Friction **8**(4): 674–683 (2020)
https://doi.org/10.1007/s40544-019-0290-6
ISSN 2223-7690
CN 10-1237/TH

RESEARCH ARTICLE

Investigation on tribological behaviors of MoS_2 and WS_2 quantum dots as lubricant additives in ionic liquids under severe conditions

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Received: 10 December 2018 / Revised: 04 March 2019 / Accepted: 18 March 2019

Abstract: Despite excellent tribological behaviors of ionic liquids (ILs) as lubricating oils, their friction-reducing and anti-wear properties must be improved when they are used under severe conditions. There are only a few reports exploring additives for ILs. Here, MoS_2 and WS_2 quantum dots (QDs, with particle size less than 10 nm) are prepared via a facile green technique, and they are dispersed in 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIm]PF_6), forming homogeneous dispersions exhibiting long-term stabilities. Tribological test results indicate that the addition of MoS_2 and WS_2 QDs in the IL can significantly enhance the friction-reducing and anti-wear abilities of the neat IL under a constant load of 500 N and a temperature of 150 °C. The exceptional tribological properties of these additives in the IL are ascribed to the formation of protective films, which are produced not only by the physical absorption of MoS_2 and WS_2 QDs at the steel/steel contact surfaces, but also by the tribochemical reaction between MoS_2 or WS_2 and the iron atoms/iron oxide species.

Keywords: MoS_2 and WS_2 quantum dots; ionic liquids; lubricant additive; friction reduction and anti-wear; severe conditions

1 Introduction

Ionic liquids (ILs), known for their extremely low volatilities, wide liquid temperature ranges, high thermal and chemical stabilities, and exceptional tribological properties, have been intensively studied as lubricants and lubricant additives in various applications [1–3]. For instance, ILs in space technology applications have attracted considerable interest over the past decade [4], and the usage of ILs as high-temperature lubricants under the conditions of high load, high speed, and elevated temperature has gained increasing attention [1, 5–8]. Numerous reports have been published on the synthesis of ILs, particular oil-miscible ILs for improving the friction reduction and anti-wear (AW) properties of lubricating oils in recent years [3, 9, 10]. Despite the excellent tribological behaviors of ILs as lubricating oils, their friction reduction and AW properties must be improved when they are used under severe conditions. There are only a few reports exploring additives for ILs. For example, 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIm][PF_6]), a commercially available IL, has been observed to exhibit superior friction reduction and AW performances compared with conventional lubricants [1, 8, 11, 12]; however, pure [BMIm][PF_6] offers poor lubricating properties when it is used under a high load condition [13, 14]. Previous reports have demonstrated that covalent modification of multi-walled carbon nanotubes (MWCNTs) with imidazolium

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cation-based ILs and brush-like poly (ionic liquids) (PILs) can remarkably improve the dispersibility of MWCNTs in [BMIm][PF_6] base oil and the tribological behaviors of this IL at high loads [13, 14], but surface functionalizations of MWCNTs with ILs and PILs are restricted by high-cost and complex synthesis processes. The development of a new type of additive with excellent tribological performances under severe conditions and low cost is required to fully exploit the advantages of ILs.

Studies have been extensively conducted for fabricating MoS_2 and WS_2 nanoparticles (NPs) for use as effective oil additives owing to their fascinating characteristics, such as high thermal and chemical stabilities, nanometric size, and exceptional lubricating properties. However, one of the major drawbacks of NPs as friction-reducing and AW additives is their poor dispersibility in lubricating oils, which limits their use in lubrication applications. To address this problem, various methods have been employed to disperse MoS_2 and WS_2 NPs in base oils. For instance, surface modification is the most promising method to promote MoS_2 dispersion [15–17]. Mixed MoS_2/graphene dispersions in base oils resist sedimentation for two weeks [18]. MoS_2 with different morphologies and sizes was fabricated and could be dispersed in different oils for a few weeks [19–21]. In particular, MoS_2 and WS_2 quantum dots (QDs, with particle size less than 10 nm) can form a homogeneous and stable dispersion in polyalkylene glycol (PAG) base oil, and can significantly enhance the friction-reducing and AW properties of neat PAG base oil at elevated temperatures [22]. Notably, the small size effect, high surface effect, and quantum size effect of MoS_2 and WS_2 QDs might play an important role in the formation of a stable dispersion of PAG base oil additized with solid NPs. These exciting physical properties have prompted us to investigate the application of MoS_2 and WS_2 QDs as additives in ILs.

Herein, MoS_2 and WS_2 QDs are fabricated by using sonication combined with solvothermal processing of bulk MoS_2 and WS_2 powder in N,N-dimethylformamide (DMF) [23], and their dispersibility in [BMIm]PF_6 is evaluated. The tribological behaviors of MoS_2 and WS_2 QDs added to the IL are investigated under high loads and high temperatures. The friction-reducing and AW mechanisms of these additives are explored using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) and X-ray photoelectron spectroscopy (XPS).

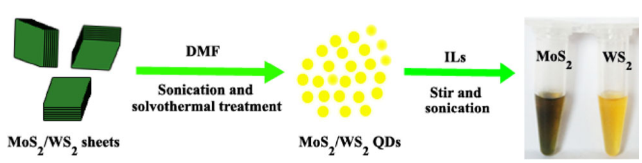
2 Experimental section

2.1 Preparation of MoS_2 and WS_2 QDs and the dispersion of IL additized with MoS_2 and WS_2 QDs

MoS_2 and WS_2 QDs were synthesized using previously reported methods [23], as shown in Scheme 1. Briefly, 1g of commercial MoS_2 and WS_2 powder with an average grain size of 500 nm (Shanghai Research Institute of Rare-Metal) was dispersed in 100 mL of DMF. The mixture was sonicated for 3 h using a sonicator (SCIENTZSB-5200D, output power 250 W), and subsequently, the resulting dispersion was transferred to a 100-mL round-bottomed flask and heated for 6 h at 140 °C under vigorous stirring. Thereafter, the black mixture was centrifuged for 10 min at 3,000 rpm to remove the solid phase. The light-yellow dispersion of MoS_2 and WS_2 QDs was evaporated. The as-synthesized MoS_2 and WS_2 QDs were added to [BMIm]PF_6 (J&K Scientific Ltd., purity≥ 99%), and thereafter, the suspension was thoroughly mixed using a magnetic stirrer for 30 min and ultrasonic mixing for 30 min. The obtained dispersion of MoS_2 and WS_2 QDs added to the IL is homogeneous and resists sedimentation for several months after preparation.

2.2 Physical and tribological characterizations

The physical characterizations were performed using high-resolution transmission electron microscopy (HR-TEM, FEI TECNAI F30: acceleration voltage, 300 kV), XPS (PHI-5702: non-monochromatic, Al Kα radiation; calibration of binding energy, C1s peak at 284.8 eV for contaminated carbon), Raman spectroscopy (Horiba, LabRAM-HR, laser wavelength 514.5 nm),



Scheme 1 Synthesis of MoS_2/WS_2 QDs by sonication and solvothermal treatment, and the digital photo of MoS_2/WS_2 QDs dispersion in ILs for two months after preparation.

X-ray powder diffraction (XRD, Bruker D8 DISCOVER: Cu Kα radiation, λ = 1.54 Å), and UV–vis spectroscopy (Hitachi U-4100).

The tribological measurements were conducted using a reciprocating friction tester (Optimal-SRV-IV) with an upper ball (Ø 10 mm, AISI 52100 steel, hardness 59–60 HRC) running against a lower stationary disk (Ø 24 mm × 7.9 mm, AISI 52100 steel, hardness 58–60 HRC) at a frequency of 25 Hz, amplitude of 1 mm, and duration of 30 min. The friction curve was recorded automatically using a computer connected to the SRV tester. The corresponding wear volume of the lower disks was obtained using a noncontact three-dimensional surface mapping profilometer (MicroXAM-3D). Three repetitive measurements were performed for each sample, and the averaged values of the friction coefficient curve and wear volume are reported in this paper. The morphology of the wear scars was measured using SEM-EDS (JSM-5600LV). The typical elemental distribution of the worn surfaces was investigated using XPS (PHI-5702).

3 Result and discussion

3.1 Physical properties of MoS_2 and WS_2 QDs

Transmission electron microscopy (TEM) was used to investigate the particle size and morphology of the MoS_2 and WS_2 QDs. As shown in Figs. 1(a) and 1(c), the median size of the MoS_2 and WS_2 QDs is approximately 3.0 nm and 3.2 nm, respectively, and the HR-TEM results (shown in Figs. 1(b) and 1(d)) indicated that the lattice spacing is 0.23 nm and 0.27 nm for MoS_2 and WS_2 QDs, respectively, corresponding to the (103) and (101) planes of MoS_2 and WS_2 crystals.

XPS established that the main compounds in the as-synthesized products were MoS_2 and WS_2, along with some MoO_3 and WO_3. The Mo 3d XPS spectrum (Fig. 2(a)) collected for the MoS_2 QDs contained peaks at 227.0 eV, 229.6 eV, and 232.8 eV, assigned to the 2s electrons of S^{2-}, Mo^{4+} 3d5/2, and Mo^{4+} 3d3/2, respectively [22]. The S 2p signal in the XPS spectrum (Fig. 2(b)) was centered at 162.2 eV and 163.4 eV, which are attributed to S^{2-} 2p3/2 and S^{2-} 2p1/2, respectively [23]. In addition, a small peak in the Mo 3d spectra at 236.3 eV is ascribed to Mo^{6+} 3d3/2 and a peak in the S 2p spectra at 168.6 eV is assigned to S^{6+} 2p3/2. The two species realized in MoO_3 and SO_4^{2-} might be generated during the synthesis process [23]. Similarly, the W 4f and S 2p XPS spectra of WS_2 QDs (Figs. 2(c) and 2(d)) demonstrated that WS_2 QDs were successfully synthesized with a relatively low content of W^{6+} and S^{6+} species (realized in WO_3 and SO_4^{2-}) in our product.

Raman and XRD spectra of the MoS_2 and WS_2 QDs

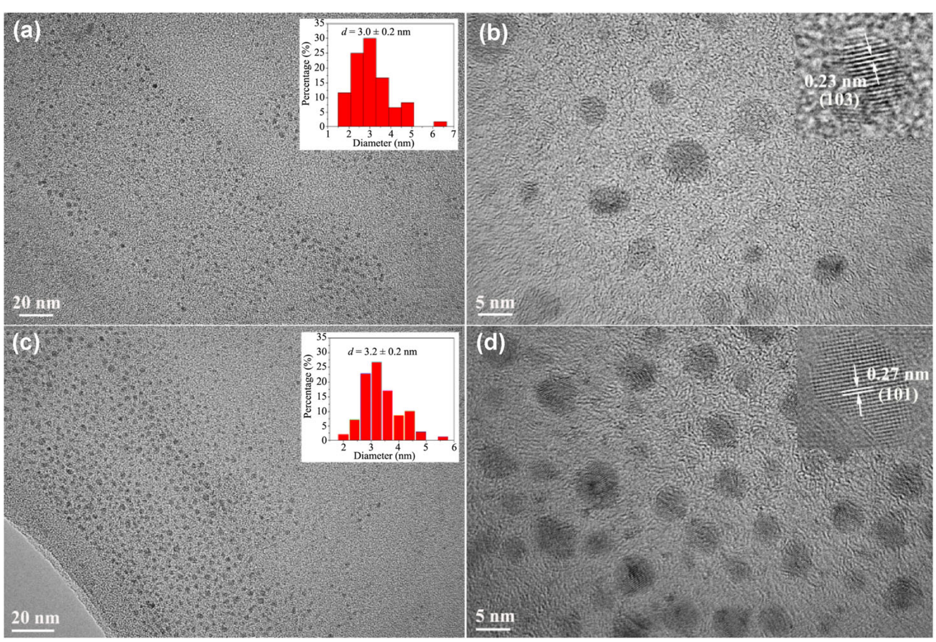


Fig. 1 TEM micrographs of (a, b) MoS_2 and (c, d) WS_2 QDs. Insets: (a, c) the size distribution and (b, d) HRTEM images of MoS_2 and WS_2 QDs.

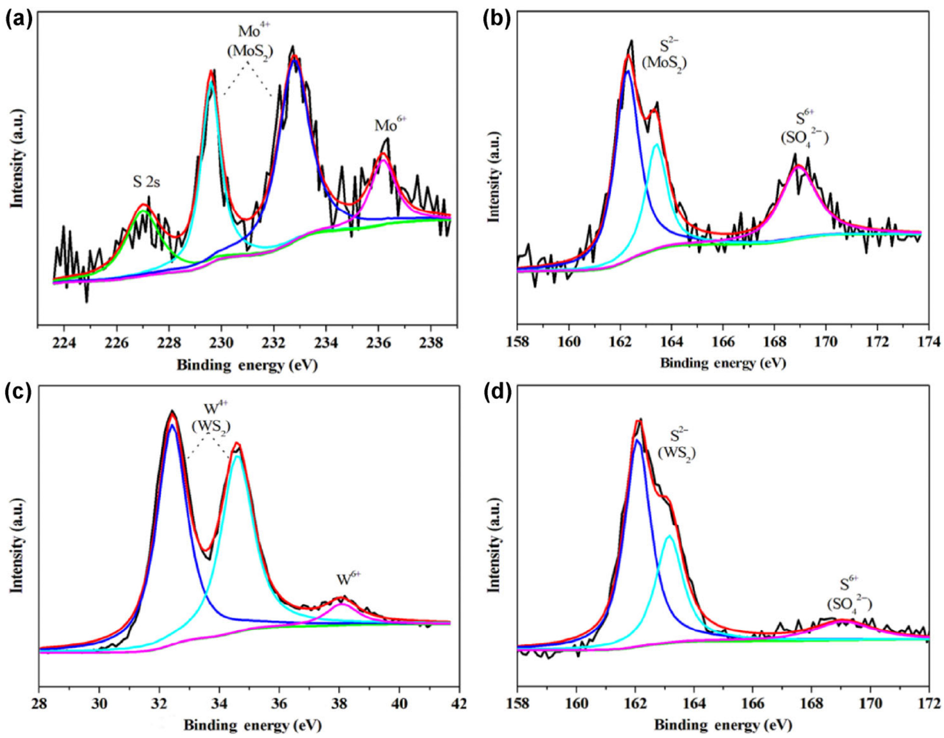


Fig. 2 XPS spectra of (a) Mo 3d and (b) S 2p of MoS_2 QDs, and (c) W 4f and (d) S 2p of WS_2 QDs.

are also obtained, and no signal or peak is observed (data not shown), which might be due to the fact that both MoS_2 and WS_2 QDs are monolayer and have no interaction with each other [24]. The UV–vis spectra of MoS_2 and WS_2 QDs in DMF are shown in Fig. 3(a). The characteristic peaks of MoS_2 and WS_2 QDs are almost the same and are located at the near-UV region (λ < 300 nm), which are attributed to the excitonic features of MoS_2 and WS_2 QDs [25]. The long-term stability of the 1% MoS_2 and WS_2 QDs dispersion in the IL was evaluated using UV–vis spectroscopy [26]. It can be observed from Fig. 3(b) that the concentrations of these dispersion systems only decreased slightly within one month, indicating excellent dispersion stability of the MoS_2 and WS_2 QDs in the IL.

3.2 Tribological performances of MoS_2 and WS_2 QDs

The friction-reducing and AW properties of the MoS_2 and WS_2 QDs added to the IL are investigated using Optimal-SRV-IV at high load and high temperature. It

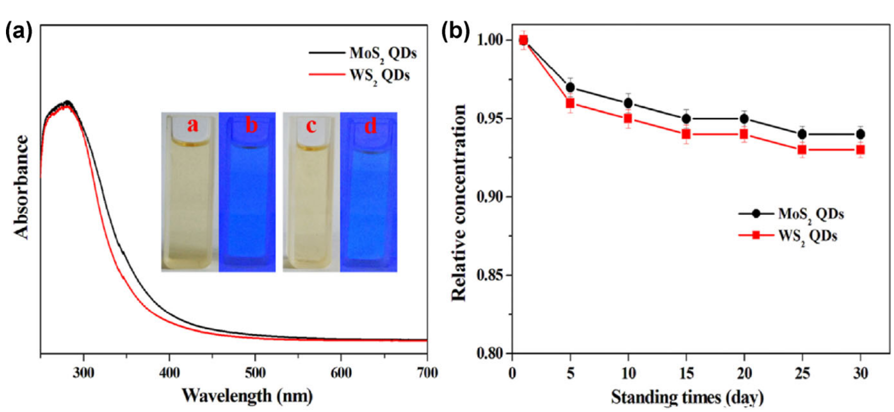


Fig. 3 (a) UV spectra of (a, b) MoS_2 and (c, d) WS_2 QDs dispersed in DMF; insets show the photographs of MoS_2 and WS_2 QDs in DMF under (a, c) visible light and (b, d) UV light. (b) Dispersion stabilities of MoS_2 and WS_2 QDs in ILs determined by UV–vis spectrophotometer.

can be observed from Fig. 4(a) that the friction curve of the IL ([BMIm]PF_6) fluctuates frequently with a relatively high friction coefficient at 150 °C and 500 N. In contrast, the addition of 1% MoS_2 QDs shows a low and stable friction coefficient, and the average friction coefficient can be reduced by approximately 22% compared with that of the IL. The addition of 1% WS_2 QDs can reduce the friction coefficient of the base oil by approximately 19%. For comparison, the inset in Fig. 4(a) shows the friction curve of pure perfluoropolyether (PFPE) base oil under the same conditions, and the friction coefficient of PFPE oil is significantly larger than those of the IL base oil and the oil additized with MoS_2 and WS_2 QDs. These results indicate that MoS_2 and WS_2 QDs have good friction-reducing property at high load and high temperature. Figure 4(b) displays the corresponding wear volume of steel disks lubricated by the IL and the IL with MoS_2 and WS_2 QDs. It shows that MoS_2 and WS_2 QDs reduce the wear volume of the base oil by approximately 91% and 89%, respectively. 3D optical microscopic images of the worn surfaces inset in Fig. 4(b) further indicate that MoS_2 and WS_2 QDs can significantly improve the AW property of ILs under high load and high temperature. The excellent tribological behaviors of these additives are probably because MoS_2 and WS_2 with particle size below 10 nm could easily enter the ball-disk contact interface and form an effective protective film. MoS_2 and WS_2 QDs will "fill" the asperity valleys and establish a smooth boundary film between the contacting surfaces. Both the films showed friction reduction and wear resistance at elevated temperatures.

The tribological performances of the MoS_2 and WS_2 QDs dispersion in the IL were also evaluated at different temperatures and a constant load of 500 N. As shown in Fig. 5(a), the addition of MoS_2 and WS_2 QDs has no effect on the friction reduction and AW behaviors of the IL at a temperature below 50 °C, whereas the two NPs can significantly reduce the friction coefficients and wear volumes of the base oil when the temperature increases from 100 to 250 °C. This conclusion is in accordance with the result of addition of MoS_2 and WS_2 QDs to PAG base oil at different temperatures [22], and the possible mechanism for the lubrication action has been reported previously. In brief, high temperature plays an active role in the viscosity of nanofluids, the free movement of NPs in ILs, and the tribochemical reaction of the lubricant during friction and wear processes, which benefits the formation of a boundary protective film. In the case of low temperatures, MoS_2 and WS_2 QDs in the contact interface cannot be complemented timely owing to the increase in the viscosity of base oil and the restriction of motion of NPs in ILs as the lubrication film is worn away.

Figure 6 shows the friction coefficient and wear volume of the IL additized with 1% MoS_2 and WS_2 QDs at various applied loads and a temperature of 150 °C. It can be observed that the addition of 1% MoS_2

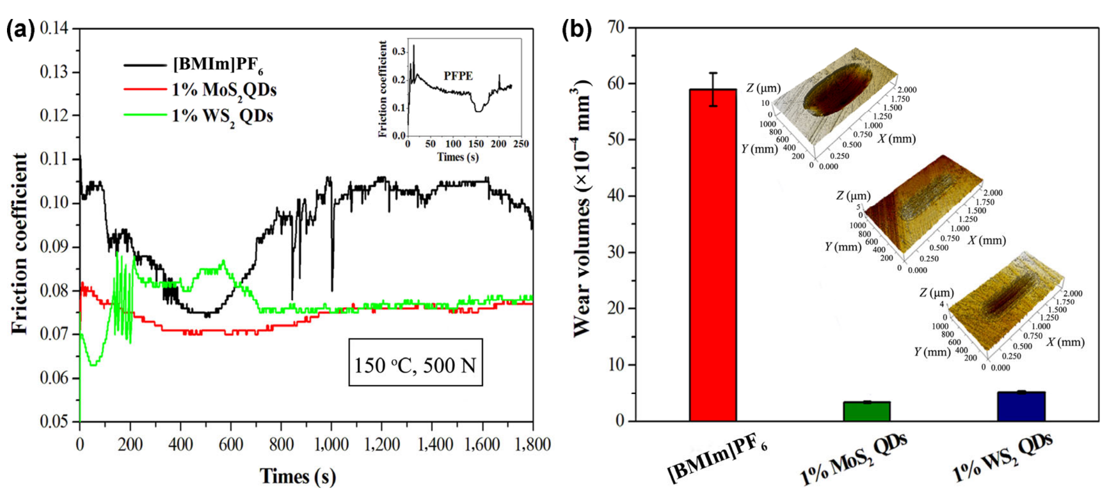


Fig. 4 (a) Friction coefficient curves for ILs ([BMIm]PF_4) and ILs additized with 1% MoS_2 and WS_2 QDs at 150 °C and 500 N. Inset of (a): Evolution of friction coefficient of PFPE under the same conditions. (b) Wear volumes and 3D images of steel disks lubricated by ILs and the dispersions of ILs with MoS_2 and WS_2 QDs.

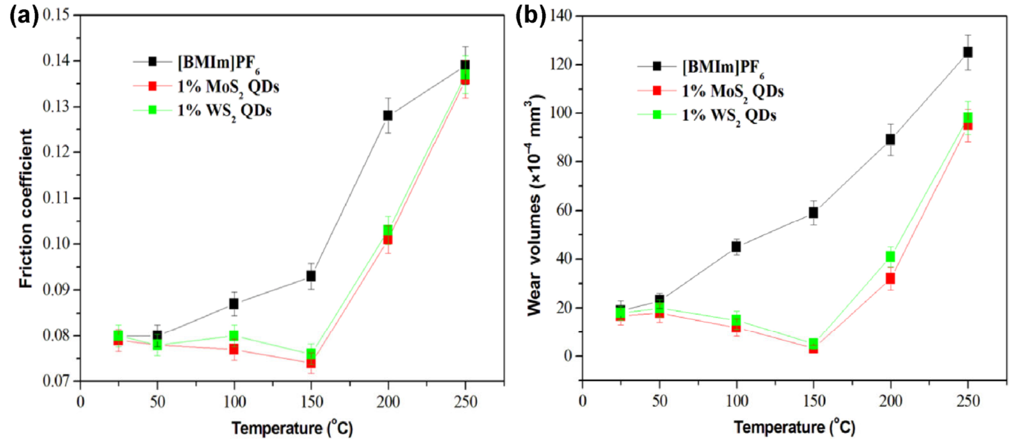


Fig. 5 Averaged values of (a) friction coefficient curves and (b) wear volumes of steel disks lubricated by ILs and ILs additized with 1 wt% MoS_2 and WS_2 QDs at different temperatures (load, 500 N; stroke, 1 mm; frequency, 25 Hz).

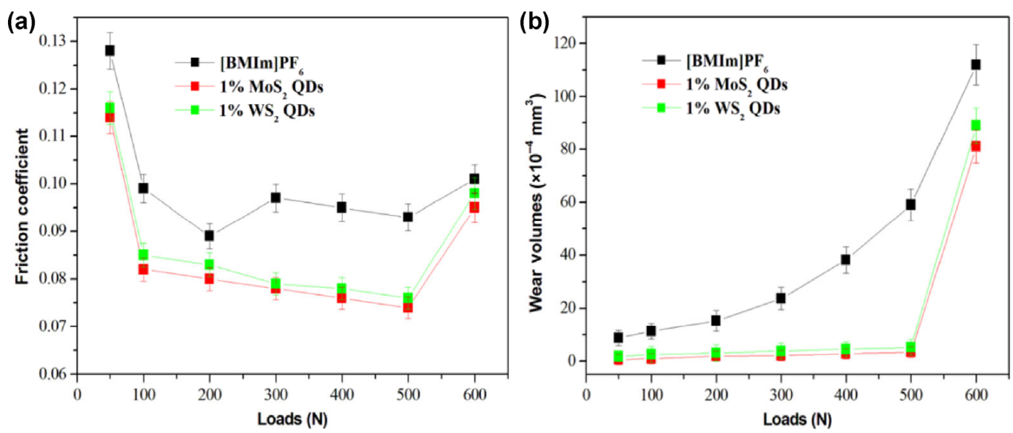


Fig. 6 The averaged values of (a) friction coefficient and (b) wear volumes of steel disks lubricated by ILs and ILs additized with 1 wt% MoS_2 and WS_2 QDs at various loads (temperature, 150 °C; stroke, 1 mm; frequency, 25 Hz).

and WS_2 QDs can improve the tribological behaviors of the IL with an increase in the load from 50 to 600 N at an elevated temperature. In particular, the friction coefficient and wear volume of the base oil are dramatically reduced when the load is 100 N, 200 N, 300 N, 400 N, and 500 N. This could be explained by the fact that the surface temperature is largely dependent on the load, and additives that might be effective at high loads may be ineffective at low loads (and vice versa) [27]. In addition, MoS_2 QDs and WS_2 QDs show similar friction-reducing and AW capacities for the ILs at different temperatures and loads.

3.3 Surface analysis

The wear surfaces of steel disks lubricated by the IL and the dispersions of the IL with 1% MoS_2 and WS_2 QDs at 150 °C and 500 N were investigated using SEM, and the tribofilms on these worn scars were analyzed using SEM-EDS. As shown in Figs. 7(a)–7(c), the wear surface under the lubrication of the pure IL shows a much wider worn scar, indicating that severe scuffing occurred in this case. However, the wear scars of the steel disks lubricated by the IL with 1% MoS_2 and WS_2 QDs evidently became narrow, suggesting that MoS_2 and WS_2 QDs can significantly improve the AW property of the IL base oil. This is consistent with the wear volume result in Fig. 3(b). The detailed views of the corresponding wear surfaces are shown in Figs. 7(a′), 7(b′), and 7(c′) (the areas designated by the red contour in Figs. 7(a), 7(b) and 7(c), respectively). The result indicates that the tribofilm on the worn surface lubricated by the pure IL contains no Mo and S (inset in Fig. 7(a′)), whereas the boundary lubrication films on the wear surfaces lubricated by the IL with

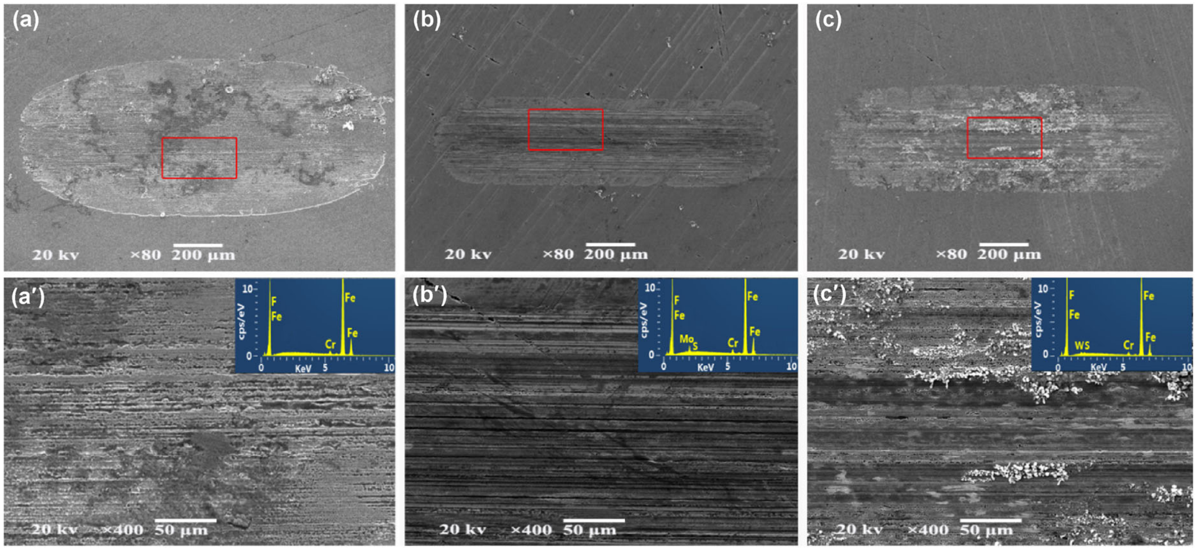


Fig. 7 SEM images of worn surfaces lubricated (a, a′) neat ILs and ILs containing 1% of (b, b′) MoS_2 QDs and (c, c′) WS_2 QDs under 150 °C and 500 N. Insets of (a′), (b′), and (c′) are the EDS spectra of tribofilms generated on the wear surfaces lubricated by ILs and ILs additized with 1% MoS_2 and WS_2 QDs. The spectra are the average of areas shown in the corresponding images in (a), (b), and (c).

MoS_2 and WS_2 QDs are rich in Mo, S and W, S (inset in Figs. 7(b′) and 7(c′)).

The friction-reducing and AW mechanisms of the IL with MoS_2 and WS_2 QDs are further explored using XPS, and the results are shown in Fig. 8. The XPS spectra of Fe 2p (Fig. 8(a)) can be deconvoluted into six peaks corresponding to FeS_2 (708.9 eV), FeO (709.7 eV), Fe_3O_4 (710.7 eV), FeOOH (711.8 eV), $FePO_4$ (712.8 eV), and $FeSO_4$ (713.6 eV) [22, 28], and the Fe 2p signals of the worn surfaces lubricated with the neat IL (a) and the IL additized with 1% MoS_2 QDs (b) and 1% WS_2 QDs (c) are similar to each other at 500 N and 150 °C, which might be attributed to the similar tribochemical reactions of the IL with the steel/steel contact surfaces. Similarly, the peaks of P 2p and F 1s of the worn surfaces under lubrication of the three types of lubricants appear at 133.7 eV and 684.9 eV (Figs. 8(b) and 8(c)), corresponding to $FePO_4$ and FeF_2 [28], respectively. The signals of S 2p of the worn scars lubricated by the dispersions of the IL with 1% MoS_2 and WS_2 QDs are located at 168.6 eV, which are assigned to $FeSO_4$ [22, 28]. The XPS spectra of Mo 3d shown in Fig. 8(e) contain three peaks corresponding to Mo^{5+} (231.4 eV), MoS_2 (232.4 eV), and Mo^{6+} (233.2 eV) [22, 23], and the spectra of W 4f shown in Fig. 8(f) are composed of three peaks corresponding to WS_2 (32.4 eV and 34.6 eV) and 37.5 eV (WO_3) [22, 23]. These results indicate that the friction-reducing and AW behaviors of the IL are attributed to the formation of a boundary lubrication film containing FeO, Fe_3O_4, FeOOH, FeF_2, and $FePO_4$, and the addition of MoS_2 or WS_2 QDs can significantly improve the tribological properties of the IL because the dispersion of the IL with MoS_2 or WS_2 QDs could form a stable protective film composed of $FeSO_4$, MoS_2 or WS_2, and the compounds generated from the tribochemical reactions of the IL with the steel/steel contacts surfaces.

4 Conclusions

In summary, MoS_2 and WS_2 QDs were investigated as friction-reducing and AW additives in an IL ([BMIm]PF_6) for the first time. They could form a homogenous and stable dispersion in the IL for several months, and significantly reduce the friction coefficient and wear volume of the IL at 500 N and 150 °C. The excellent tribological behaviors of these two additives could be explained by the fact that MoS_2 and WS_2 QDs not only formed a boundary lubrication film via physical absorption but also generated protective films during tribochemical reactions. The film is composed of MoS_2 or WS_2, $FeSO_4$, FeS_2, FeO, Fe_3O_4, FeOOH, FeF_2, and $FePO_4$, resulting in friction reduction and wear resistance at elevated temperatures.

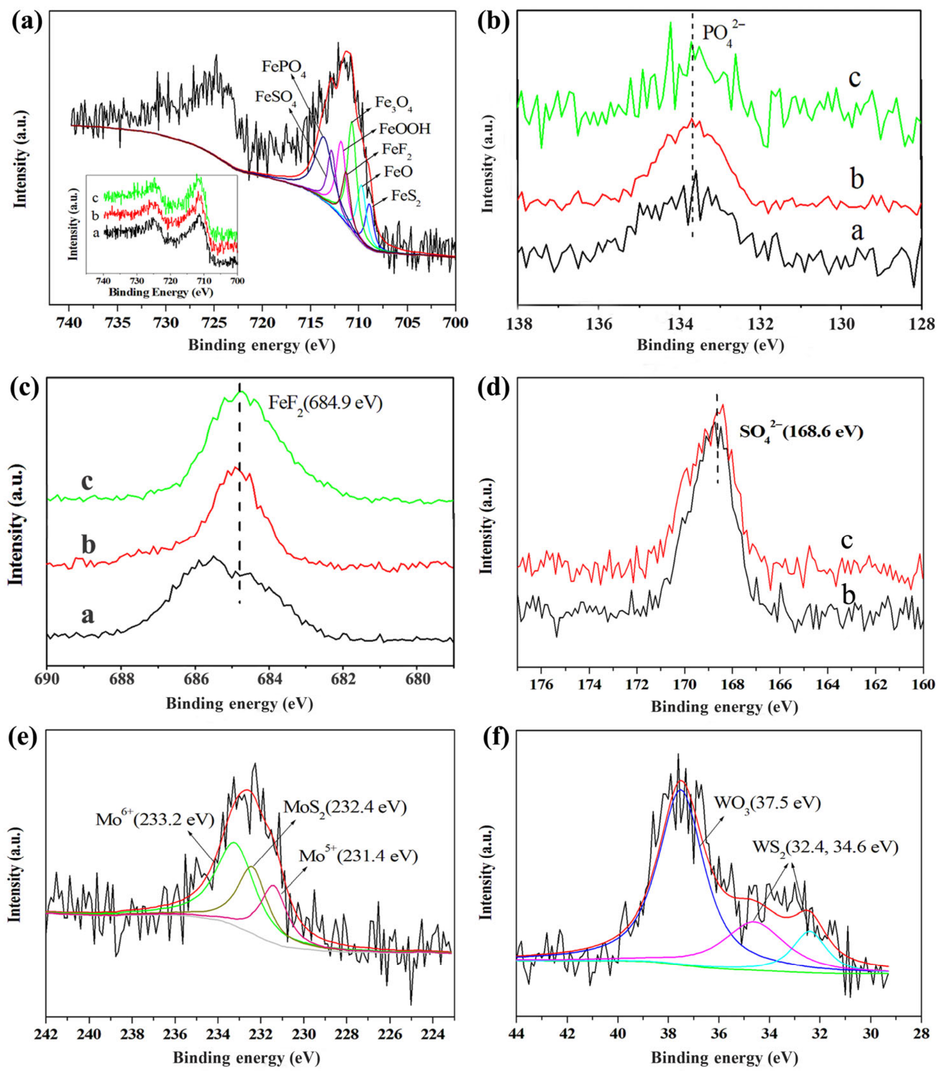


Fig. 8 XPS spectra of the elements (a, Fe 2p; b, P 2p; c, F 1s; d, S 2p; e, Mo 3d; and f, W 4f) on the wear surfaces lubricated by (a) neat ILs and ILs containing 1% of (b) MoS_2 and (c) WS_2 QDs at 150 °C, 500 N.

Acknowledgements

The authors are thankful for financial support of this work by National Key Research and Development Program of China (No. 2018YFB0703802) and National Natural Science Foundation of China (Nos. NSFC51875553 and 51775536).

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