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Tribological Properties of Nano-Scale Al_2O_3 Particles with Different Shapes as Lubricating Oil Additives

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Abstract: Enhancing lubrication across various tribological systems in the maritime industry is essential for improving safety, efficiency, and environmental sustainability. Al_2O_3 nanoparticles, employed as additives in lubricating oils, demonstrate favorable tribological properties including anti-wear and anti-friction characteristics. In this work, nano-scale γ-Al_2O_3 particles with different shapes, i.e., nanosheet, nanorod, nanosphere, and irregular-shaped nanoparticles, were prepared and calcinated forming the same crystalline phase with nanoscale size, which dispersed well in lubricating oil. The tribological properties of Al_2O_3 nanoparticles as lubricating oil additives were examined using block-on-ring wear tests, and the effects of the particle shape and particle concentration were investigated. The results indicated that the frictional properties are largely influenced by the particle shape and the concentration of the Al_2O_3 additives, with the optimal concentration being around 0.1 wt% for each shape. The lubricating oil with nanosheet additives presented the best tribological performance, followed by those with nanorod, nanosphere, and irregular-shaped Al_2O_3 nanoparticle additives. Al_2O_3 nanosheets as the lubricating oil additives reduced the stress effect on the friction surface because of their larger bearing area and are inter-particle-sheared during sliding due to the movement of friction pairs, which can further improve the tribological properties compared to other shapes.

Keywords: Al_2O_3 nanoparticles; different shapes; lubricating oil additives; tribology properties

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1. Introduction

The rapidly developing maritime transportation industry faces a series of tribological issues. Moreover, the severe marine environment demands higher tribological performance from the equipment used in maritime navigation. In particular, marine diesel engines play a pivotal role in the maritime sector, serving as the primary propulsion power source for more than 95% of ocean-going merchant vessels. Given their indispensable nature, addressing tribological challenges within the critical friction pairs of these engines is imperative to ensure vessel safety, operational efficiency, and environmental sustainability. Consequently, there is a pressing need to enhance lubrication mechanisms to mitigate fuel consumption and optimize mechanical performance, particularly in light of escalating marine fuel costs and heightened environmental concerns associated with engine operations [1,2].

High-performance lubricants can significantly reduce mechanical friction and wear, which is of great significance for stable operation and energy saving. As the machine industry faces stringent government regulations and technological competition, conventional

lubricants containing environmentally unfriendly elements such as sulfur, phosphorus, and zinc are no longer sufficient to meet the demands of practical applications [3,4]. Therefore, researchers worldwide are focusing on developing new environmentally friendly lubricant additives.

Nanomaterials with unique properties are being investigated as lubricant additives to enhance lubricant performance. Existing studies show that the addition of nanoparticles into lubricating oil can effectively improve the tribological properties of the lubricating oil [5–7]. For example, Zhou et al. [8,9] studied the impact of particle size on the friction-reducing properties of Cu nanoparticles, and discovered that nanoparticles of about 5 nm can provide better friction reduction effects in base oil because of the good dispersity and friction-reducing film-forming capability. Furthermore, Li et al. and Sun et al. [10,11] investigated two types of nanometallic particles, i.e., Cu and Ag, as lubricant additives, respectively. Various concentrations of the Cu and Ag nanoparticles on the wear resistance and load-carrying capacity of the lubricants were studied, and it was found that the addition of Cu and Ag nanoparticles in a certain range can significantly enhance the tribological properties of the lubricants. Abdullah et al. [12] reported that hBN nanoparticles used as lubricating oil additives have great potential to improve the friction performance of engines. The research indicates that an optimal concentration of 0.5% of 70 nm hBN nanoparticles added to SAE 15W-40 oil can lower the coefficient of friction (COF) and enhance wear resistance compared to classical diesel engine oil. Wu et al. [7] reported that sphere-like CuO nanoparticles added to two lubricating oils led to a decrease in the COF by 6% and 18% at 1.0 wt%, respectively, due to the rolling effect of the sphere-like nanoparticles.

These nanomaterials reduce friction and wear by forming a tribofilm on the friction pairs, and simultaneously repairing, filling, and polishing rough surfaces to a certain extent [13]. However, the nanoparticle shape is a key factor influencing lubricant performance [14]. In conventional nano additives, it is a challenge to retain their original shapes between friction pairs, which hampers the exploration of the impact of nanoparticle structure on friction performance. Al_2O_3 is well known for its excellent properties, including high hardness and chemical stability [15]. Therefore, Al_2O_3 nanoparticles can keep their original shape under high loads and provide the possibility of exploring the effect of nanoparticle shape on tribological performance. Meanwhile, Al_2O_3 nanoparticles have been reported as a highly promising lubricating additive. Luo et al. [16] studied the optimal tribological performance of 0.1 wt% Al_2O_3-nanoparticle-modified oil and confirmed the ball bearing effect of Al_2O_3 nanoparticles between the contact surfaces. Ketan Suthar et al. [17] found that the COF and wear were significantly reduced when adding 0.1 wt% Al_2O_3 nanoparticles to jojoba oil and believed that this was due to the deposition of nanoparticles on the friction pairs to form a strong tribofilm. Although there are some studies on Al_2O_3 nanoparticles of different shapes, such as sphere and rod shapes [18,19], the lubrication effects of other shapes have not been fully investigated, and the lubricant mechanism involved has not been well understood. Consequently, it is of great significance to investigate the friction and wear behavior of nanoparticles with different shapes as lubricant additives.

In this work, Al_2O_3 nanoparticles with excellent material properties including high hardness, high temperature stability, and chemical stability were studied as lubricant additives. Nano-scale γ-Al_2O_3 particles with different shapes, i.e., nanosheet, nanorod, nanosphere, and irregular-shaped nanoparticles, were prepared and calcinated to form the same crystalline phase with the nanoscale size first. The tribological performance of these Al_2O_3 nanoparticles as lubricating oil additives were studied by block-on-ring wear tests, and the effects of the particle shape and particle concentration were investigated. Finally, the lubrication mechanisms of the Al_2O_3 nanoparticle additives were discussed. This research holds great promise for advancing the efficiency and sustainability of low-speed marine diesel engines, addressing critical challenges related to fuel consumption and environmental impact in the maritime sector.

2. Experimental Methods

2.1. Preparation of Al_2O_3 Nanoparticles

γ-Al_2O_3 nanosheets were prepared via a hydrothermal synthetic method. Briefly, 10 mL of pre-prepared $Al(NO_3)_3$ solution (0.2 mol L^{-1}) and 10 mL of NaOH solution (0.2 mol L^{-1}) were mixed and stirred with a magnetic mixer for 10 min, and then the suspension was loaded into a Teflon™ reactor in an electric blast-drying oven at 180 °C for 3h. Then, it was cooled to room temperature and stirred for 30 min after adding 4.5 mL of ethanol. After that, the hydrothermal reaction was performed at 200 °C for another 8 h. Finally, the obtained solid was washed three times with DI water, dried at 80 °C, and calcined at 550 °C for 7 h.

γ-Al_2O_3 nanorods were also prepared by the hydrothermal synthetic method. Here, 0.02 g of $Al(NO_3)_3$ was dissolved in 50 mL of DI water, and a NaOH solution of 1 mol L^{-1} was added drop by drop until the pH value reached 5. Then, it was kept in a Teflon™ reactor for 24 h at 200 °C. The achieved solid was centrifugally washed with DI water and acetone 3 times, respectively. The solid was dried at 100 °C overnight and calcined at 550 °C for 7 h.

γ-Al_2O_3 nanospheres were prepared by electrostatic spinning. Aluminum isopropyl alcohol and DI water were added to a three-way flask, refluxing at 85 °C for 4 h; then, nitric acid was added until the pH value reached 5.0, and the solution was kept for another 24 h. Half of the water was evaporated by heating, and then the same amount of ethanol and appropriate amount of PVP were added, stirred for 24 h, and electrospun at 15 kV voltage. Finally, it was evaporated at 100 °C for 24 h and calcined at 550 °C for 7 h.

Irregular-shaped γ-Al_2O_3 nanoparticles were synthesized by double hydrolysis. Briefly, 2 g of partial sodium aluminate (all reagents were purchased from MackLin Reagent Company) and 2 g of aluminum sulfate hydrate were mixed in 20 mL of DI water and stirred with a magnetic mixer for 30 min. Then, it was transferred into a Teflon™ reactor and kept at 150 °C for 5 h. After that, it was washed with DI water and ethanol 3 times, respectively. After drying in a vacuum drying chamber at 80 °C for 8 h, it was calcined at 550 °C for 7 h.

Figure 1 shows the morphologies of the prepared Al_2O_3 nanoparticles. In this work, a commercially available synthetic base oil (HVIH-5, viscosity: 5cst at 100 °C) was used as a base lubricant. Figure 2a shows the viscosity–temperature curve of the base oil. The Al_2O_3 nanoparticles were added into the base oil at the different concentrations of 0.05, 0.1, 0.15, and 0.2 wt% under ambient temperature. Oleic acid was used as a surface dispersant. In order to obtain a good dispersion, the Al_2O_3 nanoparticles were mixed in the base oil by ultrasonic dispersion. The samples can be suspended stably under natural placement conditions. The dispersion stability of the Al_2O_3 nanolubricant was monitored via visual observation. Figure 2b shows the visual appearance of the Al_2O_3 nanolubricant after direct dispersion and storage for 2 and 5 days. The color and homogeneity of the Al_2O_3 nanolubricant sample showed little change throughout the storage duration. The Al_2O_3 nanolubricant samples (base oil + 0.1wt% nanosheet and base oil + 0.1wt% nanosphere) had slight sediment. Some Al_2O_3 nanoparticles could be observed at the bottom of the bottles.

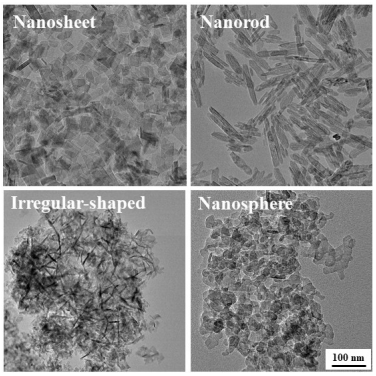


Figure 1. TEM images of Al_2O_3 nanoparticles.

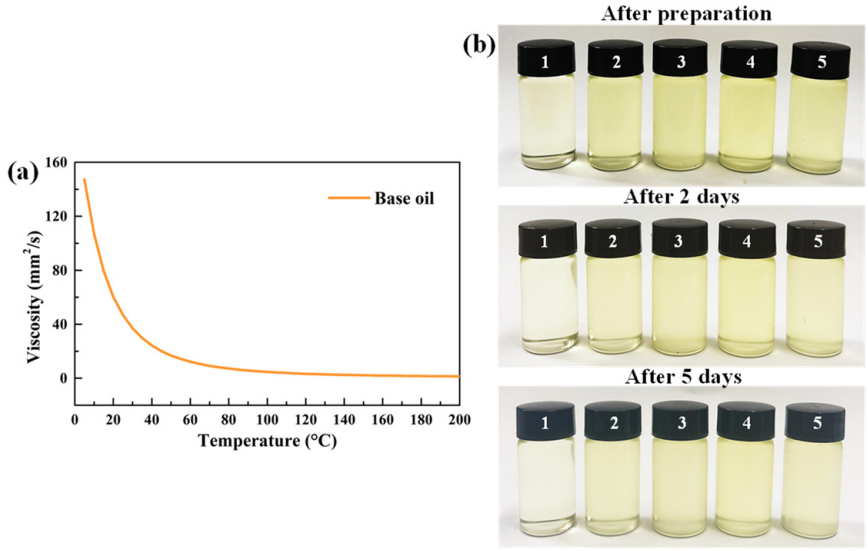


Figure 2. (**a**) Viscosity–temperature curve of base oil, and (**b**) visual appearance of the Al_2O_3 nanolubricant at different times. (1) Base oil, (2) base oil + 0.1wt% irregular-shaped nanoparticles, (3) base oil + 0.1 wt% nanorods, (4) base oil + 0.1 wt% nanosheets, (5) base oil + 0.1 wt% nanospheres.

The XRD pattern of the γ-Al_2O_3 nanoparticles with different shapes is shown in Figure 3. The strong diffraction peaks appearing at 2θ = 31.4, 37.7, 39.3, 45.5, 60.3, and 66.8° corresponded to (220), (311), (222), (400), (511), and (440) crystal planes of the face-centered cubic (fcc) structure of γ-Al_2O_3, which are in good agreement with the common JCPDS (card NO. 10-0425) and no impurity peaks are observed [20,21]. The peak intensities of all samples exhibit remarkable similarity, thereby confirming their analogous crystalline nature. The XRD analysis demonstrated that the four differently shaped samples contain 100% γ-Al_2O_3.

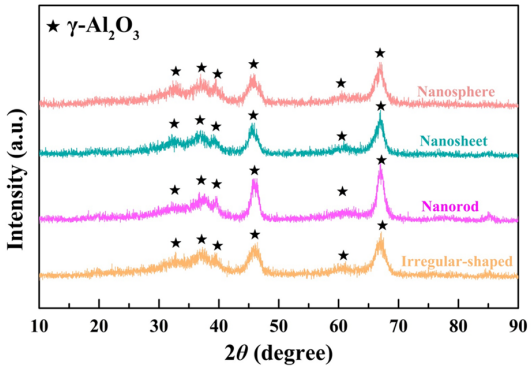


Figure 3. XRD patterns of the γ-Al_2O_3 with different shapes.

2.2. Tribological Test

Block-on-ring tests were conducted to study the tribological properties of the lubricating oil with Al_2O_3 nanoparticle additives. The testing configuration of the block—on—ring wear test machine is shown in Figure 4. The block was held in a special collet inside a pressure head. The ring was immersed in the lubricating oil, and rotated by a Servo motor. The contact form of the block and ring specimen in the tester is line contact. The block and ring materials were both GCr15, and the hardness was approximately 1433.7 $HV_{0.1}$ and 1169 $HV_{0.1}$, respectively. The ring has dimensions of Φ50 mm × 25 mm, while the block is smaller with dimensions of Φ10 mm ×10 mm. The surface roughness, Ra, of the block and ring was about 0.11 and 0.35 μm, respectively. Details regarding the operating conditions during the experiment are presented in Table 1. The block and ring specimens were ultrasonically cleaned in petrol and alcohol for around 20 min before and after the test.

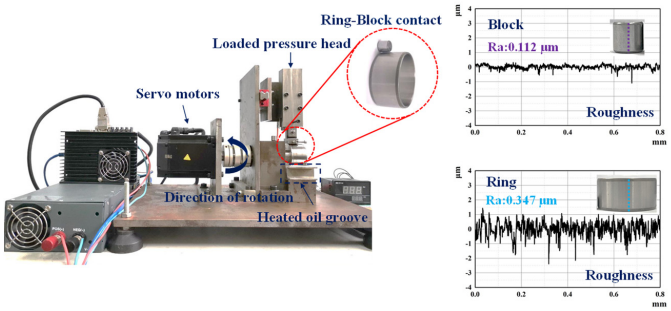


Figure 4. Block—on—ring wear tester and the testing configuration.

Table 1. Experimental parameters of block—on—ring wear tests.

Case	Parameters	Value
1	Normal load	50 N
2	Test temperature	200 °C
3	Rotational speed	10 r/s
4	Test time	45 min
5	Lubricant amount	4 mL

The coefficient of friction was recorded using a data processing system. Before and after each wear test, the morphologies of the block sample surfaces were examined by a scanning electron microscope and an optical microscope, and the wear amount was assessed by measuring the width of the wear scars. For each additive shape and concentration, three replicate experiments were performed to ensure the reliability of the obtained results.

3. Results and Discussion

3.1. Effects of Nanoparticle Shape and Concentration on Coefficient of Friction

Figure 5 shows the friction coefficient of Al_2O_3 nano-additives with different shapes at different concentrations. It can be observed that the nanoparticle shape and concentration have a significant impact on the friction behavior. The average friction coefficient is 0.091 with the addition of Al_2O_3 nanosheets, which is the lowest among the four types of Al_2O_3 additives and is 14.47% lower than that of using base oil. In addition, the minimum friction coefficient was obtained when the addition concentration was 0.1% for the four different shapes of the Al_2O_3 nanoparticles. When the concentration is lower or higher than optimal, the friction reduction properties weaken, although they remain superior to those of the base oil. It is reported that when the concentration of nanoparticles is too high, stronger interference between nanoparticles occurs and may lead to an increase in friction [22].

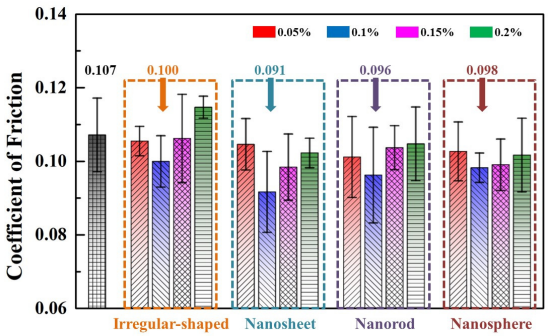


Figure 5. Friction coefficient of Al_2O_3 additives with different shapes at different concentrations.

3.2. Effects of Nanoparticle Shape and Concentration on the Wear of Block Surfaces

Figure 6 shows the wear scar width after the wear tests using the different lubricants. The wear scar width was smaller with the addition of Al_2O_3 nanoparticle additives than

that when using the base oil. When the concentration was 0.1%, the wear scar width was at its minimum for all four types of nanoparticles. The wear scar width with the addition of Al_2O_3 nanosheets was the smallest at 319 μm on average and 12.6% less than that of using the base oil.

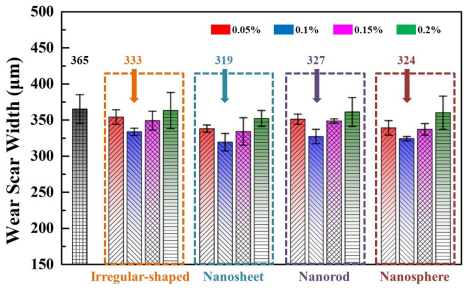


Figure 6. Corresponding wear scar width with different shapes and content of additives.

Figure 7 presents SEM micrographs of the block surfaces before and after the wear tests using the different lubricants. Figure 7a depicts the block surface before the wear test and the original cutting texture can be seen on the surface. In addition, some tiny grooves and burrs can be found on the original surface. Figure 7b shows the wear result using base oil: it can be seen that relatively severe wear occurred, leading to the almost complete disappearance of the original cutting marks. Plastic deformation along the sliding direction and the existence of some grooves can be seen on the worn surface. When the irregular-shaped nanoparticles were used as lubricating additives, slight original cutting marks were observed compared to under base oil conditions (Figure 7c), which means a milder wear occurred. However, under the nanosheet, nanorod, and nanosphere additive lubrication (Figure 7d–f), it can be seen that the original machining marks were largely retained on the block wear surface. This indicates the effective anti-friction and anti-wear properties offered by these different additive-based lubricants. Al_2O_3 nanoparticles move into worn areas and are physically adsorbed on the contact surface under the exerted compressive stress and the flow of lubricating oil. At this time, the addition of nanoparticles reduces direct contact between the friction pairs, and the presence of spheres and rods transforms the pure sliding friction into mixed sliding–rolling friction [23]. The Al_2O_3 nanosheets can be adsorbed onto the friction surfaces, resulting in easy sliding.

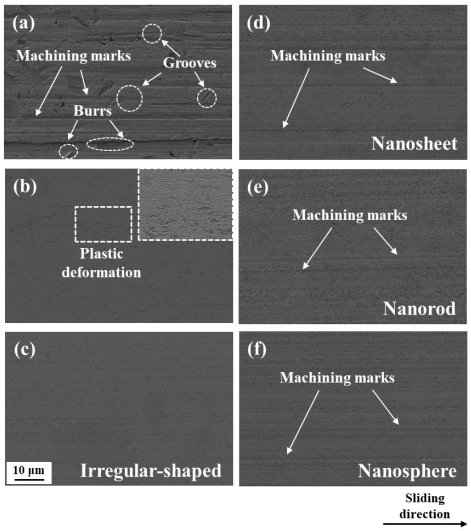


Figure 7. SEM micrographs of block surface after wear tests. (**a**) Original surface, (**b**) base oil, (**c**) base oil + 0.1wt% nanorods, (**d**) base oil + 0.1wt% nanosheets, (**e**) base oil + 0.1wt% irregular-shaped nanoparticles, and (**f**) base oil + 0.1wt% nanospheres.

3.3. Possible Lubrication Mechanism of Al_2O_3 Nanoparticle Additives

Based on the experimental findings described above, the possible lubrication mechanisms of the Al_2O_3 nanoparticle additives with different shapes were schematically plotted and are shown in Figure 8. It can be seen from Figure 8b that direct contact occurs between the friction pairs under the high load and temperature conditions, due to the limited load-carrying capacity of the pure base oil. Thus, the friction force and wear rate are greatly increased at the friction interface. When Al_2O_3 nanoparticles are added to the base oil, they prevent the direct contact of the friction pairs under boundary lubrication. Then, the Al_2O_3 nanoparticles form an isolation layer in the wear scar (Figure 8b–f). At the same time, the presence of Al_2O_3 nanoparticles on the surface changes the friction pattern. However, the irregular-shaped Al_2O_3 nanoparticles do not show a significant improvement in friction pattern (Figure 8c). The spherical and rod-shaped nanoparticles behave like nano-bearings, converting sliding friction into rolling friction (Figure 8d,e) [24]. But, there is significant difference in tribological performance between spherical and rod-shaped nanoparticles, which may be due to differences in contact. Compared with point contact, line contact increases the bearing area. Due to the high hardness of Al_2O_3 nanoparticles, the large bearing area is beneficial to decrease the contact stress of the friction surface, which increases the anti-friction and anti-wear to a certain extent. Compared to other nano-structured Al_2O_3 nanoparticles, sheet-shaped Al_2O_3 nanoparticles have a much higher specific surface area, making them easier to deposit on the friction surface [25]. They also have a larger bearing area (Figure 8j), which reduces the stress effect of the friction surface. Meanwhile, the sheet-shaped Al_2O_3 nanoparticles are inter-particle-sheared during sliding due to the movement of friction pairs, exhibiting the best tribological properties (Figure 8f).

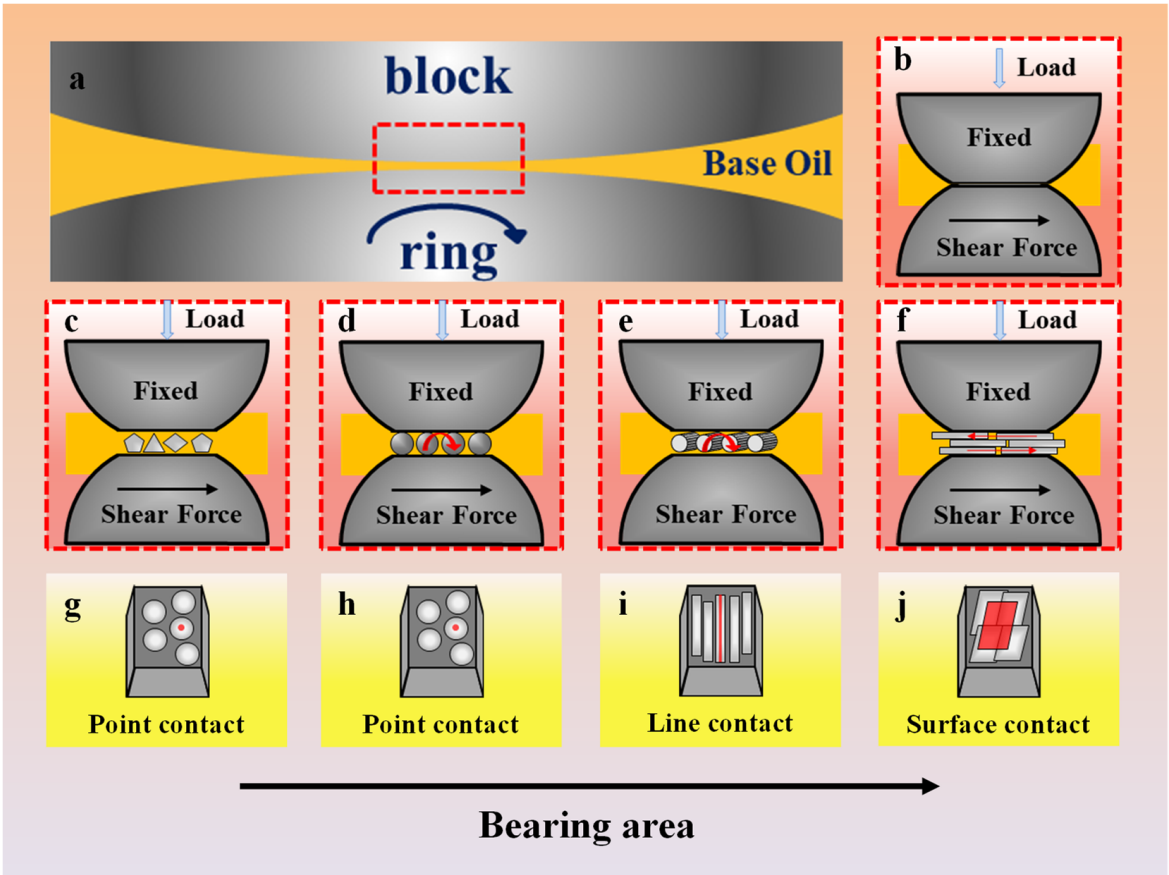


Figure 8. Schematic diagram of possible lubrication mechanisms of the Al_2O_3 nanoparticle additives. (**a**) motion state of friction pairs, (**b**–**f**) contact form of friction pairs (**g**–**j**) contact state of additives.

4. Conclusions

In this study, nano-scale γ-Al_2O_3 particles with different shapes including nanosheets, nanorods, nanospheres, and irregular-shaped nanoparticles were prepared by calcination, resulting in the same crystalline phase. Differently shaped Al_2O_3 nanoparticles can be evenly dispersed in the lubricating oil through the dispersant (oleic acid). The influence of Al_2O_3 nanoparticles on the tribological behavior of block sliding against a ring was evaluated under different nanoparticle shapes and additive concentrations. Based on the current experimental findings, the main conclusions are as follows:

(1) The addition of Al_2O_3 nanoparticles in lubricating oil can affect the tribological performance of the friction pairs. In this study, the best tribological performance was obtained when the additive concentration was 0.1 wt% regardless of the nanoparticle shapes. Al_2O_3 nanosheets presented the best anti-friction and anti-wear behaviors.

(2) Under boundary lubrication conditions, the Al_2O_3 nanoparticles deposit onto the wear scar and separate the friction pair, reducing the direct contact between the friction pair. Additionally, the presence of Al_2O_3 nanoparticles on the surface changes the sliding friction pattern. Compared to other shapes, the nanosheet particles, with their larger bearing area, reduce the stress effect on the friction surface and exhibit the best tribological properties.

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