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Effect of anion on micro/nano-tribological properties of ultra-thin imidazolium ionic liquid films on silicon wafer

Wenjie Zhao[a,b], Min Zhu[a,b], Yufei Mo[a,b], Mingwu Bai[a,*]

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

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ABSTRACT

Four kinds of room temperature ionic liquids (RTILs), as a new kind of lubricant for micro/nano-electromechanical system, with the same imidazolium cation but carrying different anions including hexafluorophosphate, tetrafluoroborate, nitrate and perchlorate were synthesized and these nano-scale films were prepared on single-crystal silicon wafer by dip-coating method. Atomic force microscopy was used to examine the morphologies of the films and evaluate their nano-friction and nano-adhesion properties. Chemical compositions of the films were characterized with a multi-functional X-ray Photoelectron Spectrometer. Micro-tribological properties of RTIL films were evaluated using a ball-on-plate micro-tribometer, and compared to that of perfluoropolyether. Results show that 3-butyl-1-methyl-imidazolium hexafluorophosphate exhibited the best anti-wear ability in comparison with the other four lubricants, so the ionic liquid films could be used as a kind of novel lubricant for application in MEMS/NEMS. Friction-reduction, adhesion resistance and anti-wear properties of RTILs were closely related to their anions. For the friction at nano-scale, the surface energy of the lubricant played a significant role, while the durability and friction-reduction of IL films strongly depend on their anions at micro-scale.

1. Introduction

Micro/nano-electromechanical systems (M/NEMS) have been increasingly demanded in many areas such as nanotechnology, high density storage, optical communication, and biomedicine. Therefore, M/NEMS related researches are being given greater attention and have obtained rapid development in the past decades due to their superior performance and low unit cost [1].

The surfaces in M/NEMS are generally separated by a couple of nanometers [2,3]. As the size of devices shrinks to micro- and nano-scales, the surface-to-volume ratio increases and the effect of body forces (gravity and inertia) becomes insignificant compared with those surface forces (van der waals, capillary, electrostatic, and chemical bonding). In M/NEMS, the gravitational body forces are negligible and adhesion becomes significant [4]. Accordingly, adhesion, stiction and friction are the major reasons that cause the failure of M/NEMS [3,5].

Perfluoropolyethers (PFPEs), which have many intrinsic properties, have been widely applied in nuclear, precision instrument, and aerospace industries as lubricating oils. They have been also commonly used as lubricating films in M/NEMS and magnetic disk drive industry to reduce the friction and wear of the interface [6–8]. However, PFPEs are catalytically degraded by strong nucleophilic agents and electropositive metals, combined with the high cost of PFPEs, their application in some fields are limited [9–11]. Therefore developing new alternatives for PFPEs is essential.

RTILs have been expected to be good candidates to replace PFPEs as versatile lubricants for different sliding pairs due to their unique characteristics including negligible vapor pressure, non-flammability, high thermal stability, low melting point, broad liquid range, and controlled miscibility with organic compounds. Ye found that RTILs can be used as a novel versatile lubricant and exhibited excellent friction-reduction, anti-wear performance and high load-bearing capacity [12], Liu and co-workers reported some tribological properties of RTILs [13–15].

Previous researches on RTILs as a lubricant were mainly focused on the characterization of various types of RTILs and synthesizing novel functionalized RTILs [12–17]. However, so far minimal research on the tribological properties of ultra-thin RTILs films (about 2 nm), aimed to be applied in M/NEMS has been reported [18]. The purpose of this research was to examine the tribological properties of ultra-thin films made from four kinds of RTILs with different anions. The effect of anions on the tribological properties of RTILs with the same cation was investigated.

* Corresponding author. Tel.: +86 931 4968080; fax: +86 931 4968080.
E-mail address: mwbai@lzb.ac.cn (M. Bai).

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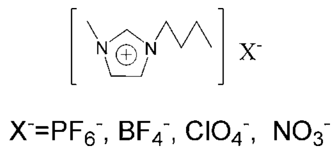

Fig. 1. Molecular structures of the ionic liquids: BMIM-PF_6^-, BMIM-BF_4^-, BMIM-ClO_4^-, BMIM-NO_3^-.

2. Experiment details

2.1. Materials

P-doped single-side polished single-crystal silicon (1 0 0) wafers (obtained from GRINM Semiconductor Materials Co. Ltd., Beijing) with a surface roughness of ∼0.2 nm and a thickness of 0.5 mm were used as substrate. Four kinds of RTILs including 1-butyl-3-methylimidazolium hexafluorophosphate, 1-butyl-3-methylimidazolium tetrafluoroborate, 1-butyl-3-methylimidazolium perchlorate, 1-butyl-3-methylimidazolium nitrate, marked as BMIM-PF_6^-, BMIM-BF_4^-, BMIM-ClO_4^-, BMIM-NO_3^-, respectively, were synthesized using the similar procedures as proposed in previous references [19,20]. All the other reagents of analytical grade were used as received. The chemical structures of the RTILs we used are given in Fig. 1. For comparison, PFPE (formula $HOCH_2CF_2O–(CF_2–CF_2O)_m–(CF_2O)_n–CF_2CH_2OH$, m and n are integers, MW = 3800, commercial name Zdol-3800), was purchased from Aldrich Chemical Company and used as received.

2.2. Film preparation

Cleaned silicon wafers were immersed in a freshly prepared Piranha solution (7:3 (v/v) mixture of 98% H_2SO_4 and 30% H_2O_2) at 90 °C for 40 min to make hydroxyl radicals on the surfaces [21]. Then the substrates were extensively rinsed with deionized water and blown dry with a stream of nitrogen. The solution of RTILs was firstly prepared in acetone with a suitable concentration, then the silicon substrate was slowly dipped into and withdrawn from a tank containing the solution with a uniform velocity of 60 μm/s, and was immersed in the solution for up to 120 s in order to obtain a uniform coating. Si wafer with RTILs film was allowed to dry in air in clean room prior to the following measurements. It could be seen that, from Fig. 2, the thickness of these films is proportional to the concentration of the corresponding RTILs solution. According to this relationship, four kinds of ionic liquid films of about 2 nm were easily prepared.

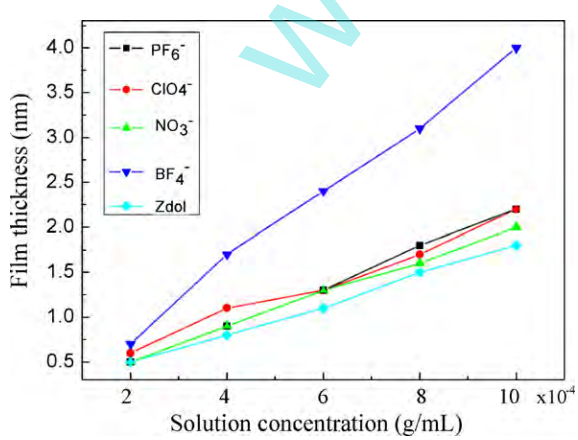

Fig. 2. Relationship between the concentration of RTILs and the thickness of the film on the silicon wafer.

2.3. Characterization of films

The film thickness was measured on a L116-C ellipsometer (Gaertner, USA) equipped with a He–Ne laser (λ = 632.8 nm) at a fixed incidence angle of 50°. Ten replicate measurements were carried out for each specimen and the thickness was recorded to be an accuracy of ±0.3 nm.

In order to identify the wettability properties of the specimen surface, the study of contact angles of the samples was on a CA-A contact angle meter (Kyowa Science Company Ltd., Japan). Contact angle was determined by averaging measurements taken from five locations on the each sample.

The chemical compositions and structures of the ionic liquid-coated surface were characterized by using a multi-functional X-ray photoelectron spectrometer (PHI-5702XPS) with a pass energy of 29.35 eV, an excitation source of Mg-Kα radiation ($h\nu$ = 1253.6 eV) and take-off angle of 36°, under a chamber pressure was about 3×10^{-8} Torr. The binding energy of adventitious carbon of C1s at 284.8 eV was used as the reference.

2.4. Measurement of micro/nano-tribological characteristics

The micro-friction properties and durability of all these films were evaluated using a commercial ball-on-plate tester. An AISI-52100 steel ball with a diameter of 3 mm moved horizontally with respect to the sample surface with a frequency of 1 Hz (10 mm/s, unless otherwise noted) and a traveling distance of 5 mm. The change in friction coefficient was monitored versus sliding times or cycles. The initiation of wear on the sample surface led to increase in friction coefficient, and a sharp increase indicated the failure of film. The friction coefficient and sliding times were recorded automatically by a computer. All the tests were conducted at room temperature and a relative humidity of 25%.

The nano-tribological behaviors of Zdol-3800 and four kinds of RTILs films were characterized with an AFM/FFM controlled by CSPM4000 electronics, using the contact mode. The measurement of frictional forces was accomplished by monitoring the lateral torsion of the cantilever as a function of applied load. The detailed procedure has been described in previous publications [22,23]. Commercially available triangular Si_3N_4 cantilever ($CSC21/Si_3N_4$/Al BS, overall Si_3N_4 coating, backside Al-coated) with a nominal force constant 2 N/m and a coated tip with a curvature radius of about 10 nm (Shanghai Haijiang NanoSci & Tech Co. Ltd.) was employed. Since the cantilever torsional force is unknown, no attempt was made to calibrate the torsional force constant, the output voltages were directly used as the relative frictional force. All the tests were conducted with the same cantilever/tip during the experiment unless specified otherwise. Each presented curve represents an average over at least 10 different measurements.

AFM has been used extensively to measure adhesive forces between surfaces at nano-scale. The adhesive force between the AFM tip and the film surfaces under ambient condition is shown in Fig. 3. The adhesive force (pull-off force) was calculated by multiplying the cantilever spring constant by the horizontal distance between points C and D [6,24]. All the experiments were performed at a relative humidity level of 25–30% at room temperature.

3. Results and discussion

3.1. Measurement of contact angle and thickness of films

Contact angle measurement is an effective way to reflect the variation of solid surface chemical composition. Table 1 lists the

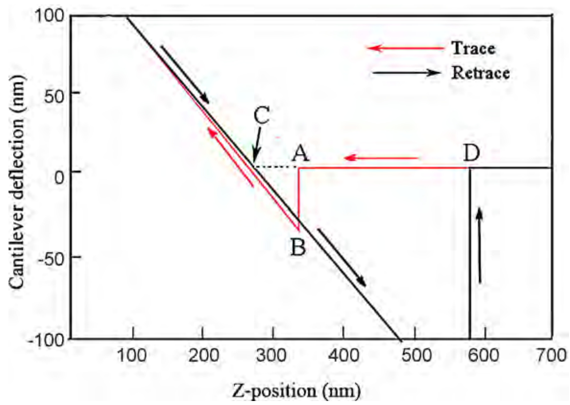

Fig. 3. A typical force–distance plot and schematic illustration for adhesion force calculation.

Table 1
Static contact angles of various films.

Sample	Contact angle (°)
SiO_2/Si	<5
Zdol-3800	100 ± 2
$[BMIM]PF_6^-$	61 ± 2
$[BMIM]BF_4^-$	21 ± 2
$[BMIM]ClO_4^-$	20 ± 2
$[BMIM]NO_3^-$	19 ± 2

ultra-pure water contact angles on hydroxylated silicon surface and various film surfaces. The hydroxylated silicon surface is hydrophilic, with the contact angle below 5°. When the RTILs and Zdol-3800 were coated onto the silicon surface, the contact angle increased, which indicated that films were formed on the silicon. In order to compare the tribological properties of different lubricants, the thickness of all the films we made in this article is about 20 ± 2 Å.

3.2. Composition and morphology

XPS is a powerful tool to clarify the surface chemical composition and structure of RTILs films. The procedure involved the measurement of F_{1s}, N_{1s}, Cl_{1s}, P_{2p}, and B_{1s} core level spectra for surfaces of these films. The XPS spectra presented in Fig. 4 show clear evidence of the formation of RTILs and Zdol-3800 on the silicon surface. The characteristic binding energy of F_{1s} at 683.8 eV, N_{1s} at 400.2 eV, Cl_{1s} at 198.2 eV, B_{1s} at 193.5 eV, P_{2p} at 133.3 eV, were observed, which indicates that Zdol-3800 and RTILs were coated successfully on the silicon surface.

AFM morphological images of Zdol-3800 and four kinds of RTILs are presented in Fig. 5. As seen from the images, all the films are quite homogenous distributed on the silicon surface. From Fig. 5a–e, the microroughness in root-mean-square (RMS) of the films were estimated to be less than 0.4 nm over an area of 1 μm × 1 μm, these observations indicated that the lubricant molecules spread evenly on the silicon surface.

3.3. Adhesive force measurements under ambient conditions

The adhesive force of Zdol-3800 and RTILs films measured by AFM are summarized in Fig. 6. As shown in Fig. 6, Zdol-3800 showed the lowest adhesive force, BMIM-NO_3^- shows the highest adhesive force. The film adhesive force increases in the sequence of Zdol-3800, BMIM-PF_6^-, BMIM-BF_4^-, BMIM-ClO_4^- and BMIM-NO_3^-. The adhesive force was related to the chemical structure and elements of the film.

It is well known that, when the lubricant films were disordered and hydrophilic, they would easily form meniscus by themselves or the adsorbed water molecules, thus they had higher adhesive force. However, when the lubricant films were hydrophobic and ordered, they would show low adhesion [5,25]. Zdol-3800 contained much more fluorine element, the film was hydrophobic and its surface energy was lower than other four kinds of ionic liquid films, it tended to form densely packed, highly ordered film, so it showed the largest contact angle and the lowest adhesive force. BMIM-BF_4^-, BMIM-ClO_4^- and BMIM-NO_3^- can dissolve in water,

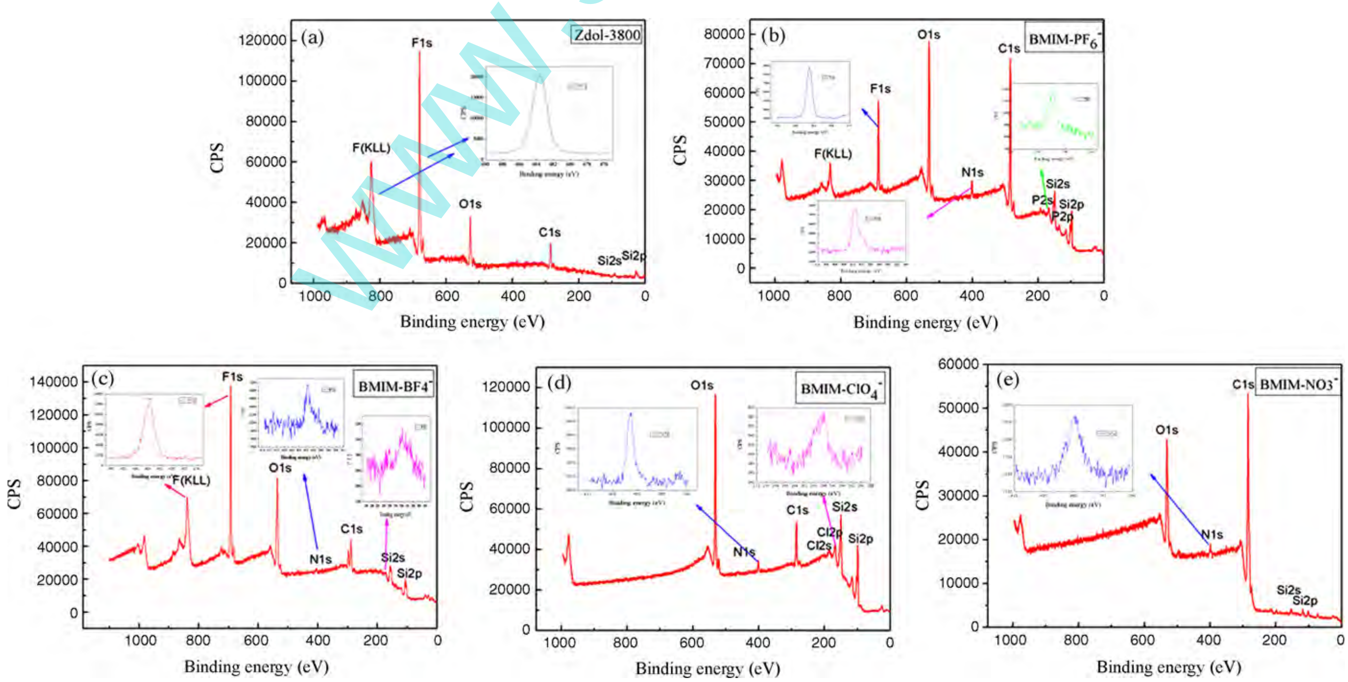

Fig. 4. XPS spectra of Zdol-3800 (a), BMIM-PF_6^- (b), BMIM-BF_4^- (c), BMIM-ClO_4^- (d), and BMIM-NO_3^- (e).

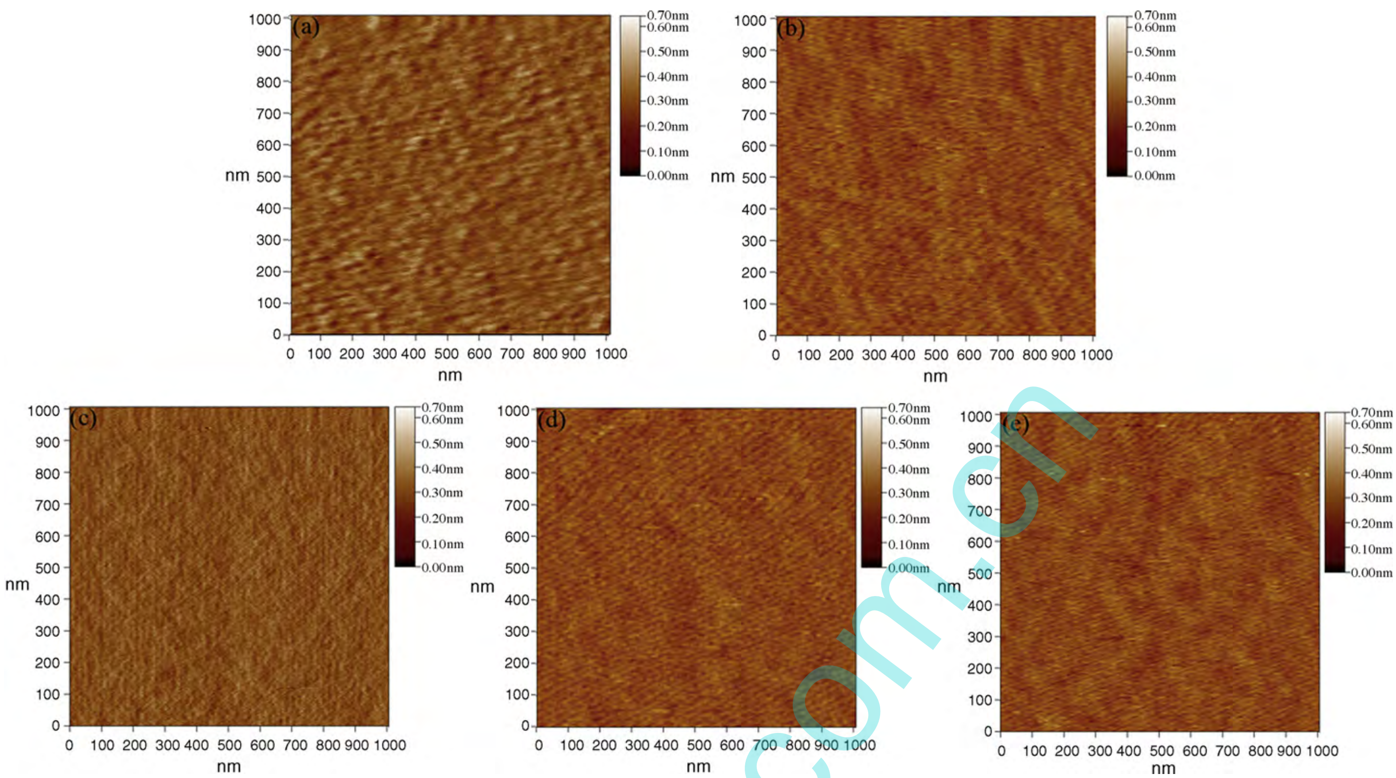

Fig. 5. 2D AFM images of Zdol-3800 (a), BMIM-PF_6^- (b), BMIM-BF_4^- (c), BMIM-ClO_4^- (d), and BMIM-NO_3^-(e).

the water molecules may be easily adsorbed onto the surface of films from the ambient environment, so their contact angles are not very large and show larger adhesive force. Compared to the above lubricants, BMIM-PF_6^- cannot dissolve in water, and also its film formed on the silicon surface may be not as order as Zdol-3800 film, so its adhesive force is between the above two.

3.4. Nano-tribological properties

To investigate the nano-friction properties of Zdol-3800 and other four kinds of RTILs films, the friction force versus normal load curves were measured in a friction measurement under increasing normal loads. As seen from Fig. 7, the nano-fiction force of the film is increasing in the sequence from Zdol, BMIM-PF_6^-, BMIM-NO_3^-, BMIM-ClO_4^-, to BMIM-BF_4^-. Compared to the above four kinds of ionic liquid films, Zdol-3800 film exhibited lowest friction. BMIM-PF_6^- film exhibited the lowest friction force among the RTILs films.

The difference in the nano-friction of the four films might be attributed to three potential factors: (1) intra-molecular energetic barriers to rotation of the rigid cycle structure; (2) long-range intermolecular steric interactions within the plane of the bulkier groups [26,27]; and (3) surface energy [28,29]. If the surface energy is much higher, it is easily to form meniscus by themselves or the adsorbed water molecules, they had higher adhesive force due to the capillary force and hydrogen bond, which would led to larger shearing strength and higher friction. As seen from the Fig. 7, all the RTILs contained rigid cycle-shape structure; they need much energy to overcome intra-molecular energetic barriers to rotate the rigid

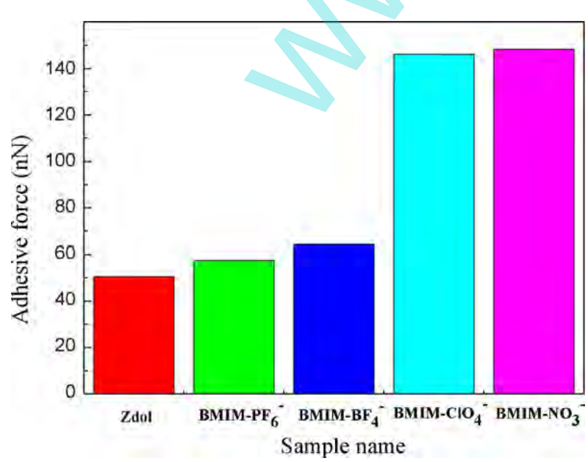

Fig. 6. Adhesion force curves of Zdol-3800, BMIM-PF_6^-, BMIM-BF_4^- and BMIM-ClO_4^-, BMIM-NO_3^- films measured in ambient air.

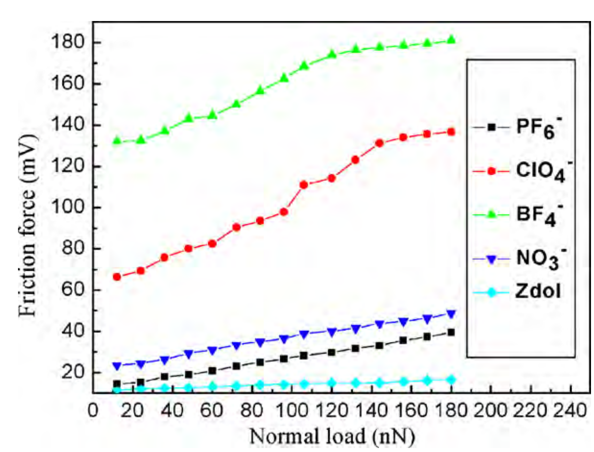

Fig. 7. Nano-friction force versus normal load curves for Zdol-3800, BMIM-PF_6^-, BMIM-BF_4^- and BMIM-ClO_4^-, BMIM-NO_3^- films at a frequency of 1 Hz.

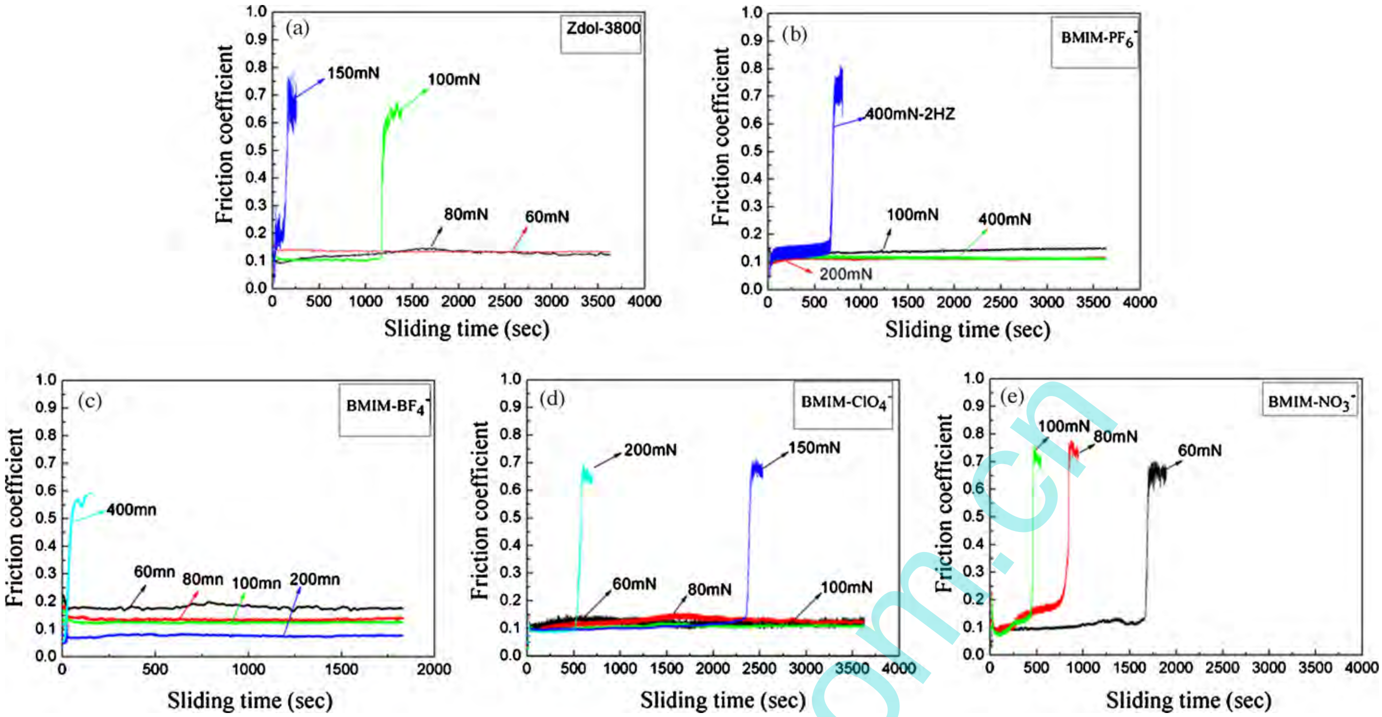

Fig. 8. Friction coefficients as function of sliding cycles for Zdol-3800 (a), BMIM-PF_6^- (b), BMIM-BF_4^- (c), BMIM-ClO_4^- (d) BMIM-NO_3^- (e), sliding against AISI-52100 steel ball at normal load between 60 and 400 mN and a sliding velocity of 1 Hz.

cycle structure, so they exhibited higher friction than Zdol-3800. The wetting/dewetting ability of ionic liquid is largely determined by the anion, so different RTILs nano-film showed different nano-friction performance due to different anions. Compared to the RTILs films, Zdol-3800 had free linear chains and it could bond strongly to the silicon, it tended to form densely packed, highly ordered film, which had good flexibility, so the friction was the least. On the other hand, the experimental observation indicated that the friction force varied roughly in the same order as the surface energy and adhesive force. It follows that the tribological properties of lubricants are determined by the flexibility and surface energy of lubricant for the friction at nano-scale. The tribological performance of RTILs is closely related to the anions.

3.5. *Micro-tribological properties*

Fig. 8 shows the friction coefficients and durability of Zdol-3800 and four kinds of RTILs films as functions of sliding cycles against steel ball.

For Zdol-3800 film, as shown in Fig. 8a, the friction coefficient was about 0.15 at the normal load of 60 mN. When the normal load rose to 100 mN, the friction coefficient decreased to about 0.1 and remained stable at sliding cycles below 1200 cycles. When sliding cycles exceeded 1250 cycles, the friction coefficient increased sharply to 0.6, which indicated that the friction-reduction effect played by Zdol-3800 film diminished under the test condition. When the normal load rose to 150 mN, sliding cycles reaching 200, the friction coefficient sharply increased to 0.7, so the durability was 200 cycles.

As shown in Fig. 8b, BMIM-PF_6^- film was recorded at a friction coefficient of 0.13, which kept almost constant with increasing sliding cycles at a load of 100 mN. When the normal load rose to 200 mN, the friction coefficient slightly decreased to 0.11, which was still almost stable under all sliding cycles, but the friction coefficient abruptly increased at a sliding load of 400 mN, a sliding frequency of 2 Hz and 750 sliding cycles, this indicated that wear of the BMIM-PF_6^- film occurred and the film failed.

Fig. 8c shows the variation of friction coefficients and durability of BMIM-BF_4^- film on Si substrates against steel ball with sliding cycles, respectively. It can be seen that BMIM-BF_4^- film was recorded at a friction coefficients about 0.07 under a load of 200 mN. With increasing normal load, durability of the ionic liquid films decreased dramatically, and it failed instantly at a load of 400 mN with just 50 sliding cycles. Fig. 8d shows the variation of friction coefficients and durability of BMIM-ClO_4^- film against steel ball with sliding cycles. It indicated that BMIM-ClO_4^- film recorded friction coefficients about 0.10 under a slight load of 150 mN, and the registered durability is 2400 cycles in this case. With the increase of normal load, durability of BMIM-ClO_4^- film decreased dramatically, and it failed instantly at a load of 200 mN with only 500 sliding cycles. At the same time, from Fig. 8e, BMIM-NO_3^- film showed very poor tribological properties under the same test conditions. At a load of 100 mN, the registered durability of BMIM-NO_3^- film was 480 cycles only.

In summary, BMIM-PF_6^- film was much superior to other four kinds of films in the test range of the loads in terms of wear resistance and load-bearing capacity in sliding against steel ball counterpart. BMIM-PF_6^- nano-film has potential application in M/NEMS which needs better durability. Zdol-3800 bonded strongly to the silicon and also had free linear chains, so it showed better tribological properties. RTILs showed different tribological performance due to different anions, some showed better tribological properties than Zdol, for example: BMIM-PF_6^-, BMIM-$BF_4^-$and BMIM-ClO_4^-; but some showed worse tribological properties than Zdol, such as BMIM-NO_3^-.

4. Conclusions

Zdol-3800 and four kinds of RTILs (with the same cation but different anions) films including BMIM-PF_6^-, BMIM-BF_4^-, BMIM-ClO_4^-, BMIM-NO_3^-, were prepared and characterized successfully. The adhesion, micro/nano-tribological properties of these films was investigated, and Zdol-3800 was used as baseline. The adhesive force property was consistent with the ultra-pure water contact angle and nano-friction properties. BMIM-PF_6^- film on hydroxyl-terminated surface showed excellent reduction-friction and anti-wear properties. The friction-reduction and anti-wear mechanism of the four kinds of RTILs were dependent on their chemical structures. From the results obtained from the experiment it is concluded that the tribological properties were close related to the flexibility and surface energy of lubricant film for the friction at nano-scale, however at micro-scale friction, tribological properties may be determined by the rigid cycle structure and different anions of RTILs. BMIM-PF_6^- nanofilm has potential application in M/NEMS which needs better durability.

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