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# An Investigation on the Tribological Performances of the SiO$_2$/MoS$_2$ Hybrid Nanofluids for Magnesium Alloy-Steel Contacts

Hongmei Xie[1,2], Bin Jiang[1,3*], Bo Liu[4], Qinghang Wang[1], Junyao Xu[1] and Fusheng Pan[1,3]

## Abstract

Hybrid nano-materials offer potential scope for an increasing numerous novel applications when engineered to deliver availably functional properties. In the present study, the SiO$_2$/MoS$_2$ hybrid nanoparticles with different mass ratios were employed as lubricant additives in the base oil, and their tribological properties were evaluated using a reciprocating ball-on-plate tribometer for magnesium alloy-steel contacts. The results demonstrate that the SiO$_2$/MoS$_2$ hybrid nanoparticles exhibit superior lubrication performances than individual nano-SiO$_2$ or nano-MoS$_2$ even in high load and diverse velocity cases. The optimal SiO$_2$/MoS$_2$ mixing ratio and the concentration of SiO$_2$/MoS$_2$ hybrid nanoparticles in the base oil are 0.25:0.75 and 1.00–1.25 wt%, respectively. The excellent lubrication properties of the SiO$_2$/MoS$_2$ hybrid nanoparticles are attributed to the physical synergistic lubricating actions of nano-SiO$_2$ and nano-MoS$_2$ during the rubbing process.

**Keywords:** SiO$_2$/MoS$_2$ hybrid nanoparticles, Lubricant additive, Magnesium alloy, Friction and wear

## Background

Magnesium and its alloys are promising materials in transportation, electronics industries, or aerospace for their excellent properties, such as low density, high specific strength, and electromagnetic compatibility [1, 2]. Nowadays, magnesium alloy products are mainly fabricated by casting and die-casting [3]. However, the poor mechanical properties of the cast alloys are not sufficient to meet the demand of most load-bearing structural components. Compared with casting products, wrought magnesium alloys fabricated by plastic deformation processes, such as rolling, extrusion, and forging, seem to be more attractive owing to their competitive productivity and performance [4]. It should be noted that the tribological interaction always takes place during forming process as two contact surfaces (tool steel and the metal) move relative to each other. These

create a great challenge to achieve high-quality product and extended tool life [5].

The use of liquid lubricants to decrease friction is an efficient way to improve energy efficiency and mechanical durability, especially in the case of magnesium and its alloys. Unfortunately, so far, there are no suitable forming fluids for the forming process of Mg alloy, even at some conditions, the forming fluid used for Al alloy forming is casually used and the result is not satisfied. The application of conventional oil-based lubricant in Al alloy forming processes relies heavily on sulfur-, chlorine-, and phosphorous-containing additives. These additives form easily sheared tribo-layers on metal surfaces, thus controlling friction and reducing wear [6]. However, the fast chemical degradation of these additives during application is accompanied by a loss of their lubrication performances. Even worse, the abovementioned additives cause negative effects on the environment even at low concentrations during the disposal of waste fluids [7]. Therefore, the exploration of the harmless lubricant with the excellent tribological performances still continues. Some efforts have already been made in order to

* Correspondence: jiangbinrong@cqu.edu.cn
[1]State Key Laboratory of Mechanical Transmissions, College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China
[3]Chongqing Academy of Science and Technology, Chongqing 401123, China
Full list of author information is available at the end of the article

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find novel lubricant additives for magnesium alloy, such as N-containing compounds [8], borates [9], and ionic liquids [10]. All of them as lubricant additives are capable of forming tribo-layers with low shear strength to protect the contacting surfaces from damage, thereby improving the lubrication performances. Nonetheless, some problems are associated with their applications. For instance, many amide-based friction reducing agents for lubricating oils, including straight-chain amides, are not liquid at low temperature [11]. The borate without active elements, i.e., nitrogen, sulfur and chlorine, cannot be used as good lubricant additive for magnesium alloy. Additionally, boron is an electron-deficient element and has a great affinity with oxygen borate esters susceptible to hydrolysis in the presence of moisture [12]. The high cost and tedious procedure for the preparation of ionic liquids are main problems to put them into industrial application after 10 years of extensive development [13]. These disadvantages are key problems to replace the traditional phosphors- and chlorine-containing additives with the aforementioned additives.

Over the past few years, the addition of nanoparticles as lubricant additives into base fluids is a rapidly progressing field of research because nanoparticles are different from traditional bulk materials due to their extremely small size, high specific surface area, and variety of particle chemistries [14, 15]. Meanwhile, due to the tiny usage of nanoparticle additive, the negative influence on the environment is greatly suppressed [16–18]. Among of the nanoparticles, the layered nanomaterials with different shapes and morphologies, such as $MoS_2$ nanoparticles, are widely used as additives to liquid lubricants [19]. Previous studies have shown that $MoS_2$ nanoparticles can effectively reduce the friction in the boundary lubrication for steel/steel contacts and titanium alloy/steel pairs based on the formation of diverse types of tribo-film [20, 21]. Moreover, it was also reported that $MoS_2$ nanoparticles can decrease the friction and wear even on relatively inert surfaces, such as diamond-like carbon (DLC) film [22, 23]. Despite many attempts, the combination of nano-$MoS_2$ with other nanoparticles or compounds is of particular significance in terms of their application as lubricant additives because the combinations usually exhibit more prominent lubrication performances in contrast with individual nanoparticles attributed to the synergistic effect among two or more components. In this respect, Kunhong Hu et al. [24] synthesized $TiO_2$/$MoS_2$ nano-clusters and the tribological properties of the as-prepared $TiO_2$/$MoS_2$ nano-clusters as lubricant additive were investigated using a four-ball tribometer. It was found that the $MoS_2$/$TiO_2$ (2:1) nano-clusters achieve the lowest friction coefficient ($\mu = 0.045$), which is 30.8 and 40 % lower than pure $MoS_2$ and pure $TiO_2$, respectively. Yanbin Zhang et al. [25] reported the use of carbon nanotubes (CNT)/$MoS_2$ hybrid nanoparticles in minimum quantity lubrication for Ni-based alloy grinding. The results demonstrated that lower grinding forces and better ground surface were achieved by CNT/$MoS_2$ hybrid nanofluids. Moreover, the optimal $MoS_2$/CNT mixing ratio and nanofluid concentration are 2:1 and 6 wt%, respectively. Yufu Xu et al. [26] investigated the tribological behaviors of esterified bio-oil (EBO) and EBO containing graphene or/and $MoS_2$ for steel/steel pairs by a point contact undirectional sliding tribometer. A synergistic effect on friction reduction and wear protection was observed with the graphene/$MoS_2$ hybrids when added into EBO. These researches primarily demonstrate that $MoS_2$-based hybrid nanoparticles have great potential as lubricant additives and are worth carrying out further study. As compared with the aforementioned nanoparticles, the $SiO_2$ nanoparticles have attracted a great deal of research attention as a lubricant additive owing to excellent tribological performances, low cost, and facile preparation. Previous investigations about the lubrication properties of $SiO_2$ nanoparticles mainly focused on Al alloy forming process, such as Al alloy machining and drilling [27, 28]. Experimental results showed that $SiO_2$ nanofluids cause a decrease in friction coefficient and increase in surface quality of workpiece during machining process. This effect has been attributed to the rolling action of nanoparticles between the contact surfaces. In our early work, the tribological behaviors of nano-$SiO_2$ and nano-$MoS_2$ as lubricant additives for magnesium alloy/steel pairs have been studied and found that nano-$MoS_2$ achieves better anti-wear behavior than nano-$SiO_2$, while nano-$SiO_2$ possesses better friction reducing behavior than nano-$MoS_2$ [29]. In view of their respective special tribological characteristics, it is worth to study how the two particles behave together as lubricant additive and try to further optimize the comprehensive tribological performances.

The aim of the current study is to provide an effective $SiO_2$/$MoS_2$ hybrid nanoparticles additive for the development of magnesium alloys forming fluid. The tribological behaviors of the $SiO_2$/$MoS_2$ hybrid nanoparticles were investigated by considering the friction coefficient and wear volume. Further, the lubrication mechanism was discussed in detail.

## Methods
### Materials
EOT5# engine oil (5.11 mm$^2$/s at 40 °C and 0.856 g/cm$^3$ at 15 °C) was adopted as base oil in the tribological investigation because it is typical oil for nonferrous metal cold forming applications. According to the supplier (Hasitai Lubricant Co., Ltd Shanghai, China), the EOT5# lubricant oil is free from sulfur, phosphorus, chlorine, and other additives. The authors deliberately chose to use a lubrication of free additives as base oil, so as to isolate the effects of the nanoparticles additives. Moreover, the use of

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sulfur-, chlorine-, and phosphorus-containing compounds should be restricted for environmental reasons. The nano-MoS₂ and nano-SiO₂ used in this study were procured commercially from Nanjing Emperor Nano Material Co. Ltd, Nanjing, China. The morphology of the two samples was investigated with Zeiss AURIGA field emission scanning electron microscope (FESEM) and JEM 1200EX transmission electron microscopy (TEM) as shown in Fig. 1. It can be observed that the nano-$MoS_2$ possesses a flake-like shape mainly with above 300 nm in length and about 90 nm in thickness, and the nano-$SiO_2$ keeps a spherical micro-structure with a mean diameter of 30 nm. The nano-fluids were prepared as follows: nano-$SiO_2$, nano-$MoS_2$ and their hybrids, with different concentrations (mass fraction) of nano-$SiO_2$ and nano-$MoS_2$, were separately dispersed into the base oil through ultrasonication for 2 h to obtain a series of homogeneous suspensions.

### Friction and Wear Test

The friction reduction and anti-wear abilities of nano-fluids were evaluated using a reciprocating ball-on-plate friction and wear tester (CSM Instruments, Peseux, Switzerland). Throughout the test, the upper AISI 52100 steel ball with diameter of 6 mm and surface roughness (Ra) of 0.05 μm slides against the lower fixed AZ31 magnesium alloy plates. The starting billet of commercial

as-cast AZ31 magnesium alloy (Mg-3.07Al-0.78Zn-0.38Mn in wt%) with 82 mm in diameter was homogenized at 380 °C for 2 h. Subsequently, this ingot was extruded to the sheet of 3-mm thickness (normal direction, ND) and 56-mm width (transverse direction (TD)) at 380 °C. Meanwhile, the speed of extrusion process was 20 mm/s with the extrusion ratio of 34:1. The mechanical properties of the extruded AZ31 Mg alloy are provided in Table 1.

The as-extruded sheet was cut into samples with dimensions of 10 mm (TD) × 20 mm (ED) × 3 mm (ND). Prior to examination, the samples were polished with 1000 grit silicon carbide paper to a mean surface roughness of Ra ~0.08 μm. The ball slides at a stroke of 6 mm back and forth along extrusion direction. In the first group of experiments, the influence of the $SiO_2/MoS_2$ mixing ratio on the lubrication behaviors of nanofluids was discussed. The normal load during the tribological tests was 8 N corresponding to maximum Hertzian contact stress 446 MPa of at least 50 % higher than the yield strength of magnesium alloy sheets. The sliding speed was 30 mm/s, and the sliding test duration was 1.5 h. Lubricant oil was supplied to the top of the plate before testing, covering the entire surface. Friction coefficient and wear volume were used as characterization parameters. Five different $SiO_2/MoS_2$ mixing ratios were designed: pure $MoS_2$, $SiO_2/MoS_2$ (0.25:0.75), $SiO_2/MoS_2$ (0.5:0.5), $SiO_2/$
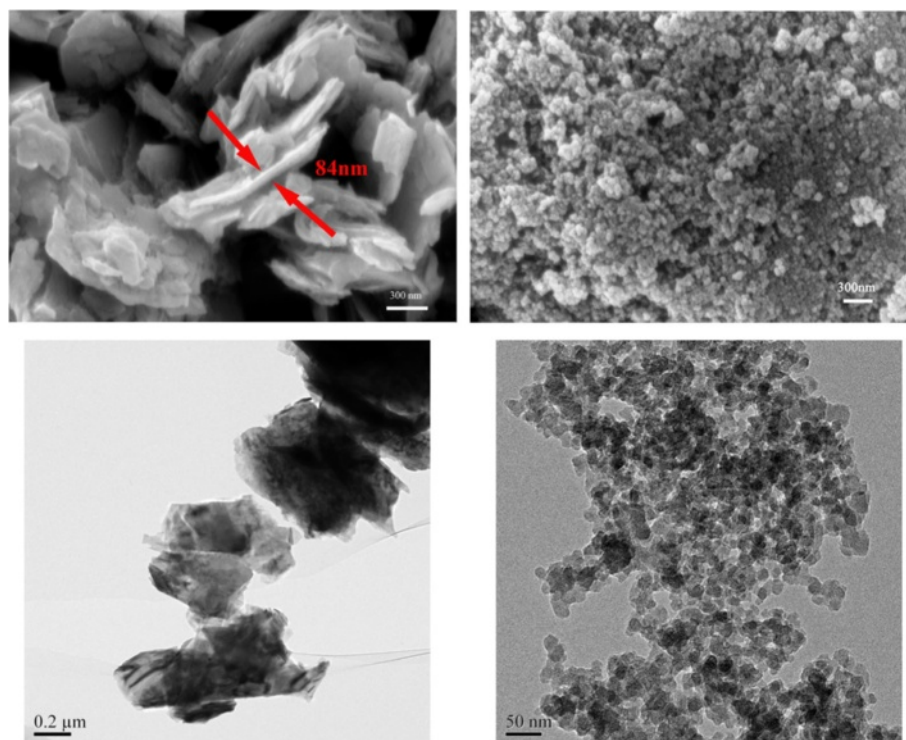


**Fig. 1** FESEM and TEM images of nano-MoS₂ (*left*) and nano-SiO₂ (*right*)

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**Table 1** Mechanical properties of extruded AZ31 magnesium alloy used in this study

| Material | 0.2 % YS/MPa | UTS/MPa | Elongation/% | $HV_{0.01}$ |
|----------|--------------|---------|--------------|-------------|
| Extruded AZ31 | 142.1 | 305 | 18.5 | 66.7 |

$MoS_2$ (0.75:0.25), and pure $SiO_2$. The concentration of these additives in the base oil was 1 wt%. The optimal $SiO_2/MoS_2$ mixing ratio was obtained from the first group of experiments. However, the concentration of nanoparticles in the base fluids is another fundamental issue because excessive concentration will make nanoparticles agglomeration and thereby destroy lubrication properties. In the second group of experiments, the influence of the concentration of $SiO_2/MoS_2$ hybrid nanoparticles on lubrication properties was discussed by changing the mass fraction of the $SiO_2/MoS_2$ hybrid nanoparticles in the base oil. The nanofluids were prepared as 0.25, 0.50, 0.75, 1.00, 1.25, 1.50, and 2.00 % by weight of $SiO_2/MoS_2$ hybrid nanoparticles into the base oil. The optimal concentration was determined. In the third group of experiments, the influence of normal load and sliding speed on the lubrication properties of the fluids was performed with a normal load varying from 1 to 10 N (corresponding maximum Hertzian contact stress from 223 to 480 MPa) and a sliding velocity ranging from 10 to 80 mm/s for 30 min. The friction coefficient was recorded by the tribometer in real time. Three sets of tests at the same normal pressure and sliding velocity were conducted to obtain each datum point to verify the repeatability and accuracy of the test results. Using nanofluids, the wear of the AISI 52100 steel ball was not measurable and there was almost no variation in the roughness measurements. For this reason, the wear volume of AZ31 magnesium alloy plate was calculated to evaluate the lubricant effectiveness in wear reduction in the overall lubricating system.

### Characterization

The morphologies of the worn surfaces were determined by Zeiss AURIGA FESEM. The wear depth and wear track profiles after the friction tests were obtained by a noncontact 3D surface profiler (Olympus OLS4000), and the wear volume of the plates was calculated from the wear depth. The final values quoted for the wear volume of the specimen were averages of three tests results. The chemical compositions of the worn surfaces were characterized by a VG model Escalab 250 X-ray photoelectron spectroscopy (XPS) with Al-Kα radiation as the excitation source. And the binding energy of C1s at 284.6 eV was utilized as the reference. Prior to the analysis, the specimens were cleaned ultrasonically for 5 min with acetone, in order to eliminate the residual lubricant.

## Results and Discussion
### Effect of $SiO_2/MoS_2$ Mixing Ratio on Lubrication Performance

Figures 2 and 3 display the friction and wear results of the $SiO_2/MoS_2$ hybrid nanofluids for magnesium alloy/steel contacts at 8 N and 30 mm/s for 1.5 h, compared with those of pure nanofluids and neat oil without nanoparticles tested in the same conditions. Among the six prepared fluids, the base oil gives the highest friction coefficient ($\mu = 0.103$). The changes in sliding time have significant effect on the friction coefficient of the base oil. It can be seen that the friction coefficient of the base oil shows considerable oscillation during the test. The friction coefficient of pure $MoS_2$ nanofluids and pure $SiO_2$ nanofluids are 0.085 and 0.078, respectively. It is evident from these measurements that pure nanoparticles as lubricant additive have positive functions for improving the friction reduction performance of the base oil. As the increase of test duration, the friction coefficient of pure $SiO_2$ nanofluids varies in the range of 0.07–0.09, accompanied by severe oscillation. While for the pure $MoS_2$ nanofluids, the friction coefficient decreases from an initial value of ~0.11 to ~0.08 after rubbing for 1000 seconds and remained essentially constant thereafter. This result proves that the pure $MoS_2$ nanofluids have much better friction reduction properties than pure $SiO_2$ nanofluids at a longer test time. However, the reduction of friction coefficient is more significant for the three $SiO_2/MoS_2$ hybrid nanofluids when compared to that found with the pure nanofluids. The improvement of lubrication performances should be ascribed to the synergistic lubricating effect of the hybrid nanoparticles during sliding. The friction coefficient of the $SiO_2/MoS_2$ hybrid nanofluids decreases with increasing mixing ratio of $SiO_2$ to $MoS_2$ until a mass ratio of 0.25:0.75 ($SiO_2/MoS_2$) is achieved. With the further increase of mixing ratio of $SiO_2$ to $MoS_2$, the friction coefficient gradually increases. The $SiO_2/MoS_2$ (0.25:0.75) hybrids achieve the lowest friction coefficient ($\mu = 0.055$), which is 35.3 and 29.5 % lower compared with pure $MoS_2$ and pure $SiO_2$, respectively. In addition, the friction coefficient of the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids shows a slightly fluctuation at the initial stage of the test and then decreases and stabilizes gradually in the latter part of the curve. The excellent lubricating performances of $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids indicate that the synergistic lubricating effect of nano-$SiO_2$ and nano-$MoS_2$ are closely related to the mixing ratio of $SiO_2$ to $MoS_2$ in the base oil.

Figure 3 shows average wear volume and 3D optical microscopic images of the wear tracks lubricated with different samples. The variation of wear volume is similar to the change in friction coefficient in Fig. 2. In this respect, the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids also outperform the rest samples. In comparison with the base oil, the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids lead to a
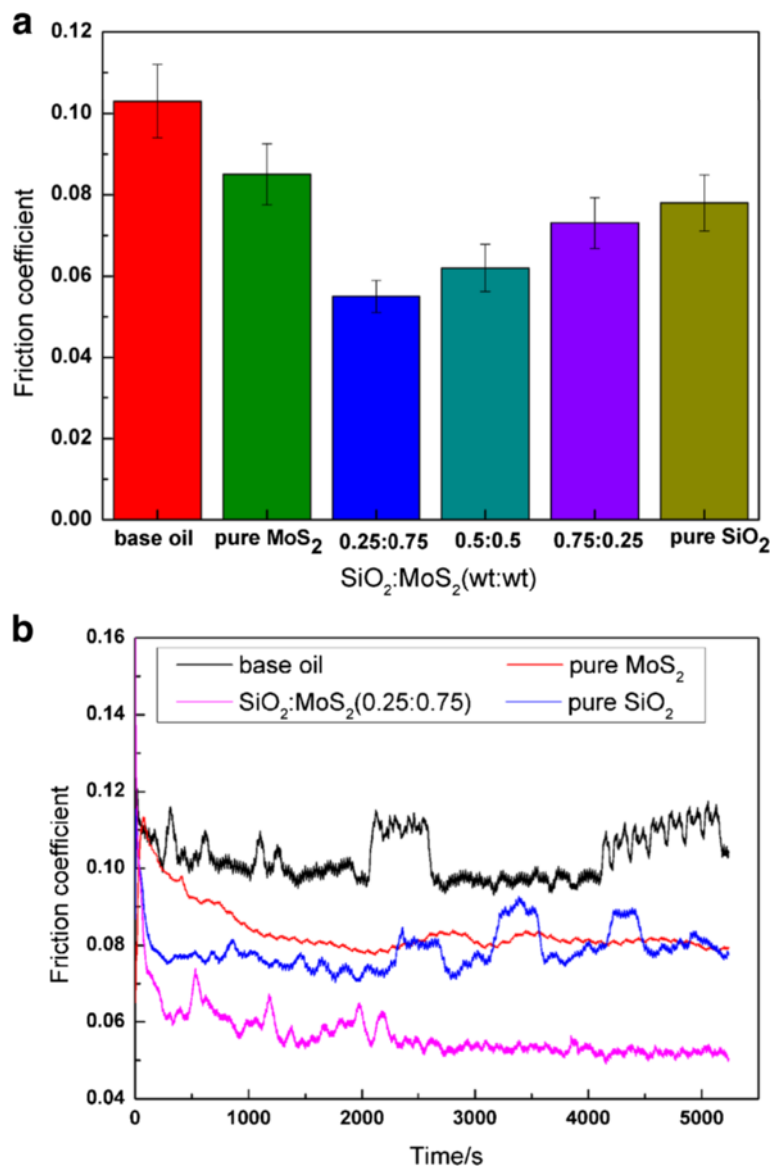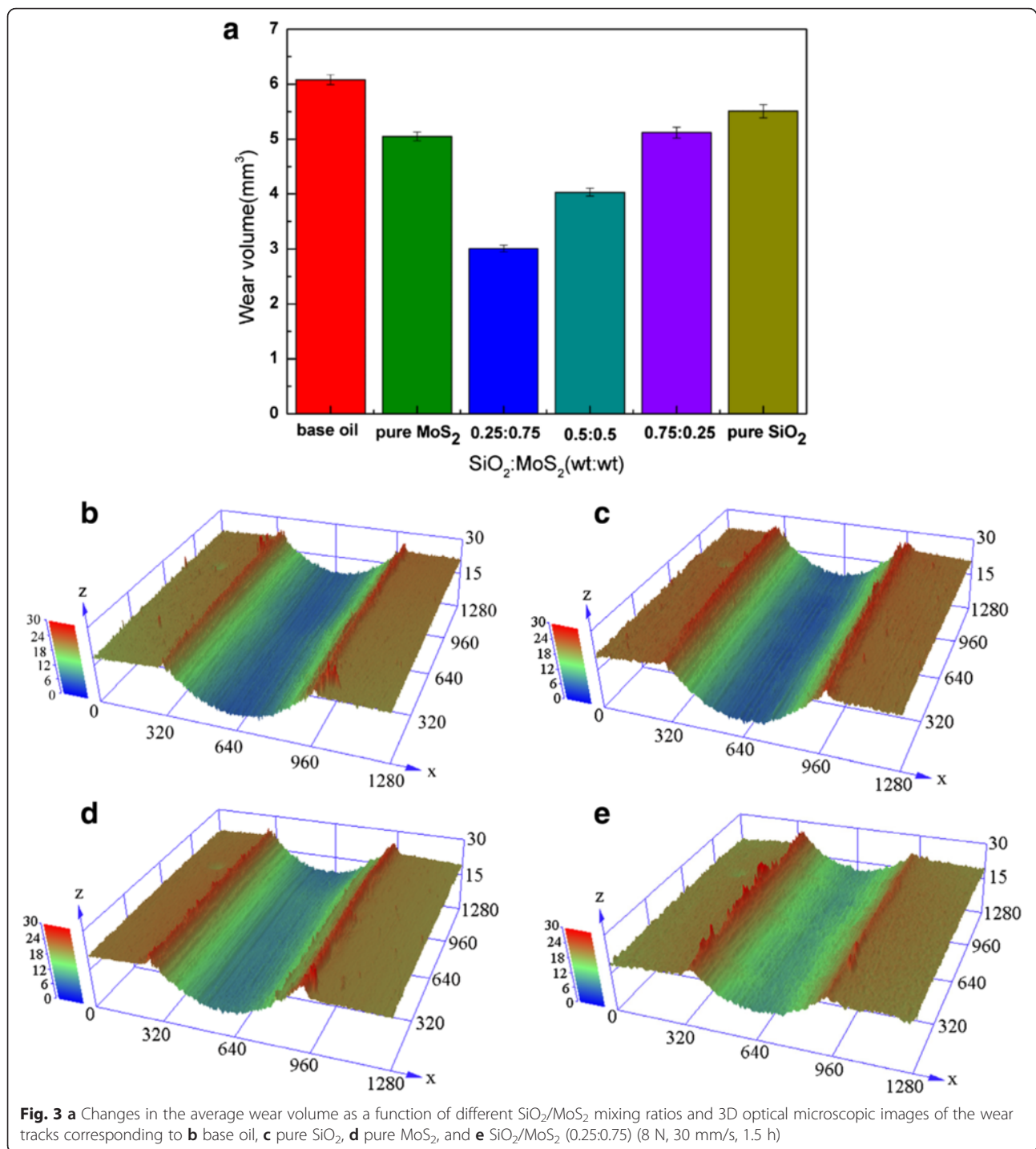
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**Fig. 2 a** Changes in the average friction coefficient as a function of different $SiO_2/MoS_2$ mixing ratios. **b** Evolution of the friction coefficient with the sliding time (8 N, 30 mm/s, 1.5 h)

significant reduction of wear volume by 50.5 % (from 6.08 to 3.01 $mm^3$ in mean value). In contrast, the pure $MoS_2$ nanofluids and $SiO_2$ nanofluids are less effective by offering 17 and 9.4 % wear volume reduction in contrast with the base oil (from 6.08 to 5.05 and 5.51 $mm^3$ in mean value), respectively. Comparative results of friction coefficient and wear volume under different $SiO_2/MoS_2$ mass ratio further prove the lubrication advantages of the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids.

### Effect of the Concentration of Hybrid Nanoparticles on the Lubrication performance

It can be concluded from the previous experiments that 0.25:0.75 is the optimal $SiO_2/MoS_2$ mixing ratio of hybrid nanoparticles. In this group of experiments, the effect of the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanoparticles concentration on the lubrication properties was investigated by changing the mass fraction (from 0.25 to 2 wt%) of the $SiO_2/MoS_2$ hybrid nanoparticles in the base oil. Figure 4 shows variations in the friction coefficient and wear volume with concentration of the $SiO_2/MoS_2$ (0.25:0.75) hybrid nanoparticles in the base oil at 8 N and 30 mm/s for 1.5 h. It can be observed that both friction coefficient and wear volume illustrate a nearly similar pattern with increasing concentration of the $SiO_2/MoS_2$ hybrid nanoparticles in the base oil. Both friction coefficient and wear volume decrease initially and then increase with increasing concentration of the $SiO_2/MoS_2$ hybrid nanoparticles. The lowest

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**Fig. 3 a** Changes in the average wear volume as a function of different $SiO_2/MoS_2$ mixing ratios and 3D optical microscopic images of the wear tracks corresponding to **b** base oil, **c** pure $SiO_2$, **d** pure $MoS_2$, and **e** $SiO_2/MoS_2$ (0.25:0.75) (8 N, 30 mm/s, 1.5 h)

friction coefficient emerges at the concentration of 1.25 wt%, and the smallest wear volume appears at the concentration of 1.00 wt%. Therefore, the optimal concentration of $SiO_2/MoS_2$ hybrid nanoparticles in the base oil is 1.00–1.25 wt%. Nanoparticle agglomeration occurs once this optimum concentration is exceeded, which will influence the lubrication efficacy [30]. On the one hand, nanoparticle agglomeration destroys the integrity of the lubrication film. On the other hand, it is also considered that the increased number of nanoparticles clusters sliding against each other can have increasing influence on friction coefficient and wear volume [31]. Based on the observations on the tribological behaviors of the $SiO_2/MoS_2$ hybrid nanofluids and cost savings, 1.00 wt%, concentration of the $SiO_2/MoS_2$ hybrid nanoparticles in the base oil was chosen for further testing.
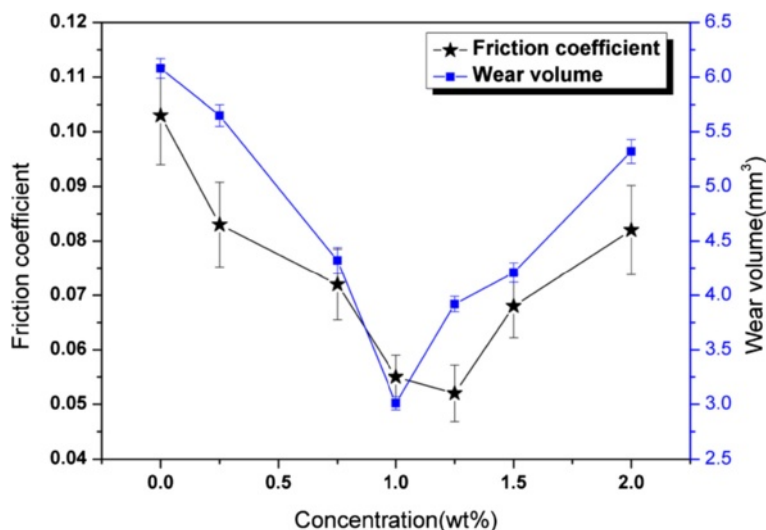
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**Fig. 4** Variations of the friction coefficients and wear volume of the plates lubricated with the SiO$_2$/MoS$_2$ (0.25:0.75) hybrid nanofluids as a function of SiO$_2$/MoS$_2$ (0.25:0.75) hybrid nanoparticles concentration (8 N, 30 mm/s, 1.5 h)

## Effect of Normal Load and Sliding Speed on Lubrication Performance

The effect of the normal load (from 1 to 10 N) on the lubrication properties of the 1.00 wt% SiO$_2$/MoS$_2$ (0.25:0.75) hybrid nanofluids was studied for magnesium alloy/steel contacts at 80 mm/s for 30 min, compared with those of pure nanofluids and neat oil without nanoparticles tested in the same conditions. The mean friction coefficient and wear volume are reported in Fig. 5. It can be observed that the average friction coefficient for base oil and pure SiO$_2$ nanofluids increases gradually with the increase of load. Higher loads can lead to smaller micro-intervals between the friction pairs. There is less base oil at a molecular level that could be drawn into the frictional interface, thus increasing friction coefficient [32]. In case of pure SiO$_2$ nanofluids, spherical SiO$_2$ nanoparticles are more likely to roll between the rubbing surfaces, thus reducing the friction coefficient [33]. However, it is necessary to emphasize that the rolling effect of spherical nanoparticles during rubbing is closely related to the thickness of the lubricant film. It has been reported in the literature [34] that when the thickness of the lubricant film is close to the size of the spherical nanoparticles, the shape of the nanoparticles is preserved. In our study, the corresponding minimum film thickness between two surfaces can be approximately predicted by the Hamrock-Dowson equation [35]. The thickness of lubricant film at the point of contact is 44, 35, 31, 28, and 26 nm for 1, 3, 5, 8, and 10 N, respectively. This would indicate that the thickness of the lubricant film between the friction pairs decreases with the increase of the load and is getting close to the size of the spherical SiO$_2$ nanoparticles. The shape of the

nanoparticles is preserved during sliding, thereby increasing friction coefficient. The friction behaviors of the pure MoS$_2$ nanofluids and the SiO$_2$/MoS$_2$ hybrid nanofluids are found to be quite different from the base oil and pure SiO$_2$ nanofluids under the same test conditions. After the nano-MoS$_2$ or SiO$_2$/MoS$_2$ hybrid nanoparticles are added into the base oil, the average friction coefficient decreases gradually with load increasing. During sliding process, part of nano-MoS$_2$ diffused into the friction region and took tribo-chemistry reaction with rubbing surface to generate a tribo-chemical reaction films which can protect magnesium alloy surface effectively. The forming velocity of tribo-chemical reaction film is closely related to the energy supplied during the rubbing process. With the load increasing, more energy will be provided and the nano-MoS$_2$ becomes easier to react with the new-exposed friction pair surfaces. This contributes to the formation of the tribo-chemical reaction film on the surface, and thus resulting in friction reduction [36]. However, the wear volume of all samples maintains a similar trend, increasing gradually with the increase in normal load. At lower loads, the anti-wear behavior is marginally different with all the nanofluids reporting similar wear volume. However, with the increased load the difference in the anti-wear behavior is substantial. Moreover, the wear volume of the SiO$_2$/MoS$_2$ hybrid nanofluids is significantly lower than that of the rest samples at high load. The different friction coefficient and wear volume can be attributed to the formation of disparate adsorption film and/or tribo-chemical reaction film on the rubbing metal surface. Detailed information on the composition of the films
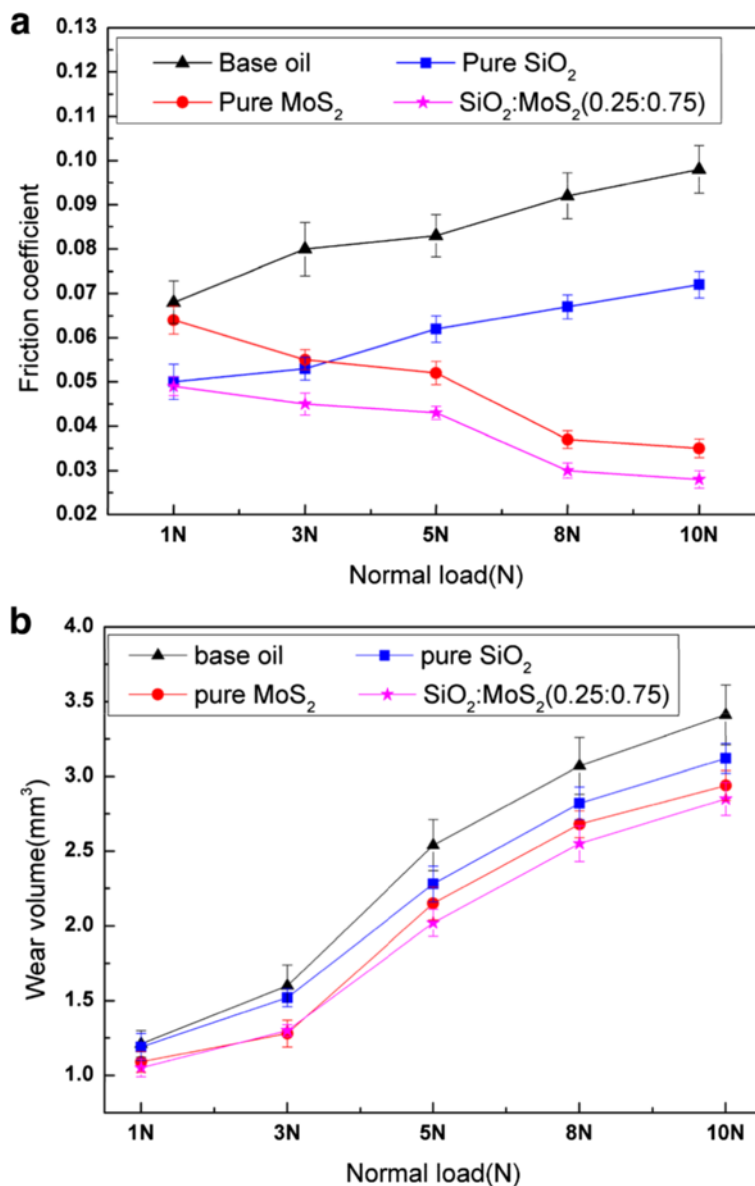
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**Fig. 5** Effects of loads on **a** average friction coefficient and **b** wear volume of magnesium alloy specimens lubricated by the base oil with and without additives (sliding speed 80 mm/s; testing time 0.5 h)

formed on the rubbing surfaces is presented in the "Rubbing Surface Analyses section."

During the tests, the sliding speed can make another significant impact on the lubrication performances. Figure 6 describes the variations of the friction coefficients and wear volumes of the plates lubricated with the $SiO_2/MoS_2$ hybrid nanofluids as a function of the sliding speed, compared with those of pure nanofluids and neat oil without nanoparticles tested in the same conditions. It can be observed that the friction coefficients of all the fluids gradually decreased with an increase in the sliding velocity over a wide range of 10 to 80 mm/s at 5 N for 30 min. With an increase in the sliding speed, a thicker liquid layer would be formed

between the shearing surfaces, thus reducing the actual contact area and the friction force. From Fig. 6b, it can be seen that the wear of magnesium alloy specimens increased with the increase in sliding speed when lubricated by all the fluids. This can be explained by the fact that there is a longer sliding distance within the same test duration at higher sliding speeds. The most significant improvement of lubricity is obtained by the $SiO_2/MoS_2$ hybrid nanofluids for the entire range of sliding velocities. The lubricating mechanism for the $SiO_2/MoS_2$ hybrid nanofluids is further analyzed in the "Lubrication Mechanism of the $SiO_2/MoS_2$ Hybrid Nanofluids" section.

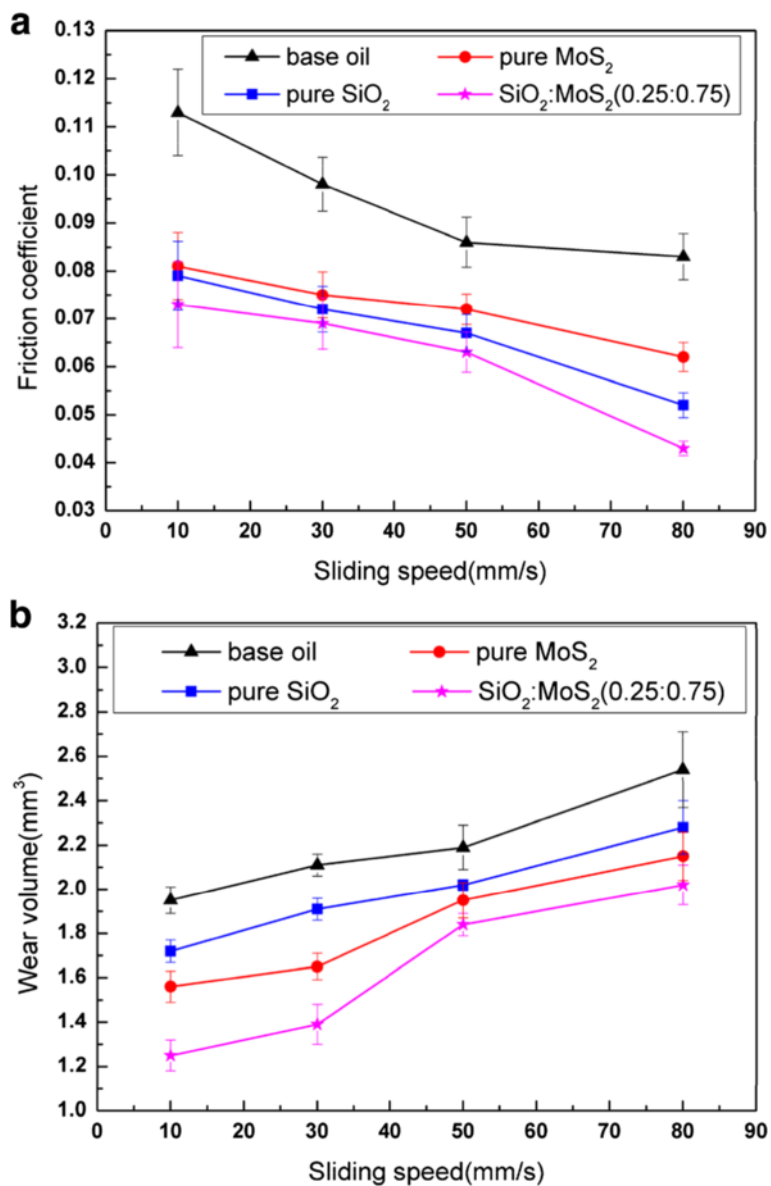Xie *et al. Nanoscale Research Letters* (2016) 11:329

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**Fig. 6** Effects of sliding speeds on **a** average friction coefficient and **b** wear volume of magnesium alloy specimens lubricated by the base oil with and without additives (normal load 5 N; testing time 0.5 h)

## Rubbing Surface Analyses

Figure 7 displays the FESEM morphologies of the worn surfaces of the magnesium alloy lubricated with base oil, pure $MoS_2$ nanofluids, pure $SiO_2$ nanofluids, and $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids under the load of 8 N and the speed of 30 mm/s for 1.5 h. It is clearly seen that the worn surface lubricated with the base oil (Fig. 7a) displays severe scuffing accompanied with serious delamination. Thus, severe wear occurs in this case. Moreover, the severely deformed material layers (Fig. 7b) are extruded along the sliding direction, forming flares and sizeable bulbs at the periphery of the sliding track. However, it should be mentioned that the severely deformed material layers are not observed at the periphery of the sliding track lubricated with nanofluids (Fig. 7d, f, h). Even so, the worn surfaces under the lubrication of various nanofluids are very diverse. In the presence of nano-$SiO_2$ (Fig. 7c), the wear process slows down. The region of the worn surface is clean with several thin furrows, which indicates that $SiO_2$ nanophases can make the lubricant system more energy efficient in contrast with the base oil. Figure 7e shows the worn surface lubricated with pure $MoS_2$ nanofluids. There are a lot of small furrows and scratches on the friction surface, and a partial coverage of protection film is formed during sliding process. In contrast, the worn surface lubricated with the $SiO_2/MoS_2$ hybrid nanofluids
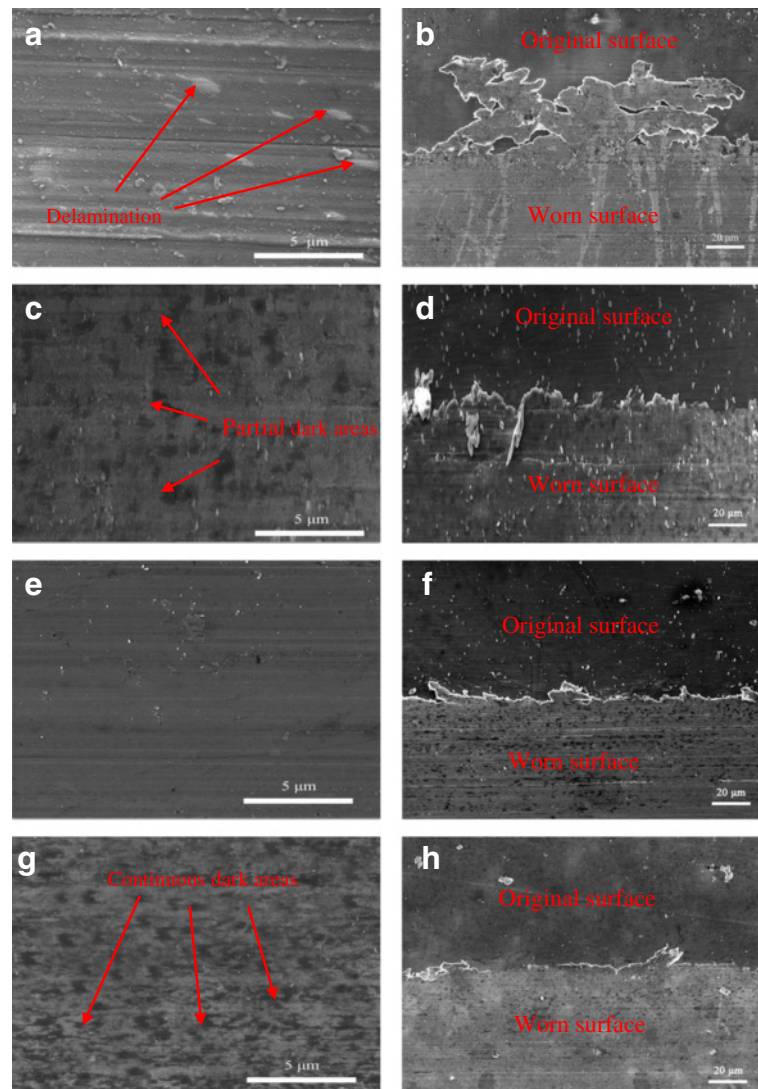
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**Fig. 7** The worn surface morphologies and the edge analysis of the worn surface lubricated with **a**, **b** base oil, **c**, **d** pure MoS₂ nanofluids, **e**, **f** pure SiO₂ nanofluids, and **g**, **h** SiO₂/MoS₂ (0.25:0.75) hybrid nanofluids (8 N, 30 mm/s, 1.5 h)

(Fig. 7g) is quite smooth and shows only slight grooves. More importantly, the continuous dark areas can be found throughout the wear track, indicating the formation of a compact protection film. This compact protection film is believed to be responsible for the SiO₂/MoS₂ hybrid nanofluids' friction reduction and anti-wear functionality. The FESEM images of worn surface were in good agreement with the measured tribological behaviors.

The XPS analyses of Mo3d, S2p, O1s, Fe2p, and Si2p on the wear tracks lubricated with pure MoS₂ nanofluids or SiO₂/MoS₂ hybrid nanofluids were further carried out to monitor the chemical changes. As shown in Fig. 8a, c, e, g concerning the rubbed surface lubricated with pure MoS₂ nanofluids, Mo3d exists as MoS₂ with peaks at 230.6 and 229.2 eV [37]. This may indicate that nano-MoS₂ have physical adsorption on the worn surfaces.

The additional contribution of Mo3d at higher energy, in association with the O1s peak at 530.5 eV, can be attributed to oxygenated Mo (VI) species. It is reasonable to presume that the adsorbed MoS₂ platelets were oxidized partly into oxides of molybdenum during the rubbing process [38]. The XPS peak of the S2p spectrum appears at 163.2 eV, which corresponds to MoS₂. Apart from MoS₂, the weak S2p peak at higher binding energy (169.5 eV) is ascribed to MgSO₄. Even so, the O1s signal assigned to MgSO₄ is not observed in Fig. 8e. A possible reason for this phenomenon is that the amount of MgSO₄ is not enough to affect the XPS spectra of O1s. Besides, the peak of S2p at 161.9 eV is difficult to be clarified because the binding energy of iron sulfide species (FeS) and molybdenum disulfide (MoS₂) is both between 161 and 162 eV [37]. In order to determine the actual type of
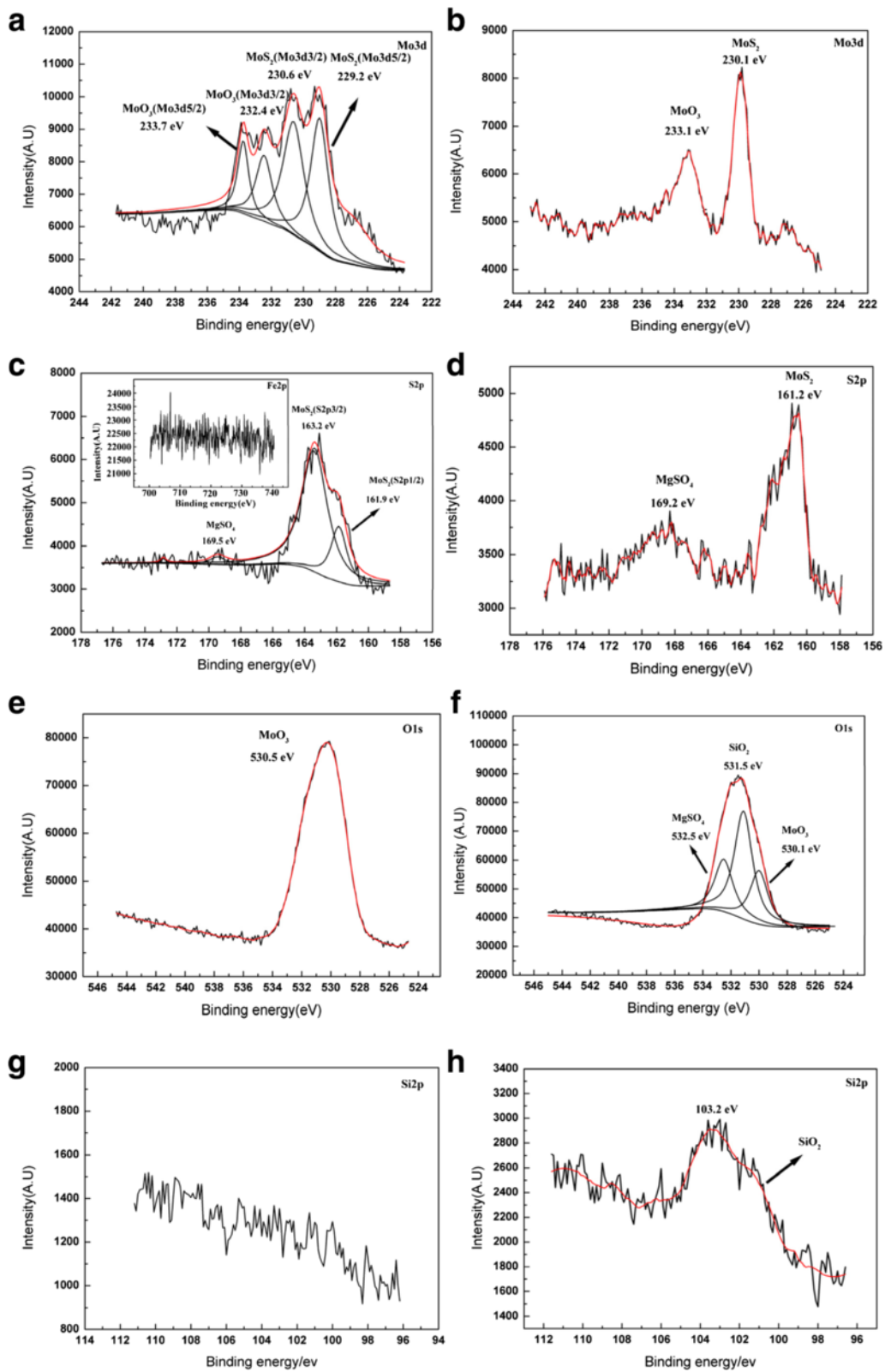
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**Fig. 8** X-ray photoelectron spectra of the elements (Mo3d, S2p, O1s, Fe2p (inset of Fig. 11c), and Si2p) on the worn surface lubricated with pure MoS$_2$ nanofluids (**a**, **c**, **e**, **g**) and SiO$_2$/MoS$_2$ (0.25:0.75) hybrid nanofluids (**b**, **d**, **f**, **h**) (8 N, 30 mm/s, 1.5 h)

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compound, the XPS of Fe2p was studied as shown in the inset of Fig. 8c. It can be seen that the signal of iron was not detected. Therefore, we argue that the peak of S2p at 161.9 eV comes from $MoS_2$ and not FeS. For the $SiO_2/MoS_2$ hybrid nanofluids, the Mo3d spectrum appears at approximately 230.1 and 233.1 eV (Fig. 8b), which correspond to $MoS_2$ (in association with S2p peak at 161.2 eV) and $MoO_3$ (in combination with O1s peak at 530.4 eV), respectively. There are some differences between the samples with respect to the distribution of the oxides of molybdenum. The sample lubricated with the $SiO_2/MoS_2$ hybrid nanofluids shows less oxide present compared to the sample lubricated with pure $MoS_2$ nanofluids. It suggests that the introduction of nano-$SiO_2$ prevents the oxidation of the $MoS_2$ on the specimen surface. The S2p spectrum for the $SiO_2/MoS_2$ hybrid nanofluids consists of two peaks (Fig. 8d). The peak at 161.2 eV is attributed to $MoS_2$, and the peak located at 169.2 eV is assigned to $MgSO_4$ (in association with the O1s peak at 532.5 eV). Interestingly, it can be seen from the spectra that the wear tracks lubricated with $SiO_2/MoS_2$ hybrid nanofluids show a much stronger signal for $MgSO_4$ in contrast to the wear tracks lubricated with pure $MoS_2$ nanofluids. The higher intensity for the $SiO_2/MoS_2$ hybrid nanofluids may lie in the fact that $MoS_2$ mixed with $SiO_2$ can easily react with magnesium alloy, indicating that more compact tribochemical film is obtained on the worn surface lubricated with the $SiO_2/MoS_2$ hybrid nanofluids. The result is also confirmed by the FESEM images in Fig. 7c. The other interesting difference is that Si2p at 103.2 eV (Fig. 8h) ascribed to $SiO_2$ is found on the worn surface lubricated with the $SiO_2/MoS_2$ hybrid nanofluids [39], whereas at the same position, there are no clear signal peaks when lubricated by pure $MoS_2$ nanofluids (Fig. 8g). Therefore, it can be concluded that the nano-$SiO_2$ participated in the formation of the lubricating film and collaborated with the nano-$MoS_2$ to lubricate the magnesium alloy surface. These results confirm that the positive effect of the $SiO_2/MoS_2$ hybrid nanofluids is more pronounced in terms of the lubrication properties when compared to that found with the pure $MoS_2$ nanofluids.

## Lubrication Mechanism of the $SiO_2/MoS_2$ Hybrid Nanofluids

According to the friction tests run on a reciprocation ball-on-plate friction tester, it can be found that the $SiO_2/MoS_2$ hybrid nanoparticles as lubricant additive exhibit superior lubricating performances than individual nanopartcles. To understand the mechanism that is underlying the trends seen in the lubrication responses, the high magnification FESEM-EDS spectra of the worn surface lubricated with the 1.00 wt% $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids is chosen for detailed analyses in Fig. 9. The schematic explanation of the corresponding lubricating mechanisms is summarized in Fig. 10. Figure 9 clearly shows that nano-$MoS_2$ protects the surface of magnesium alloy in two ways. At first, nano-$MoS_2$ is physically adsorbed on the contact surface via the flow of lubricating oil and compressive stress. Intrinsically, hexagonal lamella of nano-$MoS_2$ is stacked by weak van der Waals force between layers. Low resistance to shear between the weakly interacted lamella under the sliding contact stress reduced the friction coefficient. Besides that, when a normal force exists, the $MoS_2$ nanoparticle will extend into thin physical films in the contact zone. As the test time goes on, the applied load during sliding will produce high temperature and pressure. The deposited nano-$MoS_2$ on the surfaces will undergo complex tribo-chemical reactions, resulting in forming a new chemical transfer film on the lubricated metal surface. Combined with the XPS spectra (Fig. 8), the tribo-chemical reaction film is composed of $MoO_3$ and $MgSO_4$. The tribo-chemical reaction film strongly combines with the workpiece material, which is essential for efficient long-term lubrication. The mechanism of the tribo-chemical reaction can be directly compared to those obtained in the literature concerning the interactions between $MoS_2$ nanoparticles and steel surface [37]. Figure 11 is drawn to explain the possible binding mechanism of $MoS_2$ on the magnesium alloy surface. Two possible mechanisms were proposed for the interactions between $MoS_2$ nanoparticles and steel surface. The first is associated with a tribo-film adhesion through the bonding of molybdenum with the oxygen that was found in the native surface oxide layer of the steel. Correspondingly, a possible chemical reaction between the molybdenum and the oxygen from MgO is chemically reasonable. This is due to the fact that chemical reaction occurs between MgO and the nano-$MoS_2$ could be explained by a lower bonding energy between Mg and O than between Fe and O on the basis of Pearson hardness (Fig. 11a). Another possibility is that the oxide layer would be removed during the first cycles of the friction test and the reaction between the nano-$MoS_2$ and the Mg metal atoms could occur (Fig. 11b). At this stage, the precise role of the oxide layer in the mechanism of adhesion of the tribo-film on the worn surface is still extremely difficult to judge from the experiments results. Even so, it was shown in this study that a tribo-chemical reaction between the nano-$MoS_2$ platelets could occur via either the native oxide layer or the metal Mg atoms during the frictional process. Besides that, further understanding the role of the protect film at disparate stage of the whole test is crucial and necessary. As shown in Fig. 2, it is apparent that the friction coefficient of pure $MoS_2$ nanofluids is higher during the first 1000 s and then decreases slowly until reaches a steady state. However, this high friction coefficient period was not observable with the pure $SiO_2$ nanofluids and the $SiO_2/MoS_2$ hybrid nanofluids. Similar evolution of friction coefficient with time for a
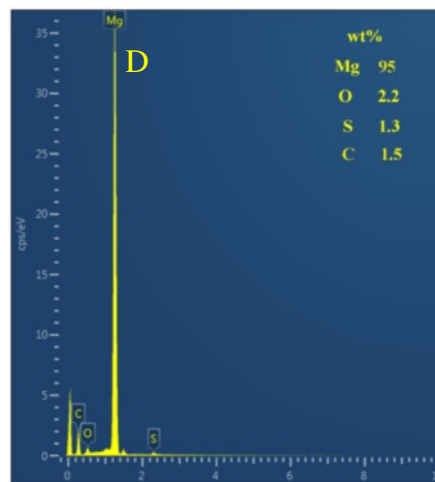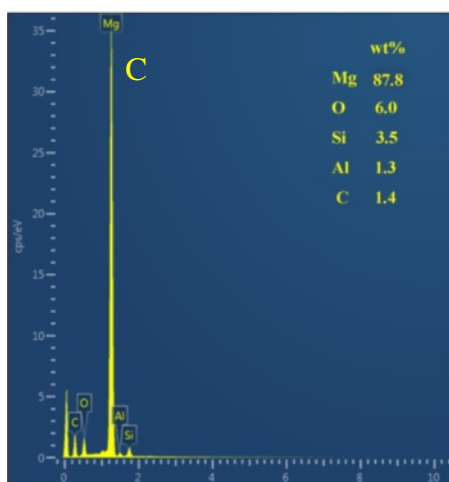
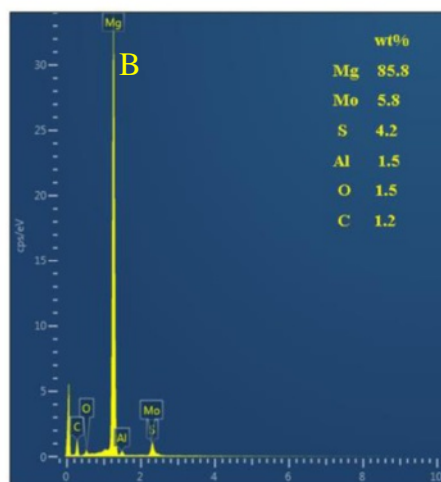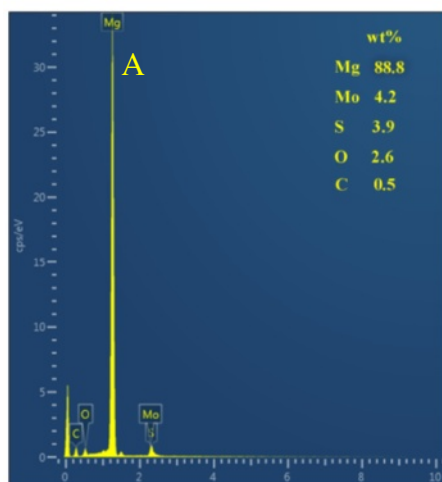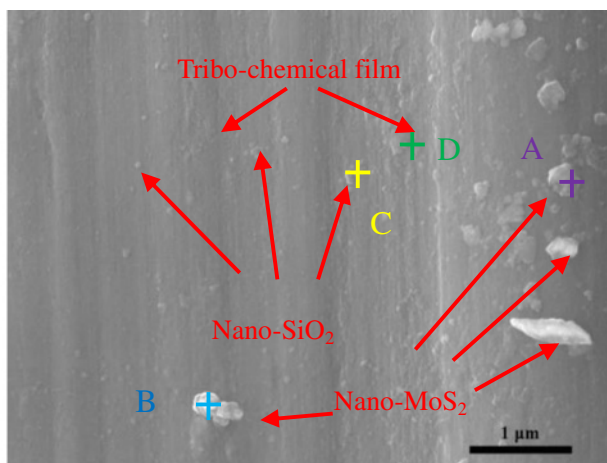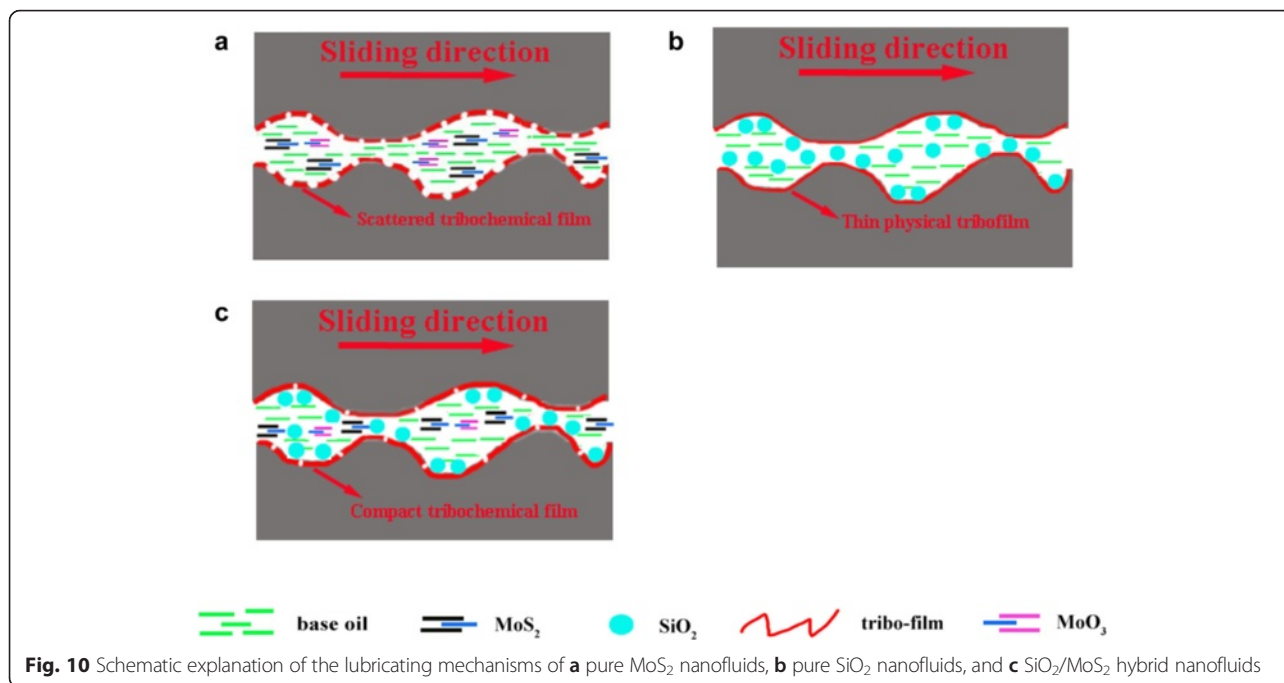Xie *et al. Nanoscale Research Letters* (2016) 11:329

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**Fig. 9** High-magnification FESEM-EDS spectra of worn plate surfaces lubricated with the 1.00 wt% $SiO_2/MoS_2$ (0.25:0.75) hybrid nanofluids (8 N, 30 mm/s, 1.5 h)

steel-steel contact lubricated with pure $MoS_2$ nanofluids, where a higher friction coefficient at the early stage of the test can be seen, was reported by L. Cizaire et al [40]. These authors explain that the friction coefficient is higher at the
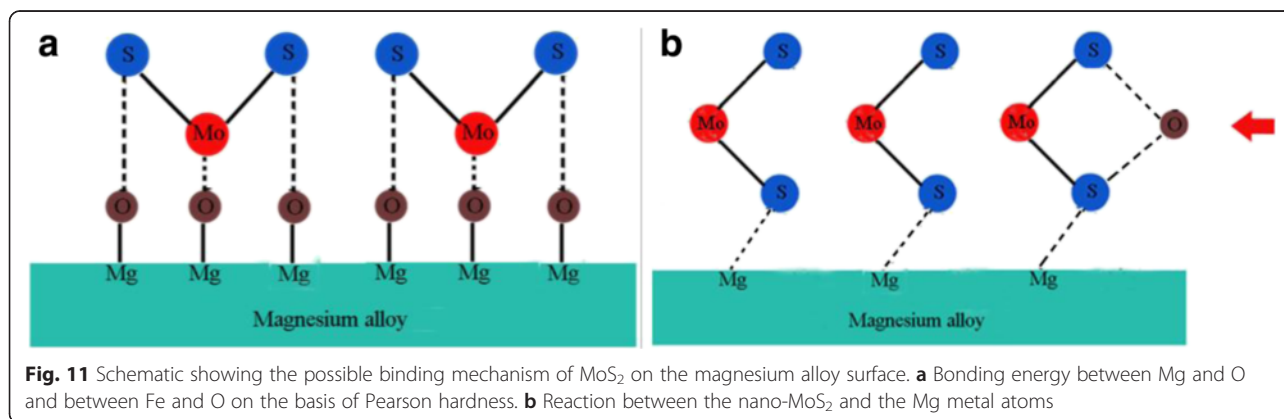
beginning of the test owing to the absent of the tribo-chemical film. However, the friction coefficient gradually decreased once the tribo-chemical film formed during the rubbing process [41]. In comparison with pure nano-$MoS_2$

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**Fig. 10** Schematic explanation of the lubricating mechanisms of **a** pure MoS$_2$ nanofluids, **b** pure SiO$_2$ nanofluids, and **c** SiO$_2$/MoS$_2$ hybrid nanofluids

additive (Fig. 10a), the SiO$_2$/MoS$_2$ hybrid nanoparticles additive with better catalytic activity may catalyze the oxidation of S$^{2-}$ more quickly during the sliding [42]. So the running-in time required to obtain very low friction values for SiO$_2$/MoS$_2$ hybrid nanoflids is shorter than pure MoS$_2$ nanofluids. Even under static conditions, the chemical reaction does not stop and more and more tribo-chemical compound is generated, leading to the formation of more compact and efficient tribo-chemical film on the worn surface (Fig. 10c).

In contrast, the lubricity mechanism of spherical nano-SiO$_2$ is quite different from that of nano-MoS$_2$ platelets. The higher-magnification image of the worn surface in Fig. 9a and corresponding EDS reveal the presence of nano-SiO$_2$ on the worn surface. Unlike nano-MoS$_2$, nano-SiO$_2$ possesses nano-scale size and excellent dispersion in

the base lubricant. These characteristics allow the nano-SiO$_2$ to easily enter the contact area, thereby resulting in faster running-in conditions (Fig. 2). The rolling effect was often proposed as a lubrication mechanism for spherical nanoparticles, although little research has been conducted on the direct visualization of the rolling behavior of the nano-SiO$_2$ into the contact interfaces [33, 43]. In this way, nanoparticles decrease the shear stress and lead to the reduction in friction coefficient. Besides, even though the surface of the magnesium alloy plate was looking smooth, when we observed its micro meter and nanoscale image, the surface was complicated with ridges and valleys. When nanoparticles were added into the base oil, the filling of valleys of the contacting asperities will occur. This is also supported by the high magnification FESEM spectra (Fig. 9), which shows the distribution of nanoparticles on



**Fig. 11** Schematic showing the possible binding mechanism of MoS$_2$ on the magnesium alloy surface. **a** Bonding energy between Mg and O and between Fe and O on the basis of Pearson hardness. **b** Reaction between the nano-MoS$_2$ and the Mg metal atoms

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the worn surface. As shown in Fig. 9, it is clearly seen that nano-$SiO_2$ fills up the grooves of the rubbing surfaces, while nano-$MoS_2$ deposits on the flat of the worn surfaces. It demonstrates that the difference in the distribution of nanoparticles on the worn surface is substantial. This can be explained by the model proposed by Mosleh Mohsen [21]. The authors suggested that the nanoparticles can fill up valleys and smooth the surface if the characteristic length $l$ of flaky nanoparticles is smaller than the peak-to-valley roughness of the harder surface which is equal to 4 Ra, i.e., the condition of $l < 4$ Ra applies. With respect to spherical nanoparticles, the diameter $d$ of the particle should satisfy $d < 0.67$ Ra. Comparatively, nano-$SiO_2$ with an average size of about 30 nm acts as a third body material filling in the valley of surface with 0.08-μm surface roughness, increasing real contact area. As a result, the load can be distributed over a larger contact area, so that the effective contact pressure decreases and consequently the wear also reduce. However, nano-$MoS_2$ platelets may be difficult to fill in the cavities because its length (more than 300 nm) is larger than 4 Ra in our study. In comparison with pure nano-$SiO_2$ additive (Fig. 10b), the micro-cooperation for $SiO_2$/$MoS_2$ hybrid nanoparticles additive occurred in succession between the shearing-sliding of the nano-$MoS_2$ platelets structure and the filling of nano-$SiO_2$ particles during sliding process, resulting in excellent lubrication performances (Fig. 10c). However, the efficiency of the $SiO_2$/$MoS_2$ hybrid nanofluids was influenced by the mass ratio of $SiO_2$/$MoS_2$. For instance, the lubrication performances of $SiO_2$/$MoS_2$ (0.5:0.5) and $SiO_2$/$MoS_2$ (0.75:0.25) were a little inferior to that of the $SiO_2$/$MoS_2$ (0.25:0.75) at the test condition. Namely, there was an optimum dose of nano-$SiO_2$ in the $SiO_2$/$MoS_2$ hybrid nanofluids. If excess nano-$SiO_2$ was added into the $SiO_2$/$MoS_2$ hybrid nanofluids, the contact area was saturated with nano-$SiO_2$. Any more nano-$SiO_2$ took up the space of nano-$MoS_2$ and disturbed its lubrication. The nano-$SiO_2$ has outstanding mechanical properties especially in terms of hardness (Vickers hardness—1000 kgf/mm$^2$); therefore, the excess hard nano-$SiO_2$ as an abrasive plows the soft magnesium alloy surfaces (Vickers hardness—66.7 kgf/mm$^2$) under the applied load. It will facilitate the abrasion of the magnesium alloy surface during rubbing process and thus results in the worse anti-wear property. This was confirmed by the poor anti-wear behaviors of the samples with high concentrations of nano-$SiO_2$, such as $SiO_2$/$MoS_2$ (0.5:0.5), $SiO_2$/$MoS_2$ (0.75:0.25) and pure nano-$SiO_2$ as shown in Fig. 3a. Therefore, the nano-$SiO_2$ and nano-$MoS_2$ mass ratio of 0.25:0.75 is the best proportion for synergetic lubrication effect under the selected testing conditions. In addition, from the XPS results of the worn magnesium alloy surface, nano-$SiO_2$ is shown to prevent nano-$MoS_2$ from oxidation, resulting in increased oxidation resistance of nano-$MoS_2$ during rubbing process. The oxidation phenomenon may provide an important role in the sense. It could provide a "soft" oxide-like $MoO_3$ which could serve to tether the nanoparticles to the magnesium alloy surface. However, if nano-$MoS_2$ is overmuch oxidized into $MoO_3$, the oxidation destroys the lubrication structure of layered $MoS_2$ and thus results in the worse lubrication property [37]. Therefore, the synergistic lubricating effect enables the $SiO_2$/$MoS_2$ hybrid nanofluids to integrate the advantages and eradicate the disadvantages of $SiO_2$ and $MoS_2$ nanoparticles at optimal ratio. So the $SiO_2$/$MoS_2$ (0.25:0.75) hybrid nanoparticles exhibit superior lubricating performances than individual nano-$SiO_2$ or nano-$MoS_2$ even in high load and diverse velocity cases. Previous work on the combination of $MoS_2$ with other nanoparticles as lubricant additives has reported improvements in their lubricating performance, such as nano-$TiO_2$ [24], carbon nanotubes (CNT) [25], and graphene [26]. Since the testing method and analysis are completely different from the present study, comparable comparison between the results cannot be made. Even so, nano-$SiO_2$ is less costly and facile preparation in contrast with the aforementioned nanoparticles. In conclusion, the $SiO_2$/$MoS_2$ hybrid nanoparticles are suggested as economic and environment friendly lubricant additives for the applications in forming process of magnesium alloy.

## Conclusions

In the present study, the tribological properties of the $SiO_2$/$MoS_2$ hybrid nanofluids to be used in metal forming fluids for magnesium alloy were investigated by a reciprocating sliding ball-on-plate contact configuration. Based on the experimental results, the following conclusions were drawn:

(1) Compared with pure nanofluids, the $SiO_2$/$MoS_2$ hybrid nanofluids achieve lower friction coefficient and wear volume even in high load and diverse velocity cases for magnesium alloy/steel contacts under the test conditions.

(2) The optimal $SiO_2$/$MoS_2$ mixing ratio and nanofluids concentration are 0.25:0.75 and 1.00–1.25 wt%, respectively. The 1.00 wt% $SiO_2$/$MoS_2$ (0.25:0.75) hybrid nanoparticles addition into the base oil shows reduction of friction coefficient by 46.6 % and reduction of wear volume by 50.5 % in contrast with the base oil.

(3) The excellent lubrication properties of the $SiO_2$/$MoS_2$ hybrid nanoparticles are ascribed to the physical synergistic lubricating actions of nano-$SiO_2$ and nano-$MoS_2$ during the rubbing process. The synergistic lubricating effect enables the $SiO_2$/$MoS_2$ hybrid nanofluids to integrate the advantages and eliminate the disadvantages of $SiO_2$ and $MoS_2$ nanoparticles at optimal ratio.

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## Authors' Contributions

HMX and BJ carried out the design and drafted the manuscript. BL and QHW prepared samples and carried out tribo-tests. JYX and FSP commented on the results and revised the manuscript. All authors read and approved the final version of the manuscript.

## Author details

[1]State Key Laboratory of Mechanical Transmissions, College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China. [2]College of Mechanical and Electrical Engineering, Yangtze Normal University, Chongqing 408100, China. [3]Chongqing Academy of Science and Technology, Chongqing 401123, China. [4]Chongqing Chang-an Automobile Co., Ltd, Chongqing 400023, China.

## References

1. Md FK, Panigrahi SK (2015) Age hardening, fracture behavior and mechanical properties of QE22 Mg alloy. J Magnes Alloy 3:210–217
2. Sunil BR, Ganesh KV, Pavan P, Vadapalli G, Swarnalatha C, Swapna P, Bindukumar P, Pradeep Kumar Reddy G (2016) Effect of aluminum content on machining characteristics of AZ31 and AZ91 magnesium alloys during drilling. J Magnes Alloy 4:15–21
3. Griffiths D (2015) Explaining texture weakening and improved formability in magnesium rare earth alloys. Mater Sci Technol 31:10–24
4. Selvam B, Marimuthu P, Narayanasamy R, Senthilkumar V, Tun KS, Gupta M (2015) Effect of temperature and strain rate on compressive response of extruded magnesium nano-composite. J Magnes Alloy 3:224–230
5. Heinrichs J, Jacobson S (2010) Laboratory test simulation of aluminium cold forming—influence from PVD tool coatings on the tendency to galling. Surf Coat Tech 204:3606–3613
6. Njiwa P, Hadj-Aïssa A, Afanasiev P, Geantet C, Bosselet F, Vacher B, Belin M, Le Mogne T, Dassenoy F (2014) Tribological properties of new MoS$_2$ nanoparticles prepared by seed-assisted solution technique. Tribol Lett 55:473–481
7. Dennis JES, Jin K, John VT, Pesika NS (2011) Carbon microspheres as ball bearings in aqueous-based lubrication. Acs Appl Mater Inter 3:2215–2218
8. Huang WJ, Du CH, Li ZF, Liu M, Liu WM (2006) Tribological characteristics of magnesium alloy using N-containing compounds as lubricating additives during sliding. Wear 260:140–148
9. Huang WJ, Fu Y, Wang J, Li ZF, Liu WM (2005) Effect of chemical structure of borates on the tribological characteristics of magnesium alloy during sliding. Tribol Int 38:775–780
10. Xia YQ, Jia ZF, Jia JH (2010) Tribological behavior of AZ91D magnesium alloy against SAE52100 steel under ionic liquid lubricated conditions. In: Advanced Tribology, Springer., pp 896–898
11. Tang ZL, Li SH (2014) A review of recent developments of friction modifiers for liquid lubricants (2007–present). Curr Opin Solid ST M 18:119–139
12. Li ZP, Li XF, Zhang YW, Ren TH, Zhao YD, Zeng XQ, van der Heide E (2014) Tribological study of a highly hydrolytically stable phenylboronic acid ester containing benzothiazolyl in mineral oil. Appl Surf Sci 308:91–99
13. Somers AE, Khemchandani B, Howlett PC, Sun J, MacFarlane DR, Forsyth M (2013) Ionic liquids as antiwear additives in base oils: influence of structure on miscibility and antiwear performance for steel on aluminum. Acs Appl Mater Inter 5:11544–11553
14. Fan XQ, Wang LP, Li W, Wan SH (2015) Improving tribological properties of multialkylated cyclopentanes under simulated space environment: two feasible approaches. Acs Appl Mater Inter 7:14359–14368
15. Fan XQ, Wang LP (2015) High-performance lubricant additives based on modified graphene oxide by ionic liquids. J Colloid Interf Sci 452:98–108
16. Rahmati B, Sarhan AAD, Sayuti M (2014) Morphology of surface generated by end milling AL6061-T6 using molybdenum disulfide (MoS$_2$) nanolubrication in end milling machining. J Clean Prod 66:685–691
17. Deorsola FA, Russo N, Blengini GA, Fino D (2012) Synthesis, characterization and environmental assessment of nanosized MoS$_2$ particles for lubricants applications. Chem Eng J 195–196:1–6
18. Chen Q, Wang X, Wang ZT, Liu Y, You TZ (2013) Preparation of water-soluble nanographite and its application in water-based cutting fluid. Nanoscale Res Lett 8:52–60
19. Fan XQ, Wang LP, Li W (2015) In situ fabrication of low-friction sandwich sheets through functionalized graphene crosslinked by ionic liquids. Tribol Lett 58:1–12
20. Kogovšek J, Remškar M, Mrzel A, Kalin M (2013) Influence of surface roughness and running-in on the lubrication of steel surfaces with oil containing MoS$_2$ nanotubes in all lubrication regimes. Tribol Int 61:40–47
21. Mosleh M, Atnafu ND, Belk JH, Nobles OM (2009) Modification of sheet metal forming fluids with dispersed nanoparticles for improved lubrication. Wear 267:1220–1225
22. Kogovšek J, Remškar M, Kalin M (2013) Lubrication of DLC-coated surfaces with MoS$_2$ nanotubes in all lubrication regimes: surface roughness and running-in effects. Wear 303:361–370
23. Kalin M, Kogovšek J, Kovač J, Remškar M (2014) The formation of tribofilms of MoS$_2$ nanotubes on steel and DLC-coated surfaces. Tribol Lett 55:381–391
24. Hu KH, Huang F, Hu XG, Xu YF, Zhou YQ (2011) Synergistic effect of nano-MoS$_2$ and anatase nano-TiO$_2$ on the lubrication properties of MoS$_2$/TiO$_2$ nano-clusters. Tribol Lett 43:77–87
25. Zhang YB, Li CH, Jia DZ, Zhang DK, Zhang XW (2015) Experimental evaluation of the lubrication performance of MoS$_2$/CNT nanofluid for minimal quantity lubrication in Ni-based alloy grinding. Int J Mach Tool Manu 99:19–33
26. Xu YF, Peng YB, Dearn KD, Zheng XJ, Yao L, Hu XG (2015) Synergistic lubricating behaviors of graphene and MoS$_2$ dispersed in esterified bio-oil for steel/steel contact. Wear 342–343:297–309
27. Pujante J, Pelcastre L, Vilaseca M, Casellas D, Prakash B (2013) Investigations into wear and galling mechanism of aluminium alloy-tool steel tribopair at different temperatures. Wear 308:193–198
28. Sayuti M, Sarhan AAD, Hamdi M (2012) An investigation of optimum SiO$_2$ nanolubrication parameters in end milling of aerospace Al6061-T6 alloy. Int J Adv Manuf Technol 67:833–849
29. Xie HM, Jiang B, He JJ, Xia XS, Pan FS (2016) Lubrication performance of MoS$_2$ and SiO$_2$ nanoparticles as lubricant additives in magnesium alloy-steel contacts. Tribol Int 93:63–70
30. Rudenko P, Bandyopadhyay A (2013) Talc as friction reducing additive to lubricating oil. Appl Surf Sci 276:383–389
31. Singh VK, Elomaa O, Johansson L-S, Hannula S-P, Koskinen J (2014) Lubricating properties of silica/graphene oxide composite powders. Carbon 79:227–235
32. Xu YF, Zheng XJ, Yin YG, Huang J, Hu XG (2014) Comparison and analysis of the influence of test conditions on the tribological properties of emulsified bio-oil. Tribol Lett 55:543–552
33. Ge X, Xia Y, Cao Z (2015) Tribological properties and insulation effect of nanometer TiO$_2$ and nanometer SiO$_2$ as additives in grease. Tribol Int 92:454–461
34. Rapoport L, Leshchinsky V, Lapsker I, Volovik Y, Nepomnyashchy O, Lvovsky M, Popovitz-Biro R, Feldman Y, Tenne R (2003) Tribological properties of WS$_2$ nanoparticles under mixed lubrication. Wear 255:785–793
35. Zhang LL, Pu JB, Wang L, Xue QJ (2015) Synergistic effect of hybrid carbon nanotube-graphene oxide as nanoadditive enhancing the frictional properties of ionic liquids in high vacuum. Acs Appl Mater Inter 7:8592–8600
36. Nan F, Xu Y, Xu BS, Gao F, Wu YX, Tang XH (2014) Effect of natural attapulgite powders as lubrication additive on the friction and wear performance of a steel tribo-pair. Appl Surf Sci 307:86–91
37. Tannous J, Dassenoy F, Lahouij I, Le Mogne T, Vacher B, Bruhács A, Tremel W (2011) Understanding the tribochemical mechanisms of IF-MoS$_2$ nanoparticles under boundary lubrication. Tribol Lett 41:55–64
38. Koroteev VO, Bulusheva LG, Okotrub AV, Yudanov NF, Vyalikh DV (2011) Formation of MoS$_2$ nanoparticles on the surface of reduced graphite oxide. Phys Status Solidi B 248:2740–2743
39. Li XH, Cao Z, Zhang ZJ, Dang HX (2006) Surface-modification in situ of nano-SiO$_2$ and its structure and tribological properties. Appl Surf Sci 252:7856–7861
40. Cizaire L, Vacher B, Le Mogne T, Martin JM, Rapoport L, Margolin A, Tenne R (2002) Mechanisms of ultra-low friction by hollow inorganic fullerene-like MoS$_2$ nanoparticles. Surf Coat Tech 160:282–287

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41.  Koshy CP, Rajendrakumar PK, Thottackkad MV (2015) Evaluation of the tribological and thermo-physical properties of coconut oil added with $MoS_2$ nanoparticles at elevated temperatures. Wear 330–331:288–308

42.  Han XH, Wang AJ, Wang XS, Li X, Wang Y, Hu YK (2013) Catalytic performance of P-modified $MoO_3/SiO_2$ in oxidative desulfurization by cumene hydroperoxide. Catal Commun 42:6–9

43.  Sia SY, Sarhan AAD (2014) Morphology investigation of worn bearing surfaces using $SiO_2$ nanolubrication system. Int J Adv Manuf Technol 70:1063–1071