Study on the enhanced tribological performance for titanium alloys by PEG oil/Zn-nanoparticles

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Abstract
This work developed an effective and promising lubricant containing Zn nanoparticles and polyethylene glycol (PEG) base oil to deal with the tough problem of boundary lubrication for steel/Ti6Al4V. Tribological tests were carried out to investigate the boundary lubrication performances and the generation process of boundary protective films on the worn surface. Results showed that low and steady friction coefficient curves could be obtained by PEG suspensions with Zn nanoparticles after a very short ‘run-in’ period, and the main wear volume of Ti6Al4V was occurred during the ‘run-in’ period. The ZnO boundary protective films generated on the counterfaces of the Ti6Al4V disk and steel ball during the friction process. This strong and steady oxide films could effectively prevent direct contact between frictional pairs and provide excellent boundary lubrication performances. This work will contribute to the development of high performance lubricants for titanium alloys

1. Introduction

Titanium alloys are considered as outstanding structural materials in modern industry, primarily due to their high specific strength and excellent corrosion resistance [1, 2]. Besides the success in the aerospace and the chemical industries, other areas such as vehicle engineering, biomedical engineering, and transportation are meeting increased application of titanium alloys. Of these, the classical alloy Ti6Al4V covers more than 50% of the commercial market. For example, the titanium alloy connecting rods have been repeatedly used in the market of super sports cars, such as Porsche or Ferrari [3]. However, because of their low elastic modulus and low wear resistance, more applications of titanium alloys such as crankshaft, piston pin, camshaft field, can rarely get prior consideration [4, 5]. The main reason is that the strong disorder in their lattice structure of titanium alloys causes the rapid growth of titanium oxides, but no protective oxide scales could form on the surfaces. As a result, the lack of wear resistance and susceptibility to galling may greatly limit the use of titanium alloys as moving parts.

Up to now, the main way to enhance the wear resistance of titanium alloys is surface treatment, including laser remelting [6, 7], deposition coating [8], thermal diffusion [9], electroplating [10, 11], ion implantation [12, 13] and nanostructured surface [14]. The major deficiency of surface treatment is that the functional layer will always confront material consumption during the whole moving process, leading to that the layer could not ensure the long-term lubrication of titanium alloys. Another effective way to obviate the wear and galling is liquid lubrication. Unfortunately, there are few universal lubricants for the lubrication of titanium alloys, because most commercial lubricants and additives show poor compatibility with titanium alloys. Halogenated hydrocarbons were once considered as the promised lubricants as a sliding titanium interface, because the formation of charge transfer complexes of halogen and aromatic compounds on the surface of titanium alloy [15, 16]. Wu et al [17] investigated the aqueous lubrication performances of dodecyl polyethyleneoxy phosphate with various metallic cations for titanium alloy and found that Ba-containing alkoxyl phosphate gave best
lubrication performance, which could be ascribed to the tribological synergism between Ba$^{2+}$ and Ti$^{4+}$. Bi et al. [18] found that containing O, O-$\text{di-n-octyldithiophosphate}$ (Cu-DTP) could increase the lubricating properties of rapeseed oil for steel/Ti6Al4V, which is mainly ascribed to the tribochemical reaction film containing S and P at low applied load and a conjunct effect of the compact Cu nanoparticle deposited film and tribochemical reaction film containing S and P at high-applied load. Bermúdez’s group [19–21] have investigated ionic liquids (ILs) as lubricants of titanium/steel and found that the best lubrication performance for ILs at 100 °C is due to adsorption and surface interactions, with formation of fluorine and phosphorus-containing wear debris, but without decomposition or tribocorrosion. Deionized water, physiological saline and bovine serum as lubricants of Ti6Al4V have been studied by Luo et al. [22] They found three lubricants had the effect on reducing the friction and wear of titanium alloy, and bovine serum was relatively obvious. Sehgal’s group [23] studied the sliding friction and wear behavior of steel/Ti6Al4V lubricated by Hydrol-68 oil and found that considerable reduction in friction and wear was achieved. Zhang et al. [24] introduced two phosphate esters into water-based lubricating additives for titanium alloy machining and found that phosphate ester solutions showed better lubricating effect than commercial cutting fluid of titanium alloy.

Until now, few effective additives were raised for the boundary lubrication of Ti6Al4V against steel. The aim of this paper was to supply a nanoparticle additive with good dispersion in base oil and excellent antiwear and friction-reduction properties for Ti6Al4V and also to investigate the acting mechanism of selected additives.

### 2. Materials and methods

#### 2.1. Materials and reagents

Two kinds of alloy materials, Ti6Al4V and AISI 52100 steel, were tested in this work. The compositions and characteristics of these alloys were listed in table 1. The anti-wear and friction-reduction properties of base oil with and without additives for steel/Ti6Al4V were tested.

Zn nanoparticles, as lubricating additives, were supplied by Shanghai ChaoWei Nanotechnology Co., Ltd. The JEM-1200EX/S Transmission Electron Microscopy (TEM) was used to examine the morphology of Zn nanoparticles and the results were presented in figure 1. It can be found that Zn nanoparticles were near spherical and the particle sizes distributed at 20–100 nm. Polyethylene glycol 200 (PEG), as base oil, was
purchased from Guangdong Xilong Chemical Regent Company. PEG suspensions with different concentrations of Zn nanoparticles: 0.5, 1.0, 1.5, 2.0 wt% were prepared for the following tribological experiments.

The dynamic viscosity of the nanofluid has been measured at temperatures between 20 °C to 60 °C. The results were listed in table 2.

2.2. Dispersion stability of Zn nanofluid

Figure 2 as below presents the digital images of ultrasonically-dispersed 1.0% Zn nanoparticle dispersion in 24 h. Without dispersant, most of the Zn nanoparticle could remain dispersed stably in the PEG for 6 h, suggesting that the suspension was stable during the tribo-test (60 min). In addition, the oscillation of the upper ball may prevent the sedimentation of Zn nanoparticle.

The surface modifiers and dispersers of nanoparticles are beneficial in preserving lubricating properties for long term use. However, the lubricating mechanisms of modifiers and dispersers in the boundary lubrication of Ti6Al4V are still unclear. Thus, the dispersant for Zn nanoparticle are not considered in current study.

2.3. Tribological properties test and calculation

The friction and wear tests were carried out on a ball-on-disk tester, SRV-IV, under 100 N with amplitude of 1 mm and frequency of 25 Hz for the duration of 60 min. All the friction tests were conducted in ambient air at 25 °C and the relative humidity of 20%–30%. The Ti6Al4V disk was mechanically abraded and polished to gain a smooth surface. Both the Ti6Al4V disk and steel ball were ultrasonically cleaned in acetone and petroleum ether, respectively, and then dried in nitrogen flow at room temperature. The minimum oil film thickness \( h_{\text{min}} \) were calculated based on the initial contact conditions through the Грубин formula:

\[
\frac{h_{\text{b}}}{R} = 1.73 \left( \frac{U h_0 \alpha}{R} \right)^{5/7} \left( \frac{E R^2}{W} \right)^{1/21}
\]

\[
h_{\text{min}} = 0.75 h_{\text{b}}
\]

In these formulae, the dynamic viscosity of PEG oil \( \eta_0 = 24.6 \text{ mPa} \cdot \text{s} \), the pressure-viscosity coefficient of PEG oil \( \alpha = 2.2 \times 10^{-8} \text{ GPa}^{-1} \) [25]. Results show that \( h_{\text{min}} = 0.022 \mu\text{m} \) under the initial contact conditions. The lubrication regime can be judged by the parameter \( \lambda \) ratio (film thickness to composite surface roughness, \( h_{\text{min}} / R_0 \)). Based on the steel ball surface roughness of about 0.025 \( \mu\text{m} \) and the Ti6Al4V disk surface roughness of about 0.035 – 0.055 \( \mu\text{m} \), all the friction tests were conducted in the boundary lubrication regime (\( \lambda < 1 \)). The friction coefficient was monitored automatically by a load cell-based force measurement system. The wear volume of the Ti6Al4V disk was measured using a MicroXAM-3D noncontact surface mapping microscope profilometer. At least three replicates were carried out for each test condition to minimize data scattering.
2.4. Characterization
A JSM-5600LV scanning electron microscope (SEM) equipped with a Kevex energy-dispersive x-ray Spectrometer (EDS) attachment were used for the analyses of the morphologies and chemical composition of worn surfaces. The chemical states of typical elements on the worn surfaces of the lower disk and the upper ball were analyzed on a K-Alpha x-ray photoelectron spectrometer (XPS) using Al Kα radiation as the exciting source. The binding energy of the target elements was determined at a pass energy of contaminated carbon (C1s: 284.8 eV) as the reference.

3. Results and discussion
3.1. Effects of Zn Nanoparticle Additives
The friction-reduction and anti-wear effects of Zn nanoparticle additives in PEG oil were firstly investigated and results were shown in figure 3. It can be seen from figure 3(a) that PEG base oil exhibits a relatively high and fluctuant friction coefficient curve during the sliding process. After the addition of Zn nanoparticle additives, there is no obvious difference between two oil samples on their friction behaviors at the very beginning of the test. The friction coefficient curve began to decrease sharply at 60 s, and this decreasing process lasted for about 200 s. Finally, relatively low and steady friction coefficient curve was obtained by PEG suspension with Zn nanoparticles. In figure 3(b), Zn nanoparticle additives exhibited remarkable friction-reduction and anti-wear properties in PEG oil. Comparing with PEG base oil, when adding Zn nanoparticles, the steady friction coefficient and wear volume decreased by 55% and 92%, respectively. It can be concluded that PEG suspension with Zn nanoparticles provides effective lubrication for steel/Ti6Al4V contact.

Figure 4 shows the analyses of worn surfaces affected by the addition of Zn nanoparticle additives in PEG oil. Smooth worn surfaces of Ti6Al4V disks with slight scratches were obtained by both PEG base oil and PEG suspension with Zn nanoparticles in figures 4(a) and (b). It can be seen from figures 4(c) and (d) that both the worn surfaces of steel balls were smooth and regular in shape, but the area of wear scar lubricated by PEG base oil was much larger. In figures 4(c1), (d1) and (d2), it can be found that lots of Ti-containing materials were transferred from Ti6Al4V disk to the counterface of steel ball when lubricated by PEG base oil. However, after the addition of Zn nanoparticle additives in PEG oil, materials containing Zn element covered the wear scar surface of steel ball. It means that the transfer tendency of Ti-containing materials to the counterface of steel ball was obviously restrained. Furtherly, Zn elements could also be detected on the worn surface of Ti6Al4V disk lubricated by PEG suspension with Zn nanoparticles from figure 4(e). It can be concluded that a boundary protective film containing Zn elements generated on the both worn surfaces of Ti6Al4V disk and steel ball, which restrained the transfer tendency of Ti-containing materials from Ti6Al4V disk to the counterface of steel ball obviously and effectively avoided the direct contact between Ti6Al4V disk and steel ball.

3.2. Generation process of protective films
For a deeper understanding of the generation process for the boundary protective films containing Zn, the specific lubrication state of PEG suspension with Zn nanoparticles at different typical stages during the sliding process was investigated. Figure 5 shows the wear volumes of Ti6Al4V disk at different times corresponding to the friction coefficient curve. It can be seen that the high friction and severe wear behaviors occurred at the initial stage of 360 s, which then could be named as ‘run-in’ period. Comparing to the wear volume produced during
the entire 3600 s rubbing period, it produced 89% of the wear volume during this initial 360 s period. Correspondingly, very little material loss of Ti6Al4V disk occurred due to the low friction and low wear behaviors during this low friction period (360 s to 3600 s).

Figure 6 shows the analyses of worn surfaces at different typical stages. At 60 s, in the ‘run-in’ period, signs of adhesive wear and few Zn elements could be seen on the wear scar of Ti6Al4V disk in figure 6(a). It can be seen from figures 6(e) and (e1) that because of the low concentration of Zn nanoparticle additives in PEG oil, no materials containing Zn element but only Ti-containing materials were transferred to the counterface of steel ball. Figure 6(b) shows that at 360 s, the end of ‘run-in’ period, fewer signs of adhesive wear and a few Zn elements could be detected on the wear scar surface of Ti6Al4V disk. In figures 6(f) and (f1), a thin protective film containing Zn element generated on the worn surface of steel ball. At 1800 s and 3600 s, in the steady friction period, no sign of adhesive wear and more Zn elements could be found on the worn surfaces of Ti6Al4V disks in
As is shown in figures 6(g), (h), (g₁) and (h₁), on the worn surfaces of steel balls, the element content of Zn becomes higher than the beginning stages while that of Ti element becomes lower. It can be deduced that a continuous protective film containing Zn element generated, which replaced the Ti-containing materials on the worn surface of steel ball. Therefore, the boundary protective film containing Zn element generated gradually on the worn surface of Ti6Al4V disk and steel ball during the ‘run-in’ period. In the steady friction period, the boundary protective film became completer and more continuous. Furtherly, this protective film could effectively restrain the situation of material transfer from Ti6Al4V disk to the counterface of steel ball.

3.3. Effects of additive concentration

Figure 7 shows additive concentration effects of Zn nanoparticles in PEG oil for steel/Ti6Al4V contact. Figure 7(a) presents the evolution of friction coefficient as a function of time. It can be seen that at low concentration of Zn nanoparticles (0.5%), it experienced a long ‘run-in’ period for about 500 s, and the steady friction coefficient was low. At medium concentration (1.0%), it experienced a shorter ‘run-in’ period for about 360 s, and the friction coefficient was low and steady. At high concentrations (1.5% and 2.0%), both ‘run-in’ periods were extremely short for about 60 s, but they gave higher and relatively fluctuant friction coefficient during the steady friction stage. In figure 7(b), as the concentration of Zn nanoparticle increased from 0.5% to 2.0%, the value of steady friction coefficient showed minor increase while the wear volume remarkably decreased. It can be concluded that the increase of additive concentration in PEG oil could effectively shorten the ‘run-in’ period, slightly increase the friction coefficient, and improve the anti-wear property obviously.

Figure 8 shows surface analyses for lower disks and upper balls lubricated by oil samples with low and high concentrations of Zn nanoparticles. In figure 8(a), it can be seen that at low additive concentration, high content of Ti element and low Zn element were detected on the worn surface of steel ball. It indicated that the boundary protective films containing Zn element were thin and intermittent, which resulted in the material transfer from Ti6Al4V disk to the counterface of steel ball continuously. In figure 8(b), at high additive concentration, excessive materials containing Zn stacked around the wear scar of steel ball, and more Zn element and less Ti element were detected on the worn surface of steel ball. It indicated that the continuous boundary protective film containing Zn element could effectively restrain the transfer of Ti-containing materials. Figures 8(c) and (d) showed that at both low and high additive concentrations of Zn nanoparticles, smooth worn surfaces with slight scratches were obtained on Ti6Al4V disk. Additionally, more Zn elements were detected on the worn surfaces of Ti6Al4V disk at high additive concentration. As a conclusion of figures 7 and 6, at low additive concentration, thin and intermittent boundary protective films containing Zn element were generated on the counterfaces of Ti6Al4V disk and steel ball, which produced lower friction coefficient but caused the Ti6Al4V disk more material loss. At high additive concentration, excessive materials containing Zn element stacked in and around the worn surface of steel ball, which would effectively restrain the material loss of Ti6Al4V disk due to the adhesive wear, but might disturb the stability of friction coefficient.
Figure 6. The analyses of worn surfaces lubricated by PEG + 1.0% Zn at different stages: (a) wear scar on Ti6Al4V disk at 60 s; (b) wear scar on Ti6Al4V disk at 360 s; (c) wear scar on Ti6Al4V disk at 1800 s; (d) wear scar on Ti6Al4V disk at 3600 s, the insert tables are typical elements content of (a)–(d); (e) wear scar on steel ball at 60 s; (f) wear scar on steel ball at 360 s; (g) wear scar on steel ball at 1800 s; (h) wear scar on steel ball at 3600 s, the insert curves are typical elements content of selected areas by dotted boxes in (e)–(h); (e1)–(h1) Zn element distributions on surfaces (e)–(h).

Figure 7. The lubrication properties of different concentrations of Zn nanoparticle additives in PEG oil: (a) friction coefficient curves as function of time; (b) steady friction coefficients and wear volume.
4. Discussion of mechanism

For a deeper understanding about the boundary lubricating mechanism of Zn nanoparticles in PEG oil for steel/Ti6Al4V contact, chemical states of typical elements on the worn surfaces were gained by XPS analysis and results were shown in Figure 9. As can be seen in Figure 9(a), the binding energy of Zn2p appears at 1022.30 eV, which is in good agreement with ZnO [26], indicating that ZnO boundary protective films generated in PEG oil on both worn surfaces of upper ball and lower disk. In Figure 9(b), the binding energies of Ti2p appears at 458.50 eV and 464.30 eV, which are in good agreement with TiO2 [26]. Furtherly, it can be seen that very few TiO2 are detected on the surface of steel ball. It indicated that the formation of ZnO boundary protective films...
could avoid the direct contact between the steel ball and Ti6Al4V disk, and the material loss of Ti6Al4V disk due to severe adhesive wear was effectively restrained.

Based on the tribological investigations and surface analyses by SEM/EDX and XPS, the main achievements can be concluded as that the lubrication performance was improved and the ‘run-in’ period was obviously shortened through the addition of Zn nanoparticle into PEG oil. Furtherly, the schematic about the generation process of boundary protective films as well as the nanofluid delivery system are summarized and shown in figure 10. Before the sliding began, the steel/Ti6Al4V contact lubricated by PEG suspension with Zn nanoparticles was in boundary lubrication regime. The suspension state was the first step in delivering the Zn nanofluid to the frictional contact area. Direct contact between Ti6Al4V disk and steel ball can be seen in figure 10(a). During the ‘run-in’ period (figure 10(b)), plenty of Ti-containing materials transferred from Ti6Al4V disk to the counterface of steel ball, forming TiO2 layers, and few Zn nanoparticles substantially participated the generation of boundary films. As a result, ‘run-in’ period was still the high friction and severe wear period for steel/Ti6Al4V contact lubricated by PEG suspension with Zn nanoparticles. In this process, the delivery of nanofluid mainly depended on the adsorption of Zn nanoparticles on the nascent friction surfaces and the tribochemical reactions of nanofluid under high load. At the steady friction stage (figure 10(c)), continuous and strong ZnO boundary films generated on the counterfaces of Ti6Al4V disk and steel ball, avoiding the direct contact of steel/Ti6Al4V. With the increase of the temperature of contact region at this stage, the viscosity of nanofluid decreased. Nanofluids are more likely to be transported to the contact surfaces by oscillating to repair the damaged friction region. Therefore, the transformation of Ti-containing materials due to the severe adhesive wear could be effectively restrained.

5. Conclusions

Based on the results and discussions above, following conclusions can be obtained:

(1) The addition of Zn nanoparticles in PEG oil can effectively increase the friction-reduction and anti-wear properties for steel/Ti6Al4V contact. Furtherly, as the additive concentration increases from 0.5% to 2.0%, the ‘run-in’ period was obviously shortened.

(2) On the counterfaces of Ti6Al4V disk and steel ball, continuous and strong ZnO boundary films generated to avoid the direct contact of steel/Ti6Al4V. Furtherly, the material transfer from Ti6Al4V disk to the counterface of steel ball due the severe adhesive wear was effectively restrained.

(3) Boundary lubrication for Ti6Al4V has always been a tough problem. PEG suspension with Zn nanoparticles will be a promising lubricant to solve it.
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Conflicts of interest

The authors declare no conflict of interest.

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