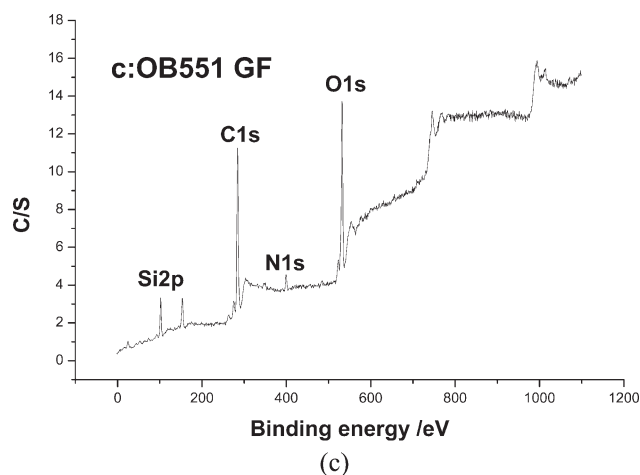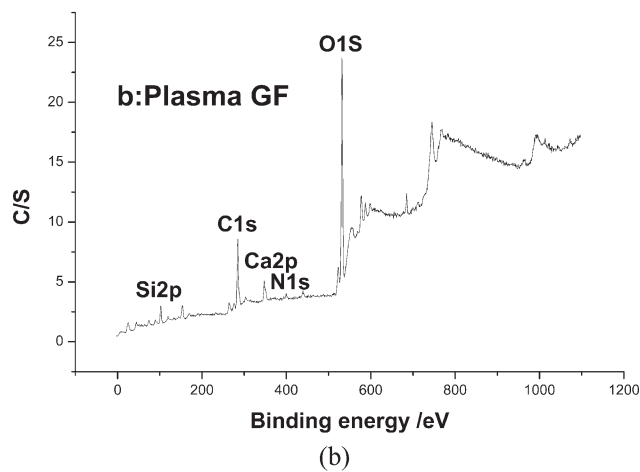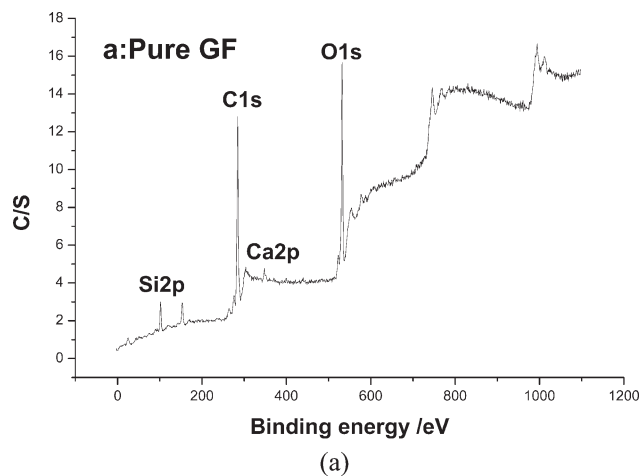

(a)

(b)

(c)

Figure 7 XPS wide scan spectra of the (a) pure, (b) plasma, and (c) OB551 GFs.

TABLE III
Relative Atomic Concentrations of the Unmodified and Modified GFs

GF	Elemental composition (wt %)					
	C	N	O	Si	Ca	Al
Pure GF	61.1	1.59	28.3	6.45	1.43	1.09
Plasma GF	30.9	1.90	55.1	6.14	3.09	2.94
OB551 GF	52.5	4.31	32.4	9.13	0.82	0.84

X-ray photoelectron spectroscopy (XPS) analysis of GF

Figure 7 shows the XPS survey spectra of the pure GF, air-plasma-bombarded (10 min) GF, and OB551 GF. The corresponding elemental compositions of the pure and modified GFs determined by the XPS analysis are listed in Table III. As shown in Figure 7, it can be concluded that the treatment of the GF with OB551 or air plasma led to changes in the chemical state of C, N, O, and Si. The air-plasma bombardment resulted in a significant decrease in the C concentration, a significant increase in the O concentration, and a slight increase in the N concentration in the GF. Combining the analytical results of Figures 6 and 7, we can infer that the air-plasma bombardment initiated chemical and physical reactions on the surface of the GF, altering the composition of the GF. Some active groups, such as carboxyl and carbonyl groups, were introduced onto the surface of the plasma GF. Comparing Figure 7(a,c), we find that 5% OB551 silanization of the GF led to a significant increase in the Si, O, and N concentrations and a slight decrease in the C concentration. Along with the analytical results from Figure 6, it can be concluded that the OB551 silane was bound onto the surface of the GF, which might have reacted with the phenolic adhesive resin during the preparation process of the GF/phenolic composite.
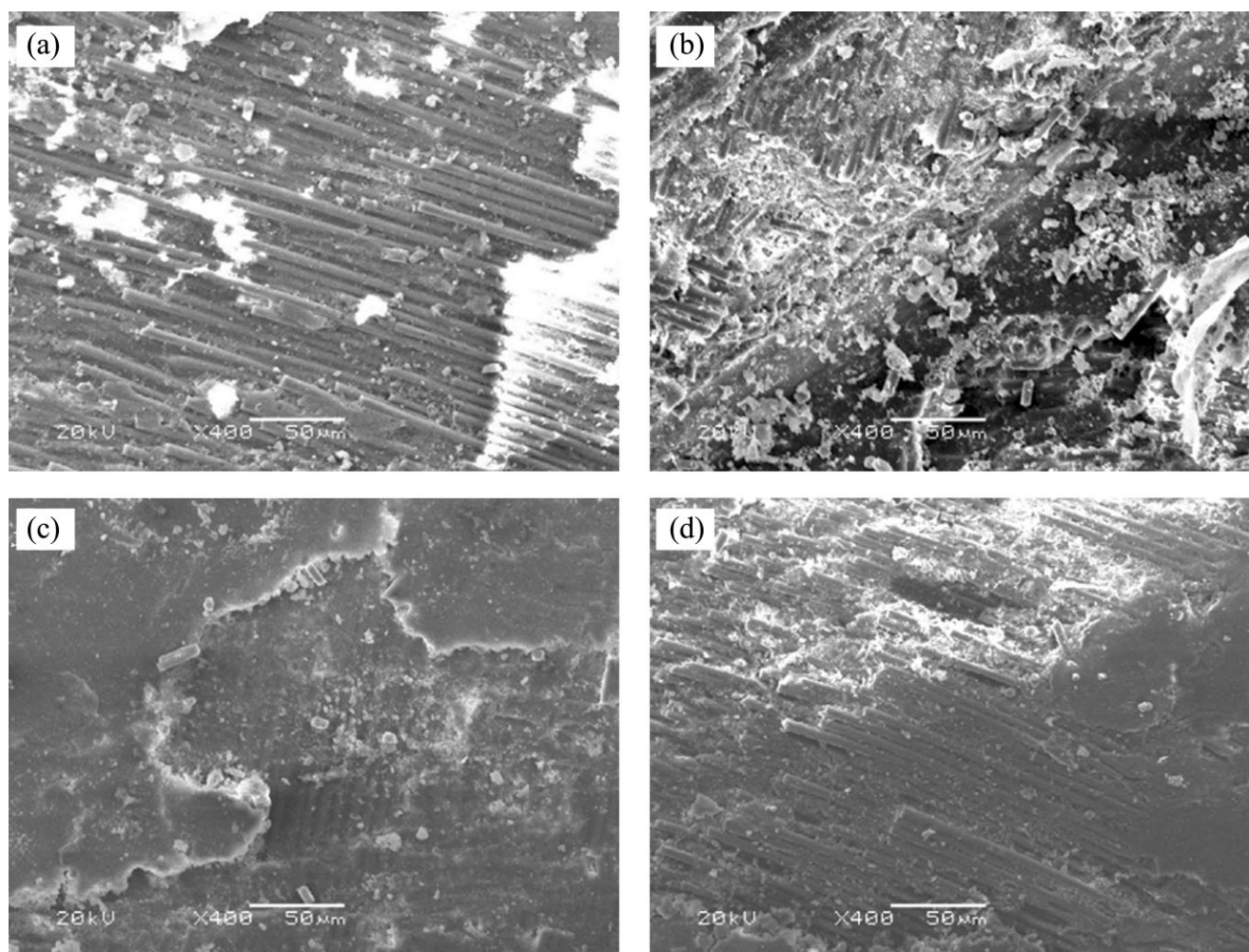

Figure 8 SEM pictures of the worn surfaces of the GF/phenolic composites made of pure, plasma, and OB551 GFs under different sliding conditions: (a) composite-U at 172.5 N, 90 min, and 20°C; (b) composite-P at 172.5 N, 120 min, and 20°C; (c) composite-S at 172.5 N, 120 min, and 20°C; and (d) composite-S at 148.9 N, 120 min, and 230°C.

Scanning electron microscopy (SEM) analysis of the worn surface

Figure 8 shows the SEM morphologies of the worn surfaces of the GF/phenolic composites composed of pure GF (composite-U), air-plasma-bombarded GF (composite-P), and OB551 GF (composite-S) under different sliding conditions. Most of the glass fibers were peeled and cut from the pure GF/phenolic composite matrix after 90 min of sliding at room temperature and 172.5 N. This indicates that the pure GF/phenolic composite experienced severe fatigue and abrasive wear under this sliding condition. If we look carefully, we can find that the glass fiber did not compactly bond with the phenolic adhesive resin; that is, interfacial debonding between the GF and phenolic adhesive resin occurred. As shown in Figure 8(b), the worn surface of the GF/phenolic composite composed of plasma GF was not very rough after 120 min of sliding at 172.5 N and room temperature in comparison with composite-U, but some glass fibers were still pulled out and cut off. In contrast to composite-U and composite-P, the worn surface of composite-S was smooth, and very few glass fibers were pulled out and exposed after 120 min of sliding at 172.5 N and room temperature [see Fig. 8(c)]. However, as the environmental temperature increased to 230°C, many glass fibers were cut and pulled out from the GF/phenolic composite composed of OB551 GF after 120 min of sliding under the low load of 148.9 N [see Fig. 8(d)], and this indicates that the composite had lower wear resistance at 230°C than at room temperature.

DISCUSSION

As illustrated in many articles,[8,10,21] the characteristics of the interfacial region between the fiber and resin, that is, the physical–chemical interactions at the fiber–resin interface, play an important role in the mechanical and tribological properties of fiber/fabric composites. As shown in Figure 9(a), the pure GF bonded poorly with the phenolic adhesive resin, and the fabric–adhesive resin interface was very large. The results indicate that the structural integrity of the GF/phenolic composite with the pure GF was bad and that the pure GF was chemically inactive. Therefore, the friction and shear stress led to interfacial debonding between the pure GF and adhesive resin as the applied load was increased to 172.5 N during the friction process. This eventually resulted in many glass fibers being pulled out and cut from the composite matrix [see Fig. 8(a)]. As a result, the tribo-performance of the GF/phenolic composite composed of the pure GF was very poor.

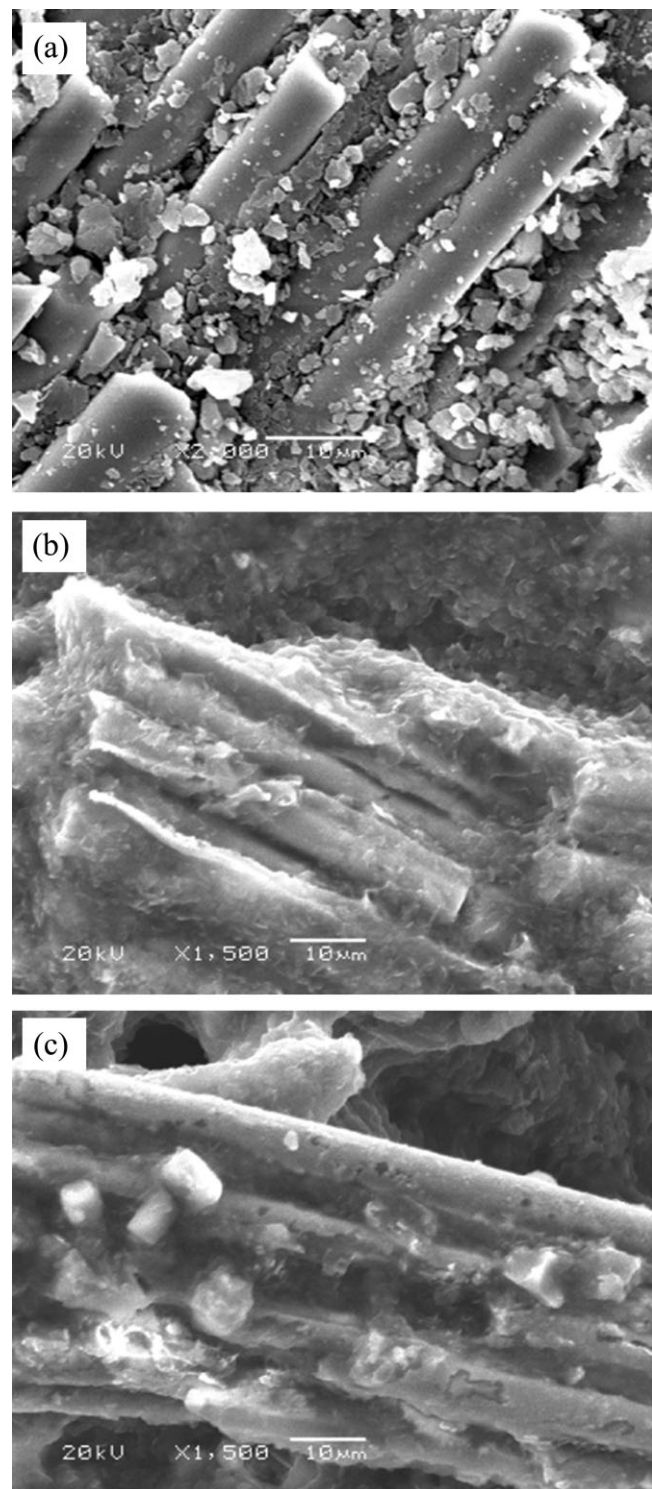However, the air-plasma bombardment of the GF initiated chemical and physical reactions on the sur-

Figure 9 SEM pictures of the interfacial region between the GF and adhesive resin of the GF/phenolic composites made of pure and treated GFs: (a) composite-U, (b) composite-P, and (c) composite-S.

face of the GF, which altered the composition of the GF. Many active chemical groups, such as carbonyl and carboxyl groups, were introduced onto the surface of the GF. During the preparation of the GF/phenolic composite, these active chemical groups

reacted with the phenolic adhesive resin and improved the bonding strength between the GF and the adhesive resin, as can be seen in Figure 9(b). As shown in Figure 9(b), the bonding between the plasma GF and the phenolic adhesive resin increased to a certain extent, but a small crack still existed in the interfacial region. As a result, a few glass fibers were pulled out and cut off from the composite matrix of the GF/phenolic composite with plasma GF [see Fig. 8(b)]. The tribo-performance of the GF/phenolic composite with plasma GF was improved in comparison with the composite composed of the pure GF.

As illustrated previously, a chemical reaction between the OB551 coupling agent (OB551) and GF occurred, and OB551 was bound to the surface of the GF. During the preparation process of the GF/phenolic composite, the adhesion between the OB551 GF and phenolic resin was substantially enhanced by chain entanglement and/or chemical bonding between the silane coupling agents bound on the GF and the phenolic adhesive resin. This can be confirmed by Figure 9(c). As shown in Figure 9(c), the OB551 GF bonded compactly with the phenolic adhesive resin, and the fabric–adhesive resin interface became blurry. The improved bonding between the OB551 GF and the phenolic adhesive resin could prevent interfacial debonding between the fabric and the adhesive resin during the sliding process at the load of 172.5 N. As a result, very few glass fibers were cut and pulled out from the composite matrix [see Fig. 8(c)] at 172.5 N.

CONCLUSIONS

The treatment of GF by OB551 silanization and air-plasma bombardment can improve the tribological property of GF/phenolic composites at various levels. A GF/phenolic composite composed of OB551 GF showed the best tribo-performance and the largest load-carrying capacity among various composites.

The difference in the tribological properties of the GF/phenolic composites made of pure GF, air-plasma-bombarded GF, and OB551 GF was mainly attributed to the different interfacial characteristic of the composites, that is, the different bonding strengths between the GF and the phenolic adhesive resin.

As the GF was bombarded with air plasma, many active chemical groups were introduced onto the surface of the GF, which improved the bonding strength between the GF and the adhesive resin. OB551 was bound to the surface of the GF, and it significantly improved the bonding strength between the OB551-treated GF and the adhesive resin by chain entanglement and/or chemical bonding.

Sliding conditions, such as the environmental temperature and the applied load, have important effects on the friction and wear properties of GF/phenolic composites.

References

1. Chen, Z. B.; Liu, X. J.; Li, T. S.; Lü, R. G. J Appl Polym Sci 2006, 102, 523.
2. Palabiyik, M.; Bahadur, S. Wear 2000, 246, 149.
3. Vassileva, E.; Friedrich, K. J Appl Polym Sci 2003, 89, 3774.
4. Lancaster, J. K.; Bay, R. G. ASTM Spec Tech Publ 1982, 769, 92.
5. Blanchet, T. A.; Kennedy, F. E. Wear 1992, 153, 229.
6. Craig, W. D. Lubr Eng 1964, 20, 456.
7. Zhang, H.; Zhang, Z.; Friedrich, K. Compos Sci Technol 2007, 67, 222.
8. Bijwe, J.; Rattan, R.; Fahim, M. Tribol Int 2007, 40, 844.
9. Su, F. H.; Zhang, Z. Z.; Liu, W. M. Compos Sci Technol 2007, 67, 102.
10. Su, F. H.; Zhang, Z. Z.; Guo, F.; Liu, W. M. Compos Sci Technol 2007, 37, 981.
11. Satapathy, B. K.; Bijwe, J. Compos A 2006, 37, 1557.
12. Park, D. C.; Kim, S. S.; Kim, B. C.; Lee, S. M.; Lee, D. G. Compos Struct 2006, 74, 89.
13. Mishra, S.; Mohanty, A. K.; Drzal, L. T.; Misra, M.; Parija, S.; Nayak, S. K.; Tripathy, S. S. Compos Sci Technol 2003, 63, 1377.
14. Yu, X. B.; Wei, C.; Xu, D.; Yu, J. H.; Lu, S. R. J Appl Polym Sci 2007, 103, 3899.
15. Cheng, X. H.; Xue, Y. J.; Xie, C. Y. Wear 2002, 253, 869.
16. Antunesa, P. V.; Ramalho, A. Tribol Int 2005, 38, 363.
17. Cech, V.; Prikryl, R.; Balkova, R.; Grycova, A.; Vanek, J. Compos A 2002, 33, 1367.
18. Zhao, F. M.; Takeda, N. Compos A 2000, 31, 1203.
19. Debnath, S.; Wunder, S. L.; McCool, J. I.; Baran, G. R. Dent Mater 2003, 19, 441.
20. Gonzaălez-Benito, J.; Baselga, J.; Aznar, A. J. J Mater Proc Technol 1999, 92, 129.
21. DiBenedetto, A. T. Mater Sci Eng A 2001, 302, 74.