Effects of Air Plasma Treatment on Tribological Properties of Hybrid PTFE/Kevlar Fabric Composite

Hui-Juan Zhang,[1,2] Zhao-Zhu Zhang,[1] Fang Guo[1,2]

[1]*State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China*
[2]*Graduate School of Chinese Academy of Sciences, Beijing 100039, China*

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ABSTRACT: Hybrid PTFE/Kevlar fabric was surface-modified by air plasma to improve the adhesion to phenolic resin. The tribological properties of fabric composite with or without plasma-treated hybrid PTFE/Kevlar fabric were investigated in detail. Friction and wear tests showed that friction-reducing and antiwear ability of hybrid PTFE/Kevlar fabric composite were remarkably improved after plasma treatment. Scanning electron microscopy and X-ray photoelectron spectroscopy results indicated that the fiber surfaces were etched and the contents of oxygen and nitrogen increased after plasma treatment, which was favorable for improved fiber/resin bonding strength. The enhancement contributed to the improved tribological properties of hybrid PTFE/Kevlar fabric composite after plasma treatment. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 3980–3986, 2009

Key words: composites; fibers; surfaces

INTRODUCTION

PTFE fiber has an important application as tribological materials because of its excellent properties in thermal stability, good resistance to solvents, and low coefficient of friction. Since Kevlar fibers came to market in 1972, it has a growing demand in material science, particularly in the areas of fiber-reinforced composites, rubber goods, ropes and cables, ballistics, pulp-reinforced friction products, gaskets, and so forth.[1,2] Kevlar fibers are good choices to high-performance composite applications, because they combine a high specific strength and modulus with a high thermal resistance and chemical inertness. Hybrid PTFE/Kevlar fabric was woven out of PTFE and Kevlar fibers. The purpose of hybridization is to construct a new material that will combine the advantages of its constituents. Thus, hybrid PTFE/Kevlar fabric combines good lubrication of PTFE fibers and good mechanical properties of Kevlar fibers, which cannot be provided by single kind of the fiber.

Fabric composites comprise two elemental parts: the fabric as matrix and adhesive resin as binder. Fabric composites can be adhered onto the metal surface and exhibit good tribological properties, therefore they are very attractive materials as advanced bearing liner materials. Moreover, they have a wide application in industry for their good self-lubrication, antiwear abilities, and low density.[3,4] In hybrid PTFE/Kevlar fabric composite system, the surface rich with PTFE fiber was used as friction surface and that rich with Kevlar fiber was used as binding surface.

When load is applied to composites, it will be distributed and transferred through fiber/resin interfaces. Therefore, adhesion properties between fabric and resin are very important to the tribological properties of fabric composites. The interfacial adhesion strength can, in general, be dominated by chemical bonding, by physical interactions, by mechanical keying, or by these combinations. However, hybrid PTFE/Kevlar fabric shows a poor bonding behavior to the phenolic resin, because of the low surface energy and chemically inert surface of the PTFE or Kevlar fibers. Various approaches have been applied to improve the bonding behavior of the fibers.[5–20] Among the methods reported, plasma treatment is considered as a clean and effective method to modify the surface of the fibers.[11–20] In this study, cold plasma treatment was used to modify the surface of the hybrid PTFE/Kevlar fabric and its effect on the tribological properties of the hybrid PTFE/Kevlar fabric composite was investigated in detail. The tribological properties of hybrid PTFE/Kevlar fabric composite with and without plasma treatment were compared and discussed.

Correspondence to: Z.-Z. Zhang (zzzhang@lzb.ac.cn).
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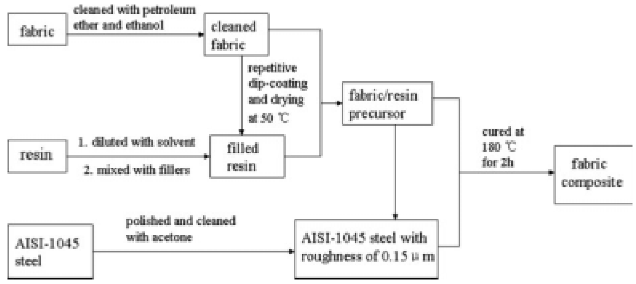
TABLE I
Properties of Phenolic Resin

Shearing strength	≥ 15 Mpa at room temperature; ≥ 6 Mpa at 200°C
Long-term working temperature	−70 to 200°C
Curing temperature	180°C
Curing time	2h



Scheme 1 Flow chart for preparing hybrid PTFE/Kevlar fabric composites.

EXPERIMENTAL

Specimen preparation

The hybrid PTFE/Kevlar fabric (weave: satin; area density: 270 g/m^2) was weaved out of PTFE fibers and Kevlar-49 fibers (Du pont, USA). The phenolic adhesive resin (204 phenolic adhesive) was purchased from Shanghai Xin-guang Chemical Plant of China (Shanghai, China). The properties of the phenolic resin are shown in Table I. The hybrid PTFE/Kevlar fabric was cleaned by Soxhlet extractor in petroleum ether and ethanol in sequence and dried for 24 h at 80°C. The hybrid PTFE/Kevlar fabric was bombarded with air-plasma at 10, 30, 50, 70, or 100 W and low vacuum pressure of 5 Pa for 5, 10, 15, 20, and 25 min on a PGT-II plasma-apparatus produced by Beijing Institute of Physics of Chinese Academy of Sciences (Beijing, China), respectively. The untreated or air-plasma treated hybrid PTFE/Kevlar fabric was then used to prepare the fabric composite by dip-coating in the phenolic resin diluted in the mixed solvent ($V_{ethanol} : V_{acetone} : V_{ethyl\ acetate} = 1 : 1 : 1$). A series of repetitive immersings and coatings of the hybrid fabric in the adhesive resin were performed to generate the composite coating with the depth of ~ 400 μm and the mass fraction of the hybrid PTFE/Kevlar fabric in the composite coating was about 70–75%. Subsequently, the composite was adhered on an AISI-1045 steel substrate (surface roughness R_a about 0.45 μm) with phenolic resin as the adhesive. Finally, the composite was heated at about 50°C for 12 h at atmospheric pressure to let the mixed solvent evaporate. The final target fabric composite was obtained by curing the composite coating on the steel substrate at 180°C and 0.15 MPa for 2 h. The roughness of the final composite was ~ 2.6 μm. The resulting fabric composite originated from the untreated or air-plasma treated hybrid PTFE/Kevlar fabric with the optimal input power and treatment time were abbreviated as composite I and II, respectively. The procedures for preparing the fabric composite are briefly shown in Scheme 1.

Friction and wear test

Sliding experiments were performed in a Xuanwu-III pin-on-disk tribometer, as shown in Figure 1. In the pin-on-disk tester, a stationary steel pin slides against a rotating steel disk, which was affixed with the hybrid PTFE/Kevlar fabric composite specimens. The flat-ended AISI-1045 pin (diameter 2 mm) was secured to the load arm with a chuck. The distance between the center of the pin and the axis was 12.5 mm. The pin stays over the disk with two degrees of freedom: a vertical one, for normal load application by direct contact with the disk, and a horizontal one, for friction measurement.

Before the tests, the pin was polished with 350, 700, and 900 grade water-proof abrasive paper to a surface roughness R_a = 0.15 μm, and then cleaned with acetone. The sliding was performed at varied temperatures, loads between 282.24 and 470.40 N, the speed of 0.26 or 0.37 m/s and over a period of 2 h under dry condition. At the end of each test, the corresponding wear volume loss (V) of the composite was obtained by measuring the depth of the wear scar on a micrometer (±0.001 mm). The wear performance was expressed by wear rate (w, m^3 (N m)$^{-1}$) as follows: $w, V=p\ L$, where V is the wear volume loss in m^3; P the load in Newton; L the sliding distance in meter.

The friction coefficient was measured from the frictional torque gained by a load cell sensor, which could be obtained directly from the computer running the friction-measure software. The contact temperature of the worn surface was monitored by a thermocouple positioned on the edge of the counterpart pin. The environmental temperature of

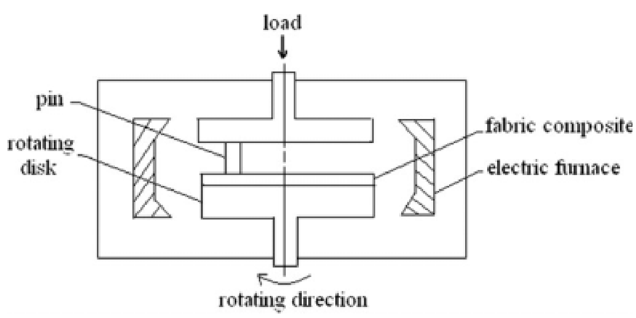


Figure 1 The contact schematic diagram for the frictional couple.

frictional condition was controlled with the electric furnace and was monitored with a thermocouple in the furnace. Each experiment was carried out three times and the average value was used.

The morphology of the fiber surface, the transfer film formed on the counterpart pin and the worn surfaces of the composites were analyzed on a JSM-5600LV scanning electron microscopy (SEM). X-ray photoelectron spectroscopy (XPS) analysis was performed on a VG ESCALAB 210 (VG Scientific) spectrometer with an Mg Kα x-ray source (1253.6 eV) to observe the effect of the air plasma treatment on the hybrid PTFE/Kevlar fabric. Water contact angle measurement was conducted in the KRÜSS DSA100 analyzer.

RESULTS AND DISCUSSION

Fiber surface morphology

Figures 2 and 3 show SEM images corresponding to the untreated and plasma-treated PTFE and Kevlar fibers under different input powers. The untreated PTFE fiber surface is quite smooth, as seen in Figure 2(a). An increase in surface roughness was observed after plasma treatment with the input power of 30 W, with some microdents substituting the smooth surfaces present on the PTFE fiber [Fig. 2(b)]. When the input power was increased to 70 W, the PTFE fiber surface was etched more vigorously. Some ripple-like structures are distributed evenly on the PTFE fiber surface [Fig. 2(c)]. This could enhance the mechanical interlocking of the polymer on the fiber surface. At higher input power of 100 W, the fiber surface was etched rather vigorously [Fig. 2(c)]. Large patches of fiber surface are damaged, which may weaken the mechanical strength of PTFE fiber.

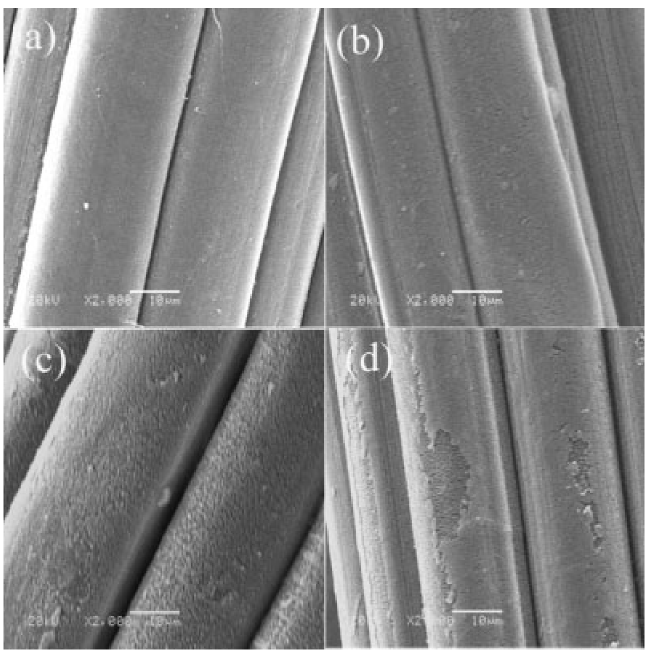


Figure 2 SEM images of PTFE fiber in untreated and plasma-treated hybrid PTFE/Kevlar fabric at different input powers. (a) Untreated PTFE fiber, (b) plasma-treated PTFE fiber at 30 W, (c) plasma-treated PTFE fiber at 70 W, and (d) plasma-treated PTFE fiber at 100 W.

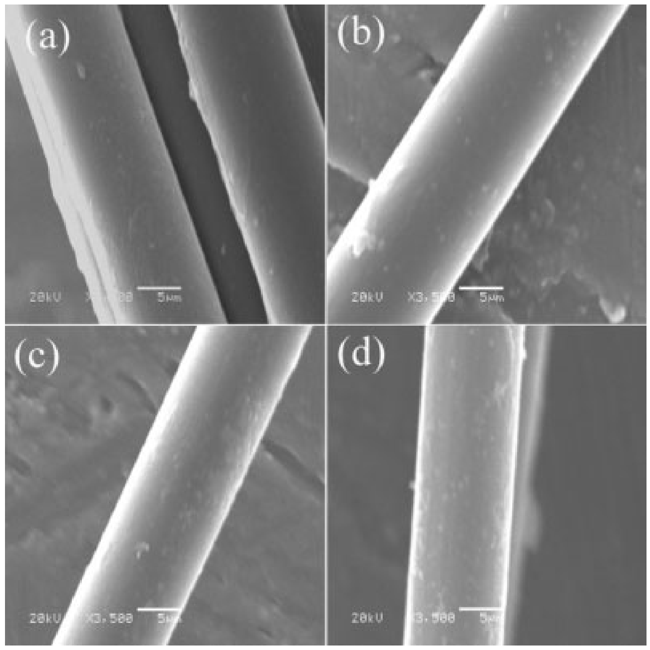


Figure 3 SEM images of Kevlar fiber in untreated and plasma-treated hybrid PTFE/Kevlar fabric at different input powers. (a) Untreated Kevlar fiber, (b) plasma-treated Kevlar fiber at 30 W, (c) plasma-treated Kevlar fiber at 70 W, and (d) plasma-treated Kevlar fiber at 100 W.

Before treatment, the Kevlar fiber surface was very smooth [Fig. 3(a)]. As the input power was 30 W, the Kevlar fiber surface structure did not change much with only some granule-like structures [Fig. 3(b)]. When the input power was increased to 70 W, the amount of granule-like structures increased and connected each into large patches [Fig. 3(c)]. This may increase the total surface area for bonding. At higher input power of 100 W, the fiber surface appeared rougher [Fig. 3(d)]. Some microcraters and granule-like structures appeared on the Kevlar fiber surface, which may damage the mechanical strength of Kevlar fiber.

XPS analysis

PTFE and Kevlar fiber surfaces in hybrid PTFE/Kevlar fabric before and after plasma treatment were analyzed by XPS. Table II summarized the atomic percentages and the atom ratios for the untreated and plasma-treated PTFE or Kevlar fiber surfaces. For the PTFE fiber, the surface content of oxygen increased from 3.394 to 9.839 and that of fluorine

TABLE II
XPS Analysis Results of Chemical Compositions for Untreated and Plasma-Treated PTFE or Kevlar Fiber

	Atomic percent			Atomic ratio	
Fiber sample	C	O	F	O/C	F/C
Untreated PTFE fiber	26.916	3.394	69.690	0.126	2.59
Air-plasma treated PTFE fiber	32.696	9.839	57.465	0.300	1.76
	C	O	N	O/C	N/C
Untreated Kevlar fiber	68.631	28.755	2.614	0.419	0.0380
Air-plasma treated Kevlar fiber	64.949	29.638	5.413	0.456	0.0833

decreased from 69.690 to 57.465 after 20 min plasma treatment at 70 W. Additionally, the low O/C atomic ratio of the untreated PTFE fiber surface increased significantly after plasma treatment. This indicated that the oxygen atoms were incorporated into the PTFE fiber surface during plasma discharge. Higher oxygen content of plasma-treated PTFE fiber was favorable for its bonding with phenolic resin. After 20 min plasma treatment at 70 W, the oxygen and nitrogen contents of Kevlar fiber increased from 28.755 to 29.638 and 2.614 to 5.413, respectively. The O/C and N/C atomic ratio also increased after plasma treatment.

Water contact angle investigation

Water contact angles of the fabric, the untreated and plasma treated, were measured to examine the surface tension properties. The results are shown in Figure 4. It can be seen that the water contact angle of the fabric surface decreased considerably from 118.8° to 25.1° because of the plasma treatment. This was probably caused by the changes in morphology (SEM images) and chemical composition (XPS results) of the plasma treated surface. This indicated that the surface free energy was increased for the air plasma treated fabric compared with that of the untreated fabric.

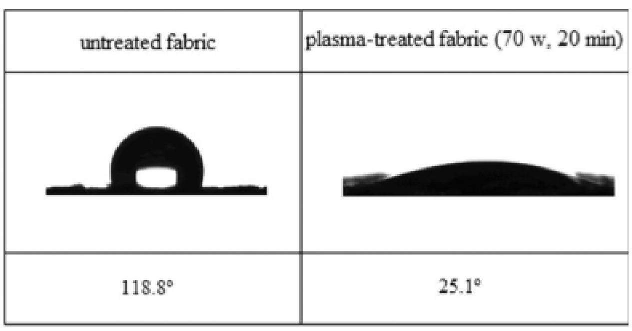


Figure 4 The contact angle images of water drops on the surface of hybrid PTFE/Kevlar fabric.

Friction and wear test

Figure 5 shows the variation of friction coefficient and wear rate of hybrid PTFE/Kevlar fabric composite with the input power at the fixed 10 min plasma treatment time under the load of 282.24 N. It can be seen that the friction coefficient of fabric composite originated from plasma-treated hybrid PTFE/Kevlar fabric did not vary much with the elevation of input power, although a comparatively lower value was obtained when the power was 70 W. With the increase of input power, the wear rate of fabric composite reduced first and then increased a lot, with a lowest value when the power was 70 W. As shown in Figure 3(b) and 4(b), when the input power was 30 W, the surface of PTFE or Kevlar fiber was only etched with some microdents or granule-like structures. As the input power elevated to 70 W, the roughness of PTFE or Kevlar fiber increased. The evenly-distributed ripple-like and increased granule-like structures on the PTFE or Kevlar fiber surface should increase the mechanical interlocking of the polymer on the fiber surface and the adhesion between fiber and resin. In this case, the tribological properties of hybrid PTFE/Kevlar fabric composite improved to a great extent. However, the high input power of 100 W may weaken the mechanical properties of hybrid fabric and contributed negatively to the tribological properties of fabric composite.[21]

The friction and wear behaviors of fabric composite originated from plasma-treated hybrid PTFE/Kevlar fabric with varied treatment time at a fixed input power of 70 W were further tested. Friction coefficient and wear rate of hybrid PTFE/Kevlar fabric composite plot as a function of the plasma treatment time under the load of 282.24 N are shown in Figure 5. The friction coefficient of fabric composite varied in a small range with the prolonging of treatment time. The wear rate

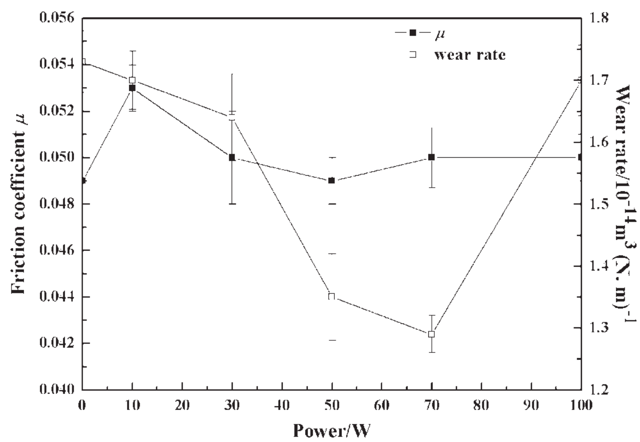


Figure 5 Variation of friction and wear rate of hybrid PTFE/Kevlar fabric composite with the increasing plasma treatment input power under the load of 282.24 N (treatment time: 10 min).

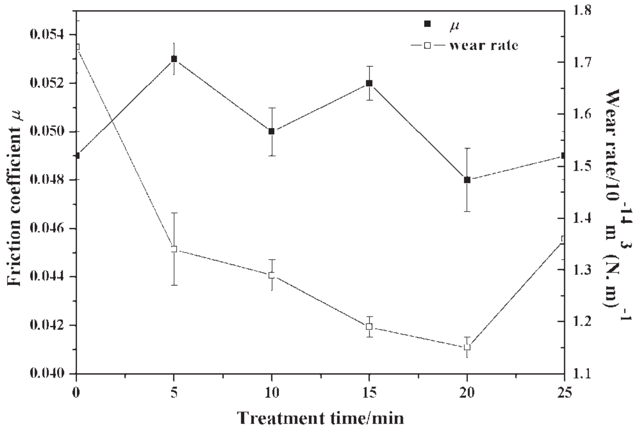


Figure 6 Variation of friction and wear rate of hybrid PTFE/Kevlar fabric composite with the increasing plasma treatment time under the load of 282.24 N (input power: 70 W).

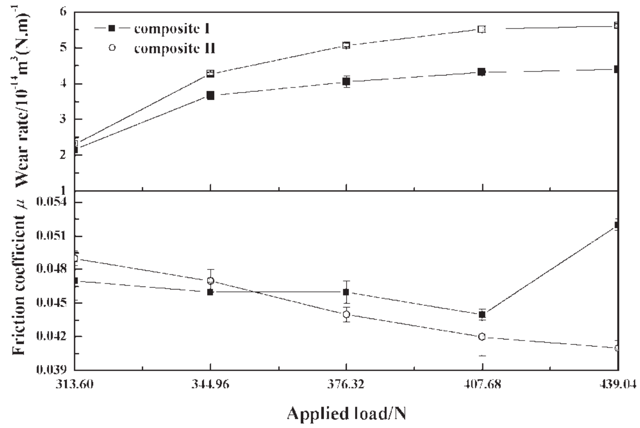


Figure 7 Effect of the applied load on friction coefficient and the wear rate of composites I and II at room temperature (sliding speed: 0.26 m/s).

of hybrid PTFE/Kevlar fabric composite decreased with the prolonging of treatment, and got a lowest value when the treatment time was 20 min. Afterward, the wear rate increased with the further increase of treatment time. Obviously, the optimal condition for treating hybrid PTFE/Kevlar fabric with plasma was under 70 W input power at 20 min. Therefore, this condition was chosen to investigate the effect of load on the tribological properties of hybrid PTFE/Kevlar fabric composite.

Figure 7 shows the effects of load on the friction coefficient and wear rate of fabric composite originated from untreated (denoted as composite I) and hybrid PTFE/Kevlar fabric treated with 20 min plasma at 70 W (denoted as composite II). The friction coefficient of composite II registers a continual decrease with the rise in the experimental applied load, whereas an abrupt increase in the friction coefficient at 439.04 N was seen in composite I (see Fig. 6). This indicated that composite I worn out under this load. A substantial decrease in the wear rate can be observed in composite II in comparison with composite I (see Fig. 7), which can be ascribed to the better interfacial adhesion between fabric and phenolic resin. On one hand, SEM analysis on the surface morphology of the fibers revealed that 20 min plasma treatment at 70 W led to a roughened fiber surface, which could increase the surface area of the PTFE or Kevlar fiber. The roughened surface of plasma-treated fabric had the advantage of mechanical interlocking with phenolic adhesive resin. On the other hand, it is anticipated that the significant increase of the surface oxygen and nitrogen contents observed in the fibers under study, as measured by XPS, would lead to a better fiber/phenolic resin interfacial adhesion. Better fiber/resin adhesion plays an important role in the improved tribological properties of hybrid PTFE/Kevlar fabric composite, because shear stress can be transferred evenly from resin to fiber. When composite II was subjected to wearing, the friction was very smooth and the damage induced by localized stress concentration was avoided to a great extent. As a result, composite II would not suffer from the severe debonding of resin and fabric and exhibited enhanced friction and wear behaviors during sliding test. That is, the tribological properties were enhanced because of the integrity of the structure of fabric composite treated with plasma.

Comparisons of the friction coefficient, the wear rate under the load of 470.68 N and the maximal load-carrying capacity of composites I and II are shown in Table III. It is seen from Table III that composite II exhibited reduced friction coefficient and wear rate but enhanced load-carrying capacity in comparison with composite I, suggesting that plasma treatment was favorable for the friction-reducing and antiwear ability of hybrid PTFE/Kevlar fabric composite. Composite II exhibited rougher surface and higher oxygen and nitrogen content and better fiber/phenolic resin bonding strength, which guaranteed a more integrated structure. During sliding test, the evenly transferred stress protected the composites from being severely destroyed and ensured enhancement of load-carrying capacity.

Hybrid PTFE/Kevlar fabric composite treated with 20 min plasma at 70 W (denoted as composite II) was

TABLE III
Comparison of the Friction Coefficient, Wear Rate Under the Load of 470.68 N and the Maximal Load-Carrying Capacity of Composites I and II

Composite	Friction coefficient μ under 407.68 N	Wear rate/10^{-14}m^3 (N m)$^{-1}$ under 407.68N	Maximal load-carrying ability/N
Composite I	0.044	5.52	439.04
Composite II	0.042	4.32	470.40

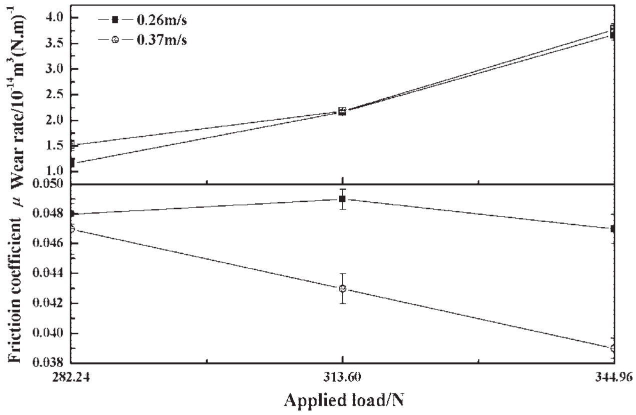


Figure 8 Effect of the load on the friction coefficient and wear rate of composite II under varied speeds.

selected to investigate the effect of load on the friction and wear behaviors under varied speeds. Friction coefficient and wear rate of hybrid PTFE/Kevlar fabric composite treated with plasma plot as a function of the load under varied speeds are shown in Figure 8. It can be seen from Figure 8 that friction coefficients of the composite under two different speeds decreased with the increasing loads, and the friction coefficient at 0.37 m/s was lower than that at 0.26 m/s. When the sliding speed was elevated, the temperature of the composite surface rose and friction heat was accumulated. During sliding test, the heat tended to cause softening of the resin so the surface was polished rapidly, which resulted in the decreasing friction coefficient. With the increasing load, the wear rate of the fabric composite under two speeds, however, showed slight differences. This implies that composite II had good wear resistance and low friction when it was applied in the sliding conditions of both low and high speeds.

Worn surface and transfer film analysis

Worn surfaces of the fabric composite with untreated or plasma-treated hybrid PTFE/Kevlar fabrics and transfer films formed on the counterpart pins were examined by SEM. The SEM images were shown in Figures 9 and 10. SEM morphologies of worn surfaces of composites I and II and transfer films formed on the counterpart pins under the load of 439.04 N are shown in Figure 9. It can be seen that a severe surface damaging was caused in composite I in the form of fiber pull-outs [see Fig. 9(a)] and the transfer film formed on the counterface was rough and damaged to a certain extent [see Fig. 9(c)]. On the contrary, the wear of composite II was less severe. The broken resin and fiber pull-outs were characteristics of the worn surface [see Fig. 9(b)]. Correspondingly, the transfer film was smooth and continuous, indicating good bonding between the transfer film and the counterpart pin [see Fig. 9(d)]. It is believed that this smooth transfer film can protect hard asperities from damaging the composite. The different wear characteristics of composites I and II can be explained by the corresponding structures. The composite II had more compact structure

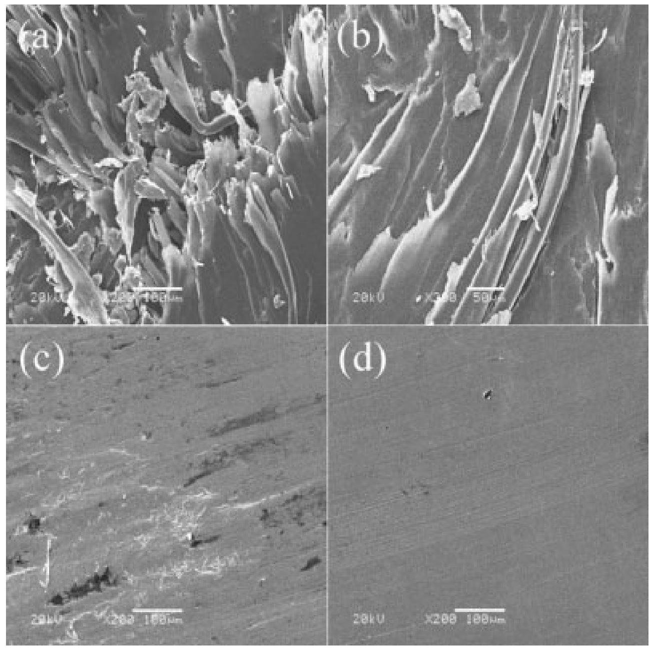


Figure 9 SEM images of the worn surfaces of (a) composite I and (b) composite II and transfer films formed on the counterpart pin sliding against (c) composite I and (d) composite II under the load of 439.04 N at room temperature (sliding speed: 0.26 m/s).

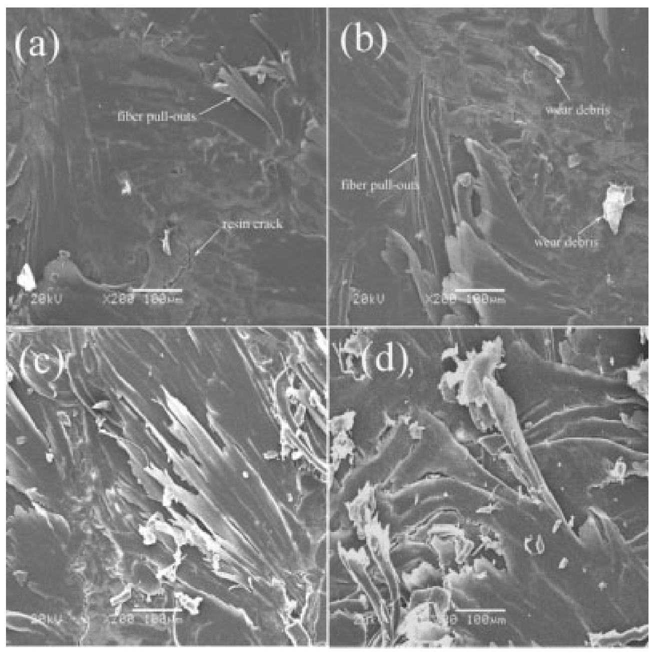


Figure 10 SEM images of worn surfaces of composite I with increasing load (a) at 313.60 N, (b) at 344.96 N, (c) at 376.32 N, and (d) at 407.68 N.

after plasma treatment. In this case, the resistance of periodic frictional stress can be greatly increased. This is important to the tribological properties of fabric composite as the integrated structure favored the stress transfer between resin and fabric. During sliding test, stress concentration caused by the structural defects can be effectively avoided in composite II and good tribological properties were obtained.

To illuminate the wear mechanism of hybrid PTFE/Kevlar fabric composite with the increasing load, SEM images of worn surfaces of composite I under a series of loads were examined and shown in Figure 10. At a load of 313.60 N, the wear of composite I was mild. The worn surface was characterized by resin crack and fiber pullouts [see Fig. 10(a)]. As the load was increased to 344.96 N, large surface damage became clear by the evidence of pulling out and the wear debris composed of cut fiber and broken adhesive resin was seen in the worn surface [see Fig 10(b)]. A lot of PTFE or Kevlar fibers were pulled out when the load was 376.32 N [see Fig. 10(c)], which indicated that composite I was severely damaged. At the high load of 407.68 N, the worn surface of composite I was destructed to a great extent. A large amount of PTFE or Kevlar fibers were cut and pulled out from the substrate [see Fig. 10(d)]. As mentioned in the earlier sections, the untreated hybrid PTFE/Kevlar fabric exhibited a smoother fiber surface and lower oxygen and nitrogen contents in fiber surface. Therefore, it can be concluded that the fiber/phenolic resin interfacial adhesion was weak and the structure of composite I was loose. With the increasing applied load, the shear stress was increased. The crack observed on the worn surfaces of composite I experienced the process of initiation, growth, and fracture. Under the higher load, the debonding between fabric and resin occurred and PTFE or Kevlar fibers were pulled out of the steel substrate. Thus, fiber pull-outs became a kind of dominant damaged form. The reason was that the stress cannot be evenly transferred from resin to fabric because of the weak adhesion and loose structure. Consequently, the wear was severe and composite I was destructed severely.

CONCLUSIONS

a. Plasma treatment resulted in changes of surface roughness and chemical compositions in the hybrid PTFE/Kevlar fabric. It is suggested that the changes can lead to improved interfacial bond strength between the hybrid PTFE/Kevlar fabric and the phenolic resin.
b. Tribological properties of the hybrid PTFE/Kevlar fabric composite are importantly dependent on the plasma treatment input power and time. The optimal tribological properties of the hybrid PTFE/Kevlar fabric composite were obtained when the hybrid fabric was treated with 20 min plasma at the input power of 70 W.
c. The fabric composite originated from plasma-treated hybrid PTFE/Kevlar fabric showed better friction-reducing and antiwear ability. This can be attributed to its better fiber/phenolic resin interfacial bond strength.

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