J Mater Sci (2009) 44:6199–6205
DOI 10.1007/s10853-009-3862-4

Enhanced wear resistance of hybrid PTFE/Kevlar fabric/phenolic composite by cryogenic treatment

Zhao-zhu Zhang · Hui-juan Zhang · Fang Guo · Kun Wang · Wei Jiang

Received: 30 April 2009 / Accepted: 29 August 2009 / Published online: 10 September 2009

Abstract Hybrid PTFE/Kevlar fabric was treated by cryogenic approach. The untreated or cryo-treated fabric was incorporated into fabric/phenolic composite for friction and wear tests. It was found that the wear resistance of the fabric/phenolic composite was improved after cryo-treatment, although the friction coefficient increased to a certain extent. SEM observations showed that the roughness of hybrid fabric increased by cryo-treatment, which may enhance the mechanical interlocking of the phenolic resin on the fiber surface. Enhanced fiber/resin adhesion was considered to contribute to the improved wear resistance of cryo-treated fabric/phenolic composite.

Introduction

Hybrid PTFE/Kevlar continuous fabric is woven out of PTFE and Kevlar fibers by a satin weaving process. When such a weaving process is finished, the PTFE/Kevlar fabric exhibits two sides of surface with different proportion between PTFE and Kevlar. The one rich in PTFE fiber was used as a friction surface while the other rich in Kevlar fiber was used as a binding surface. In this way, the low friction of PTFE and high strength of Kevlar fiber were combined to a great extent [1, 2]. Fabric/resin composites were prepared by immersing the fabric in the adhesive resin and can be adhered onto the metal surface. They exhibit good tribological properties therefore; they are very attractive materials as advanced bearing liner materials [3, 4]. Moreover, they have wide applications to the heavy-loaded key positions in the fields of aviation, astronavigation, and railways.

Unfortunately, Kevlar or PTFE fiber exhibits poor adhesion to adhesive resin due to its chemically inert surface. As a result, the tribological properties of fabric composite are usually not satisfactory under higher loads and temperatures since the fiber/resin adhesion strength is very important for obtaining good tribological properties. There are many reports related to the approaches used to modify the fiber surface [5–16]. Among those approaches, cryogenic treatment seemed to be a simple and interesting method. Zhang et al. [17, 18] treated short carbon fibers by a cryogenic treatment and investigated the mechanical and tribological properties of carbon fiber reinforced epoxy. The results showed that the mechanical and tribological properties of carbon fiber reinforced epoxy were improved after cryogenic treatment due to the enhanced fiber–matrix interfacial bonding. However, the efforts on the continuous fabrics and the performance of tribological properties of the composite have not fully made. In the present work, cryogenic treatment was applied to treat the hybrid PTFE/Kevlar fabric at various conditions. The improved wear properties of hybrid PTFE/Kevlar fabric/phenolic composite were also discussed.

Z. Zhang (✉) · H. Zhang · F. Guo · K. Wang · W. Jiang
State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China
e-mail: zzzhang@lzb.ac.cn; zhjchemistry@gmail.com

H. Zhang · F. Guo
Graduate School of Chinese Academy of Sciences, Beijing 100039, China

Experimental

Specimen preparation

The hybrid PTFE/Kevlar fabric (weave:satin) 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. The hybrid PTFE/Kevlar fabric was cleaned by Soxhlet extractor in petroleum ether and absolute alcohol in sequence and dried for 24 h at 80 °C. For cryogenic treatment (hereafter denoted as cryo-treatment), hybrid PTFE/Kevlar fabric was immersed into liquid nitrogen (−196 °C) for various minutes. The treatment details are described in Sect. "Specimen preparation". The untreated or cryo-treated hybrid PTFE/Kevlar fabrics were then used to prepare the fabric composites by dip-coating in the phenolic resin diluted by solvent ($V_{ethanol}$:$V_{acetone}$:$V_{ethyl\ acetate}$ = 1:1:1). A series of repetitive immersions and coatings of the hybrid fabric were performed to generate the composite coating with the depth of approximately 400 μm and the mass fraction of hybrid PTFE/Kevlar fabric in the composite coating was about 70–75%. Subsequently, the composites were adhered on an AISI-1045 steel substrate (size of Φ 45 mm × 8 mm, surface roughness Ra about 0.45 μm) with phenolic resin as the adhesive. Finally, the composites were heated at about 50 °C for 12 h at atmospheric pressure to let the solvent evaporate. The final target fabric composites were obtained by curing the composite coating on the steel substrate at 180 °C and 0.15 MPa for 2 h. The procedures for preparing fabric composites are briefly shown in Scheme 1. The cryo-treated hybrid PTFE/Kevlar fabric/phenolic composite for 0, 5, 10, 15, or 20 min was denoted as composite-0, composite-5, composite-10, composite-15, and composite-20, respectively.

T-peel strength

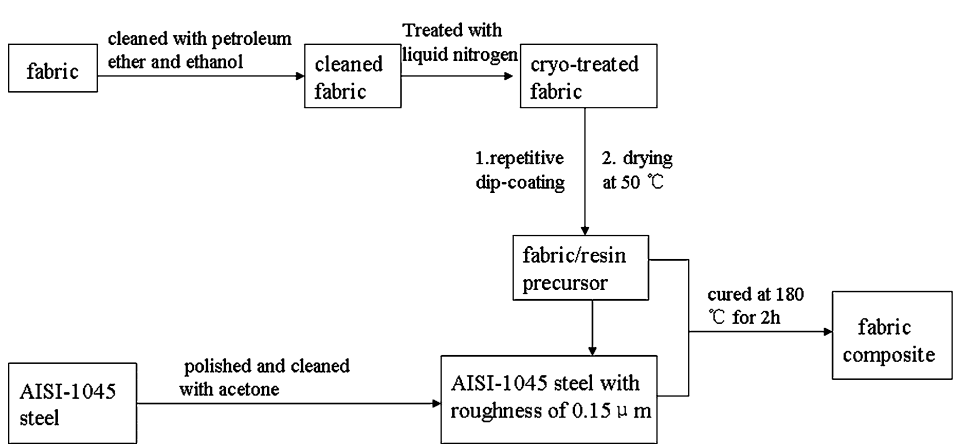
The T-peel strength between hybrid PTFE/Kevlar fabric composite and the steel substrate was determined using the T-peel test by DY35 universal materials test machine. Before the T-peel strength, the hybrid PTFE/Kevlar fabric composite was cut into a strip of 100 mm long and 20 mm wide. Then the fabric composite was affixed onto an AISI-1045 steel strip (60 mm × 2 mm) and cured at 180 °C for 2 h. The specimen for T-peel strength had a starter crack implanted between two layers. T-peel strength was carried out as follows: the specimen was fixed vertically by the clamp and pulled apart at a constant speed of 30 mm/min. The T-peel strength in gw/cm, W, is calculated from $W = F/B$, where F is the force applied to the joint in Newton and B is the width of the strip in mm. The T-peel strength was the average of 10 measurements.

Friction and wear test

Sliding experiments were performed in a Xuanwu-III pin-on-disk tribometer, as described elsewhere [19]. 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/phenolic 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 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.

Prior to the tests, the pin was polished with 350, 700, and 900 grade water-proof abrasive paper to a surface roughness Ra = 0.15 μm, and then cleaned with acetone. The sliding was performed at varied temperatures, loads between 156.80 and 407.68 N, the speed of 0.26 m/s, temperatures between room temperature and 180 °C 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:

Scheme 1 Flow chart for preparing hybrid PTFE/Kevlar fabric composite



$w = V/p{\cdot}L$, 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 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 morphologies of the fiber surface and the worn surfaces of the composites were analyzed on a JSM-5600LV scanning electron microscopy (SEM).

Results and discussion

SEM observations of fiber surface

SEM configurations of as-received and cryo-treated PTFE or Kevlar fiber in PTFE/Kevlar fabric are shown in Fig. 1. The surface of as-received PTFE or Kevlar fiber appeared to be quite smooth (Fig. 1a, b). After cryogenic treatment, the surface of PTFE or Kevlar fiber became rougher (Fig. 1c, d). Some small granules can be seen in PTFE fiber surface and some striations along the fiber axis can be observed in Kevlar fiber surface. This should increase the mechanical interlocking of phenolic resin on the fiber surface.

T-peel strength

T-peel strength is usually used to be a measurement of the bonding at the interface. In this research, the T-peel strength represented the fabric composite/steel substrate bonding strength. It was found during friction and wear tests that fabric composite was pulled out of the steel substrate if higher applied loads or higher temperatures were applied. In view of this, the T-peel strength played an important role in the wear properties of fabric composite. According to our test results, T-peel strength of composite-10 was 1.17 gw/cm and that of composite-0 was 1.08 gw/cm. The T-peel strength increased after 10 min cryo-treatment, although the degree was not high. It can thus be anticipated that composite-10 would probably exhibit a better wear resistance than that of composite-0 under the same conditions.

Friction and wear

Sliding tests were performed to investigate the effect of cryo-treatment time on the tribological properties of fabric/phenolic composite. The results are shown in Fig. 2. It is clear that the tribological properties of fabric/phenolic

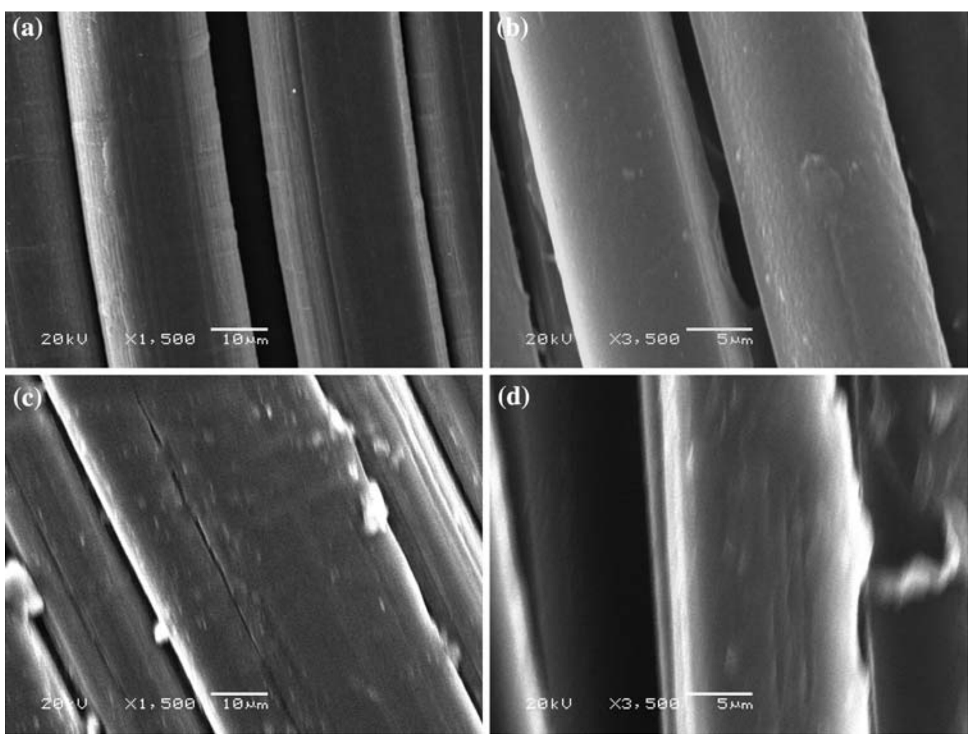


Fig. 1 SEM images of hybrid PTFE/Kevlar fabric: **a** as-received PTFE fiber; **b** as-received Kevlar fiber; **c** cryo-treated PTFE fiber for 10 min; **d** cryo-treated Kevlar fiber for 10 min

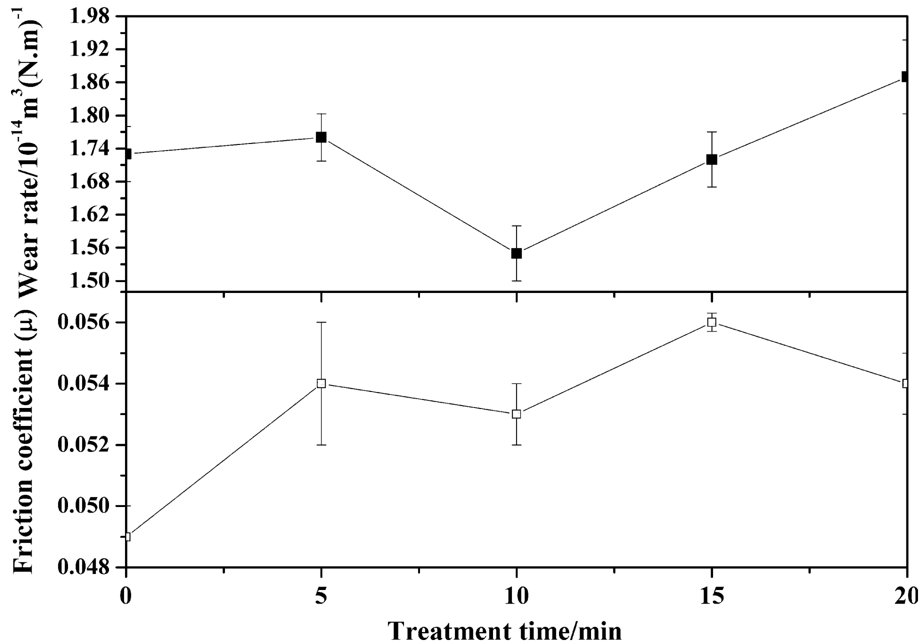


Fig. 2 Variation of friction coefficient and wear rate of fabric/phenolic composite with the cryo-treatment time (0.26 m/s; 282.24 N; room temperature)

composite depended importantly on the cryo-treatment time. When the treatment time was 5 min, the wear rate was slightly influenced in comparison with that untreated. With the treatment time extended to 10 min, a lowest wear rate was achieved. The wear rate increased, however, when the treatment time was extended to 20 min. This indicated that excessive cryo-treatment is unfavorable for the wear resistance of fabric/phenolic composite. The fibers probably suffered from damage under extended time, which will contributed negatively to the wear resistance of fabric/phenolic composite. It can, therefore, be concluded that an appropriate cryo-treatment time was favorable for the wear resistance of fabric/phenolic composite. However, excessive treatment should be avoided.

Further investigations on the effect of loads on the friction and wear of composite-0 and composite-10 were conducted, at 0.26 m/s over a period of 2 h. Friction coefficient and wear rate of composite-0 and composite-10 plot as a function of applied load are shown in Fig. 3. It can be seen that a satisfactory improvement in wear property was obtained after cryo-treatment, especially under higher loads. It is obvious that the wear rate of composite-0 was

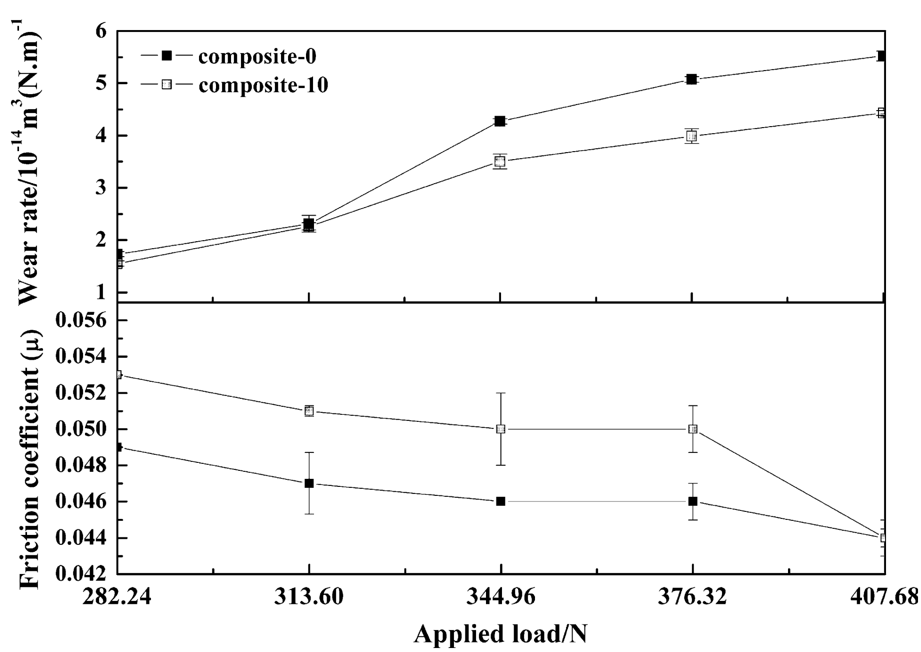


Fig. 3 Effect of applied load on the friction coefficient and wear rate of composite-0 and composite-10 (0.26 m/s; room temperature)

more sensitive to the applied load. At lower applied loads between 282.24 and 313.60 N, the difference between the wear rate of composite-0 and composite-10 was indistinct. At higher applied loads, however, the wear rate of composite-0 increased sharply. The results indicated that the adhesion strength may be weaker without cryo-treatment, which led to more fiber pull-outs at higher loads.

The effect of environmental temperatures on the tribological properties of composite-0 and composite-10 was investigated and the results were shown in Fig. 4. Environmental temperature plays a very important role on the friction and wear of polymer related composites. First, the inherent mechanical properties of polymer were weakened at elevated temperatures. That is, the decomposition and destruction of polymer were accelerated with the increase of temperature. Second, the adhesion strength between composite and steel substrate was reduced at elevated temperatures. As a result, the fabric composite tended to be pulled out of the substrate, which indicated a deterioration of anti-wear property. As seen in Fig. 4, the wear rate of composite-10 is much lower in comparison with composite-0 with the temperature elevated. And the friction coefficient of composite-10 had a small increase in a general sense. The improved wear resistance can be ascribed to the enhanced T-peel strength between composite-10 and the steel substrate as shown in Sect. “T-peel strength”.

SEM observations of worn surfaces

SEM images of worn surfaces of composite-0 and composite-10 under the same load of 282.24 N are shown in Fig. 5. Obviously, the wear of composite-0 was more severe than that of composite-10. The worn surface of composite-0 is characterized by resin crack and fiber debonding from the phenolic resin (Fig. 5a), while the worn surface of

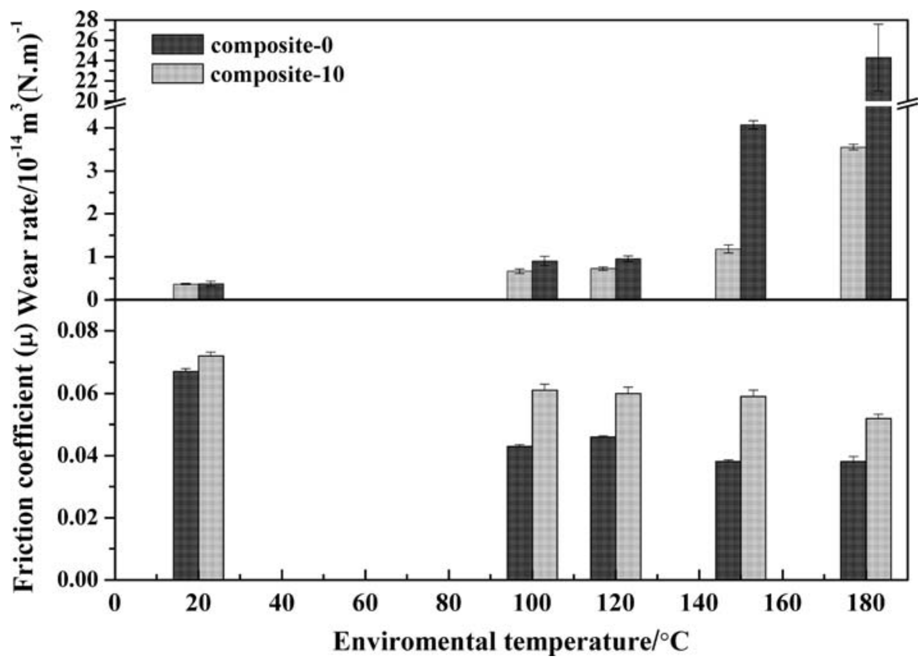


Fig. 4 Friction coefficient and wear rate of composite-0 and composite-10 plot as a function of environmental temperature (0.26 m/s; 156.80 N)

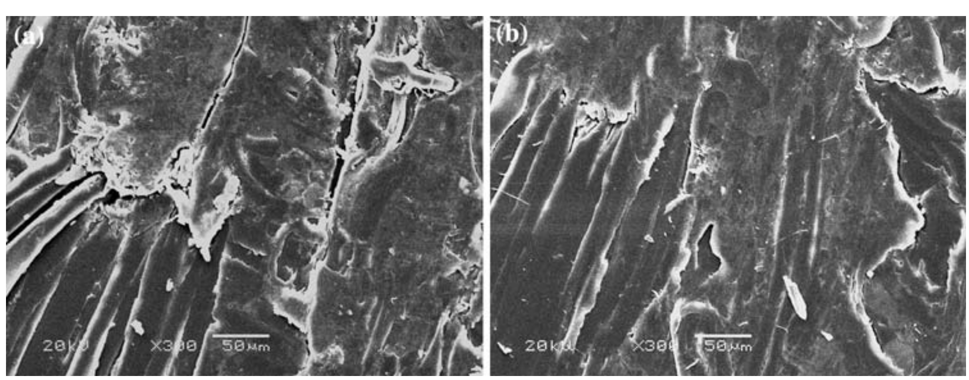


Fig. 5 SEM images of worn surfaces of **a** composite-0 and **b** composite-10 sliding under the load of 282.24 N for 2 h

composite-10 is mainly characterized by resin crack (Fig. 5b), which is an indication of fatigue wear. The observations showed that the severe wear of composite-0 was replaced by the mild fatigue wear of composite-10. It can also be seen that the fabric/phenolic resin bonding of composite-10 was stronger than that of composite-0. This can be attributed to the rougher cryo-treated PTFE or Kevlar fiber surface as shown in Fig. 1, which enhanced the mechanical interlocking of the phenolic resin on the fiber surface and hence the stronger fabric/resin adhesion. This strengthened bonding facilitate the even stress transfer of the load and protected the fabric composite from being destructed severely. The improvement of wear resistance of composite-10 was in agreement with the above observations.

The worn surfaces of composite-0 and composite-10 at varied temperatures are shown in Fig. 6. As indicated in Sect. T-peel strength, the T-peel strength of composite-10 was higher than that of composite-0. At elevated temperatures, the degree of interface debonding and fiber pull-outs of composite-10 was reduced due to the enhanced fabric composite/steel adhesion strength. Therefore, the anti-wear ability of fabric composite was improved with the elevated temperatures. As shown in Fig. 6, the worn surfaces of composite-0 at elevated temperatures showed large amounts of fiber pull-outs (Fig. 6a, c, e), which indicated fiber peel-outs occurred easily with the elevation of temperature. Comparatively, the wear of composite-10 was less severe. The fiber pull-outs were avoided to a great extent and the worn surfaces were relatively smooth (Fig. 6b, d, f). These observations confirmed the wear mechanism discussed above.

Conclusions

Optimized cryo-treatment of hybrid PTFE/Kevlar fabric can improve the wear resistance of fabric/phenolic composite. Excessive treatment, however, may negatively

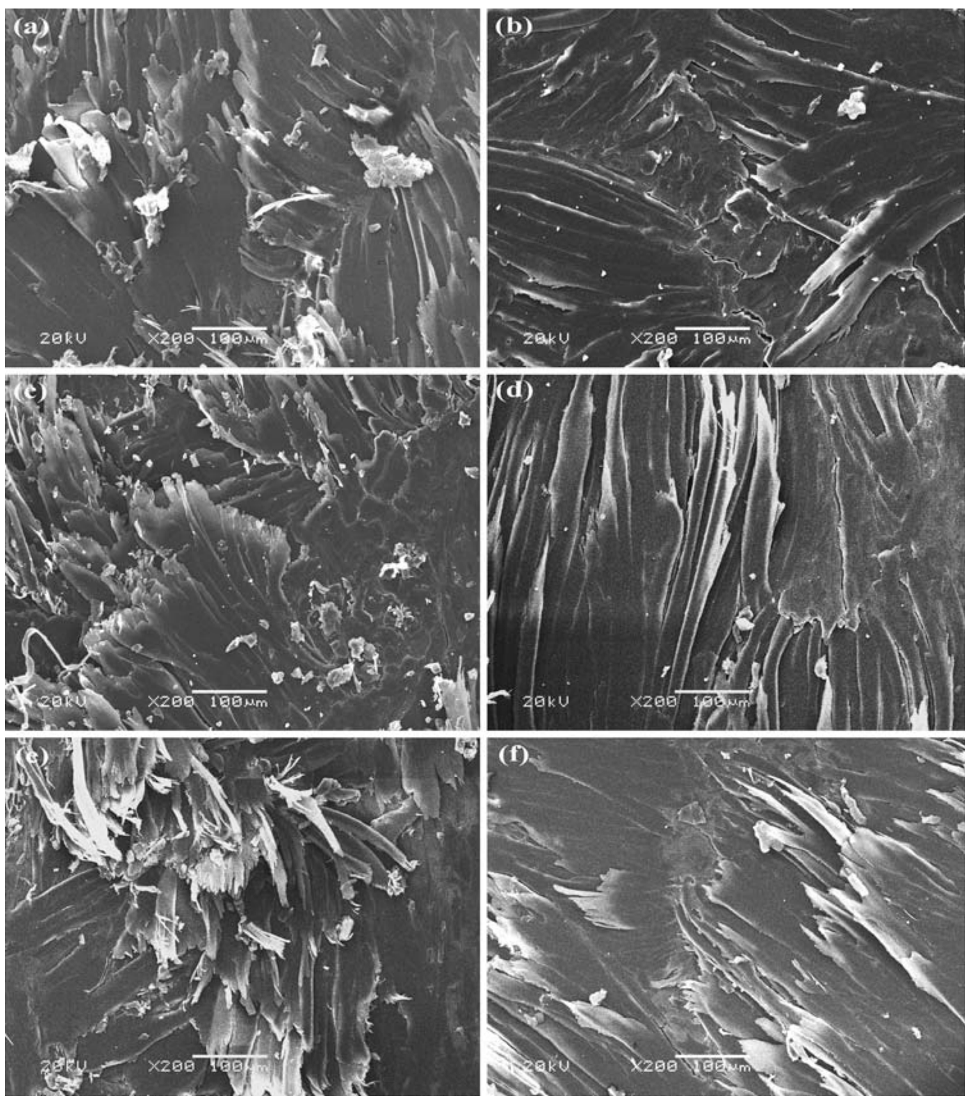


Fig. 6 SEM images of worn surfaces of **a** composite-0 at 120 °C; **b** composite-10 at 120 °C; **c** composite-0 at 150 °C; **d** composite-10 at 150 °C; **e** composite-0 at 180 °C; **f** composite-10 at 180 °C

influence the tribological properties, which may be due to the weakened fiber after over-treatment. Fabric/phenolic composite after cryo-treatment exhibited a lower wear rate under the examined loads. Additionally, fabric/phenolic composite containing cryo-treated fabric was less sensitive to the applied load. Cryo-treatment enhanced the wear resistance of fabric/phenolic composite at elevated temperatures. The improved fabric composite/steel adhesion strength may contribute to this effect.

Acknowledgements We appreciate the financial support from national Project 973 (Grant No. 2007 CB607601), Fund for Creative Research Groups (Grant NO. 50721062) and National natural science foundation (Grant NO. 50773089).

References

1. Yue CY, Sui GX, Looi HC (2000) Compos Sci Technol 60:421
2. Blanchet TA, Kennedy FE (1992) Wear 153:229
3. Craig WD (1964) Lubr Eng 20:456
4. Lancaster JK, Bay RG (1982) ASTM, STP 769:92
5. Zhang ZZ, Xue QJ, Liu WM, Shen WC (1997) Wear 210:151
6. Li F, Hu KA, Li JL, Zhao BY (2001) Wear 249:877
7. Lu XC, Wen SZ, Tong J, Chen YT, Ren LQ (1996) Wear 193:48
8. Zou XP, Kang ET, Neoh KG, Cui CQ, Lim TB (2001) Polymer 42(26):6409
9. Klaas NV, Marcus K, Kellock C (2005) Tribol Int 38(9):824
10. Combellas C, Richardson S, Shanahan MER, Thiébault A (2001) Int J Adhesion Adhesives 21(1):59
11. Cech V, Prikryl R, Balkova R, Grycova A, Vanek J (2002) Compos A 33(10):1367
12. Pappas D, Bujanda A, Demaree JD, Hirvonen JK, Kosik W, Jensen R, McKnight S (2006) Surf Coat Technol 201(7):4384
13. Huang HC, Ye DQ, Huang BC (2007) Surf Coat Technol 201(24):9533
14. Wu GM, Chang CH (2007) Vacuum 81(10):1159
15. Krump H, Hudec I, Jaššo M, Dayss E, Luyt AS (2006) Appl Surf Sci 252(12):4264
16. Indumathi J, Bijwe J, Ghosh AK, Fahim M, Krishnaraj N (1999) Wear 225–229:343
17. Zhang H, Zhang Z, Breidt C (2004) Compos Sci Technol 64:2021
18. Zhang H, Zhang Z (2004) Compos Sci Technol 64:2031
19. Guo F, Zhang ZZ, Liu WM, Su FH, Zhang HJ (2009) Tribol Int 42:243