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A study of the tribological behaviour of TiO₂ nano-additive water-based lubricants

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A study of the tribological behaviour of TiO₂ nano-additive water-based lubricants

Abstract

A ball-on-disk tribometer was employed to evaluate the lubrication performance and mechanisms of innovative TiO₂ nano-additive water-based lubricants. Two experimental methods were applied to determine the optimal mass fraction of TiO₂. In the method I, lubricants were added onto the worn disk tracks at a predetermined time interval. In the method II, the disks were immersed in the lubricants continuously during the whole process of tribological tests. The results both indicate that the water-based lubricants can significantly reduce the coefficient of friction (COF). The 0.8 wt% TiO₂ lubricant demonstrates excellent tribological properties including the lowest COF and the strongest wear resistance under all lubrication conditions. The lubrication mechanisms are attributed to the rolling and mending effects of the TiO₂ nanoparticles.

Disciplines

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Study on tribological behaviour of TiO₂ nano-additive water-based lubricants

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Abstract: A ball-on-disk tribometer was employed to evaluate the lubrication performance of novel TiO₂ nano-additive water-based lubricants and examine the role of TiO₂ nanoparticles dispersed in water. Two experimental methods in this study were conducted to determine the optimal mass fraction of TiO₂. In method I, various lubricants were added into the worn disk track at a predetermined time interval of each test. While in method II, the disk was immersed in the lubricants during the whole process of tribological test. The results both indicate that the novel water-based lubricants can significantly reduce the coefficient of friction (COF). Especially, the 0.8 wt.% TiO₂ presents comprehensive tribological properties including the lowest COF and the strongest wear resistance in all lubrication conditions. The lubrication mechanisms were dominated by rolling and mending effects of the TiO₂ nano-additive.

Keywords: TiO₂ nano-additive; Water-based lubricant; Tribology; Ball-on-disk; Coefficient of friction

1. Introduction

Friction, wear and lubrication between materials in contact are of fundamental importance in many pure and applied sciences [1]. Reduction in friction and wear is one of the most important objectives of tribological research [2]. Due to the special physical and chemical properties of nanomaterials, considerable studies have been conducted in recent years using nanoparticles as additives in lubricating oil to improve the tribological properties of lubricants [3-22]. Li et al. [8, 9] prepared well-dispersed oil-based lubricants with nano-SiO₂ as an additive to increase the anti-wear ability and reduce the COF under four-ball and ring-on-block test. A film formed by nano-SiO₂ protected the metal-on-metal surfaces and filled the wear scars. Some researchers [10, 12, 13, 16, 19] added Cu nanoparticles into the base oil to improve the anti-wear, load-carrying, and friction-reduction performances. The formation of Cu-containing boundary film benefits the separation of friction pairs from rubbing each other. Furthermore, some researchers investigated the tribological properties of fullerene nano-additive

mineral oil, and found that fullerene nanoparticles between the friction surfaces improved the lubricating performance by increasing the viscosity of lubricating oil and simultaneously preventing direct metal surface contacts [5-7, 15, 17]. Composite nanoparticles have also been proposed to act as additives in lubricating oil to obtain improvement in anti-wear, extreme pressure, and friction-reducing properties because the individual component cannot perform effectively due to the restrained physicochemical properties [20-23].

Although the nano-additive oil-based lubricants exhibit excellent lubrication performance for tribological applications, it inevitably leads to environmental pollution and is also difficult to be recycled. In addition, the nozzles which supply flow of the fluid are prone to being blocked and regular maintenance is required frequently. Therefore, development of an environment-friendly water-based lubricant with superior lubrication performance becomes a trend in the field of lubrication.

Nowadays, few researchers have applied nanoparticle as an additive in water-based lubricants and the corresponding results have been rarely reported. TiO_2 is widely used as a nano-additive in oil-based lubricants [3, 4, 11], and it is increasing attention in water-based lubricants [24-26]. However, the role of nano-additive dispersed in water and the corresponding lubrication mechanism have not been clearly understood until now.

In the present work, the water-based lubricants with varying TiO_2 nano-additive additions were synthesised, and the lubrication performance was evaluated under a ball-on-disk tribometer. The objective of this study is to understand the lubrication mechanism of as-synthesised lubricants through experimental analysis and further lubrication models.

2. Experimental

2.1 Materials

A low-carbon microalloyed steel was adopted as disk material in this study. Its chemical compositions are listed in Table 1.

Table 1 Chemical compositions of the low carbon microalloyed steel (wt.%)

C	Si	Mn	P	Cr	S	N	Nb+V+Ti
0.05	0.02	0.25	0.014	0.01	0.002	0.003	<0.01

All the disks for ball-on-disk tests were machined to 40 mm in diameter and 8 mm in thickness with grinding on the surfaces to ensure the surface roughness (R_a) was about $0.03 \mu\text{m}$, which can eliminate the influence of the original surface condition on the experimental results. The Vickers hardness of the disk material is 90 HV.

E52100 Cr steel balls with a diameter of 9.5 mm were used for ball-on-disks. The Vickers hardness of the ball material is 780 HV.

2.2 Synthesis

The TiO₂ nano-additive water-based lubricants were synthesised following the flow chart shown in Fig. 1. Firstly, TiO₂ nanoparticles (P25 sourced from Sigma- Aldrich™ with approx.20 nm in diameter) were mixed into the deionised water by mechanical stirring. Secondly, Polyethyleneimine (PEI) was added into the solution dropwise followed by a high speed centrifuge at 20,000 rpm for 30 min to prepare a dispersive solution. PEI is a cationic polymer, which acts as a surfactant of TiO₂ to improve the dispersing property of the nanoparticles. Afterwards, glycerol was added dropwise. Glycerol is a colourless, odourless, and viscous liquid, which is mainly used to improve the viscosity of solutions. The solution was then processed by ultrasonication with stirring for 10 min to break down any remaining agglomeration. The synthesised TiO₂ nano-additive water-based lubricant showed good colloid stability, and no sedimentation could be observed in 7 days of aging.

The chemical compositions of as-synthesised lubricants are outlined in Table 2. Different lubrication conditions numbered from 1 to 9 were used for tribological tests. The dry condition and water lubrication condition were used as benchmarks in comparison to the lubrication effects of as-synthesised water-based nano-additive lubricants. The water-based lubricants were composed of different mass fractions of TiO₂ nanoparticles (from 0.2 to 8.0 wt.%), and corresponding volume fractions of PEI. The concentration of glycerol was fixed to 10.0 vol. % for each type of lubricant.

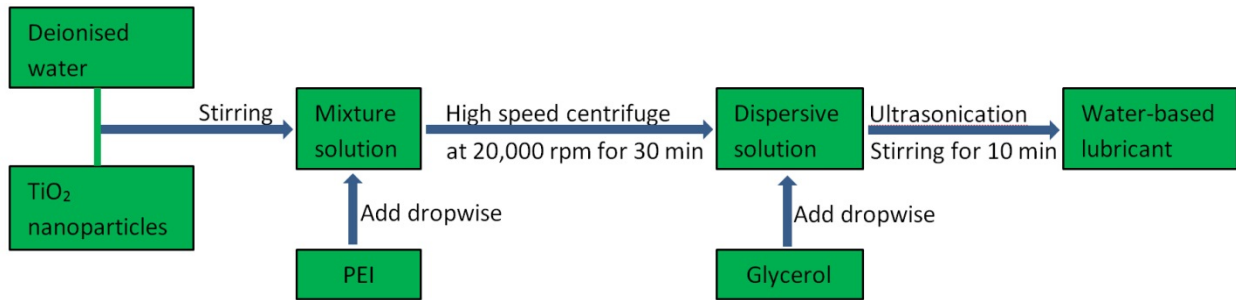


Fig. 1 Flow chart of synthesis of TiO₂ nano-additive water-based lubricants

Table 2 Chemical compositions of lubricants

Lubrication numbers	Description
1	Dry condition
2	Water
3	0.2 wt.% TiO ₂ +0.002 wt.% PEI + 10.0 vol.% glycerol + balance water
4	0.4 wt.% TiO ₂ +0.004 wt.% PEI + 10.0 vol.% glycerol + balance water
5	0.8 wt.% TiO ₂ +0.008 wt.% PEI + 10.0 vol.% glycerol + balance water
6	1.0 wt.% TiO ₂ +0.01 wt.% PEI + 10.0 vol.% glycerol + balance water
7	2.0 wt.% TiO ₂ +0.02 wt.% PEI + 10.0 vol.% glycerol + balance water
8	4.0 wt.% TiO ₂ +0.04 wt.% PEI + 10.0 vol.% glycerol + balance water
9	8.0 wt.% TiO ₂ +0.08 wt.% PEI + 10.0 vol.% glycerol + balance water

2.3 Tribological tests

COF is an important factor in evaluating the characteristics of lubricants. A Rtec MFT-5000 Multi-functional Tribometer was employed to measure the COF values by ball-on-disk tribological tests. The basic configuration of the ball-on-disk tribometer is schematically shown in Fig. 2. The ball holder with Cr

steel ball and the disk were cleaned with ethanol and then assembled prior to the tests. The disk was fastened to the disk holder by a screw and a small pin was inserted to ensure the disk can be rotated together with the holder smoothly. The arm adjusted by a bubble level should be accurately horizontal to reduce experimental error. The normal force applied on the ball holder was measured by a F_z load cell installed above a spring. While the friction force was induced by the combination of the rotating motion and the normal load and it was measured by a F_x load cell attached to the right point of the arm. The disk holder and disk which were controlled by a servo motor for rotating were located in a liquid container. Two types of lubrication methods (defined as M-I and M-II) were used in this study, and the detailed experimental conditions have been elaborated in Tables 2 and 3, respectively.

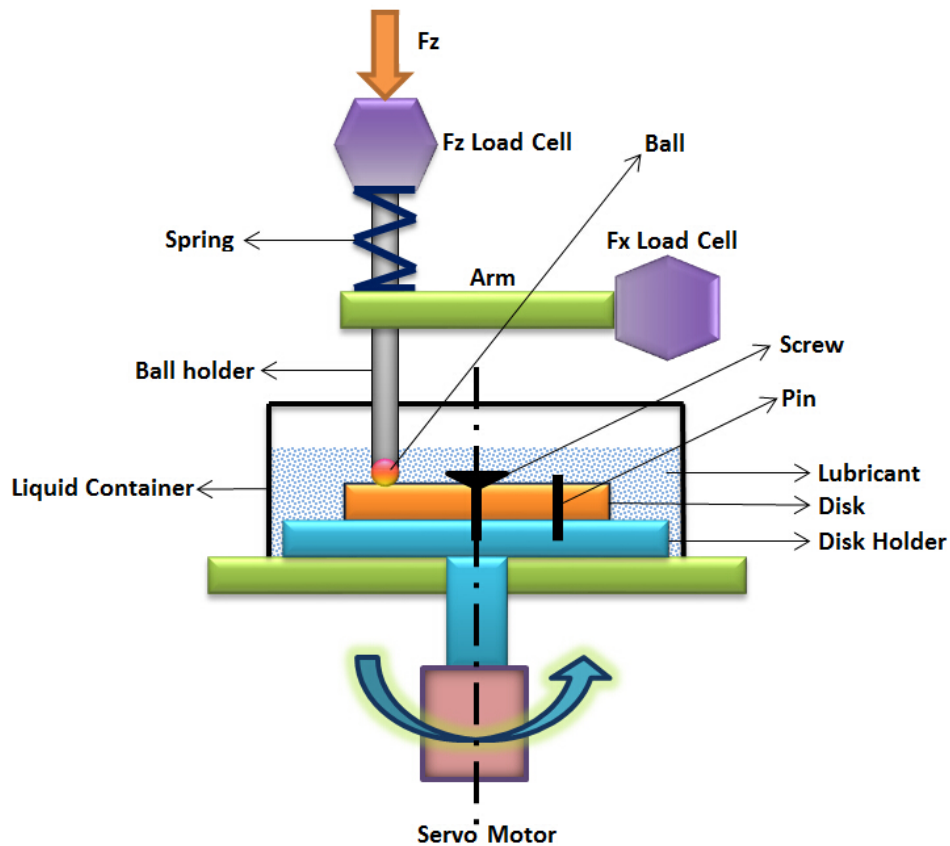


Fig. 2 Schematic of ball-on-disk tribometer used for lubrication tests

Table 2 specifies the experimental conditions of M-I. The tests were conducted at room temperature. Prior to the tests, the ball was located on the disk at a position of 14 mm in radius. Then the disk was applied a normal force of 50 N and started to rotate with a linear speed of 20 mm/s, which was restricted to reduce the hydrodynamic effect of liquid. By calculation, the rotating speed of 13.65 rpm was obtained and was input into the software in computer. The tribological tests were implemented under dry condition during the first five-minute rotation followed by continuous drops of lubricants onto the wear track in the rest five minutes. Based on this experimental process, the M-I can be defined as “dry condition plus lubricants addition”.

Table 3 lists the experimental conditions of M-II. The tests were also conducted at room temperature. Prior to the tests, the disks were immersed into the liquid bath with a normal force of 50 N applied on the ball. The rotating linear speed was also restricted to 20 mm/s to reduce the hydrodynamic effect. The tests under the same lubrication condition were carried out with three tracks at radii of 14, 15, and 16mm, respectively, to obtain mean COF value. Each track was generated following the same linear speed of 20 mm/s with corresponding rotating speeds of 13.65, 12.74, and 11.94 rpm, and each process lasted for 10 min. Similarly, the M-II can be defined as “continuous lubrication condition”.

Table 2 Experimental conditions of M-I

Normal force	Testing temperature	Rotating speed	Testing duration
50 N	room temperature	13.65 rpm	5 min plus 5 min

Table 3 Experimental conditions of M-II

Normal force	Testing temperature	Rotating speed	Testing duration
50 N	room temperature	13.65, 12.74, 11.94 rpm	10 min

2.4 Characterisation of nanoparticles

Powder X-ray diffraction (XRD) was implemented on a Philips PW1730 conventional diffraction meter with Cu-K α radiation. The XRD pattern of the nanoparticles is shown in Fig. 3, from which the phase of particles can be determined as typical P25 TiO $_2$ containing 75% of anatase and 25% of rutile by referring to the XRD standard atlas.

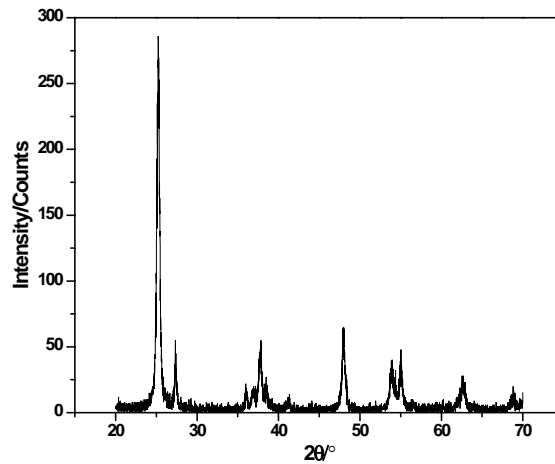


Fig. 3 XRD pattern of TiO $_2$ nanoparticles

Micrographs of the TiO $_2$ nanoparticles inside the water-based lubricants were obtained using a Hitachi model H-800 Transmission Electron Microscope (TEM) coupled with an energy-dispersive spectrometry (EDS). Fig. 4 exhibits the TEM image of TiO $_2$ nanoparticles dispersed in the as-prepared water-based lubricants. It is clearly seen that most nanoparticles are round in shape with an average diameter of 20 nm, and the nanoparticles are uniform and well-dispersed without apparent agglomeration.

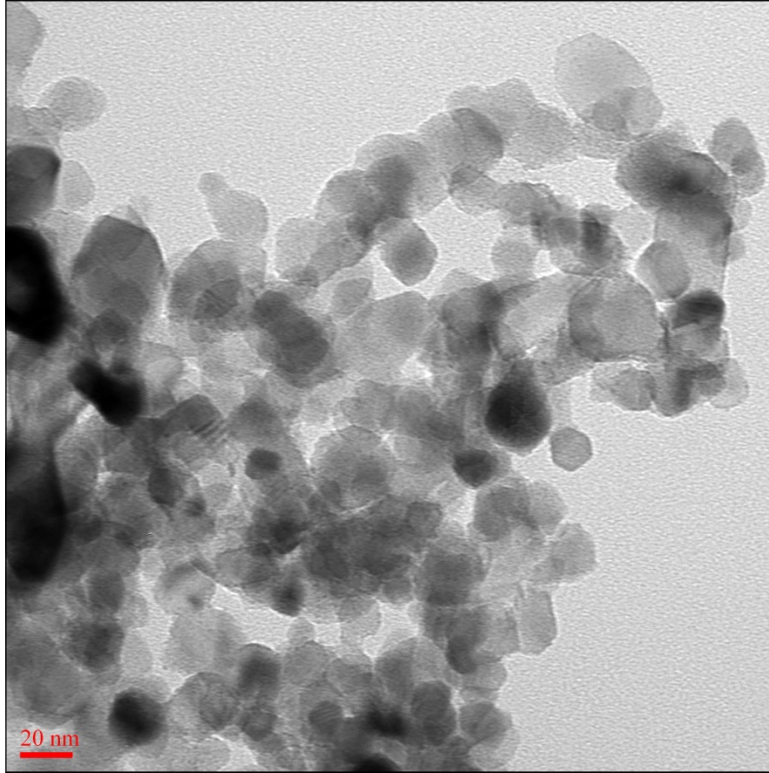


Fig. 4 TEM image of TiO_2 nanoparticles dried from water-based lubricant

The wear tracks of balls and disks after tribological tests were cleaned in an ultrasonic acetone bath and observed under KEYENCE VK-X100K 3D Laser Scanning Microscope, from which the 3D profile of the wear areas of both ball and disk were obtained. The wear scars of balls were then observed using a JEOL model JSM-6490 Scanning Electron Microscope (SEM) equipped with an EDS to evaluate the lubrication mechanism.

3. Results and discussion

3.1 Tribological properties

3.1.1 Dry condition plus lubricants addition

Fig. 5 presents variation of COF values before and after lubricants additions on disks. In the first half of the whole tribological process, dry condition leads to fluctuation at the beginning due to the severe friction between the ball and disk. Then the curves decrease to a stable stage as the friction process behaves gently on a uniform worn track. With the addition of lubricants onto the worn disk track, all the COF values reduces significantly followed by smooth curves to the end of the tests. It is clear that water-based lubricants with different TiO_2 additions show much lower COF values than that of water. However, the reduced COF curves of water-based lubricants cannot be distinguished obviously in this figure as they are quite close to each other. The detailed difference of these COF values is shown in Fig. 6(a).

Fig. 6(a) and (b) show the COF values after additions of lubricants and wear areas of balls under different lubrication conditions, respectively. Compared to the COF of dry condition, water and water-based lubricants all exhibit remarkable lubrication effects especially for the water-based lubricants, the

COF values of which decrease continuously with an increase of TiO₂ nano-additive and reach the lowest point when 0.8 wt.% TiO₂ is added, as shown in Fig. 6(a). A rising COF value is observed when TiO₂ fraction is further increased to 2.0 wt.%, and it begins to keep a constant even though the TiO₂ fraction increases to 8.0 wt.%.

The ball wear under different lubrication conditions is shown in Fig. 6(b). It can be seen that the addition of 0.8 wt.% TiO₂ induces the lowest ball wear, which is consistent with the COF values indicated in Fig. 6(a). Fig. 7 shows the 3D profile images of the ball wear areas. It can be seen that water induces wear area of 2239 μm² along z axis (depth) after calculation according to the profile curves. While, the water-based lubricants with additions of 0.4, 0.8 and 4.0 wt.% TiO₂ present much smaller wear areas than that caused by water. Among the water-based lubricants, the worn surface becomes smoother when the mass fraction of TiO₂ reaches 0.8 wt.%, indicating that 0.8 wt.% TiO₂-containing lubricant has better wear resistance as compared to other lubrication conditions. Based on the 3D profile images, abrasive ploughing should be the main wear manner for all the friction processes in this study, which will be explained later with the elaboration of lubrication mechanisms.

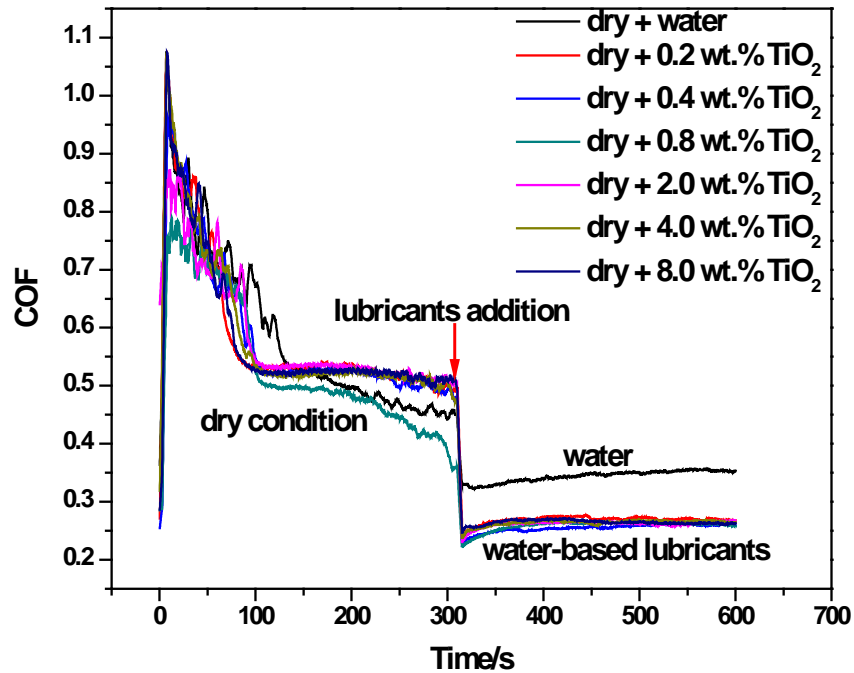


Fig. 5 Variation of COF values with addition of lubricants after 5-min dry condition friction

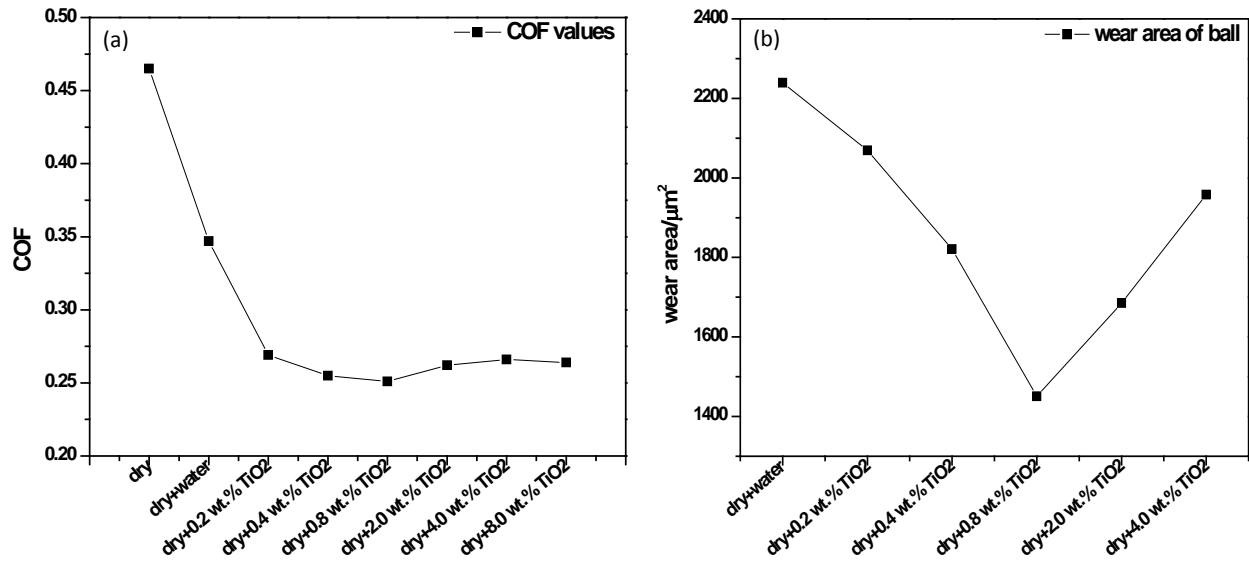


Fig. 6(a) COF values after additions of lubricants and (b) wear areas of balls under different lubrication conditions

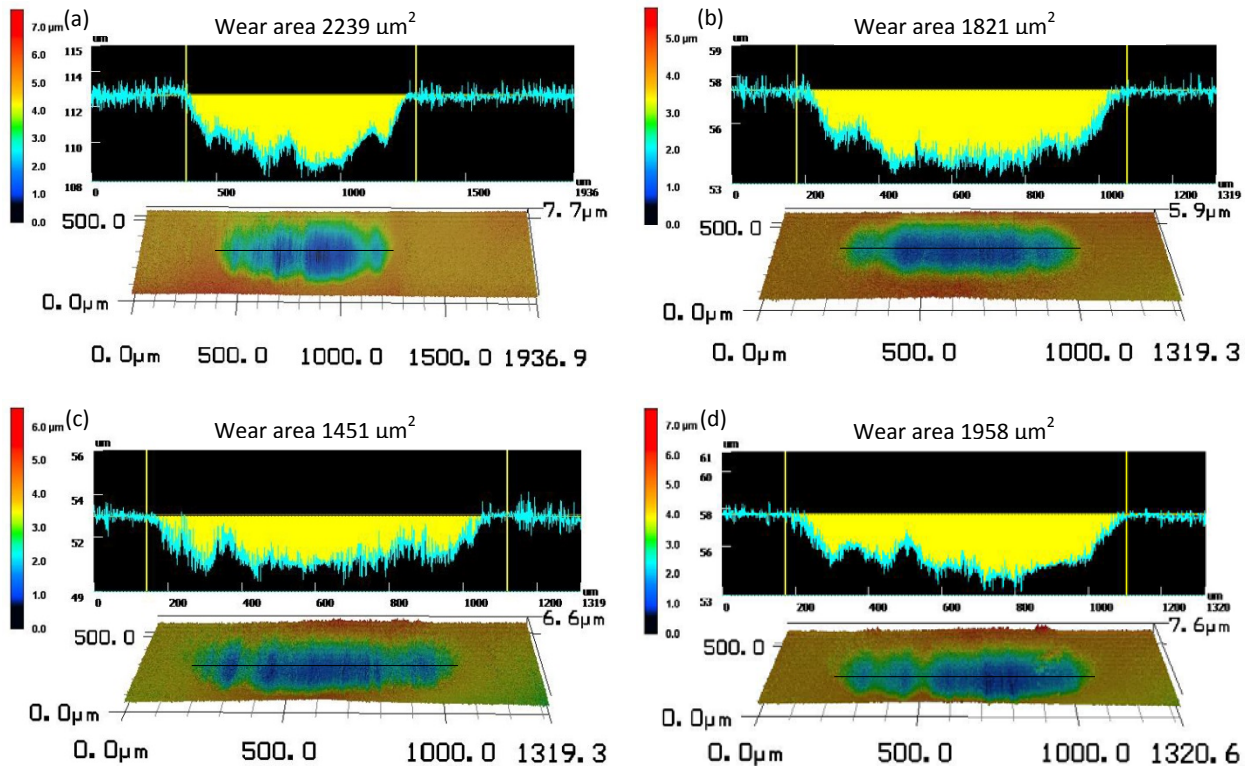


Fig. 7 3D profile images and curves of ball wear areas by (a) water, (b) 0.4 wt.% TiO₂, (c) 0.8 wt.% TiO₂, and (d) 4.0 wt.% TiO₂

3.1.2 Continuous lubrication condition

Fig. 8 shows the comprehensive tribological properties under continuous lubrication conditions with water and water-based lubricants. It is coincident to the results shown in Fig. 6 that 0.8 wt.% TiO₂ exhibits the best lubrication effects of all. Furthermore, both wear areas of the disk and ball along Z axis

confirm that 0.8 wt.% is the optimum mass fraction for TiO₂ nano-additive water based lubricants at room temperature. The 3D profile images of the disk wear areas are shown in Fig. 9. It can be seen that the water lubrication generates abrasive friction and the wear area along z axis (depth) is obtained to be 1371 μm². While with additions of TiO₂ nano-additive at lower fractions of 0.2 and 0.8 wt.%, the wear areas are decreased to around 1000 μm². However, the wear area goes up to 1977 μm² when lubricated by 4.0 wt.% TiO₂, which is even higher than those under other lubrication conditions. Furthermore, furrows exist only inside the wear tracks lubricated by 0.2 and 0.8 wt.% TiO₂. The probable reason is that the lower fraction of TiO₂ nano-additive makes particles much easier to enter the rubbing zone and take better lubrication effect in comparison to the lubricants with higher fraction such as 4.0 wt.% [27].

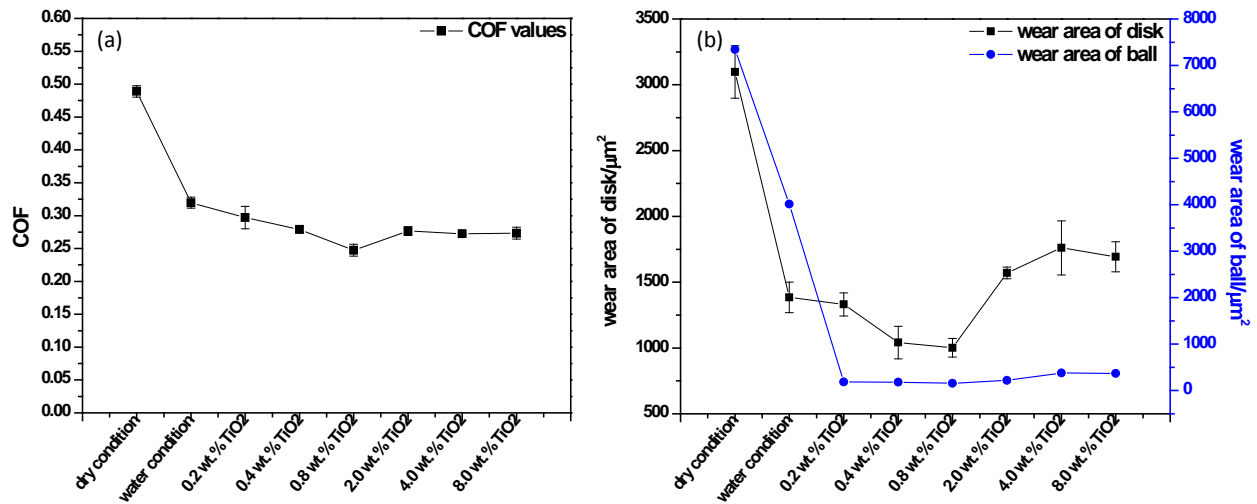
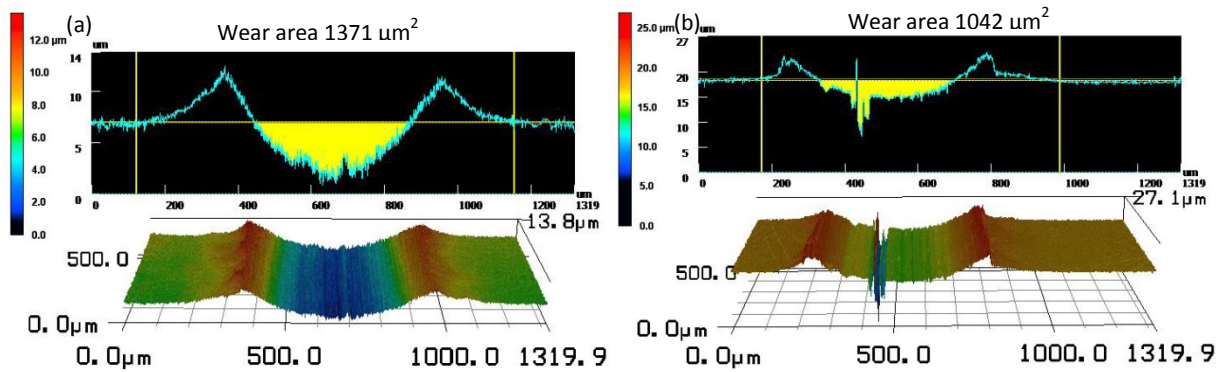


Fig. 8 (a) COF values with continuous additions of lubricants and (b) wear areas of balls and disks under different lubrication conditions



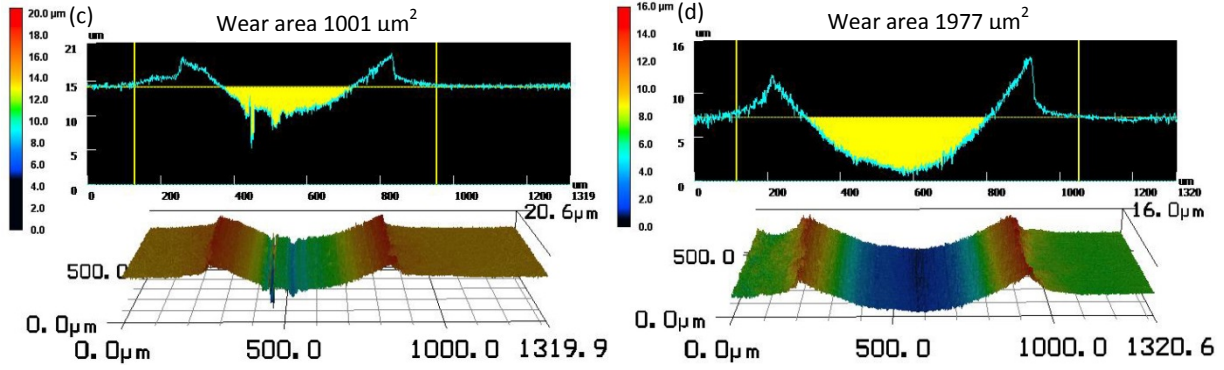


Fig. 9 3D profile images and curves of disk wear areas by (a) water, (b) 0.2 wt.% TiO₂, (c) 0.8 wt.% TiO₂, and (d) 4.0 wt.% TiO₂

3.2 Lubrication mechanism

In the past years, a number of lubrication mechanisms have been proposed to explain the improvement of lubrication effect by using various nanoparticles as additives in lubricating oil, including rolling effect [3, 17, 21, 27-30], mending effect [16], polishing effect [3, 27, 31], and protective film [4, 8, 10, 11, 13, 14, 19, 32-36], as schematically shown in Fig. 10 in terms of ball-on-disk friction. The rolling effect and protective film are mainly used to separate the friction pairs rubbing each other on COF reduction, while mending and polishing effects act on the enhancement of surface quality. In this study, the lubrication mechanisms of the TiO₂ nano-additive water-based lubricants will be addressed based on the four lubrication mechanisms.

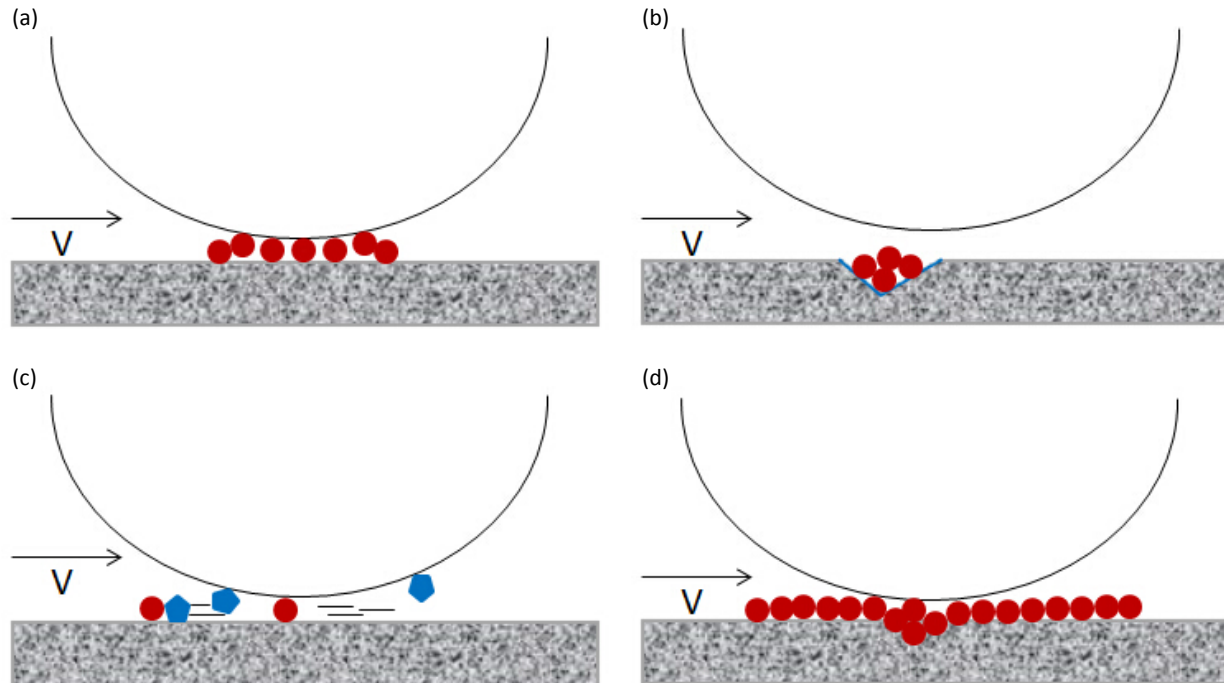


Fig. 10 The schematic of lubrication mechanisms under TiO₂ nano-additive water-based lubricants

(a) rolling effect, (b) mending effect, (c) polishing effect, and (d) protective film

Fig. 11 shows the SEM images and EDS mappings of the disk sample lubricated by 0.8 wt.% TiO₂. It can be seen that a large number of TiO₂ nanoparticles have filled in the defects of disk surfaces. In comparison to the SEM images in Fig. 11(a) and (c), the bright zones in Fig. 11(b) and (d) give a direct evidence to support this phenomenon, which is defined as “mending effect”.

Fig. 12(a)-(d) give the SEM images of disk samples lubricated by 0.2, 0.8, 2.0, and 4.0 wt.% TiO₂, respectively. Fig.12(e) and (f) give the EDS spectra to show the element titanium. It can be seen that TiO₂ nanoparticles are distributed dispersedly in the worn disk tracks, which indicates that the spherical TiO₂ nanoparticles can roll between the rubbing surfaces in the process of friction. This “rolling effect” of TiO₂ nanoparticles in water-based lubricants contributes to a significant COF reduction in comparison to that caused by water. When the mass fraction of TiO₂ is below 0.8 wt.% (such as 0.2 wt.%), as shown in Fig. 12(a), particle-depleted zones appear in the worn disk track, which implies few nanoparticles taking effect between the rubbing surfaces, and hence the COF and wear area are not reduced significantly. While, with the mass fraction of TiO₂ nanoparticles increases above 0.8 wt.%, as shown in Fig. 12(c)-(d), some nanoparticles would be agglomerated, and then the secondary particle size becomes coarse (around 50 nm for 2.0 wt.% TiO₂ and 100 nm for 4.0 wt.% TiO₂). This would aggravate the friction and wear, and therefore leads to increase of COF and wear area [21]. Differently, as shown in Fig. 12(b), the disk surface lubricated by 0.8 wt.% TiO₂ owns numerous nanoparticles distributed evenly with small size in diameter, which induces even lower COF and smaller wear area as compared to other lubricants with different TiO₂-additions. Therefore, 0.8 wt.% is considered to be the optimal mass fraction of TiO₂ as nano-additive in the as-synthesised water-based lubricant. The role of TiO₂ nanoparticles and the lubrication mechanism can be further elaborated in Fig. 13.

Fig. 13 demonstrates the lubrication mechanisms with varying TiO₂ mass fractions of 0.2, 0.8, and 4.0 wt.%. At a low TiO₂ fraction of 0.2 wt.% (Fig. 13(a)), few nanoparticles can enter the rubbing surfaces, as a result the lubrication effect is appeared to be insignificant. When the TiO₂ fraction increases to 4.0 wt.%, as shown in Fig. 13(c), a large amount of nanoparticles accumulate around the rubbing zone to be agglomerated as a barrier which decreases the continuous supply of nanoparticles to the zone for lubrication [27]. Furthermore, the large-sized particles which can still roll between the ball and disk are supposed to deteriorate the wear during friction process [21]. Differently, the 0.8 wt.% TiO₂-containing lubricant is able to feed abundant nanoparticles continuously to the rubbing zone during friction process owing to the fine size without agglomeration, as shown in Fig. 13(b). Therefore, 0.8 wt.% is thought to be the optimum mass fraction of TiO₂ to present the best tribological properties of all the as-synthesised water-based lubricants.

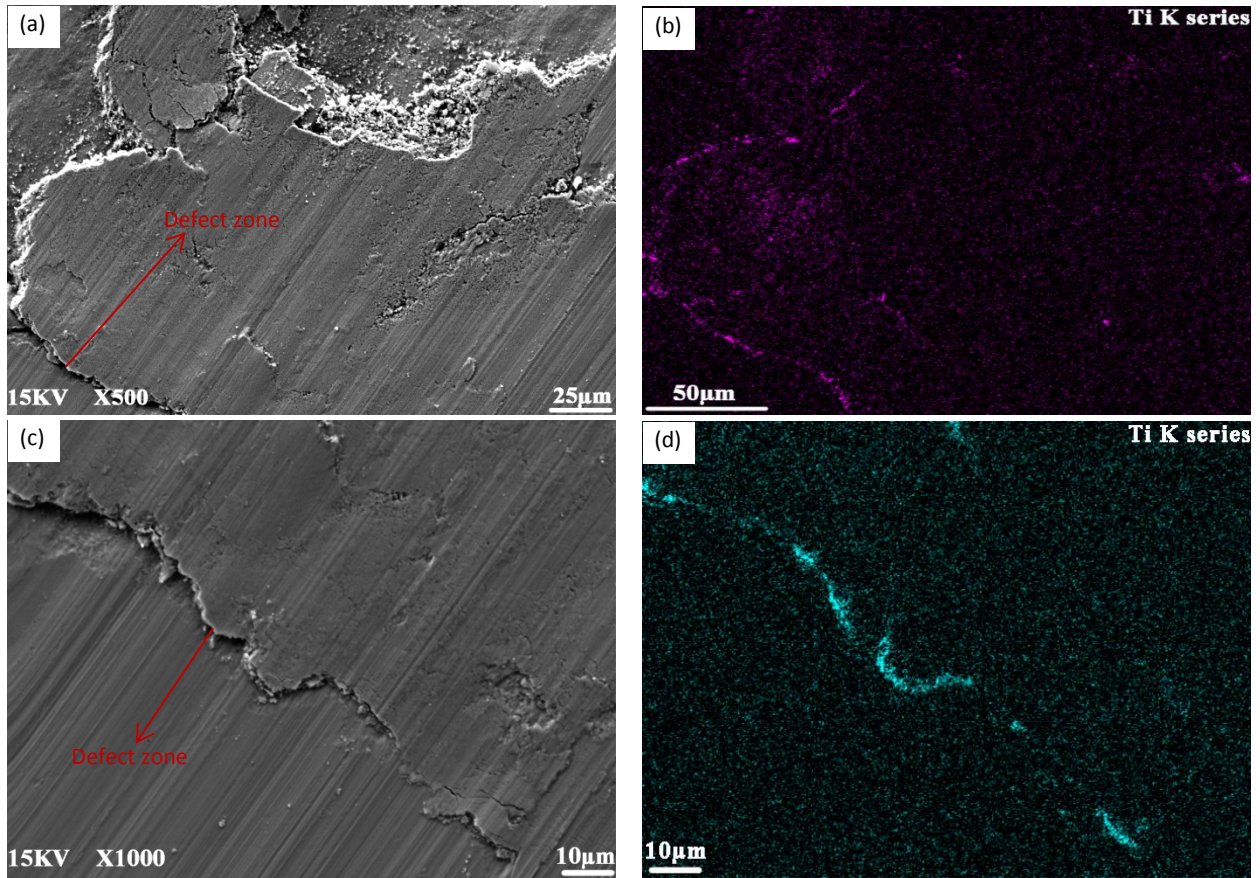
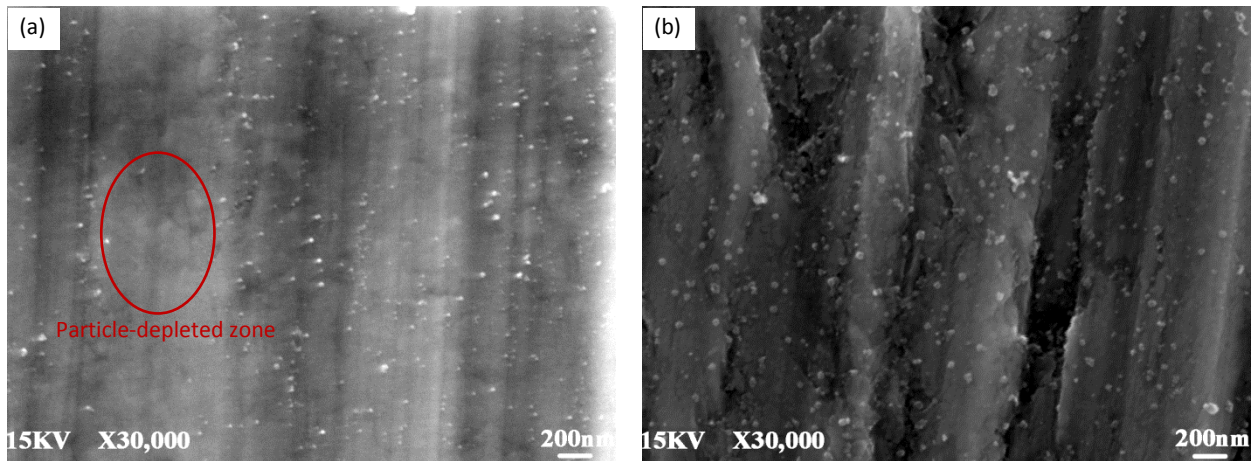


Fig. 11 SEM images and EDS mappings of the disk sample lubricated by 0.8 wt.% TiO₂: (a) SEM image, (b) EDS mapping of (a), (c) SEM image, and (d) EDS mapping of (c)



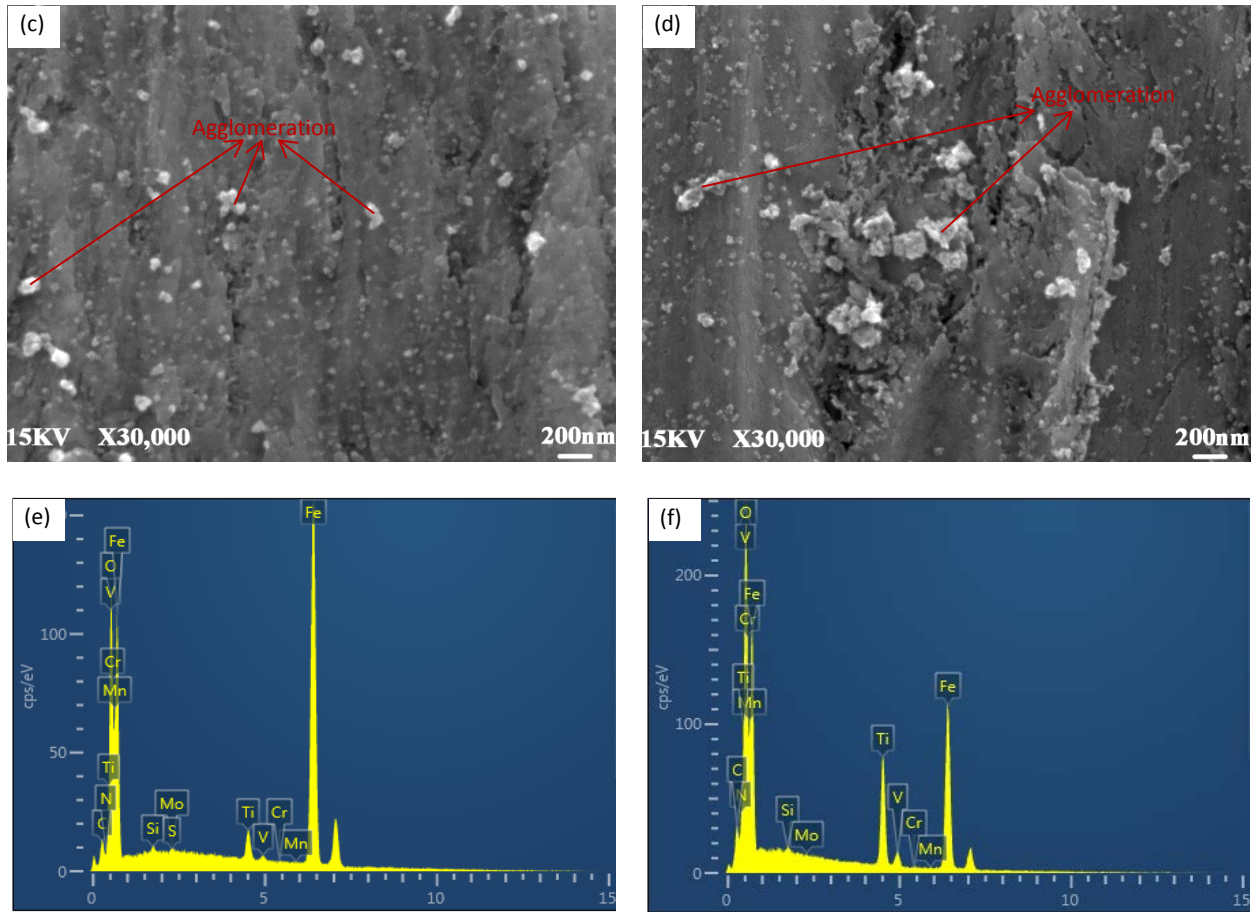
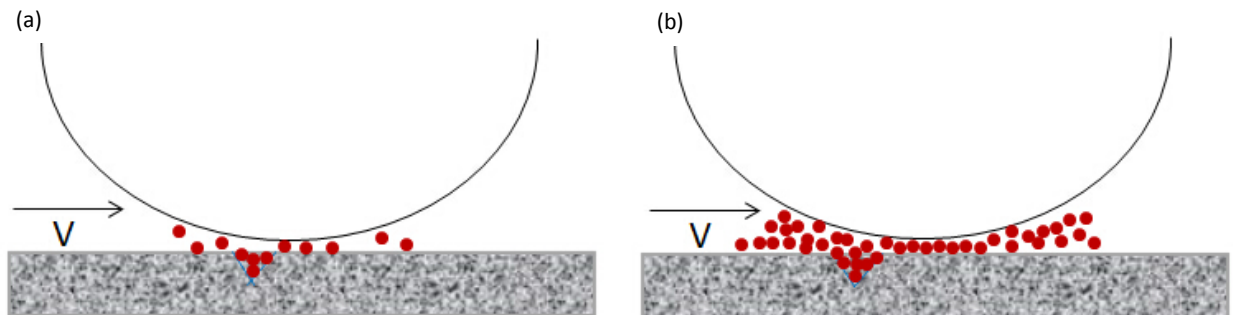


Fig. 12 SEM images of the samples lubricated by (a) 0.2 wt.% TiO₂, (b) 0.8 wt.% TiO₂, (c) 2.0 wt.% TiO₂, and (d) 4.0 wt.% TiO₂, and EDS spectra of (e) 0.8 wt.% TiO₂, and (f) 4.0 wt.% TiO₂



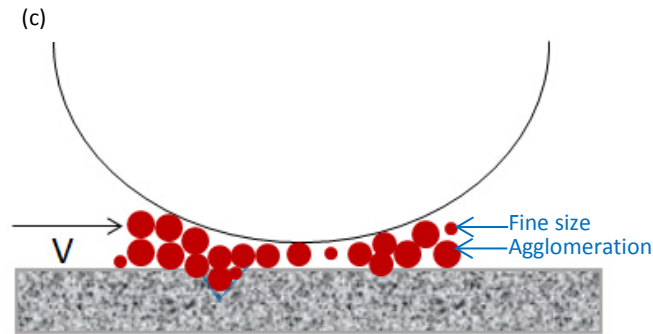


Fig. 13 The models of lubrication mechanisms by (a) 0.2 wt.% TiO₂, (b) 0.8 wt.% TiO₂, and (c) 4.0 wt.% TiO₂

4. Conclusions

The lubrication properties of TiO₂ nano-additive water-based lubricants were evaluated by ball-on-disk tests at room temperature based on two different testing methods. The following conclusions have been drawn from this study.

- (1) The addition of TiO₂ nanoparticles into water can significantly reduce the COF and improve the wear resistance. The COF and ball wear can be decreased by 49.5% and 97.8%, respectively, compared to those of dry condition.
- (2) Low fraction of TiO₂ below 0.8 wt.% leads to few nanoparticles taking effect for COF reduction; while high fraction of TiO₂ above 0.8 wt. % results in agglomeration of nanoparticles and increases COF.
- (3) 0.8 wt.% TiO₂ water-based lubricant leads to the lowest COF of all the lubrication conditions conducted by dry friction, water and water-based lubricants at room temperature.
- (4) 0.8 wt.% TiO₂ water-based lubricant induces the smallest ball and disk wear areas on all the lubricated friction pairs at room temperature.
- (5) Rolling effect and mending effect are thought to be the lubrication mechanism of TiO₂ nano-additive water-based lubricants.

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