Research Article

Tribological Performance and Lubrication Mechanism of Alkylimidazolium Dialkyl Phosphates Ionic Liquids as Lubricants for Si_3N_4 - Ti_3SiC_2 Contacts

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The tribological performance of Si_3N_4 - Ti_3SiC_2 contacts lubricated by alkylimidazolium dialkyl phosphates ionic liquids (ILs) was investigated using an Optimol SRV-IV oscillating reciprocating friction and wear tester at room temperature (25°C) and 100°C. Glycerol and tributyl phosphate (TBP) were also selected as lubricants for Si_3N_4 - Ti_3SiC_2 contacts to study the tribological properties under the same experimental conditions for comparison. Results show that the alkylimidazolium dialkyl phosphates ILs were effective in reducing the friction and wear for Si_3N_4 - Ti_3SiC_2 contacts, and their performance is superior to that of glycerol and TBP. The SEM/EDS and XPS results reveal that the excellent tribological endurance of alkylimidazolium dialkyl phosphates ILs is mainly attributed to the high load-carrying capacity of the ILs and the formation of surface protective films consisting of TiO_2 , SiO_x , titanium phosphate, amines, and nitrogen oxides by the tribochemical reactions.

1. Introduction

It is known that Ti_3SiC_2 is the most studied material in the system of $\text{M}_{n+1}\text{AX}_n$ ($n = 1-3$) phases, which has a layered crystal structure and possesses the properties of both metals and ceramics. The unique properties of Ti_3SiC_2 include high strength and modulus, good damage tolerance at room temperature (RT), excellent thermal shock resistance, good electrical and thermal conductivities, easy machinability, good oxidation resistance, and low density. These unusual combinations of the properties render it a candidate structural material for high temperature applications [1–5].

Interestingly, all these unique properties of Ti_3SiC_2 are attributed to its layered structure, which is similar to that of graphite and MoS_2 . Thus, it is proposed that Ti_3SiC_2 is a self-lubricating material and possesses low friction coefficient [6, 7], and the related research about Ti_3SiC_2 has been

reported with the measurement of its mechanical properties [8, 9]. However, limited work has been carried out to evaluate the tribological behavior of Ti_3SiC_2 with liquid lubricants, which mainly focuses on the liquid lubricants of water and alcohol [10–16]. Hibi reveals that both the friction and wear of Ti_3SiC_2 -SiC composite are much lower with the lubrication of ethanol than that of water, even under dry condition [14]. Unfortunately, water and alcohol could not be used under extreme conditions, such as wide temperature changes and high load.

Recently, a program has been started to illustrate the tribochemical reactions of Ti_3SiC_2 lubricating by several liquid lubricants which could be used under extreme conditions. Ionic liquids (ILs) as a unique member in the family of liquid lubricants have received increasing attention in the academic and industrial tribology fields owing to their intrinsic characteristics, such as negligible volatility, nonflammability, high

thermal stability, and low melting point. An explosive growth of researches on ILs has been conducted in the past few years [17–22].

Research shows that the phosphates can be used to improve the antiwear and load-carrying capacity of liquid lubricant for ceramic materials [23]. Wei and Xue found that tributyl phosphate (TBP) has a little antiwear function for lubrication of ceramic because of their physical adsorption on the rubbing surfaces [24]. ILs with functional group of phosphate also can perform good tribological properties, especially at a moderate temperature [25].

Bearing this in mind, the alkyimidazolium dialkyl phosphates ILs were synthesized and evaluated as lubricants for Si_3N_4 - Ti_3SiC_2 contacts. Glycerol and TBP were also investigated under the same conditions for comparison. The details of the wear mechanism have been studied using scanning electron microscope with a Kevex energy dispersive X-ray analyzer attachment (SEM) and X-ray photoelectron spectrometer (XPS).

2. Experimental Details

2.1. Materials Preparation. The bulk Ti_3SiC_2 sample was prepared using an in situ hot pressing/solid-liquid reaction process starting from Ti, Si, and graphite powders and Al powder (3.1 mol%) as a sintering additive [15, 26]. Three kinds of powders with stoichiometric quantities were weighed, ball-milled, and then hot-pressed at 1450°C , 25 MPa in a graphite die. The home-made Ti_3SiC_2 has the relative density of 96.4%, hardness of 4.66 GPa, and surface roughness (R_a) of $0.24\text{ }\mu\text{m}$. The composition of Ti_3SiC_2 was determined by D/Max-2400 (Japan) X-ray diffraction (XRD). The XRD spectrum of Ti_3SiC_2 sample is shown in Figure 1, and it can be seen that the structure of sample is polycrystalline with a small amount (less than 3 wt.%) of TiC impurity.

The alkyimidazolium dialkyl phosphates ILs were synthesized according to the references [27, 28], and the synthesized route and molecular structures of them are shown in Figure 2. Glycerol and TBP were selected with the analytical reagent.

Several typical physical properties of the lubricants are shown in Table 1. The kinematic viscosities of the lubricants were measured using an SYP1003-III kinematic viscosity of petroleum-product measuring apparatus at 40°C and 100°C . Thermogravimetric analysis (TGA) was performed on a Perkin-Elmer TGA-7 conducted in nitrogen atmosphere from 20°C to 600°C at a rate of $10^\circ\text{C}/\text{min}$. The volatilization losses of all used lubricants were tested at 100°C for 24 h according to the standard method of ASTM D972.

2.2. Friction and Wear Tests. Friction and wear tests were conducted on an Optimol SRV-IV oscillating reciprocating friction and wear tester with a ball-on-disc configuration at RT (25°C) and at 100°C . The upper ball was made of Si_3N_4 (G5 according to ANSI/AFBMA Std 10-1989 with surface roughness R_a of $0.02\text{ }\mu\text{m}$) with a diameter of 10 mm. The surface roughness (R_a) of the Ti_3SiC_2 disc is $0.04\text{ }\mu\text{m}$. Both the balls and discs were cleaned ultrasonically in ethanol for

FIGURE 1: XRD spectrum of Ti_3SiC_2 .

30 min and allowed to dry before testing. The contact between frictional pairs was achieved by pressing the upper running ball against the lower stationary disc. The upper running ball was reciprocated at a constant frequency and stroke.

All the SRV tests were conducted under the following condition: load of 100 N, frequency of 25 Hz, amplitude of 1 mm, duration of 30 min, and a relative humidity of 20–50%. Before the tests, 0.2 mL liquid lubricants were dropped onto the ball-disc contact area. The corresponding friction curves were recorded automatically with a charter attached to the SRV test rig. The wear volumes of lower discs were measured using a MicroXAM-3D noncontact surface mapping microscope profilometer. Three repetitive measurements were carried out under each test condition, and the average values are reported in this paper.

2.3. Measurements and Analysis. The morphologies of worn surfaces were observed by JSM-5600LV scanning electron microscopy (SEM). The X-ray photoelectron spectrometer (XPS) analysis was carried out on a PHI-5702 multifunctional XPS, using Al-Ka radiation as the exciting source. The binding energies of the target elements were determined at pass energy of 29.35 eV, and the resolution is about $\pm 0.3\text{ eV}$ with the binding energy of contaminated carbon (C 1s: 284.8 eV) as reference.

3. Results and Discussion

3.1. Physical Properties of ILs. Table 1 shows the physical properties of the alkyimidazolium dialkyl phosphates ILs. It can be seen that all the ILs compounds have a good viscosity index as well as a high decomposition temperature compared with glycerol and TBP. When the alkyl chains of anions increased, the viscosity of ILs became higher. And the order of the viscosity for these lubricants at 100°C is as follows: TBP < Glycerol < PEE < PBE < POE and the details were shown in

FIGURE 2: Synthesized route of the ILs of alkylimidazolium dialkyl phosphates (a) and molecular structures of TBP (b).

TABLE 1: Physical properties of the alkylimidazolium dialkyl phosphates ILs.

Lubricants	Kinetic viscosity/mm ² ·s ⁻¹			Properties	
	40°C	100°C	VI	Density/Kg·m ⁻³	TGA/°C (onset decomposition temperature)
PEE	138	17.1	129	1125	275
PBE	231	23.9	115	1038	281
POE	267	28.4	117	1016	274
Glycerol	246	13.7	4.9	1258	156
TBP	3.0	1.1	—	979	118

“—” Could not be calculated.

Table 1. And it is indicated that the alkylimidazolium dialkyl phosphates ILs were superior to the glycerol and TBP as lubricants for ceramic materials which can be used under wide temperature conditions.

The volatilization losses results of these lubricants are listed in Table 2. It can be seen that the PEE IL has the least weight loss (0.54%) among the alkylimidazolium dialkyl phosphates ILs. TBP has the worst thermal stability with a weight loss of 53.4% and the glycerol exhibits the thermal stability with a weight loss of 8.49%. These results illustrate that the alkylimidazolium dialkyl phosphates ILs possess better thermal stability and are more suitable to be used as high-temperature lubricants compared to glycerol and TBP.

3.2. Tribological Behavior

3.2.1. Tribological Behavior of ILs. Figures 3 and 4 exhibited the evolution of friction coefficients for Si₃N₄-Ti₃SiC₂ contacts and wear volumes of Ti₃SiC₂ discs lubricated by different ILs at 25°C and 100°C under the load of 100 N. It can be seen from Figure 3 that POE performed the highest friction coefficient among the three kinds of ILs, and the other two ILs showed the similar evolution of friction coefficients at RT. However, the lowest wear loss of ILs was obtained by the lubrication of POE, while PEE showed the highest wear loss. When the test temperature increased to 100°C, it could be found from Figure 4 that the friction coefficient decreased remarkably as the viscosity decreased. However, the higher viscosity is benefit to improve the antiwear performance of the lubricants. Moreover, POE still exhibited the highest friction coefficient and the lowest wear loss within the three kinds of ILs. It can also be seen that PEE showed lower

TABLE 2: Volatilization losses of the lubricants (100°C, 24 hours).

Lubricants	PEE	PBE	POE	TBP	Glycerol
Mass loss (%)	0.54	0.97	2.9	53.4	8.49

friction coefficient and lower wear loss compared to the other two kinds of ILs at 100°C.

In our previous work [29], it is found that anions in ILs are always involved in the tribochemical reactions, and the presence of reaction products on the worn surface makes the ILs to be good lubricants. Liu et al. also found that the structure of alkylimidazolium cation had an important effect on the tribological properties of ILs [30]. In this study, based on the aforementioned results, a conjecture can be drawn which describes that the anion with shorter alkyl chain shows higher reactivity than the anion with longer alkyl chain in the molecular structure of ILs. It also shows that lower viscosity would produce lower friction coefficient and higher wear loss under the tested conditions. Accordingly, the anion with longer alkyl chain in the molecular structure of ILs would show higher friction coefficient and the lower wear loss. In addition, ILs with longer alkyl chains in cations show the higher viscosity, which might cause higher friction coefficient and lower wear loss. Moreover, it can be seen that ILs with higher viscosity would show stronger load-carrying capacity under boundary lubrication condition from the above tribological results, which means reduction in friction coefficient depended on lower viscosity of ILs and ILs with higher viscosity showed good antiwear performance. Thus, it can also be found that the above mentioned two effects, high

FIGURE 3: Evolution of friction coefficients (a) and wear volumes (b) of Ti_3SiC_2 discs lubricated by different ILs at 25°C under 100 N.

FIGURE 4: Evolution of friction coefficients (a) and wear volumes (b) of Ti_3SiC_2 discs lubricated by different ILs at 100°C under 100 N.

reactivity and high viscosity, have exhibited more obviously synergistic effects while the temperature increased.

3.2.2. Comparison of Tribological Behavior with Glycerol and TBP. To verify the hypothesis that is mentioned above, the tribological experiments of TBP were also done under the same conditions, and the molecular structure of TBP is similar to the part of anion in ILs of PBE. Glycerol, which was an extensively investigated lubricant for Ti_3SiC_2 , was also tested as a comparison with ILs. The compared results of the two kinds of lubricants with PBE were summarized in Figures 5 and 6. It can be found that TBP showed much higher friction coefficient and higher wear loss both at 25°C and at 100°C, which is attributed to the simple molecular structure and poor load-carrying capacity of TBP compared to that

of PBE. The results were consistent to the above hypothesis that ILs with longer alkyl chains in cations would show greater load-carrying capacity under boundary lubrication condition, which means reduction in friction coefficient and wear loss. Compared with glycerol, it could be found from Figures 5 and 6 that PBE exhibited more steady friction coefficient and lower wear loss.

As a result, the high thermal stability and excellent tribological property of alkylimidazolium dialkyl phosphates ILs made it an attractive alternative to liquid lubricants for Si_3N_4 - Ti_3SiC_2 contacts.

3.3. Surface Analysis. SEM and three-dimensional (3D) non-contact surface measurement were employed to examine the morphologies of the wear tracks on the Ti_3SiC_2 discs. Figure 7

FIGURE 5: Evolution of friction coefficients (a) and wear volumes (b) of Ti_3SiC_2 discs lubricated by PBE, TBP, and glycerol at 25°C under 100 N.

FIGURE 6: Evolution of friction coefficients (a) and wear volumes (b) of Ti_3SiC_2 discs lubricated by PBE, TBP, and glycerol at 100°C under 100 N.

shows the SEM and 3D optical microscopic images of the worn surfaces lubricated by POE and glycerol under a load of 100 N at 100°C. It can be seen that the worn surfaces under the lubrication of glycerol presented severe fracture and oxidation wear, whereas the worn surfaces lubricated by POE exhibited slight wear. These results highly agreed with those of high-temperature experimental results and further indicated that POE could effectively improve the wear-resistance ability for Si_3N_4 - Ti_3SiC_2 contacts. Figures 7(c) and 7(f) show the 3D optical microscopic images of the corresponding wear scars, which could clearly observe the wear scenario under the lubrication of different lubricants. The worn surfaces lubricated by glycerol (Figure 7(f)) exhibited considerably wider wear scars; however, the wear scar that is lubricated

by POE (Figure 7(c)) was narrow and deeper. This result further confirms the excellent antiwear properties of POE, corresponding with the results of SEM.

To gain further insights into the complex tribochemistry of the Si_3N_4 -ILs- Ti_3SiC_2 interface, the worn surfaces of the Ti_3SiC_2 disc lubricated with ILs were analyzed by XPS (Figure 8). The XPS analyses of P2p and N1s for neat POE were conducted before the tribo-tests (A), and the XPS spectra of Ti2p, Si2p, P2p, and N1s for POE were obtained after the tribo-tests (B). It can be seen that the XPS spectra of Ti2p peak at 454.50 eV correspond to Ti_3SiC_2 , and the binding energy of Ti2p at 458.00 eV and 464.10 eV would be ascribed to TiO_2 [16]. In addition, titanium phosphate (458.80 eV) [31] could be generated on the worn surface

FIGURE 7: SEM and 3D morphologies of worn surfaces lubricated by POE and glycerol (SRV: 100 N, 100°C).

during the friction process because of the interaction between the substrate and the IL lubricant.

However, a decrease for the main binding energy of the Si2p peaks occurs after the friction tests, which is obtained from the comparison with the previous reported result [16]. It can be seen that the peak at 99.50 eV corresponds to SiO_2 , and the peak at 100.60 eV is referred to Si_3N_4 . There is also SiO_x present on the worn surface.

The binding energies of N1s undergo some changes compared with neat phosphate IL (the binding energy of N1s for POE appears at 401.20 eV). The appearance of N1s peaks at 398.70 eV after friction can be ascribed to Si_3N_4 , which is in agreement with the Si2p peak at 100.60 eV. Amines and nitrogen oxides (400.90 eV) [32] are produced because of the high reactivity of cation moiety. However, there is little difference for the main binding energy of the P2p peaks of phosphate IL which occurred after the friction tests. Based on the binding energy of P2p at 133.25 eV, we can infer that phosphate anions are capable of reacting with Ti to form $\text{Ti}_3(\text{PO}_4)_4$.

Based on the above analysis, it can be concluded that ILs are easily adsorbed on the sliding surface of frictional pairs to form strongly ordered adsorbed films. Tribochemical reactions occur during the friction process, thus forming effective boundary-lubricating films which consisted of TiO_2 , SiO_x , titanium phosphate, amines, and nitrogen oxides. Hence, the friction and wear of Ti_3SiC_2 materials are reduced.

4. Conclusions

Sliding friction experiments were carried out with the lubrication of alkylimidazolium dialkyl phosphates ILs. Based on the above experimental results, the following conclusions are drawn.

- (1) The alkylimidazolium dialkyl phosphates ILs were effective for Si_3N_4 - Ti_3SiC_2 contacts lubrication and are superior to the glycerol and TBP both at 25°C and 100°C; the high thermal stability and excellent tribological properties of alkylimidazolium dialkyl phosphates ILs made it an attractive alternative to liquid lubricants for Si_3N_4 - Ti_3SiC_2 contacts.
- (2) The friction coefficient and wear loss for Si_3N_4 - Ti_3SiC_2 contacts decreased, which is attributed to the shorter alkyl chains of anion and the longer alkyl chains of cation in alkylimidazolium dialkyl phosphate ILs.
- (3) The tribological mechanisms of alkylimidazolium dialkyl phosphates ILs were mainly attributed to the load-carrying capacity of the ILs and the formation of surface protective films which consisted of TiO_2 , SiO_x , titanium phosphate, amines, and nitrogen oxides by the tribochemical reactions.

FIGURE 8: XPS spectra of the worn Ti_3SiC_2 surfaces lubricated by POE under 100 N and 100°C (SRV: 100 N, 100°C).

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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