J Mater Sci (2011) 46:6673–6681
DOI 10.1007/s10853-011-5620-7

Friction and wear behavior of the polyurethane composites reinforced with potassium titanate whiskers under dry sliding and water lubrication

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Received: 30 March 2011 / Accepted: 9 May 2011 / Published online: 19 May 2011

Abstract A series of polyurethane (PU)/potassium titanate whiskers (PTW) composites modified by a high molecular weight hydroxyl-terminated polydimethylsiloxane (HTPDMS) were prepared. The PTW is modified by 2,4-diisocyanatotoluene (2,4-TDI). The effect of the PTW content on the mechanical and tribological properties of the PU composites was studied. Tensile strength of the PU composites increased with the addition of PTW. The friction and wear experiments were tested on a MRH-3 model ring-on-block test rig at different sliding speeds and loads under dry sliding and water lubrication. Experimental results revealed that the small content of PTW contributed to largely improve the tribological properties of the PU composites. The coefficient of friction (COF) of the composites increased and the wear rate value decreased with increasing PTW. Scanning electron microscopic (SEM) investigations showed that the worn surfaces of the PTW-reinforced PU composites was smoother than pure polyurethane under given load and sliding speed.

Introduction

Composite materials are among the most rapidly growing classes of materials. Polyurethane (PU) is a versatile polymer that can be prepared by a simple polyaddition reaction of polyol, isocyanate, and a chain extender [1–3]. Its properties can be tailored upon requests by different modifiers, fillers. Compared with metals and ceramics, polyurethane composites exhibit good resistance to wear and excellent tribological property, which make them to be an important class of composite materials for marine and tribological applications. But conventional PU exhibits little resistance to heat, and this limits its applications without lubrication. Its structure may be destroyed during the stage of friction. Therefore, it is important to improve the thermal stability of PU by chemical modification and physical blending. Exactly, potassium titanate whisker (PTW, $K_2Ti_6O_{13}$) have high thermo-oxidative stability till ~500 °C without any substantial degradation loss, high melting point (1250–1310 °C), extremely high tensile strength close to the binding forces of the adjacent atoms, excellent reinforcing ability and a good friction-wear performance till 204 °C [4]. But, PTW often combines composites with low interfacial bonding between whiskers and polymer matrix when used without any surface treatment, which accordingly affects most of the other mechanical properties of the composites [5]. Therefore, it is necessary to modify the surface of the potassium titanate whiskers by grafting active group. At the same time, numerous amount of scientific work have been developed over the years to modify the whiskers surface and thus enhance the ability to establish strong interaction between whiskers and polymer matrix [6–12]. Many researchers have studied the tribological properties of the polymer reinforced with potassium titanate whiskers. Xie [13] studied the tribological behavior of PEEK/PTFE composites reinforced with potassium titanate whiskers. Mu [14] compared the tribological properties of different fibers-reinforced PTFE/PEEK composites.

Relative systematic studies about polyurethane have been carried out to modify or fill PU for coatings and

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medical applications, but not for elastomers as tribological materials. Zhang [15–21] studied the properties of many kinds of polyurethane coatings by chemical modification or fiber reinforcement or nanoparticle filled. Zhou [22] studied the friction and wear behavior of three-dimensional braided carbon fiber/epoxy composites under lubricated sliding conditions. Many researchers have also shown how the friction and wear of PU composites depend on fiber, inorganic fillers, sliding conditions, etc. [23–25]. And investigations on tribological behavior of polymers in aqueous environments have also reported by many researchers. Evans et al. [26, 27] reported that the introduction of water into a polymer-metal sliding combination generally reduced the friction coefficient. Of course, aqueous medium can effectively take away the friction heat, as a result, the adherence and plastic deformation are inhibited greatly [28, 29]. The research and development of water-lubricated materials are urgent demands for modern technology. They have involved either the modification of the chemical structure by blending or copolymerization or grafting to more thermally stable polymers such as polydimethylsiloxane (PDMS) [30] and polyimides [31]. Surprisingly, little is reported on the wear performance of whiskers reinforcing PU composites.

In the present work, the main objective of this study was to investigate the friction and wear characteristics of the PTW/PU composites under dry sliding and water lubrication. Emphasis was placed on the enhancement effect of the PU composites with adding small content PTW and the best conditions for use. For this purpose, wear test of the PU composites with varying amount of PTW was performed at various testing conditions (different speed under water lubrication at the same load).

Experimental

Materials

In the present study, hydroxyl-terminated polydimethylsiloxane (HTPDMS) with a number average molecular weight of 5,600 was obtained from Aldrich. 2,4-diisocyanatotoluene (2,4-TDI) was provided by China Medicine Shanghai Chemical Reagent Corp. Dibutyltinlaureate (DBTDL), supplied by Aldrich, was used as a catalyst. Polyethylene glycol1000(PEG1000; number average molecular weight = 1,000), provided by Sinopharm Chemical Reagent Co. Ltd., was dried for removing the traces of moisture in vacuum oven at 110 °C for 24 h. 4,4-methylenebis (2-chloroaniline), used as chain extender, was obtained from Laizhou Jiecheng Chemical Co., Ltd. (Shandong, China). Potassium titanate whiskers (PTW, $K_2Ti_6O_{13}$) were obtained from Shanghai Whiskers Composites Manufacturing Co., Ltd., China. The morphology of the PTW is shown in Fig. 1. The average diameter was 1–5 μm and specific density 3.13 g/cm^3. HTPDMS and PTW were dried under vacuum before use. Other solvents such as toluene and dimethylformamide were dried with calcium hydride before use.

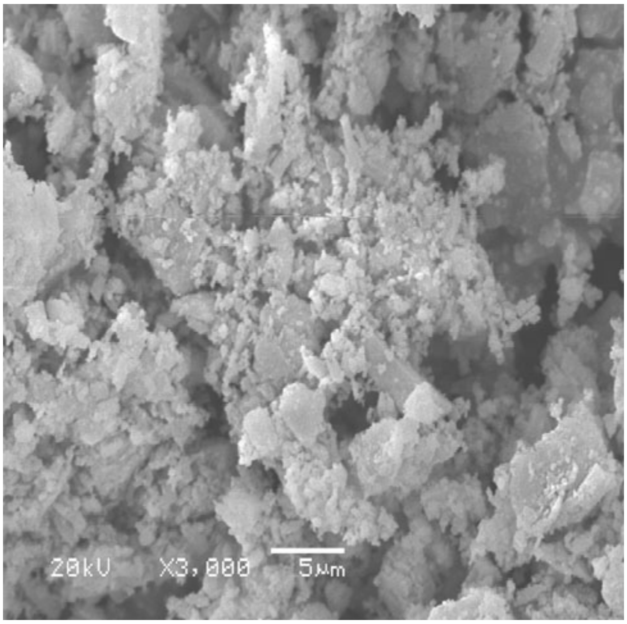

Fig. 1 SEM images of the morphology of the potassium titanate whiskers

Surface treatment of PTW

Figure 2 shows the chemical modification of the PTW. The typical grafting of PTW proceeded as follows. A 500 mL round-bottomed flask was charged with 10 g of the PTW, 200 mL toluene, 20 mL 2,4-TDI, and 2–3 drops of dibutyltindilaurate. The mixture was treated with an ultrasonic both for 30 min and stirred for 24 h at room temperature. Then, the mixture was vacuum filtered through 0.2 μm Millipote polyvinylidene fluoride membrane and washed with toluene for three times. The filtered solid was dried under vacuum for 24 h at 100 °C, obtaining PTW-2,4-TDI.

Preparation of PTW/PU composites

In this study, polyurethane composites were synthesized by a pre-polymer method. The 2,4-diisocyanatotoluene was

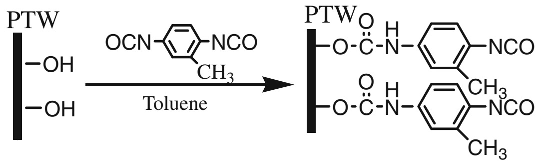

Fig. 2 Chemical route for the modification of the PTW

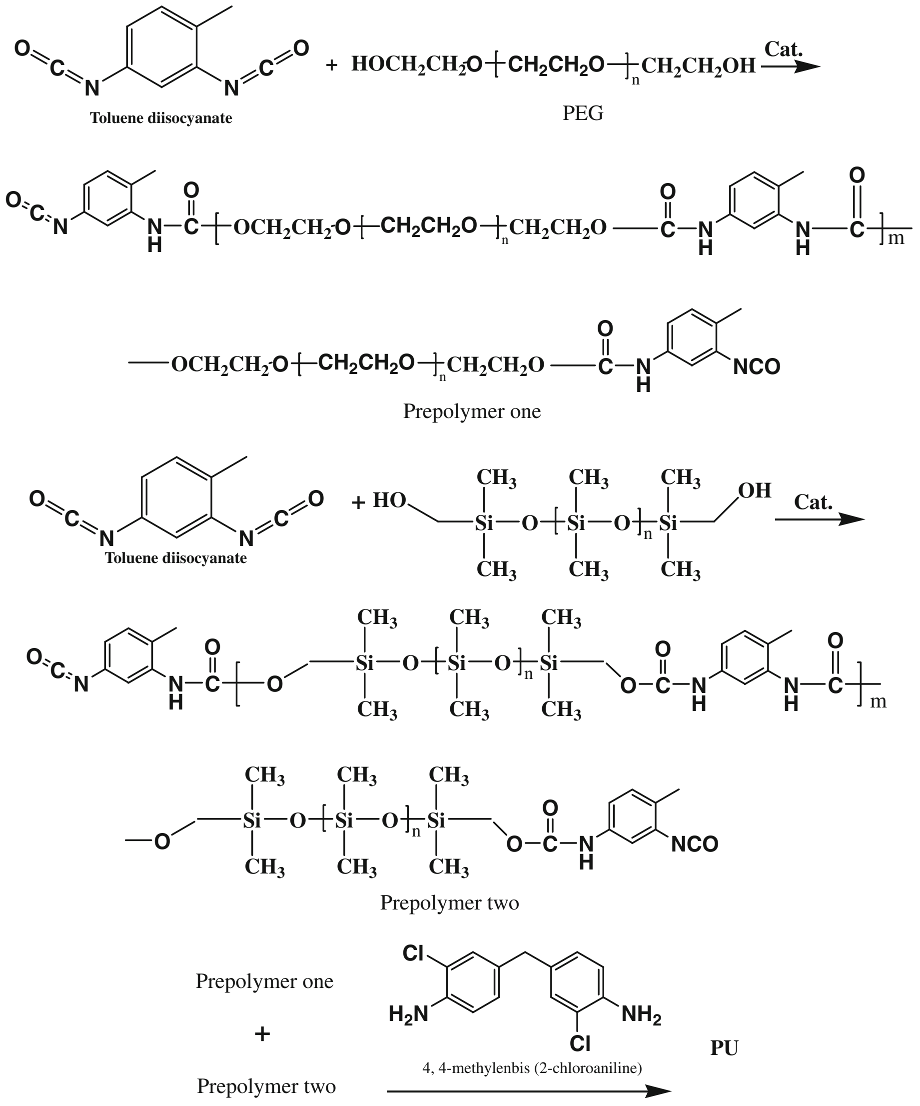

Fig. 3 Synthesis of polyurethane composites with 2,4-TDI, PEG1000, HTPDMS, and 4,4-methylenebis (2-chloroaniline)

used as hard segments, the polyethylene glycol and HTPDMS as soft segments, and the 4,4-methylenebis (2-chloroaniline) as a chain extender. Figure 3 shows the synthesis of polyurethane matrix with 2,4-TDI, PEG1000, HTPDMS and 4,4-methylenebis (2-chloroaniline). Firstly, the PEG1000 was dried for removing the traces of moisture in a vacuum oven at 110 °C for 24 h. The superfluous 2,4-TDI was mixed with PEG1000 and HTPDMS with stirring in a three-neck round bottom flask under nitrogen sweep. The temperature was maintained at 80 °C. This reaction was catalyzed by dibutyltinlaureate about 1–2 drops. After stirring for 120 min, air bladders were removed from the pre-polymer with a vacuum pump for 30 min. Then, PTW was added in predetermined amounts (Listed in Table 1) to the pre-polymer and stirred quickly. The chain extension reaction was carried out by fast adding the 4,4-methylenebis (2-chloroaniline). Finally, the PTW/PU composites were vulcanized in an oven at 80 °C for 24 h. In the present study, six polyurethane composites were prepared and compared.

Characterization

Fourier transform infrared spectroscopy (FTIR) measurements were recorded on a Bruker IFS/66 V spectrometer (Bruker Instruments, Germany) in the wave number range 4,000–500 cm^{-1}. The PTW specimen for FTIR measurements were prepared by KBr tabletting method. The PU composite specimens for FTIR measurements were prepared by a solvent method, dissolving specimen in *N*,*N*-dimethylformamide. The morphologies of worn surface were examined on a JEM-5600LV scanning electron microscope (JEOL, Japan) operated at 20 kV. In order to increase the resolution for the SEM observation, the tested polymeric samples were plated with gold coating to render them electrically conductive.

Table 1 Preparation of the polyurethane composites with varied amount of PTW (unit: g)

Sample	PEG1000	HTPDMS5600	2,4-TDI	MOCA	PTW
PU	1,000n	0	348.3n	267.0n	0
PU + 7%HTPDMS	1,000n	392n	372.7n	285.7n	0
PU + 7%HTPDMS + 1%PTW	1,000n	392n	372.7n	285.7n	20.5n
PU + 7%HTPDMS + 3%PTW	1,000n	392n	372.7n	285.7n	61.5n
PU + 7%HTPDMS + 5%PTW	1,000n	392n	372.7n	285.7n	102.5n
PU + 7%HTPDMS + 7%PTW	1,000n	392n	372.7n	285.7n	143.5n

n number of moles for each components; i.e., if 1 mol of PEG1000 was used, 1,000 g PEG1000 were added

Mechanical testing

Tensile strength was carried out on an Electron Omnipotence Experiment Machine SANS-CMT5105 (Shenzhen Sans Testing Machine Co., Ltd, China), according to Chinese Standard GB/T1040.2/1A-2006 (ISO527-2/1A: 1993) using a standard sample size (ISO527-2/1BA). Five specimens were tested for each sample.

Testing procedure

The friction and wear behavior of polyurethane composites were evaluated on a MRH-3 (made by Jinan Yihua Testing Machine Factory, China) model ring-on-block test rig. The contact schematic diagram of the frictional couple is shown in Fig. 4. The blocks were the polymeric samples. The ring of Φ49.22 mm × 13.06 mm was made of SnCu alloy, which has excellent mechanical properties and corrosion resistance in water and is potentially applicable for bearing materials in a marine environment.

The friction and wear tests were carried out at different velocities with a fixed duration of 120 min. For slide wear test, the samples of size 12.32 × 12.32 × 19.05 mm were cast on a steel mould. Before each test, the SnCu alloy ring was polished with abrasive papers to a surface roughness (Ra) of about 0.10 μm. Then, the ring and blocks were polished with acetone followed by drying. The water lubrication between the sliding surfaces was realized by continuously dropping water onto the sliding surface at a rate of 60–70 drops per minute (180–210 mL per hour). The length of the wear track was measured with a digital-reading microscope to an accuracy of 0.01 mm. Then, the wear volume loss ΔV of the specimen was calculated from the following relationship:

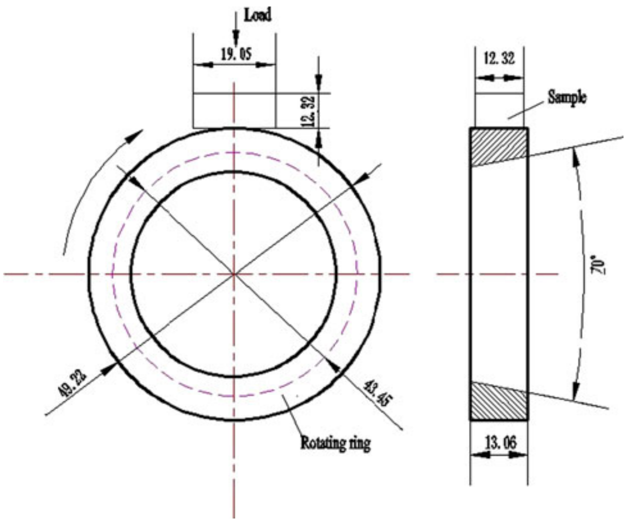

Fig. 4 Contact schematic diagram for the frictional couple (unit: mm)

$$\Delta V = \left[R^2 \arcsin\frac{b}{2R} - \frac{b\sqrt{R^2 - (b/2)^2}}{2} \right] B$$

where ΔV is the wear volume loss (mm^3), R is the diameter of the ring (49.22 mm), b is the length of the wear trace (mm), B is the width of the sample (12.32 mm). The specific wear rate K (mm^3/N·m) was calculated from the volume loss using the following equation:

$$K = \frac{\Delta V}{PL}$$

where P is the load (N), L is the sliding distance (m). The average of two replicate tests results is reported. All the friction and wear tests were carried out at 20–25 °C and a relative humidity of 40–60%.

Results and discussion

Mechanical properties

As described in the experimental section, the mechanical property of PTW/PU composites was examined by tensile strength tests at room temperature and the results were summarized in Fig. 5. With increasing of the content of the PTW, the tensile strength of the PU composite increased sharply. Compared to pure polyurethane, the tensile strength increased from 1.71 to 7.41 MPa, 333% increased

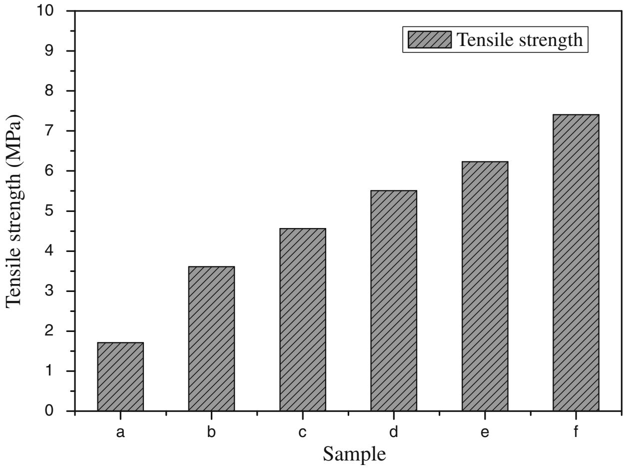

Fig. 5 Tensile strength of the PU/PTW composites: **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7%HTPDMS + 3%PTW, **e** PU + 7%HTPDMS +5%PTW, **f** PU + 7%HTPDMS + 7%PTW

Table 2 Tensile strength of the PU composites

Sample	Tensile strength (MPa)	Shore D hardness
PU	1.71	10
PU + 7%HTPDMS	3.61	21
PU + 7%HTPDMS + 1%PTW	4.56	28
PU + 7%HTPDMS + 3%PTW	5.51	30
PU + 7%HTPDMS + 5%PTW	6.23	31
PU + 7%HTPDMS + 7%PTW	7.41	33

(Table 2). The possible reason was attributed to the adhesion properties between PTW and PU matrix in the composite was excellent and the dispersion degree of PTW in the composite was high. The covalent bond is stronger than physical attraction.

Structural characterization of the PTW-reinforced PU composites

Figure 6 shows the spectra of PTW, PTW-2,4-TDI, and PTW/PU. The PTW have the special peak at 1,617 cm^{-1}. After modification with 2,4-TDI, the characteristic peaks of the 2,4-TDI are observed at about 2,271 cm^{-1}, respectively. The new peak 2,271 cm^{-1} is attributed to –NCO stretching mode. Together with the FTIR results, it was concluded that the surface of PTW was coated with 2,4-TDI through covalent bond and physical attraction. It can be seen from Fig. 6 that the peak of –NCO (2,271 cm^{-1}) was appeared in the PTW/PU. This indicated that graft reaction was complete.

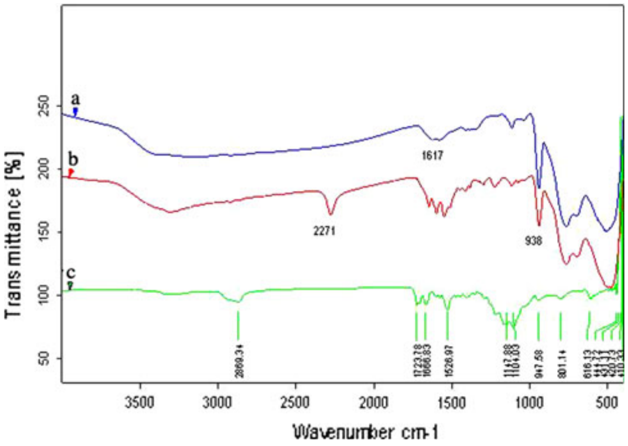

Fig. 6 The FTIR results of the treated PTW: **a** PTW, **b** PTW-TDI, **c** PU/PTW

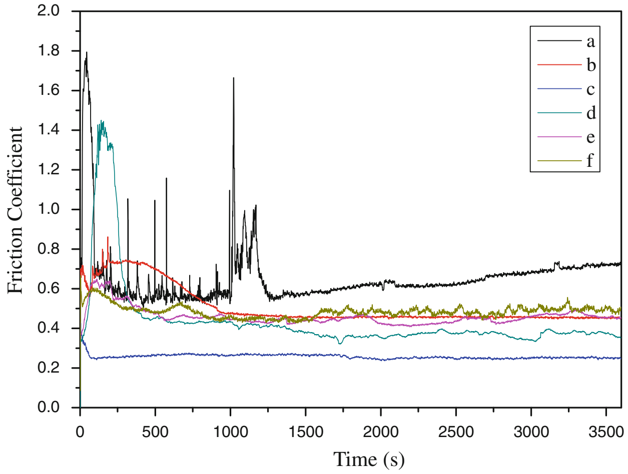

Fig. 7 Variation in dry COF with time for various samples: **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7%HTPDMS + 3%PTW, **e** PU + 7%HTPDMS + 5%PTW, **f** PU + 7%HTPDMS + 7%PTW (Load: 20 N, Sliding Speed: 1.031 m/s, time: 120 min)

Friction and wear behavior of the PTW-reinforced PU composites

Figure 7 shows the variation of the COF with sliding time at a normal load of 20 N and sliding speed of 1.031 m/s under dry sliding for PU composites reinforced with different proportion of PTW. The composite exhibited a lower COF compared to pure PU. However, with the addition of PTW, the COF increased. For example, when the PTW content was 1%, the COF was the lowest, decreased 62% compared to pure PU. It indicated that the small content of PTW helped decreasing the COF of the PU. As for the sliding wear of polymers against metallic counterparts, the friction component resulting from adhesion equals the product of the real contact area and the shear stress of

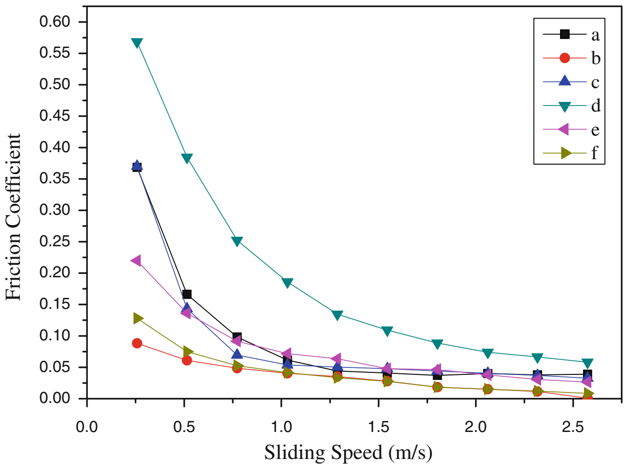

Fig. 8 Variations in the COF of the PU composites under water lubrication (20 N and different sliding speeds): **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7% HTPDMS + 3%PTW, **e** PU + 7%HTPDMS +5%PTW, **f** PU + 7%HTPDMS + 7%PTW

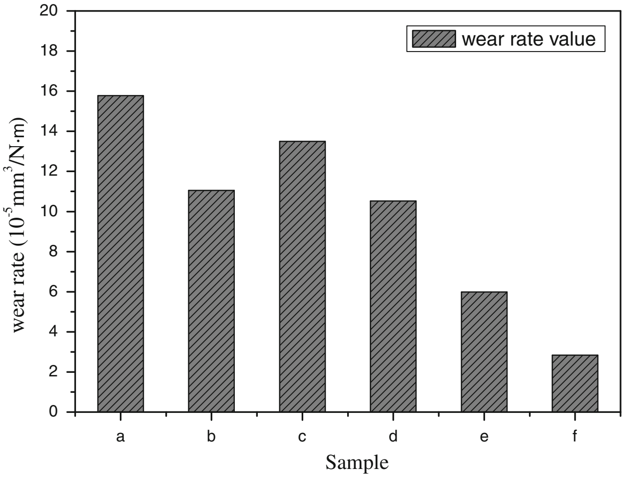

Fig. 9 Variations in the wear rate value of the PU composites: **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7%HTPDMS + 3%PTW, **e** PU + 7%HTPDMS + 5%PTW, **f** PU + 7%HTPDMS + 7%PTW

the softer material [32]. However, the PTW always increase the abrasive force and decrease during the real contact area during the friction process [33, 34]. Therefore, the COF increased with the increasing of the PTW contents. At preliminary stage of friction, the COF oscillated up and down. The reason for this phenomenon was that the surface of metal ring was not very smooth and the ploughed function on friction test caused by the bulge on the surface of the ring was large. At the beginning, the transfer film was not formed between the metal counterpart and the polymer composites. However, as the time of test went by, the stable transfer film was formed; the COF declined and tended to stable stage.

Figure 8 shows the variations in the COF of the PU composites under water lubrication with different sliding speeds at a normal load of 20 N. Under water lubrication, the COF gradually decreased with increasing the sliding speed. For example, the COF of 1%PTW/PU sharply decreased from 0.3703 to 0.0696 (decreased 81%) at the stage of low speeds, especially from 0.25 to 0.75 m/s. This is due to the effect of boundary lubrication. Water is not only a lubricant medium but also exports a large quantity of frictional heat. It can also be explained by the wettability of SnCu for water. With the sliding speed increasing, the COF tended to smooth, which was highly related to the wettability of friction surface. The fluid film, as a lubricant medium, gradually formed during the wear process. It is clear from experimental results, HTPDMS/PU and 7%PTW/PU have the lowest COF under water environment. It can be explained by the wettability and the hardness of the PU composite. As it is known, HTPDMS has the effect of hydrophobic which prevents the corrosion of water for PU matrix during the wear process. On the other hand, in aqueous environments, polymer plasticization often occurs and leads to the decrease in the hardness of polymer because of medium absorption. Therefore, the 7%PTW/PU, which was the hardest of the PU composites, exhibited the lowest COF.

Table 3 Dry COF and wear rate value of the PU composites

Sample	Dry COF	Dry wear rate (10^{-5}mm^3/N·m)
PU	0.66	15.783
PU + 7%HTPDMS	0.46	11.053
PU + 7%HTPDMS + 1%PTW	0.25	13.495
PU + 7%HTPDMS + 3%PTW	0.36	10.526
PU + 7%HTPDMS + 5%PTW	0.45	5.984
PU + 7%HTPDMS + 7%PTW	0.51	2.836

Figure 9 shows the variations in the wear rate value of the PU composites under dry sliding. Detailed data were listed in Table 3. It can be seen that the HTPDMS/PU and all PTW/PU composites hold the lower wear rate value than pure PU. It is well known that the chemical modification plays an important role on the tribological properties of the polyurethane. Besides, the wear rate value of PTW/PU reduced greatly with the increasing content of the PTW. This was due to the enhancement of PTW which exhibited higher strength and better worn-resistance. The possible reason was that the adhesion property between PTW and PU matrix was excellent and the dispersion degree of PTW in the composite was high.

Figure 10 shows the SEM micrographs of the worn surfaces of the PU composites under dry sliding. As seen from Fig. 10a, much debris appeared on the worn surface of the

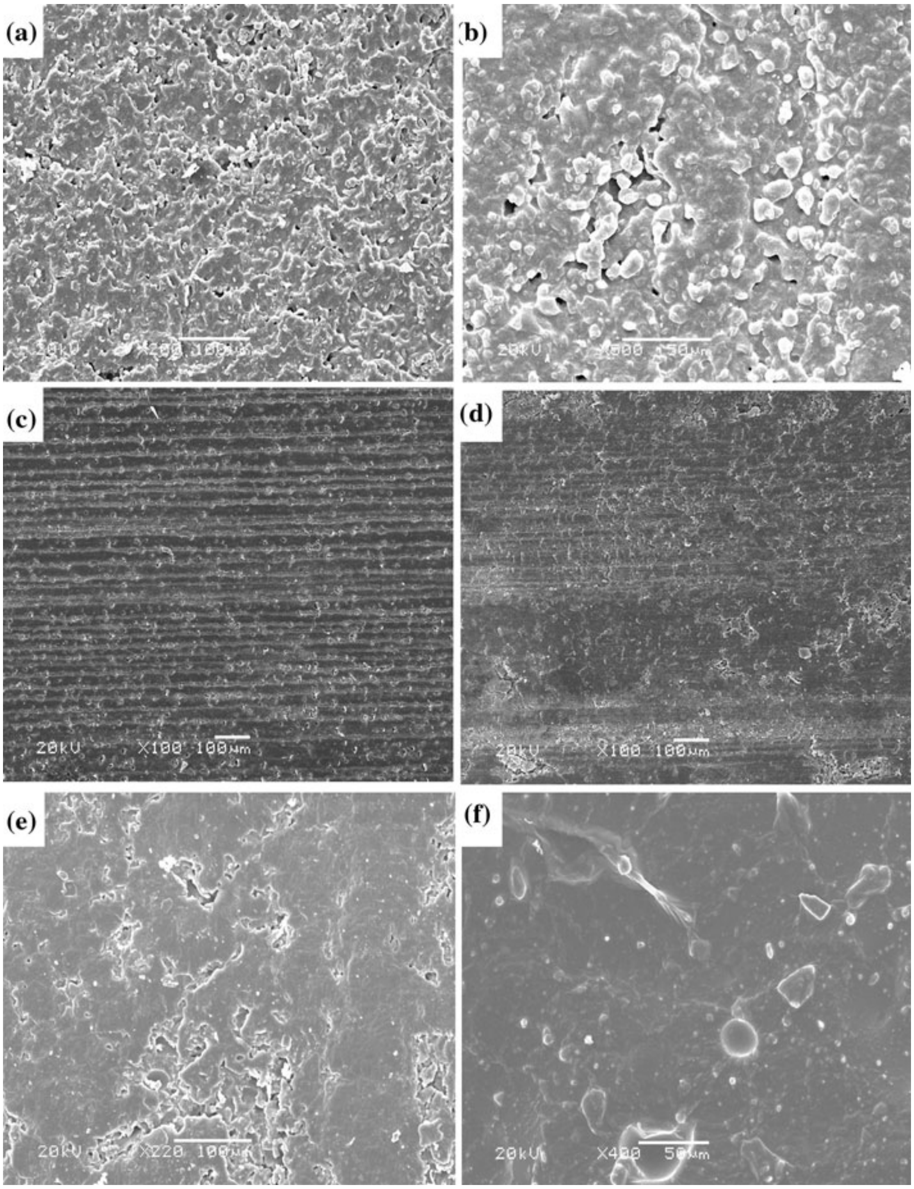

Fig. 10 SEM images of the worn surface of the PU composites. **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7%HTPDMS + 3%PTW, **e** PU + 7%HTPDMS + 5%PTW, **f** PU + 7%HTPDMS + 7%PTW

pure polyurethane, which indicated that fatigue wear played an important role. It was related to the heating effect of friction which promoted a temperature rise, softening the polymer surface, and as a result, the wear regime became a severe surface with wrinkling occurrence. When PU was modified by HTPDMS, the worn surface of the composite exhibited smoother. It was due to the thermal stability of HTPDMS [35] and chemical reaction with polyurethane. It is clear that the worn surface of PTW/PU composites became smoother with increasing content of PTW. With the addition of PTW, shown from Fig. 10c–f, the plucked and pit marks gradually appeared on the surface of the composite. This indicated that adhesive wear was primary wear mechanism which can explain the friction and wear phenomenon. It can be attributed to the mechanical properties of the composites improved by PTW. The PTW, which exposed to the surface of the composite, changed the hardness of the composites and played an important role of sharing shear pressure.

Figure 11 shows the SEM micrographs of the worn surfaces of the PU composites under water lubrication. As seen from Fig. 11, the worn surfaces of the PU composites under water lubrication were smoother than under dry sliding because of the lubrication and cooling effect of water medium. As shown in the SEM results, Pure PU and 7% HTPDMS-modified PU exhibited smoothing worn surface. It indicated that water had little corrosion effect on

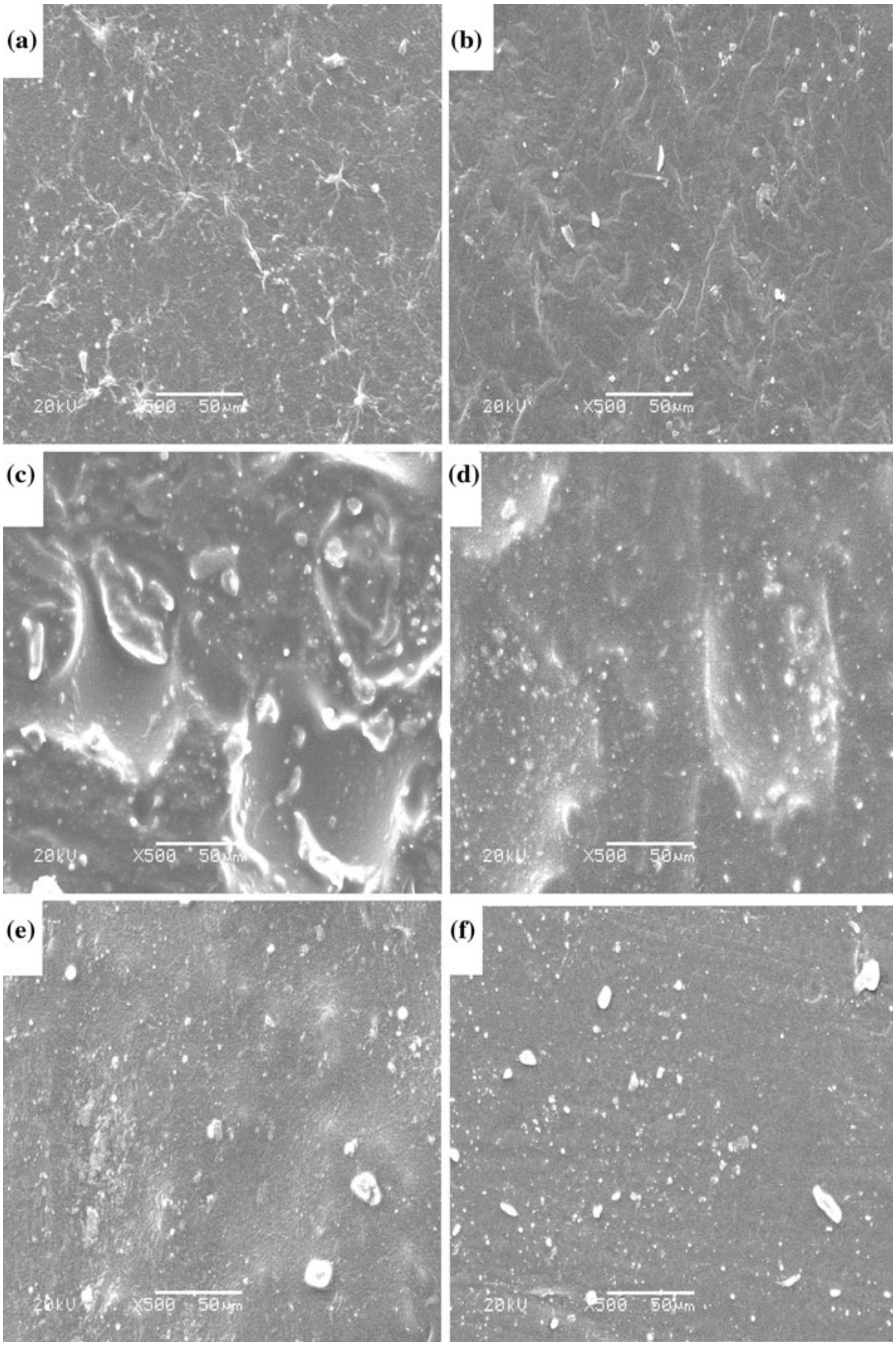

Fig. 11 SEM images of the worn surface of the PU composites under water lubrication. **a** PU, **b** PU + 7%HTPDMS, **c** PU + 7%HTPDMS + 1%PTW, **d** PU + 7%HTPDMS + 3%PTW, **e** PU + 7%HTPDMS + 5%PTW, **f** PU + 7%HTPDMS + 7%PTW

the pure PU. However, PTW-filled PU composites had much debris on the worn surface. It can be attributed to the hydrolysate and corrosion during the wear process which resulted in separation between PTW and PU matrix. As a result, the formation of the water films became difficult because the surface roughness of the counterface increased. The smoother the friction surface is, the more beneficial the formation of the fluid film is. In a word, compared with dry sliding, PTW/PU composites exhibited good friction and wear properties under water lubrication.

Conclusions

When the polyurethane was reinforced with chemical treatment PTW or modified by HTPDMS, the tensile strength of the PU composites increased. The influence on the COF and wear rate value of the PU composites reinforced by PTW was studied in this work. With the increasing of the PTW, the COF of the PU composites increased, but wear rate value clearly decreased. With increasing of sliding speed under water lubrication, the COF of the composites decreased. The sliding speed showed stronger effect on the COF of the composites under water lubrication at the same load. This made them to be used under water environment at high speed. Under dry sliding, worn surface of the tested materials showed that the fatigue wear and adhesive wear played an important role for the composites together. Under water lubrication, the wear mechanism was a combination of boundary lubrication and elastic fluid theory. Importantly, chemical treatment for PTW reduced phase separation between PTW and polymer matrix in the composite. Therefore, the tensile strength of the PU composites, which reinforced with PTW, improved greatly.

Acknowledgements The authors would like to acknowledge the financial support of the National Science Foundation for Distinguished Young Scholars of China (Grant No. 51025517), the Innovative Group Foundation of NSFC (Grant No. 50721062) and the financial support of the National 973 project of China (2007CB607606).

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