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Article

Investigation on the Lubrication Performance of γ -Al₂O₃/ZnO Hybrid Nanofluids for Titanium Alloy

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Abstract Titanium alloys are difficult to machine and have poor tribological properties. This paper investigates the lubricating performance of γ -Al₂O₃/ZnO hybrid nanofluids for Ti-6Al-4V. The pure and hybrid nanofluid are compared, and the effects of γ -Al₂O₃/ZnO ratios are studied. The results show that γ -Al₂O₃/ZnO hybrid nanofluids outperform pure nanofluid in terms of lower friction coefficients and better surface quality. Moreover, the hybrid nanofluid with a mass ratio of Al₂O₃ to ZnO of 2:1 demonstrates the best lubrication performance with a reduced friction coefficient of up to 22.1% compared to the base solution, resulting in improved surface quality. Al₂O₃ nanoparticles can adhere to the surface of ZnO nanoparticles and work as a coating, which further enhances the lubrication performance of the water-based nanofluid.

Keywords: hybrid nanofluids; titanium alloy; lubricating properties; synergistic lubrication

1. Introduction

Titanium alloys are difficult to machine due to low thermal conductivity, low elastic modulus, and high chemical reactivity. High cutting temperatures and severe built-up edge formation during machining lead to excessive tool wear and poor surface quality [1][2]. Traditional lubricants like mineral oil, vegetable oil, and grease fail to lubricate titanium alloys well [3-5]. Effective water-based lubricants for titanium alloys have been investigated by researchers [6-8]. Castor oil sulfated sodium salt (CSS) solution can decrease friction coefficient and adhesive wear and can be used as a good water-based lubricant base stock for the design of cutting fluid for titanium alloys [9]. However, the lubricating and cooling performance of CSS solution for titanium alloys still needs to be improved for efficient application.

Nanoparticles are added to lubricants to create nanofluids that possess excellent frictional properties and high load-carrying capacity. These nanofluids are then sprayed onto the workpiece surface in the form of high-speed droplets. Rahmati et al. [10] investigated the effects of MoS₂ nanoparticles suspended in nano lubricants on machined surface morphology. Experimental results showed that MoS₂ nanoparticles with a concentration of 0.5 wt% led to superior machined surface quality compared to pure oil or other nanoparticle concentrations. Marcon et al. [11] reported that the addition of graphite nanoplatelets significantly reduced cutting forces and friction in the micro-milling of hardened steel. As to the mechanism, nanoparticles have a small size but a large surface area, and their thermal conductivity is much higher than that of millimeter- or micrometer-sized particles of the same volume. Nanoparticles alter the structure of the liquid, turning it into a liquid-solid two-phase suspension, which affects the energy transfer process within the nanofluid [12][13]. The randomly dispersed nanoparticles in the liquid promote micro-disturbances within the fluid, thereby enhancing the rate of energy transfer between the nanoparticles and the base liquid [14]. Due to the increased thermal conductivity and other mechanisms such as the thermal Brownian motion of nanoparticles, the use of nanofluids can significantly improve heat transfer performance [15].

Hybrid nanofluids, which are mixtures of two or more types of nanoparticles, exhibit better lubrication and heat transfer properties than single nanoparticle additives due to the variations in molecular structures and shapes. Zhang ^{[16][17]} prepared a hybrid nanofluid consisting of MoS₂ with good lubrication effect and CNTs with high heat conductivity coefficient. The effects of grinding force, coefficient of friction, and workpiece surface quality for Ni-based alloy grinding were investigated and MoS₂/CNT hybrid nanofluids achieved better lubrication than single nanoparticle. Kalita ^[18] found that MoS₂/Al₂O₃ hybrid nanofluid provided better lubrication than dry grinding and soybean oil pouring lubrication methods. Setti ^[19] investigated the surface grinding process of Ti-6Al-4V under different Al₂O₃/CuO hybrid nanofluid lubrication and found that the tangential force and grinding temperature were reduced with the hybrid nanofluid.

In summary, while scholars have achieved significant progress in the field of hybrid nanofluid lubrication, there are limited researches on the application of hybrid nanofluids in lubricating titanium alloys and on the micro-scale mechanisms of hybrid nanofluids. Therefore, the objective of this study is to evaluate the effectiveness of hybrid nanofluid lubrication for Ti-6Al-4V and to analyze the lubrication mechanisms of hybrid water-based nanofluids.

Using Al₂O₃ as a lubricant additive in nanofluid can enhance the lubrication performance, effectively reducing the friction coefficient and wear rate. Eltaggaz^[20] investigated the impact of Al₂O₃ nanofluid on tool wear, surface finish, and power consumption in the Ti-6Al-4V machining process. The results showed that nanofluid minimal lubrication had a positive effect on tool life and surface finish quality. However, existing research on the lubrication performance of hybrid nanofluids containing Al₂O₃ mostly focuses on more common α -Al₂O₃, rarely discussed γ -Al₂O₃ for its lower thermal conductivity. Studies have shown that ZnO exhibits good thermal conductivity in aqueous solution ^[21]. Furthermore, the hybrid nanofluid is expected to yield better thermal conductivity compared to individual nanofluids due to synergistic effect ^[22]. So a hybrid nanofluid is prepared in this paper by mixing γ -Al₂O₃ and ZnO at certain mass fractions which combines the advantages of both and results in superior lubrication performance. The lubricating properties of γ -Al₂O₃/ZnO hybrid nanofluids for Ti-6Al-4V with CSS aqueous solution as the base stock are investigated to improve the working performance of cutting fluid.

2. Experiment

2.1. Experimental setup and materials

Friction tests were conducted using a ball-on-disc device (CFT-I, Licp, Lanzhou, China) under various lubrication conditions. The schematic diagram of the testing instrument is shown in Figure 1. The lower specimen was a Ti-6Al-4V disc with a hardness of HRC 35. Table 1 displays the chemical composition of Ti-6Al-4V titanium alloy, while Table 2 shows its mechanical parameters. Before the friction tests, all samples underwent automated polishing and grinding to achieve a surface roughness (Sa) of less than 40nm.

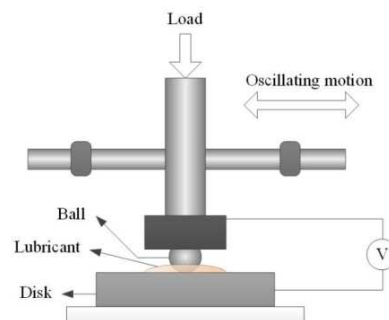


Figure 1. The schematic diagram of CFT-I tribo-tester.

Table 1. Chemical compositions of Ti-6Al-4V titanium alloy (wt.%).

N	C	H	Fe	O	Al	V	Ti
0.05	0.08	0.015	0.4	0.2	5.5-6.75	3.5-4.5	Remaining

Table 2. The mechanical parameters of Ti-6Al-4V titanium alloy.

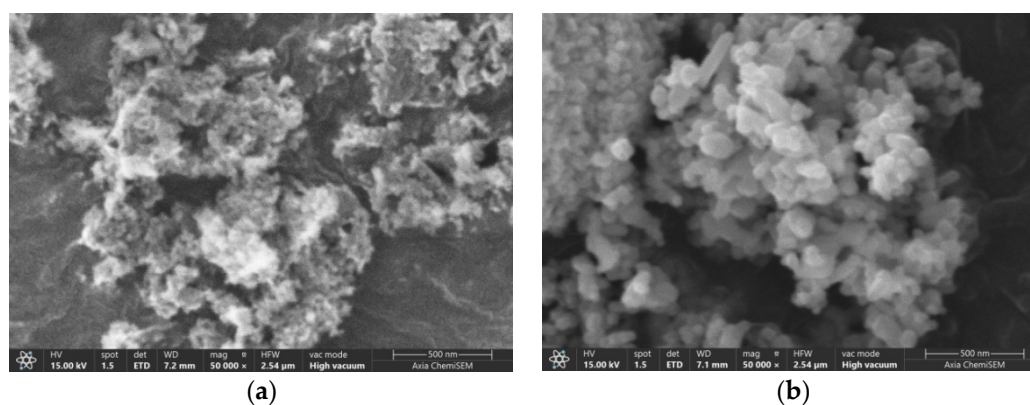
Tensile strength (MPa)	Yield Strength (MPa)	Hardness (VHN)	Young's Modulus (GPa at 20 °C)	Poisson's Ratio
1230	1060	315	113.8	0.34

Cemented carbide is the optimal tool material for machining Ti-6Al-4V titanium alloy. The upper specimen is a YG8 (WC-Co) ball with a hardness of 89HRA, a diameter of 10mm, and a surface roughness (Sa) of 25nm. Each specimen was cleaned with acetone and ethanol, followed by ultrasonic cleaning with deionized water for 10 minutes. The upper ball reciprocated on the stationary disc with a 5mm amplitude and a 5Hz frequency for 10 minutes. Before the reciprocating motion, 0.2 ml lubricant was dropped onto the disc surface. A normal load of 50N was applied, and the maximum Hertz contact pressure was 7.5 GPa.

After the frictional test, the worn specimens were cleaned with acetone for 30 minutes and then dried. The wear volume was calculated by LEXTTMOLS5100 laser scanning confocal microscope (Olympus, Tokyo, Japan). The microtopography of the worn surface was observed using a scanning electron microscope (FEI, Hillsboro, OR, USA) with Energy-dispersive X-ray spectroscopy (EDS).

2.1. Nanofluids preparation

Aluminum oxide (Al₂O₃) and zinc oxide (ZnO) are selected as nano-additives for lubricating titanium alloys, with Castor oil sulfated sodium salt (CSS) as the base stock. Table 3 presents the relevant parameters of the nanoparticles, and their microscopic morphology is depicted in Figure 2. It is worth noting that although crystal structures of both nanoparticles are cubic, γ -Al₂O₃ has a unique porous structure and therefore has a high specific surface area. The nanofluids were prepared by a two-step method to disperse the nanoparticles in a water-based lubricant. The components used were γ -Al₂O₃ and ZnO nanoparticles and a 10% CSS aqueous solution. The mixture was ultrasonically vibrated for 20 minutes. The mass fraction of Al₂O₃ and ZnO nanoparticles was given by mix(x:y), and seven groups of hybrid nanofluids were prepared to investigate the effects of the nanoparticles' content ratio, including pure Al₂O₃, pure ZnO, mix(1:1), mix(1:2), mix(2:1), mix(1:4), and mix(4:1).

**Figure 2.** The microtopography of the two kinds of nanoparticles (a) Al₂O₃ (b) ZnO.**Table 3.** Properties of the nanoparticles as provided by the manufacturer.

Property	γ -Al ₂ O ₃	ZnO
Purity	99%	99%

Average particle size (nm)	20	50
Specific surface area (m ² /g)	120	21.5
Crystal structure	cubic	cubic
Thermal conductivity (W/mK)	25	47

3. Results and Discussion

3.1. Lubricating Properties of the nanofluids

The friction coefficient curves for seven groups of nanofluids and one control group (CSS aqueous solution without nanoparticles) were presented in Figure 3. During the running-in period, the friction coefficients of pure Al₂O₃ and ZnO nanofluids increased to 0.38 and then decreased with fluctuations due to factors such as lubricating film rupture. Then the friction coefficients reached a steady state after about six minutes. The COF of pure Al₂O₃ nanofluid in the steady state was about 0.162, while it was 0.168 for pure ZnO nanofluid. When the mass fraction ratio of Al₂O₃ to ZnO nanoparticles was 4:1, the friction coefficient rapidly increased to 0.38 during the running-in period and then gradually decreased. For the other hybrid nanofluids, the maximum friction coefficient during the running-in period decreased to below 0.35.

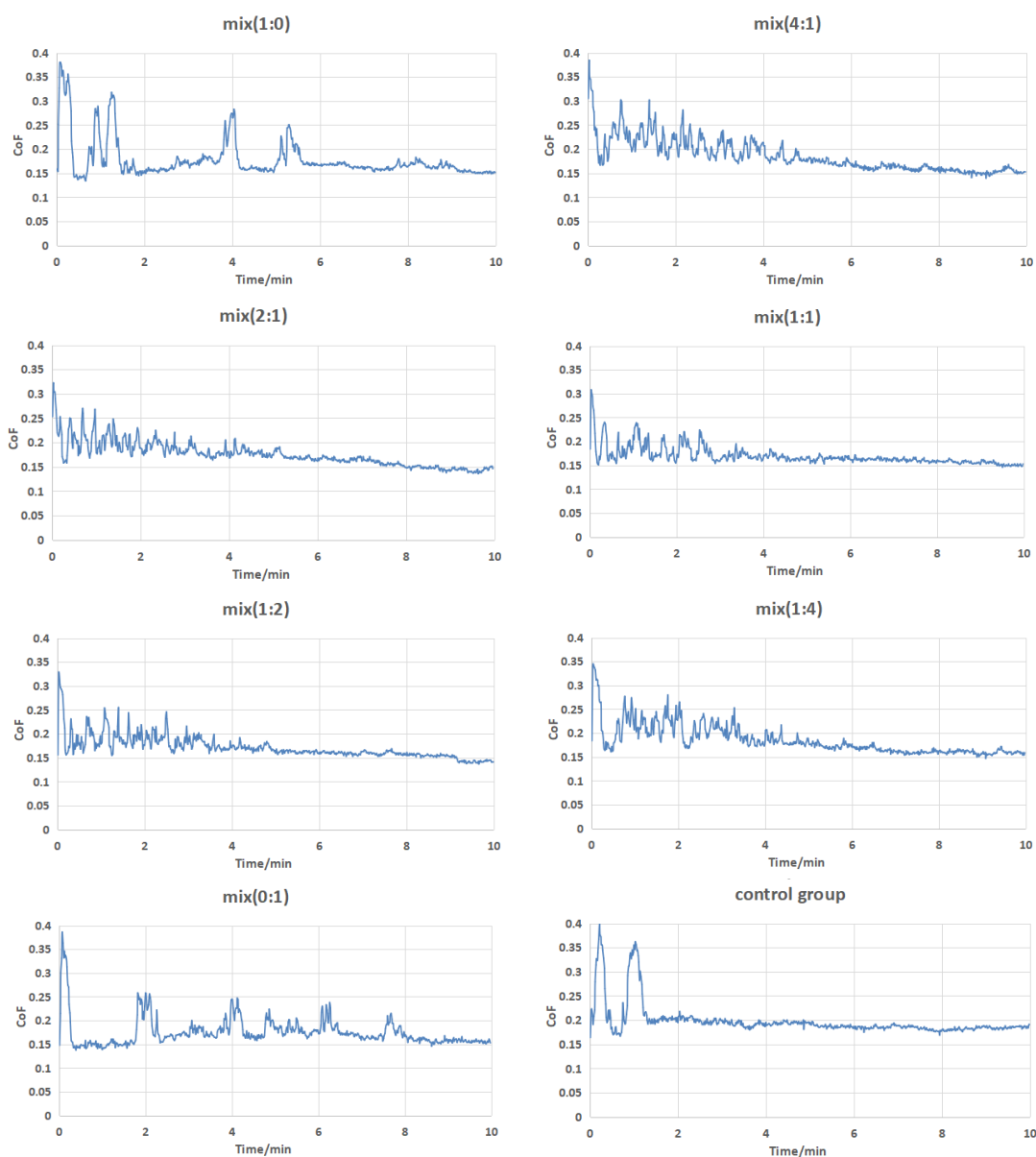
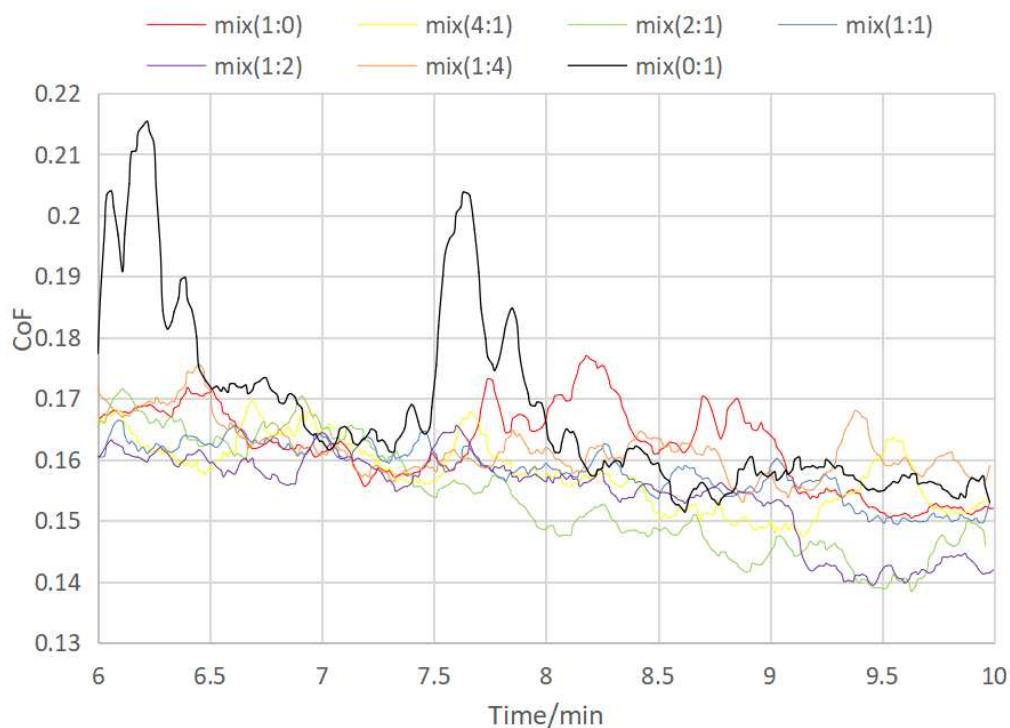


Figure 3. Friction coefficient of seven nanofluids and control group.

After six minutes, the stabilized COF values were compared and listed in Figure 4. The average friction coefficients at different stages for various nanofluids were shown in Table 4. All the nanofluids significantly reduced the friction coefficients at different stages for titanium alloy. Combining Figure 4 with Table 4, it can be concluded that mix(1:2) and mix(2:1) showed the most significant friction reduction for water-based nanofluid lubrication of titanium alloy. The lowest average friction coefficient was 0.144, which was 22.1% lower than the CSS solution without nanoparticles. The friction coefficients of mix(4:1), mix(1:1), and mix(1:4) solutions were similar to pure Al_2O_3 nanofluid, but all lower than pure ZnO nanofluid. Compared with the control group, their friction coefficients could be reduced by up to 16.7%, 17.3%, and 14.1% respectively.

**Figure 4.** Comparison diagram of friction coefficients in steady state.**Table 4.** Average friction coefficient of seven nanofluids.

Time/min	pure Al_2O_3	mix(4:1)	mix(2:1)	mix(1:1)	mix(1:2)	mix(1:4)	pure ZnO	control group
1	0.228	0.237	0.215	0.192	0.201	0.226	0.186	0.247
2	0.192	0.222	0.202	0.186	0.196	0.220	0.167	0.227
3	0.162	0.209	0.191	0.182	0.188	0.208	0.180	0.200
4	0.189	0.202	0.180	0.169	0.178	0.194	0.180	0.192
5	0.171	0.184	0.179	0.167	0.171	0.184	0.189	0.194
6	0.189	0.174	0.171	0.164	0.163	0.174	0.179	0.187
7	0.166	0.164	0.166	0.163	0.160	0.167	0.183	0.185
8	0.162	0.161	0.158	0.161	0.160	0.160	0.174	0.183
9	0.168	0.154	0.148	0.157	0.155	0.160	0.159	0.182
10	0.154	0.154	0.144	0.153	0.144	0.159	0.157	0.185
Average	0.178	0.186	0.175	0.169	0.172	0.185	0.175	0.198
Average in steady	0.162	0.158	0.154	0.158	0.155	0.162	0.168	0.184

The worn surface of the titanium discs after frictional tests was observed and the wear volume was shown in Figure 5. It can be seen that several hybrid nanofluids increased the wear volume to some extent compared to pure Al_2O_3 nanofluid. What's more, the increase in wear volume was positively correlated with the content of ZnO nanoparticles. Compared to pure ZnO nanoparticles, mix(4:1), mix(2:1), mix(1:1), and mix(1:2) showed a reduction in wear volume by 23.5%, 18.9%, 21.2%, and 9.6% respectively. It is worth noting that the wear volume of mix(1:4) even exceeded that of pure ZnO nanofluid, which is related to the interaction between nanoparticles.

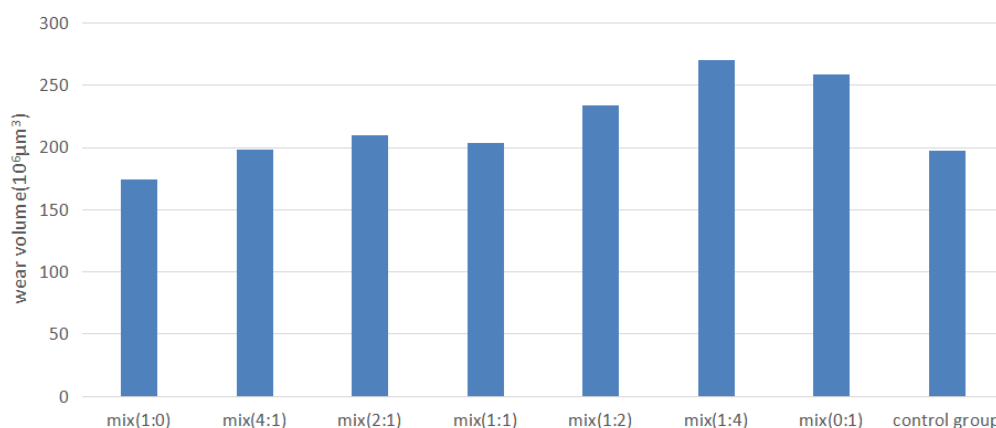


Figure 5. Wear volume of the discs lubricated by different lubricants.

On one hand, pure Al_2O_3 nanofluid exhibits better lubricating effect than pure ZnO nanofluid due to its different physical properties and shape characteristics. On the other hand, the friction coefficients of $\gamma\text{-Al}_2\text{O}_3/\text{ZnO}$ hybrid nanofluids are generally lower than those of pure nanofluids, indicating that the hybrid nanofluids have better anti-friction effects compared to pure nanofluids under the experimental conditions. This indicates that different nanoparticles interact with each other to improve the lubrication performance of hybrid nanofluids. Furthermore, the lubrication performance of different hybrid nanofluids varies. Mix(1:2) and mix(2:1) demonstrate the lowest friction coefficients. Based on the comparison among multiple hybrid nanofluids, the wear rate of hybrid nanofluids is higher than that of pure Al_2O_3 nanofluid, and the ZnO content affects the wear rate of hybrid nanofluids' lubrication. Generally, a higher ZnO content leads to poorer lubrication performance of the hybrid nanofluids. These findings indicate that the synergistic effect of hybrid nanoparticles can be influenced by adjusting the proportion of the hybrid nanofluids.

3.2. Microtopography of the worn titanium discs under various lubrication condition

The microtopography of the worn titanium discs were observed using scanning electron microscopy (SEM). The SEM images under different lubricant conditions are shown in Figure 6. As seen from Figure 6(a)(b)(c), among the various nanofluids, the surfaces of three nanofluids, mix(4:1), mix(2:1), and pure Al_2O_3 , exhibit the smoothest surfaces without obvious grooves and scratches. The worn surface under mix(2:1) hybrid nanofluid lubrication shows particularly excellent surface quality, which is likely attributed to the synergistic effect of Al_2O_3 and ZnO nanoparticles. As shown in Figure 6(e), the worn surface of the mix(1:2) lubrication is relatively smooth, but there are some plow furrows on the surface, indicating that the lubricating effect under the separate action of ZnO is relatively poor compared to the other hybrid nanofluids. As shown in Figure 6(d)(f), obvious scratches and plow furrows can be observed on the worn surfaces under the mix(1:1) and mix(1:4) lubrication, and their surface quality is even worse compared to mix(1:2) and pure ZnO nanofluid. The possible reason is that the excess ZnO nanoparticles plowed the surface of the titanium alloy workpiece. However, due to their limited cutting ability, they cannot cut undivided chips from the disk completely and effectively, resulting in significant agglomeration on the friction surface. Compared to the surface under the lubrication of pure ZnO nanofluid, the relatively smooth worn

surface is not conducive to the high storage capacity of the nanofluid, thus unable to form a friction film and instead reducing the surface quality of the titanium alloy.

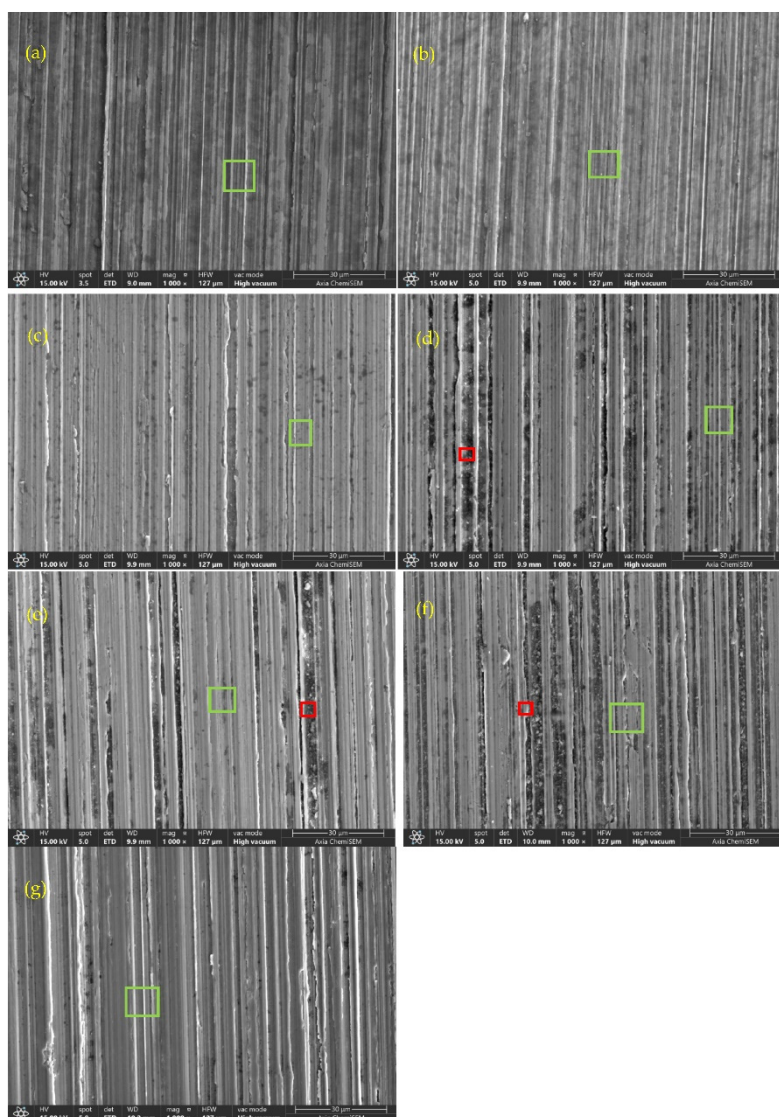
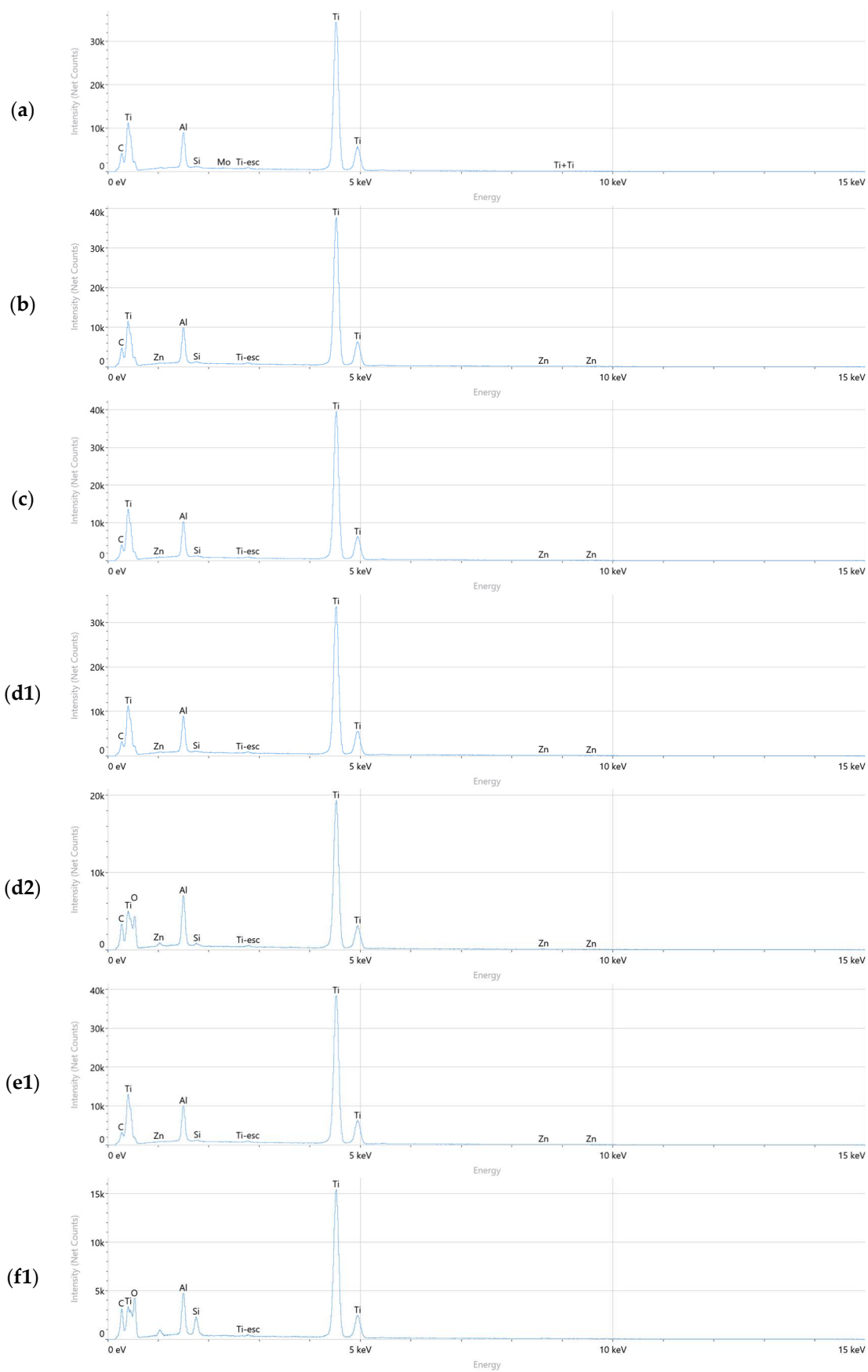


Figure 6. SEM morphology of the tracks on Ti-6Al-4V lubricated by (a) pure Al_2O_3 (b) mix(4:1) (c) mix(2:1) (d) mix(1:1) (e) mix(1:2) (f) mix(1:4) (g) pure ZnO.

EDS analysis was conducted on the worn surface to determine the elemental distribution of the product. The data in Figure 7 and Table 5 show that the elemental content does not significantly change with the proportion of hybrid nanofluids, suggesting that the synergistic effect between Al_2O_3 and ZnO nanoparticles is predominantly physical while the frictional chemical reactions are not dominant. The physical synergistic effect of Al_2O_3 and ZnO nanoparticles provides better lubrication than the individual nanoparticles. In addition, there are dark accumulations on the friction surfaces of mix(1:1), mix(1:2), and mix(1:4). The accumulated portion on the friction surface contains some Zn and O elements that were not previously detected seen from Figure 7(d2)(f2)(g2). This suggests the presence of residual nanoparticles and intermediate products from the cutting process. The content of Ti and Al elements significantly decreased compared to previous results, indicating the presence of many other impurities besides undivided chips. Additionally, there is a significant fluctuation in the O element content among the three EDS analyses. ZnO and Al_2O_3 nanoparticles aggregate in some form, and when the content of ZnO is high, it has a certain impact on its machined surface and remains on the machined surface. The aggregation effect of nanoparticles may have contributed to this result, considering the relatively small sampling range.



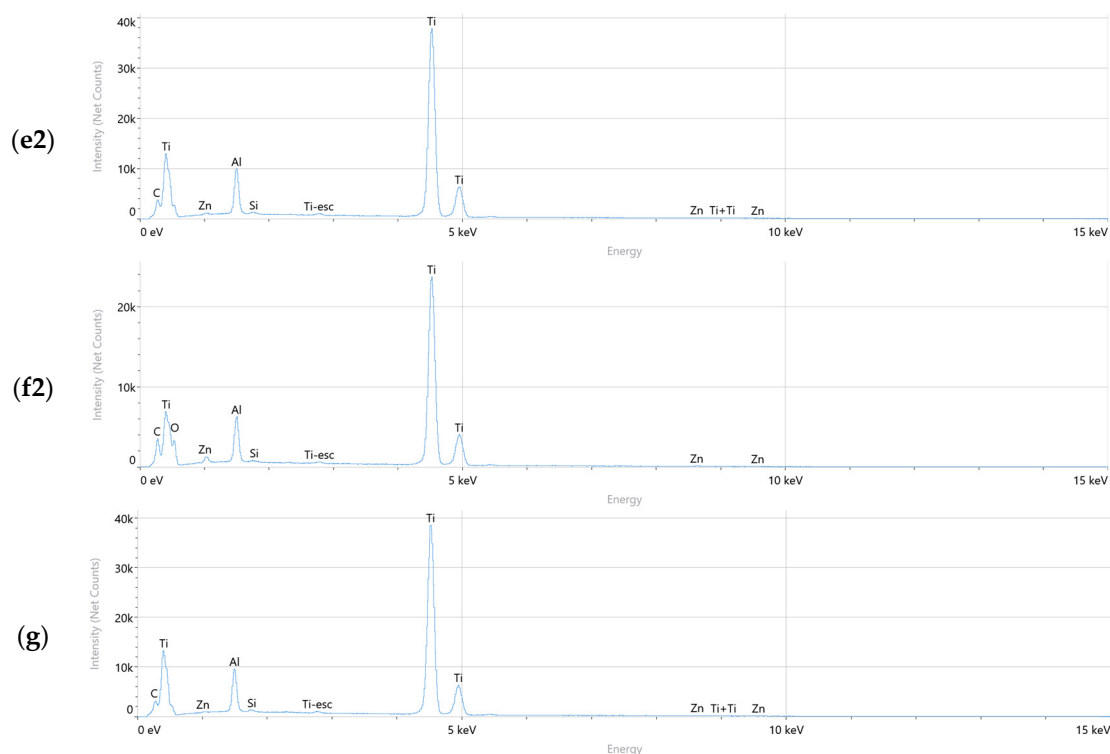


Figure 7. EDS spectrum of the green box in (a)pure Al_2O_3 (b)mix(4:1) (c)mix(2:1) (d1)mix(1:1) (e1)mix(1:2) (f1)mix(1:4) and (g)pure ZnO and the red box in (d2)mix(1:1) (e2)mix(1:2) (f2)mix(1:4).

Table 5. The elements content of the above EDS analysis.

Element(wt.%)	C	Al	Si	Ti	O	Zn
mix(1: 0)	3.5	6.0	0.3	89.9	0	0
mix(4: 1)	4.0	6.0	0.2	89.4	0	0
mix(2: 1)	3.0	6.2	0.2	90.6	0	0
mix(1: 1)(e1)	2.9	6.2	0.2	90.3	0	0
mix(1: 1)(e2)	5.7	7.0	0.4	74.4	11.8	0.8
mix(1: 2)(f1)	2.2	6.2	0.2	91.0	0	0
mix(1: 2)(f2)	6.1	5.4	0.5	69.3	16.9	1.1
mix(1: 4)(g1)	3.1	6.2	0.3	90.1	0	0
mix(1: 4)(g2)	5.4	5.9	0.2	85.9	1.4	1.2
mix(0: 1)	2.3	5.9	0.3	91.1	0	0

3.3. Lubrication mechanisms of $\text{Al}_2\text{O}_3/\text{ZnO}$ hybrid nanofluids

Nanoparticles can fill in micro-pits and damages on the surface, thus playing a repairing role. Some particles embed into the machined surface after collision. Their shape is changed due to shear compression and the fragments continue to assist in cutting [24]. Part of the embedded particles is ploughed off by new nanoparticles, and some continue to polish the surface. The rolling nanoparticles generate a lubricating film that is easy to shear. As a result, the surface is polished leading to better surface quality [24-26].

Figure 2(a) depicts the shape and structure of Al_2O_3 nanoparticles. In our experiment, $\gamma\text{-Al}_2\text{O}_3$ is utilized, which has uniform particle size, high purity, excellent dispersion, high specific surface area, inertness to high temperatures, high reactivity. $\gamma\text{-Al}_2\text{O}_3$ is classified as active alumina with porosity, high hardness, and good dimensional stability. The oxygen anions in $\gamma\text{-Al}_2\text{O}_3$ have the same cubic-close-packing arrangement as in spinel (e.g. MgAl_2O_4), and the cations have an averaged cubic spinel structure [27]. The spinel structure can be described as a sequence of layers of close-packed oxygen anions stacked in ABC order (cubic close packing, ccp) [28]. Therefore, porous Al_2O_3 nanoparticles

expand the coverage area of the lubricating oil film formed between the interfaces of nanofluids. Al_2O_3 nanoparticles can repair the oil film through their porous structure [29]. This leads to a more stable development of the lubricating oil film and consequently produces a good lubrication effect. Due to the different shape characteristics of Al_2O_3 and ZnO nanoparticles, hybrid nanofluids with corresponding mass fraction ratios have different lubrication effects. ZnO nanoparticles act as rolling balls at the workpiece interface. When there are relatively few ZnO nanoparticles, it provides only limited ZnO nanoparticles to take effect, while the lubricant with a high ZnO concentration indicates the ZnO nanoparticles to agglomerate. On the other hand, the agglomeration of ZnO nanoparticles operates as a barrier to hinder the perpetual supplies of fine nanoparticles to the contact zone for lubrication [30].

According to the experimental results above, the friction coefficients of the five hybrid nanofluids are generally lower than those of pure Al_2O_3 and pure ZnO nanofluids. Due to the synergistic effect between nanoparticles, hybrid nanofluids exhibit better lubrication performance. Figure 8 shows the lubrication mechanism diagram of $\text{Al}_2\text{O}_3/\text{ZnO}$ hybrid nanofluids. Al_2O_3 nanoparticles have a porous structure and excellent adsorption ability. As a result, they can adhere to the surface of ZnO nanoparticles, creating a "physical coating". The adhesion of Al_2O_3 nanoparticles to the surface of ZnO nanoparticles improves the dispersion stability of nanoparticles in water-based lubricants. When the proportion of ZnO in the hybrid nanofluids is very small, local agglomeration occurs and Al_2O_3 nanoparticles coat the surface of ZnO nanoparticles. Each group of such locally agglomerated nanoparticles forms a "big bearing" which further enhances the lubrication performance of the water-based nanofluid. Compared with pure Al_2O_3 nanofluids, as the content of ZnO in the hybrid nanofluids increases, the number of adhered local agglomerations also increases. This is reflected in the experimental results that mix(2:1) achieves an excellent friction coefficient. However, as the content of ZnO in the hybrid nanofluids increases, Al_2O_3 nanoparticles are unable to cover all ZnO nanoparticles. The unadhered ZnO nanoparticles act independently and improve the lubricating effect of the water-based lubricants to some extent, achieving an excellent friction coefficient for mix(1:2) nanofluid. Nevertheless, ZnO nanoparticles act as independent bearings for lubrication, which may cause some degree of plowing on the surface of the titanium alloy workpiece. Therefore, as the mass fraction of ZnO nanoparticles increases, the wear of the titanium alloy under water-based hybrid nanofluids shows an upward trend. In summary, a certain amount of local agglomeration or a certain number of ZnO nanoparticles can significantly reduce the friction coefficient. The lower content of ZnO and higher content of Al_2O_3 improve the lubrication of water-based hybrid nanofluids.

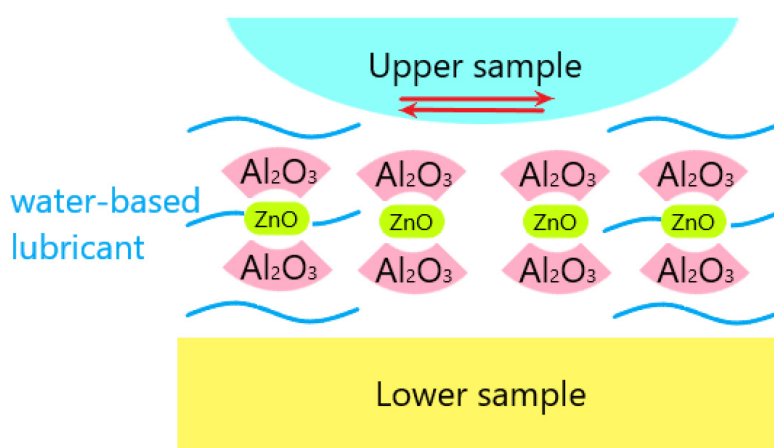


Figure 8. The schematic diagram of physical synergy of Al_2O_3 and ZnO.

Conclusion

The lubrication performance of Al₂O₃/ZnO hybrid nanofluid in water-based lubrication for titanium alloys is investigated and several conclusions are drawn as follows:

Pure Al₂O₃ nanoparticle nanofluid exhibits lower friction coefficient and wear volume indicating better lubrication performance than pure ZnO. It can be attributed to the unique lamellar structure and high porosity of Al₂O₃ nanoparticles.

The Al₂O₃/ZnO hybrid nanofluid outperforms pure nanofluid lubrication for titanium alloy. Hybrid nanofluids with different ratios consistently achieve lower friction coefficients and better surface quality. The coating of Al₂O₃ nanoparticles on the surface of ZnO nanoparticles improve the dispersion stability of ZnO nanoparticles, thus enhancing the lubrication performance.

The five hybrid nanofluids with different Al₂O₃/ZnO ratio exhibit different lubrication effects. The hybrid nanofluid with a mass ratio of Al₂O₃ to ZnO of 2:1 demonstrates the best lubrication performance with a reduced friction coefficient of up to 22.1% compared to the base solution, resulting in improved surface quality. The mix(2:1) hybrid nanofluid can be used for the cutting fluid of titanium alloys.

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