

## Research Article

# Tribological Behavior of $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$ Contacts Lubricated by Lithium-Based Ionic Liquids

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Received 28 March 2014; Accepted 6 August 2014; Published 27 August 2014

Academic Editor: Peter Majewski

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The tribological performance of  $\text{Si}_3\text{N}_4$  ball sliding against  $\text{Ti}_3\text{SiC}_2$  disc lubricated by lithium-based ionic liquids (ILs) was investigated using an Optimal SRV-IV oscillating reciprocating friction and wear tester at room temperature (RT) and elevated temperature (100°C). Glycerol and the conventional imidazolium-based IL 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (L-F106) were used as references under the same experimental conditions. The results show that the lithium-based ILs had higher thermal stabilities than glycerol and lower costs associated with IL preparation than L-F106. The tribotest results show that the lithium-based ILs were effective in reducing the friction and wear of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts. [Li(urea)]TFSI even produced better tribological properties than glycerol and L-F106 both at RT and 100°C. The SEM/EDS and XPS results reveal that the excellent tribological endurance of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts lubricated by lithium-based ILs was mainly attributed to the formation of surface protective films composed of various tribochemical products.

## 1. Introduction

Titanium silicon carbide ( $\text{Ti}_3\text{SiC}_2$ ) is the most studied material in the  $M_{n+1}AX_n$  ( $n = 1$  to  $3$ ) phase system, which combines the prominent properties of both metal and ceramic. Similar to metal,  $\text{Ti}_3\text{SiC}_2$  is electrically and thermally conductive, is relatively soft, and can be easily machined with standard tools. Similar to ceramic,  $\text{Ti}_3\text{SiC}_2$  is elastically stiff with relatively low hardness and light weight and can resist thermal shock, corrosion, and chemical attack [1–5]. Several researchers have studied the two-stage sliding friction and wear process using  $\text{Ti}_3\text{SiC}_2$  against various counterparts under dry sliding condition or its tribological behaviors under various solutions. For example, Crossley and coworkers measured ultralow friction coefficients of ceramic  $\text{Ti}_3\text{SiC}_2$  using lateral force microscopy and showed that  $\text{Ti}_3\text{SiC}_2$  is an exceptional solid lubricant [6]. Zhang et al. reported the dry sliding friction coefficient of the self-mated  $\text{Ti}_3\text{SiC}_2$  tribocouple, which ranges from 1.16 to 1.43, and that of

$\text{Ti}_3\text{SiC}_2/\text{diamond}$ , which is below 0.1 under varying loads [7]. Ren et al. investigated the tribocorrosion behaviors of  $\text{Ti}_3\text{SiC}_2/\text{Si}_3\text{N}_4$  in hydrochloric acid and sodium hydroxide solutions and reported that oxide films form on the worn surfaces of both  $\text{Ti}_3\text{SiC}_2$  and  $\text{Si}_3\text{N}_4$  by corrosive oxidation and/or tribooxidation [8]. Hibi et al. obtained a low friction coefficient of 0.2 for the self-mated  $\text{Ti}_3\text{SiC}_2/\text{SiC}$  during the initial friction and wear period under dry sliding conditions, as well as in water and alcohol [9]. Nevertheless, there are few studies on  $\text{Ti}_3\text{SiC}_2$  samples lubricated by ionic liquids (ILs).

It is well known that ILs have dipolar structure and unique physicochemical properties such as extremely low volatility, nonflammability, high thermal stability, and low melting points, which make them versatile tribomaterials with excellent friction-reducing and antiwear properties in lubrication engineering [10, 11]. When IL is introduced between two friction pairs, liquid films (physical adsorption films or tribochemical films) are developed between the contact surfaces and then prevent direct contact between

the friction pairs and reduce friction and wear. ILs can be used not only as high-performance neat lubricants for various materials such as metals, alloys, polymers, and hard coatings but also as lubricant additives and thin films [12–15]. In recent years, widespread attention has been devoted to ILs based on lithium salts because of their wide application in electrochemical devices, such as lithium batteries, electrochemical mechanical actuators, high-energy density batteries, photoelectrochemical solar cells, supercapacitors, and electrodeposits [16–25]. Our group first reported the application of lithium-based ILs as lubricants in the field of tribology [26–28]. These lubricants are synthesized by blending an appropriate molar ratio of lithium salts with organic compounds bearing acylamino groups (such as 2-oxazolidinone (OZO) and urea), synthetic esters (such as bis(2-ethylhexyl) adipate and pentaerythritol oleate), or oligoethers (such as triglyme (G3), tetraglyme (G4), and polyethylene glycol). This process significantly reduces the cost associated with IL preparation (the synthesis and purification of the most reported ILs are difficult and costly because of their complicated structures). In addition, these lithium-based ILs have similar physicochemical properties to conventional ILs, such as wide liquid-phase range, high thermal stability, negligible vapor pressure, nontoxicity, nonflammability, high ionic conductivity, and high electrochemical stability [18, 29, 30]. Our previous reports also showed that lithium-based ILs have excellent friction-reducing and antiwear properties [26–28]. Thus, this type of IL has potential application as lubricants or lubricant additives.

Our present work aimed to investigate the tribological characteristics of  $\text{Si}_3\text{N}_4$  sliding against  $\text{Ti}_3\text{SiC}_2$  under lubrication of lithium-based ILs, namely,  $[\text{Li}(\text{OZO})]\text{TFSI}$ ,  $[\text{Li}(\text{urea})]\text{TFSI}$ ,  $[\text{Li}(\text{G3})]\text{TFSI}$ , and  $[\text{Li}(\text{G4})]\text{TFSI}$ . Glycerol and the conventional imidazolium-based IL 1-hexyl-3-methyl-imidazolium bis(trifluoromethylsulfonyl)imide (L-F106) were selected for comparisons under the same experimental conditions. The lubrication mechanism was also demonstrated based on the data of scanning electron microscopy (SEM) and X-ray photoelectron spectrometry (XPS).

## 2. Experimental Details

**2.1. Preparation of Lubricants.** The chemical structures of LiTFSI, OZO, urea, G3, G4, and L-F106 are shown in Figure 1. L-F106 was prepared as previously described [31]. All other reagents were used as received without further purification. LiTFSI (99 wt. %), urea (99 wt. %), and G3 (99 wt. %) were purchased from J&K Chemicals Ltd. OZO (99 wt. %) was purchased from Alfa Aesar. G4 (98 wt. %) was purchased from Fluka. All lithium-based ILs, namely,  $[\text{Li}(\text{OZO})]\text{TFSI}$ ,  $[\text{Li}(\text{urea})]\text{TFSI}$ ,  $[\text{Li}(\text{G3})]\text{TFSI}$ , and  $[\text{Li}(\text{G4})]\text{TFSI}$ , were synthesized according to previously reported literatures [16–18]. They were prepared simply by blending an appropriate molar ratio of OZO, urea, G3 or G4, and LiTFSI together and then stirring at approximately 70°C until homogeneous liquids formed. The molar ratios for LiTFSI/OZO, LiTFSI/urea, LiTFSI/G3, and LiTFSI/G4

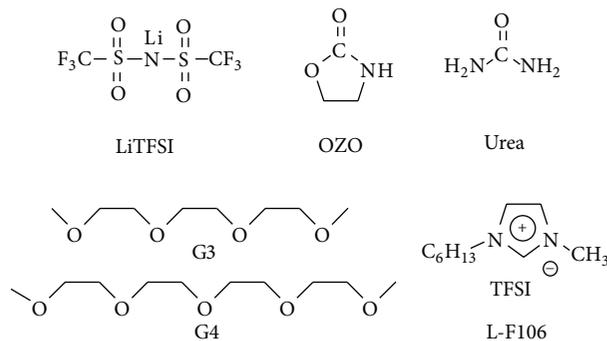


FIGURE 1: Molecular structures of LiTFSI, OZO, urea, G3, G4, and L-F106.

are 1.0/1.5, 1.0/3.0, 1.0/1.0, and 1.0/1.0, respectively [16–18]. For example,  $[\text{Li}(\text{OZO})]\text{TFSI}$  can be obtained by stirring 1.0 mol LiTFSI and 1.5 mol OZO together at 70°C until a homogeneous liquid ( $[\text{Li}(\text{OZO})]\text{TFSI}$ ) forms.

**2.2. Preparation of  $\text{Ti}_3\text{SiC}_2$ .** The bulk  $\text{Ti}_3\text{SiC}_2$  was prepared according to the previous reported literature [5]. It was homemade from the powders of Ti, Si, and C by solid-liquid reaction and simultaneous *in situ* hot pressing process. Three kinds of powders with stoichiometric quantities were weighed, ball-milled, and hot-pressed at 1450°C and 25 MPa in a graphite die. Al powder (3.1 mol %) was used to produce a liquid phase during sintering. The homemade  $\text{Ti}_3\text{SiC}_2$  had a relative density of 96.4%, hardness of 4.66 GPa, and surface roughness  $R_a$  of 0.24  $\mu\text{m}$ . The composition of  $\text{Ti}_3\text{SiC}_2$  was determined by D/Max-2400 (Japan) X-ray diffraction (XRD), and a sample with the polycrystalline structure was obtained with a small amount (less than 3 wt.%) of TiC impurity. Details of the  $\text{Ti}_3\text{SiC}_2$  sample were described in our previous work [32].

**2.3. Characterization.** The kinematic viscosities of the lubricants were measured using a SYP1003-III kinematic viscosity of petroleum-product measuring apparatus at 40°C and 100°C. Thermal behaviors were studied using STA 449C Jupiter-simultaneous thermogravimetry (TG) and differential scanning calorimetry. The samples were heated from room temperature (RT) to 600°C or 700°C at a rate of 10°C/min in air. The volatilization losses of all used lubricants were tested at 100°C for 24 h according to the method of ASTM D972.

**2.4. Friction and Wear Test.** 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 and 100°C. The upper specimen was a commercial  $\text{Si}_3\text{N}_4$  ball (G5 according to ANSI/AFBMA Std. 10-1989 with a surface roughness  $R_a$  of 0.02  $\mu\text{m}$ ) with 10 mm diameter. The lower stationary disc (24 mm  $\times$  7.9 mm) was homemade  $\text{Ti}_3\text{SiC}_2$  (98.9 wt.%) with hardness of 4.66 GPa and surface roughness  $R_a$  of 0.24  $\mu\text{m}$ . Contact between frictional pairs was achieved by pressing the upper running ball against the

TABLE 1: Physical properties of different lubricants.

Lubricants	Kinematic viscosity (mm <sup>2</sup> /s)		Properties	
	40°C	100°C	Viscosity index	TGA (Onset decomposition temperature °C)
Glycerol	246.4	13.7	4.9	155.8
[Li(OZO)]TFSI	999.3	39.5	65.9	237.3
[Li(urea)]TFSI	399.0	22.5	62.4	210.3
[Li(G3)]TFSI	74.6	11.2	140.7	194.1
[Li(G4)]TFSI	41.5	7.3	139.4	194.5

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

Lubricants	Glycerol	L-F106	[Li(OZO)]TFSI	[Li(urea)]TFSI	[Li(G3)]TFSI	[Li(G4)]TFSI
Mass loss	8.49%	0.0078%	0.041%	0.18%	2.73%	0.56%

lower stationary disc. The upper running ball reciprocated at an invariable frequency and displacement. The friction and wear tests in this experiment were conducted at amplitude of 1 mm, frequency of 25 Hz, test duration of 30 min, and relative humidity of 20% to 50%. Prior to the friction and wear test, a moderate amount of lubricant was placed onto the ball-disc contact area. The corresponding friction curves were automatically recorded by the SRV test rig. The wear volume losses on the lower discs were measured using a MicroXAM-3D noncontact surface mapping microscope profilometer. At least three replicates were carried out at each test condition. The average values are presented in this paper.

JSM-5600LV SEM was used to analyze the morphologies of the worn surfaces. XPS measurements were performed using a PHI-5702 multifunctional XPS, with Al-K $\alpha$  radiation as the excitation source. The binding energies of the target elements were determined at a pass energy of 29.35 eV and resolution of  $\pm 0.3$  eV, with the binding energy of contaminated carbon (C1s: 284.8 eV) as reference.

### 3. Results and Discussion

**3.1. Physical Properties.** Table 1 shows the physical properties of these lubricants. It can be seen that ILs of [Li(OZO)]TFSI and [Li(urea)]TFSI had relatively high kinematic viscosities at 40°C and 100°C, but moderate viscosity indexes (65.9 and 62.4, resp.). By contrast, [Li(G3)]TFSI and [Li(G4)]TFSI had moderate kinematic viscosities at 40°C and 100°C, but high viscosity indexes (140.7 and 139.4, resp.). Thus, lithium-based ILs from LiTFSI and oligoether compounds had better viscosity-temperature characteristics than those generated from LiTFSI and organic compounds bearing acylamino groups. For glycerol, its viscosity index was the lowest, which corresponded to the worst viscosity-temperature characteristics among the five lubricants. It also can be seen that the decomposition temperatures of these lithium-based ILs were significantly higher than the glycerol.

Table 2 shows the volatilization loss results of these liquid lubricants at 100°C for 24 h. It can be seen that the conventional IL L-F106 had the least weight loss (0.0078%). Lithium-based ILs, namely, [Li(OZO)]TFSI, [Li(urea)]TFSI, [Li(G3)]TFSI, and [Li(G4)]TFSI, showed

moderate volatilization losses from 0.041% to 2.73%. Glycerol exhibited the worst thermal stability with a weight loss of 8.49%. These results were consistent with the thermal properties of the compounds and further illustrate that L-F106 and lithium-based ILs were more suitable as high-temperature lubricants than glycerol.

### 3.2. Friction and Wear Test

**3.2.1. Room-Temperature Test.** The results of ball-on-disc tribotest (evolution of friction coefficients and wear volume losses) at RT are summarized in Figures 2(a) and 2(b). The experiments were carried out under a load of 100 N, amplitude of 1 mm, frequency of 25 Hz, and test duration of 30 min.

Figure 2(a) shows that all the lithium-based ILs synthesized in the present work effectively reduced the friction of Si<sub>3</sub>N<sub>4</sub>/Ti<sub>3</sub>SiC<sub>2</sub> contacts. These ILs exhibited slightly larger friction coefficients than glycerol under the same test conditions, but more stable friction traces during the entire sliding process. Compared with L-F106, [Li(urea)]TFSI registered a smaller friction coefficient, and other lithium-based ILs showed either similar or slightly larger friction coefficients. However, for the wear volume losses of Ti<sub>3</sub>SiC<sub>2</sub> discs under the lubrication of all lubricants shown in Figure 2(b), [Li(urea)]TFSI produced more than 50% less wear for Ti<sub>3</sub>SiC<sub>2</sub> disc than glycerol and L-F106. [Li(OZO)]TFSI also showed slightly better antiwear property than glycerol and L-F106, whereas [Li(G3)]TFSI and [Li(G4)]TFSI exhibited slightly larger wear volume losses among the six lubricants. The difference in tribological performances for the titled lithium-based ILs may be attributed to their cationic moiety. [Li(OZO)]<sup>+</sup> and [Li(urea)]<sup>+</sup> had more important functions in reducing friction and wear than [Li(G3)]<sup>+</sup> and [Li(G4)]<sup>+</sup> because of the heterocyclic or acylamino structures, which may provide firmer protective films on the sliding surfaces and further reduce friction and wear to some extent. The aforementioned results confirm the excellent tribological behaviors of lithium-based ILs, especially [Li(urea)]TFSI. From a practical perspective, these lithium-based ILs were more qualified for use as lubricants for Si<sub>3</sub>N<sub>4</sub>/Ti<sub>3</sub>SiC<sub>2</sub> contacts than L-F106 because the lithium-based ILs had lower

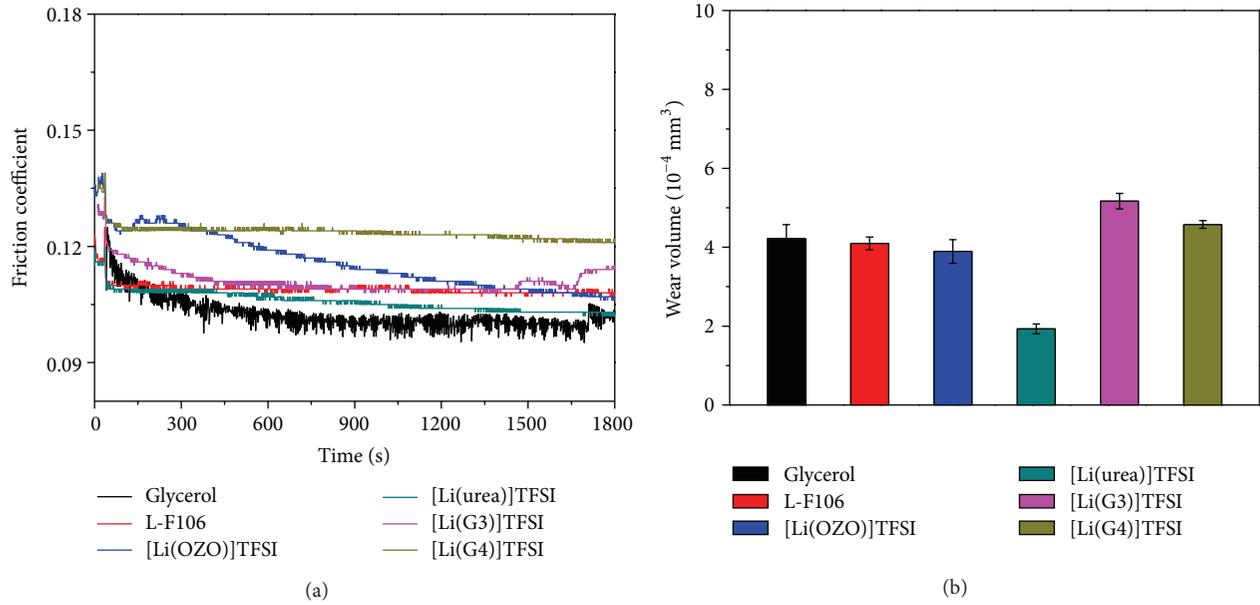


FIGURE 2: Evolution of friction coefficients (a) and wear volume losses (b) for  $\text{Ti}_3\text{SiC}_2$  discs lubricated by different lubricants at RT (load: 100 N, stroke: 1 mm, frequency: 25 Hz, and duration: 30 min).

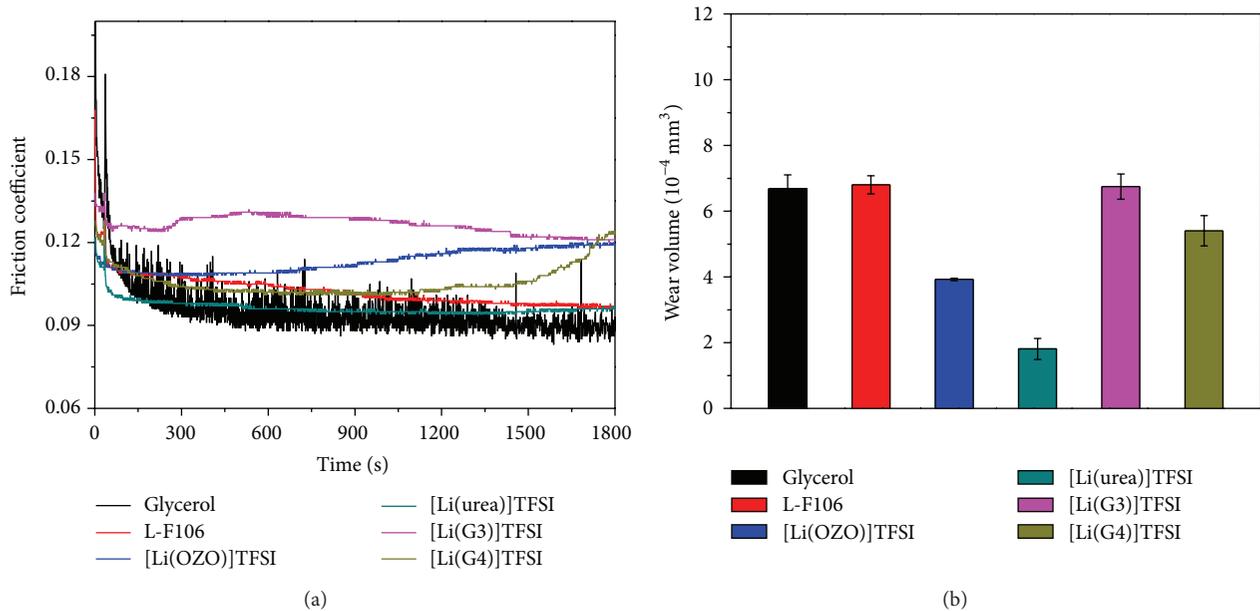


FIGURE 3: Evolution of friction coefficients (a) and wear volume losses (b) for  $\text{Ti}_3\text{SiC}_2$  discs lubricated by different lubricants at  $100^\circ\text{C}$  (load: 100 N, stroke: 1 mm, frequency: 25 Hz, and duration: 30 min).

costs associated with their preparation. The high thermal stability and excellent tribological property of [Li(urea)]TFSI also made it a qualified replacement for glycerol.

**3.2.2. High-Temperature Test.** The titled lithium-based ILs were expected to be suitable for the lubrication of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts at high temperature because of their high thermal stabilities. Thus, the tribotests were also carried out at  $100^\circ\text{C}$ .

Figure 3 shows the variations in the friction coefficients and wear volume losses of the  $\text{Si}_3\text{N}_4$  ball sliding against the  $\text{Ti}_3\text{SiC}_2$  disc under the lubrication of glycerol and ILs, respectively. Glycerol produced a significantly more unstable friction coefficient during the entire test process than that at RT. The friction-reducing and antiwear traces of all ILs were highly similar to those of RT test, which showed very stable friction behavior. [Li(urea)]TFSI also registered better friction-reducing and antiwear properties than glycerol and

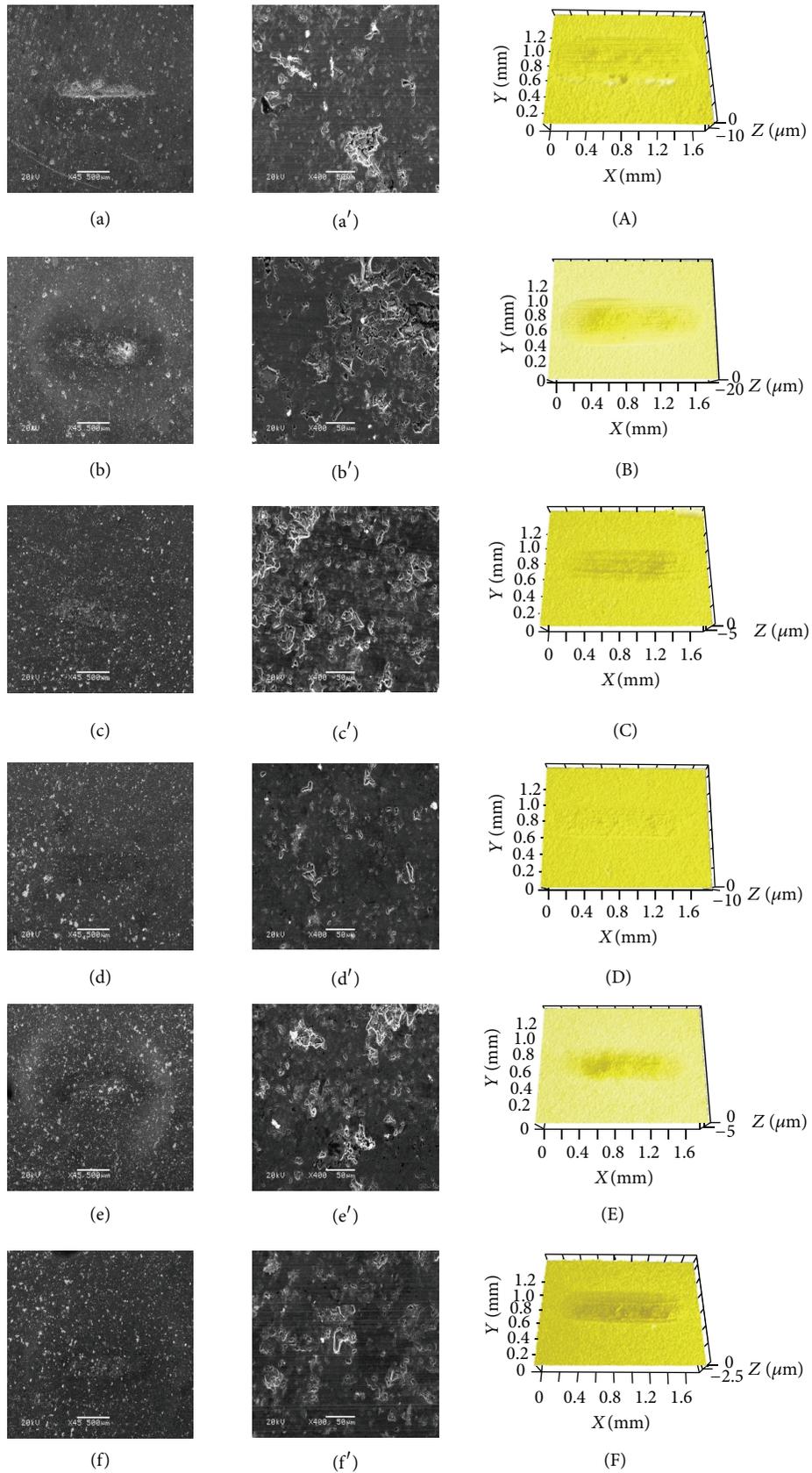


FIGURE 4: SEM and 3D morphologies of worn  $Ti_3SiC_2$  surfaces lubricated by different lubricants: ((a), (a'), and (A)) glycerol, ((b), (b'), and (B)) L-FI06, ((c), (c'), and (C)) [Li(OZO)]TFSI, ((d), (d'), and (D)) [Li(urea)]TFSI, ((e), (e'), and (E)) [Li(G3)]TFSI, and ((f), (f'), and (F)) [Li(G4)]TFSI (SEM magnification: 45x for the upper ones and 400x for the lower ones, load: 100 N, stroke: 1 mm, frequency: 25 Hz, duration: 30 min, and temperature: 100°C).

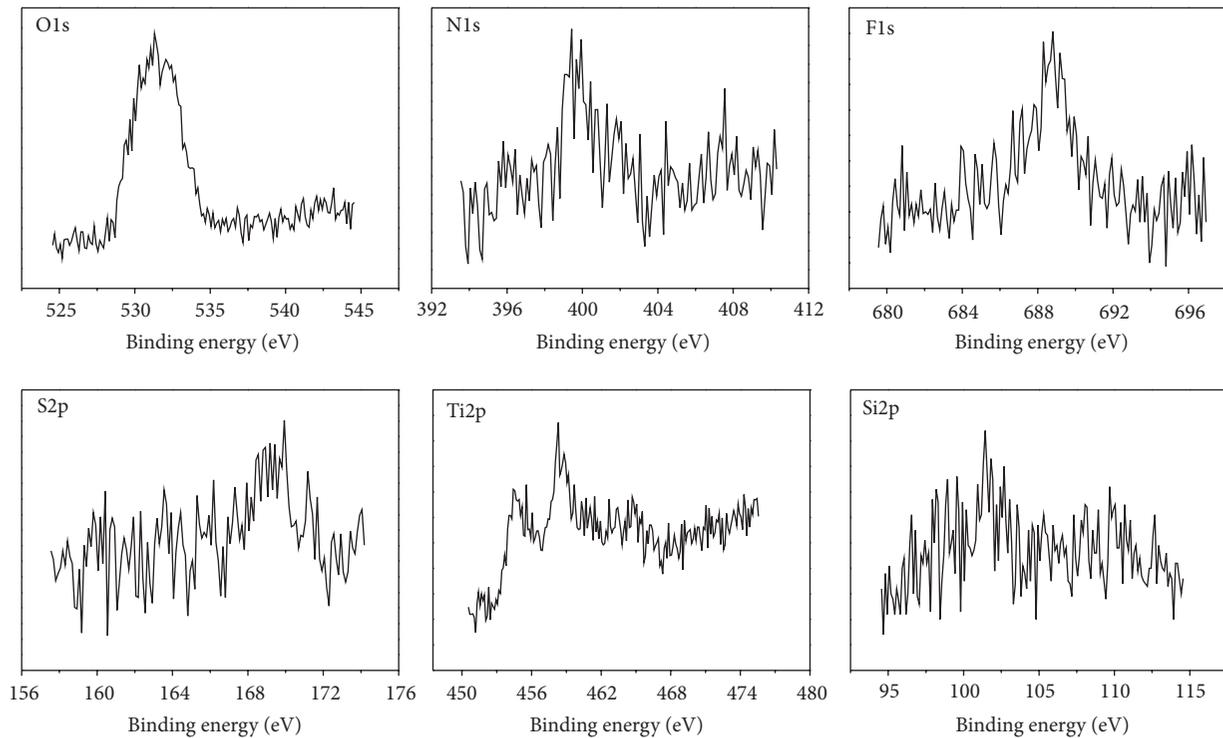


FIGURE 5: XPS spectra of the worn surfaces of  $\text{Ti}_3\text{SiC}_2$  disc lubricated by [Li(urea)]TFSI under 100 N and at  $100^\circ\text{C}$ .

other ILs. The results of RT and high-temperature tests show that [Li(urea)]TFSI could be used as an alternative lubricant to L-F106 and glycerol for the lubrication of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts.

**3.3. Surface Analysis.** SEM and three-dimensional (3D) noncontact surface measurement were employed to examine the morphologies of the wear tracks on the  $\text{Ti}_3\text{SiC}_2$  discs. Figure 4 shows the SEM and 3D optical microscopic images of the worn surfaces lubricated by glycerol, L-F106, [Li(OZO)]TFSI, [Li(urea)]TFSI, [Li(G3)]TFSI, and [Li(G4)]TFSI under a load of 100 N at  $100^\circ\text{C}$ . All the images were obtained at the same operating conditions. As shown in Figures 4(a) to 4(f), wear scars were observed on the worn surfaces lubricated by glycerol, L-F106, and [Li(G3)]TFSI (Figures 4(a), 4(b), and 4(e)). However, the worn surfaces under the lubrication of [Li(OZO)]TFSI, [Li(urea)]TFSI, and [Li(G4)]TFSI showed relatively slight signs of wear (Figures 4(c), 4(d), and 4(f)). Figures 4(a') to 4(f') show that the worn surfaces under the lubrication of glycerol, L-F106, [Li(OZO)]TFSI, [Li(G3)]TFSI, and [Li(G4)]TFSI presented severe fracture and pullout of grains (Figures 4(a'), 4(b'), 4(c'), 4(e'), and 4(f')), whereas that lubricated by [Li(urea)]TFSI was smoother but numerous small pits were visible after undergoing the same process (Figure 4(d')). These results highly agreed with those of high-temperature tribotests and further indicate that [Li(urea)]TFSI could effectively improve the wear-resistance ability of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts. Figures 4(A) to 4(F) show

the 3D optical microscopic images of the corresponding wear scars, which clearly show the wear scenario under the lubrication of different lubricants. The worn surfaces lubricated by glycerol and L-F106 (Figures 4(A) and 4(B)) exhibited considerably wider wear scars. By contrast, others (Figures 4(C), 4(D), 4(E), and 4(F)), especially for [Li(urea)]TFSI (Figure 4(D)), were relatively narrow and shallow; thus, scuffing was greatly alleviated in this case. This result further confirms the results of SEM and indicates that the lithium-based ILs, especially [Li(urea)]TFSI, possess the excellent antiwear properties.

XPS analysis of the worn  $\text{Ti}_3\text{SiC}_2$  surfaces was performed to verify the chemical states of typical elements after the lubrication of lithium-based ILs. The XPS spectra of O, N, F, S, Ti, and Si for the worn surfaces lubricated by [Li(urea)]TFSI are given as an example (Figure 5). The characteristic binding energy of O1s on the worn surfaces between 530.3 and 532.5 eV corresponds to the oxidation products [33]. The peak at 399.7 eV in the N1s spectrum indicates the presence of nitride. The binding energy of F1s at approximately 688.7 eV corresponds to  $\text{F}^-$ , possibly because of the formation of ionic compounds, such as  $\text{TiF}_4$ . The peak of S2p mainly appears at 169.3 eV and can be identified as  $\text{SO}_4^{2-}$ . The peaks of Ti2p on the worn surfaces lubricated by [Li(urea)]TFSI at 454.8 and 458.5 eV, combined with the binding energy of O1s, are assigned to  $\text{Ti}_3\text{SiC}_2$  and TiO or  $\text{TiO}_2$  [34]. On the XPS spectrum of Si2p, the peak at approximately 101.4 eV, combined with the binding energies of O1s and N1s, corresponds to  $\text{SiO}_2$  or  $\text{Si}_3\text{N}_4$ . Based on the aforementioned XPS results, complicated tribochemical reaction occurred during

the rubbing process. The excellent tribological behavior of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts under the lubrication of lithium-based ILs was attributed to the formation of a surface protective film composed of fluoride, sulfate,  $\text{TiO}$ ,  $\text{TiO}_2$ ,  $\text{SiO}_2$ , or/and  $\text{Si}_3\text{N}_4$ , which had an important function in reducing friction and wear of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts.

#### 4. Conclusions

In this work, we studied the friction and wear behavior of  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts under the lubrication of lithium-based ILs with glycerol and L-F106 as comparisons under the same experimental conditions. The results show that the lithium-based ILs were effective lubricants for  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts.  $[\text{Li}(\text{urea})]\text{TFSI}$  even produced better tribological properties than glycerol and L-F106 both at RT and  $100^\circ\text{C}$ . Moreover, lithium-based ILs exhibited higher thermal stabilities than glycerol and lower costs associated with IL preparation than L-F106. Thus, lithium-based ILs, especially  $[\text{Li}(\text{urea})]\text{TFSI}$ , could be used as novel lubricant agents for  $\text{Si}_3\text{N}_4/\text{Ti}_3\text{SiC}_2$  contacts.

#### Conflict of Interests

The authors declare that they have no conflict of interests regarding the publication of this paper.

#### Acknowledgments

The authors acknowledge financial support from the National Key Basic Research Program of China (973) (2013CB632301) and the National Natural Science Foundation of China (51175492, 51105353, and 21125316).

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