Effect of h-BN Nanoparticles on the Tribological Properties and Lubricating Mechanism of Compound Lithium Grease

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Keywords: h-BN Nanoparticles; Compound Lithium Grease; Friction and Wear; Lubricating Mechanism.

ABSTRACT

Lithium base grease is a fundamental lubricating material that has been used widely in industrial equipment. However, its tribological performance is not adequate for the rapid development of advanced equipment. In this work, the influence of hexagonal boron nitride (h-BN) nanoparticles on the tribological performance of compound lithium grease was systematically examined. Comprehensive friction and wear tests were conducted utilizing a four-ball tribometer. The findings revealed that the lithium grease with 0.2wt% h-BN nanoparticles exhibited superior tribological properties, and the coefficient of friction and wear scar diameter decreased by 24.2% and 14.2%, respectively. To investigate the lubricating mechanism, the steel ball wear scars were analyzed through scanning electron microscopy (SEM), confocal laser scanning microscopy (CLSM), and X-ray photoelectron spectroscopy (XPS). The results indicated that h-BN nanoparticles infiltrate the wear surfaces during the friction process, forming a protective B_2O_3 oxide film.

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INTRODUCTION

Grease is extensively utilized in wheel bearings and is often referred to as the "fifth element" due to its critical role in friction. The grease forms a lubricating film that protects the contact surfaces during wheel bearing operation, thereby reducing wear and significantly impacting the service life of wheel bearings (Jain et al., 2024). However, as wheel bearings evolve towards higher speeds, greater reliability, and longer lifespans, traditional grease no longer meets performance requirements. Thus, developing high-performance wheel bearing grease is of paramount importance (Ghatage et al., 2020).

In recent years, nanomaterials have demonstrated significant application potential across various fields (Mateti et al., 2018; Merlo et al., 2018). Compared to conventional additives, nanomaterials exhibit superior physicochemical properties (Li et al., 2018) and are increasingly utilized in lubrication. Currently, carbon nanomaterials (graphite, graphene, carbon nanotubes, etc.) (Kumar et al., 2020; Rawat et al., 2020; Senatore et al., 2021), metal nanomaterials (Cu, Ag, Al, etc.) (Muhammad et al., 2023; Ma et al., 2009; Peng et al., 2010), oxide nanomaterials (CuO, Al_2O_3, SiO_2, etc.) (Zheng et al., 2020; Radice et al., 2006; Rylski et al., 2019), and sulfide nanomaterials (WS_2, MoS_2, etc.) (Rawat et al., 2019; Kang et al., 2008) are extensively employed as grease additives.

Hexagonal boron nitride (h-BN) has a layered structure akin to graphite, and its excellent high-temperature stability and anti-wear properties make it a widely used grease additive (Li et al., 2022; Wen et al., 2015). Wang et al. (2020) examined the influence of varying mass fractions of boron nitride nanoparticles on the tribological properties of lithium grease. The results indicated that the grease with 0.6wt% BN nanoparticles exhibited the best anti-friction properties, with BN nanoparticles forming a lubrication film to protect the friction pair.

Cheng et al. (2023) investigated the friction and anti-friction properties of h-BN nanoparticles as additives in lithium grease under sliding conditions. The results demonstrated that the grease containing 3wt% 60 nm h-BN particles and 1wt% 500 nm h-BN particles reduced wear scar diameters by 22.34% and 20.18%, respectively. Wu et al. (2022) examined the effects of h-BN and calcium carbonate nanoparticles on the tribological and vibration performance of polyurea grease. Their findings indicated that polyurea grease with 1wt% h-BN and 5wt% $CaCO_3$ nanoparticles exhibit optimal tribological and vibration suppression properties. Kumar et al. (2023) explored the impact of h-BN particle size on grease performance. The results revealed that smaller h-BN particles more readily penetrate the interstitial spaces of the friction pair, promoting the formation of a protective friction film. These studies collectively indicate that h-BN nanoparticles enhance the tribological properties of grease. However, the mechanism underlying h-BN's friction-reducing capabilities remains unclear.

This study investigated the influence of h-BN nanoparticles on the tribological performance of composite lithium grease utilizing a four-ball tribometer. Furthermore, the friction-reducing mechanism of h-BN particles was elucidated through observations of the wear scar surfaces via scanning electron microscopy (SEM), confocal laser scanning microscopy (CLSM), and X-ray photoelectron spectroscopy (XPS).

EXPERIMENT

Materials and characterization techniques

Hexagonal boron nitride (h-BN) nanoparticles were sourced from Sahn Chemical Technology (Shanghai) Co., Ltd. These nanoparticles appear as white powder with a particle size of 1 μm and a purity of 99.5%. The compound lithium-based grease used in this study was developed by the PetroChina Lanzhou Lubricating Oil Research and Development Center. The base oils are PAO40 and 150BS, the thickener is lithium dodecahydroxystearate, the viscosity at room temperature is 220 mm^2/s, and the dropping point is 280 °C. The surface morphology of h-BN was characterized using tungsten filament scanning electron microscopy (EVO10, Zeiss, Germany), phase analysis was conducted with an X-ray diffractometer (XRD-6100, Shimadzu, Japan), and particle size was analyzed by a nanoparticle size and zeta potential analyzer (Zetasizer Nano ZSE, Malvern, UK).

Preparation of h-BN nano-grease

An electronic balance was used to measure a specific mass of base grease, which was then placed into a beaker. The corresponding mass of h-BN nanoparticles was also measured using an electronic balance and added to the beaker. The mixture was mechanically stirred for 10 minutes to achieve initial homogeneity, followed by grinding with an S65 three-roll grinder for 30 minutes. This process produced h-BN nanoparticle grease with mass fractions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5%.

Friction and wear testing

A four-ball tribometer (MRS-10W, Jinan Liantai Testing Machine Co., Ltd.) was employed to conduct the friction and wear tests on the grease. The test conditions were in accordance with ASTM D2266-23: test temperature of 75 °C, rotational speed of 1200 r/min, load of 392 N, and duration of 30 minutes. The steel balls used in the test were GCr15 bearing steel balls with a diameter of 12.7 mm and hardness of 64~66 HRC. To ensure the accuracy of the test results, each sample was tested in duplicate.

The four-ball tribometer is a widely utilized experimental apparatus to assess the tribological characteristics of lubricating substances. The device primarily comprises four spheres (Figure 1(a)), where three bottom spheres are stationary, immersed in lubricant, and the fourth sphere rotates to exert pressure and induce friction. The interaction between the rotating sphere, the lubricant, and the stationary spheres enables the simulation of friction and wear processes akin to real-world conditions, allowing for the measurement of parameters such as the friction coefficient, wear volume, and wear morphology.

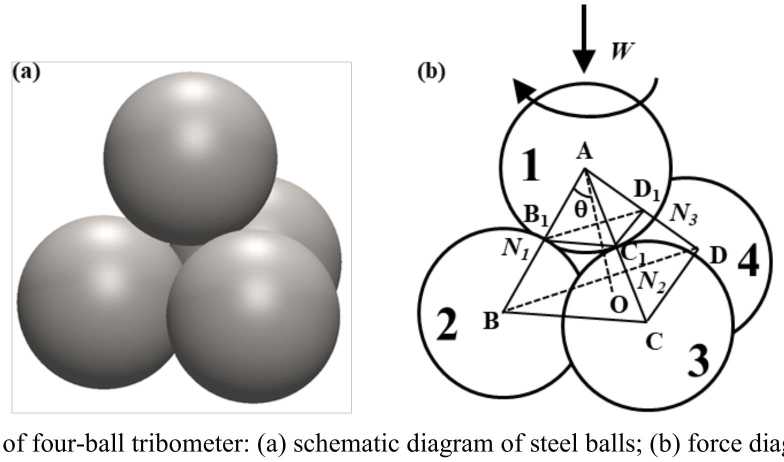

Fig. 1. Steel balls of four-ball tribometer: (a) schematic diagram of steel balls; (b) force diagram of steel balls

Fig. 1(b) shows the force diagram of the steel balls, and the linear velocities of the contact points of the bottom three steel balls and the top steel ball is:

$$V = \frac{2\pi n}{60}\frac{1}{\sqrt{3}}R \quad (1)$$

Where n and R represents spindle speed and steel ball radius.

The vertical force loaded on ball 1 is W. The forces on the bottom three steel balls at the point of contact with the top steel ball are N_1, N_2, N_3. Construct the equations for balancing the forces:

$$N_1\cos\theta + N_2\cos\theta + N_3\cos\theta = W \quad (2)$$

Where θ is the angle between the reaction forces N_1, N_2, N_3 and the vertical line from point A at the center of the upper ball, and $\theta = arc\sin\frac{\sqrt{3}}{3}$ 。

Since the forces on the bottom three steel balls are equal in magnitude and equal in angle to the plumb line:

$$N = N_1 = N_2 = N_3 = \frac{W}{3\cos\theta} = 0.40825W \quad (3)$$

Under the action of normal pressure N, the steel ball approximates a circle of radius a due to elastic deformation, which is available according to the Hertzian contact equation:

$$a = \sqrt[3]{\frac{3N}{4}\times\frac{\frac{1-\mu_1^2}{E_1}+\frac{1-\mu_2^2}{E_2}}{\frac{1}{R_1}+\frac{1}{R_2}}} \quad (4)$$

Where E_1, E_2 represent the modulus of elasticity of the two sphere materials, μ_1, μ_2 represent Poisson's ratio and R_1, R_2 represent the radius of the two spheres.

Since the four steel balls have the same material and radius, the above equation can be simplified to:

$$a = \sqrt[3]{\frac{3NR\left(1-\mu^2\right)}{4E}} \quad (5)$$

Where E = 207 GPa, $\mu = 0.29$ and R = 6.35 mm.

$a = \sqrt[3]{N \times 2.10723 \times 10^{-14}}$ (m)

The contact stress is:

$$P = \frac{N}{\pi a^2} = 309.52\times W^{\frac{1}{3}}(\text{MPa}) \quad (6)$$

Wear surface characterization techniques

After testing, the three steel balls were immersed in anhydrous ethanol and ultrasonically cleaned for 5 minutes. An optical microscope (CX40M, Suzhou Lefeng Precision Instrument Co., Ltd.) was used to measure the wear scar diameter on the steel balls. A tungsten filament scanning electron microscope (EVO10, Zeiss, Germany) was used to observe the surface morphology of the wear scars. The morphology of the wear scars was further analyzed using confocal laser scanning microscopy (Leica, Germany), and the contour curves were obtained. An X-ray photoelectron spectrometer (ESCALAB Xi+, Thermo Fisher Technologies) was employed to detect the elemental composition on the surface of the wear scars.

ERESULT AND DISCUSSION

Characterization of h-BN nanoparticles

Figure 2 presents the SEM image of h-BN nanoparticles. The image reveals that the h-BN nanoparticles exhibit a lamellar, overlapping, and slightly loose structure. The average particle diameter is approximately 1 μm.

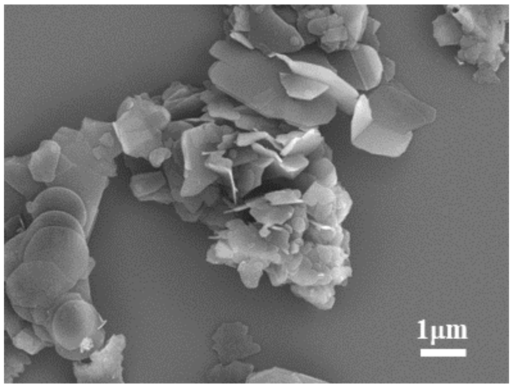

Fig. 2. SEM image of h-BN nanoparticles

Fig. 3 displays the XRD pattern of h-BN nanoparticles without further treatment. The 2θ peaks at 26.6°, 41.5°, 43.7°, 50°, 54.8°, and 75.8° correspond to the characteristic diffraction peaks of the (002), (100), (101), (102), (004), and (110) crystal planes of h-BN. This confirms that the detected nanoparticles are h-BN, and the strong diffraction peaks indicate an excellent crystal structure.

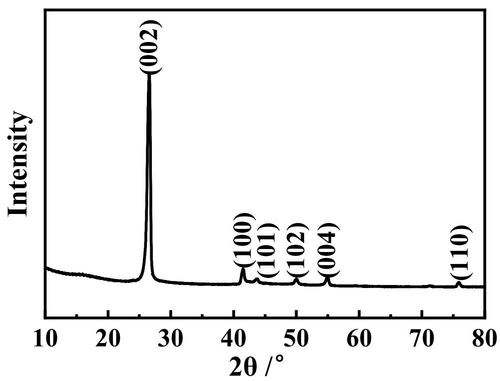

Fig. 3. XRD pattern of h-BN nanoparticles

Fig. 4 shows the particle size distribution of h-BN. h-BN particle sizes were mainly distributed between 531.2 and 1281.3 nm, and the average particle size was 813.8 nm.

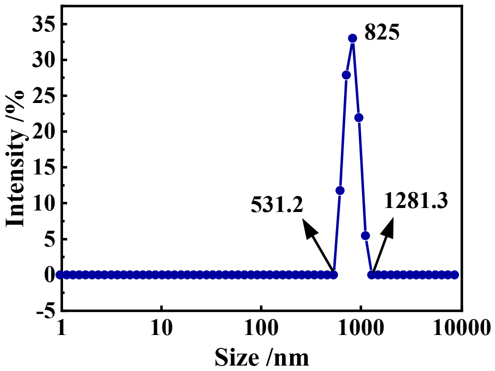

Fig. 4. Size distribution of h-BN nanoparticles

Figure 5(a) presents the friction coefficient curves of h-BN greases with varying mass fractions. For the base grease, the friction coefficient increases sharply during the initial stage, and the peak occurs at around 300 s. This process is termed the unstable wear stage. Subsequently, the friction coefficient gradually decreases and stabilizes, indicating the onset of the stable wear stage. For the mass fractions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5%, the friction coefficient reaches its maximum initially, then gradually decreases to a stable state. This suggests that h-BN nanoparticles fill the wear surface, significantly shortening the unstable wear time and allowing the friction pair to quickly enter the stable wear stage.

In general, the addition of nanoparticles can enhance anti-friction. Fig. 5(b) illustrates the relationship between the average friction coefficient and h-BN mass fraction. The average friction coefficient of the base grease is 0.070. As the h-BN mass fraction increases, the average friction coefficient initially decreases and then increases. Overall, the average friction coefficient of nearly all h-BN greases is lower than that of the base grease. At a mass fraction of 0.2%, the grease exhibits the lowest average friction coefficient of 0.053, which is 24.2% lower than that of the base grease.

Figure 6 illustrates the relationship between the average wear scar diameter and h-BN mass fraction. The average wear scar diameter of the base grease is 466 μm. As the h-BN mass fraction increases, the average wear scar diameter initially decreases and then increases. The average wear scar diameter of nearly all h-BN greases is smaller than that of the base grease, as shown in Fig. 6(g). The smallest average wear scar diameter is observed at a mass fraction of 0.2%, measuring 400 μm, which is 14.2% lower than that of the base grease. The test results are consistent with the friction coefficient, indicating that the grease exhibits the best tribological properties at the mass fraction of 0.2%.

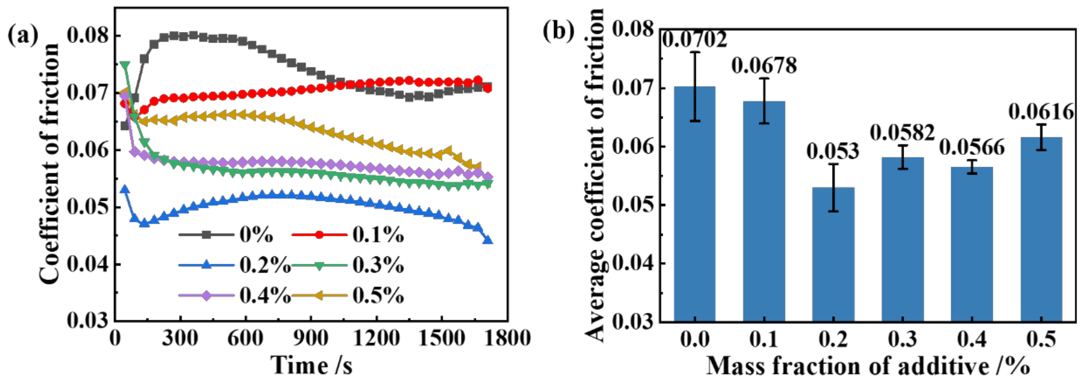

Fig. 5. Friction coefficient of grease with h-BN: (a) evolution of the friction coefficient with time; (b) the relationship between the average friction coefficient and the h-BN mass fraction

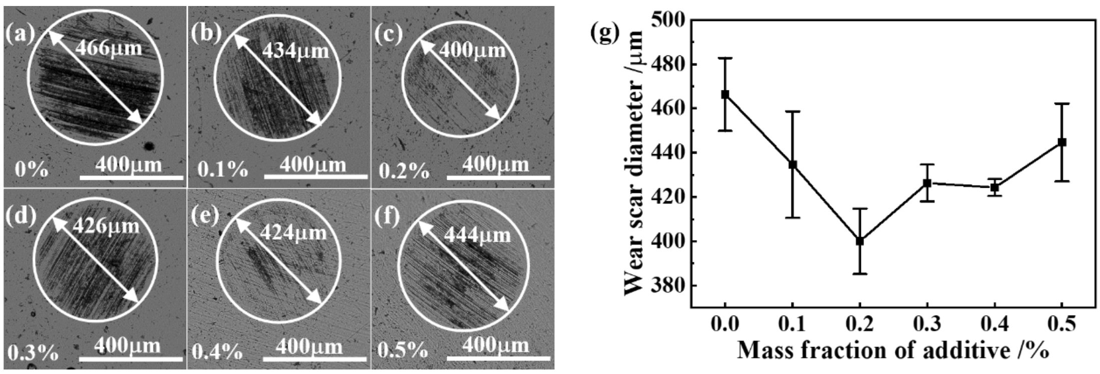

Fig. 6 (a)-(f) optical microscope images of wear scar diameters of grease with different h-BN mass fractions; (g) the relationship between average wear scar diameters and h-BN mass fraction

Morphology analysis of wear surfaces

Figure 7 presents the SEM images of the wear surfaces under varying h-BN mass fractions. The SEM image of the wear surface of the base grease is depicted in Fig. 7(a). The wear surface exhibits pronounced furrows, numerous spalling pits, and attached wear particles, resulting from adhesive and abrasive wear during the friction process. The furrows on the surface are significantly reduced when the h-BN mass fraction is 0.1%, though numerous spalling pits remain, as shown in Fig. 7(b). At an h-BN mass fraction of 0.2% (Fig. 7(c)), the grinding wear surface appears relatively smooth, with fewer and shallower furrows, and only a few spalling pits. As the h-BN mass fraction increases from 0.3% to 0.5% (Fig. 7(d)-(f)), the furrow width on the wear surface progressively increases. Furthermore, at a mass fraction of 0.5%, a large number of wear particles are present on the wear surface. This is because an excessive amount of h-BN nanoparticles act as wear particles, intensifying surface wear.

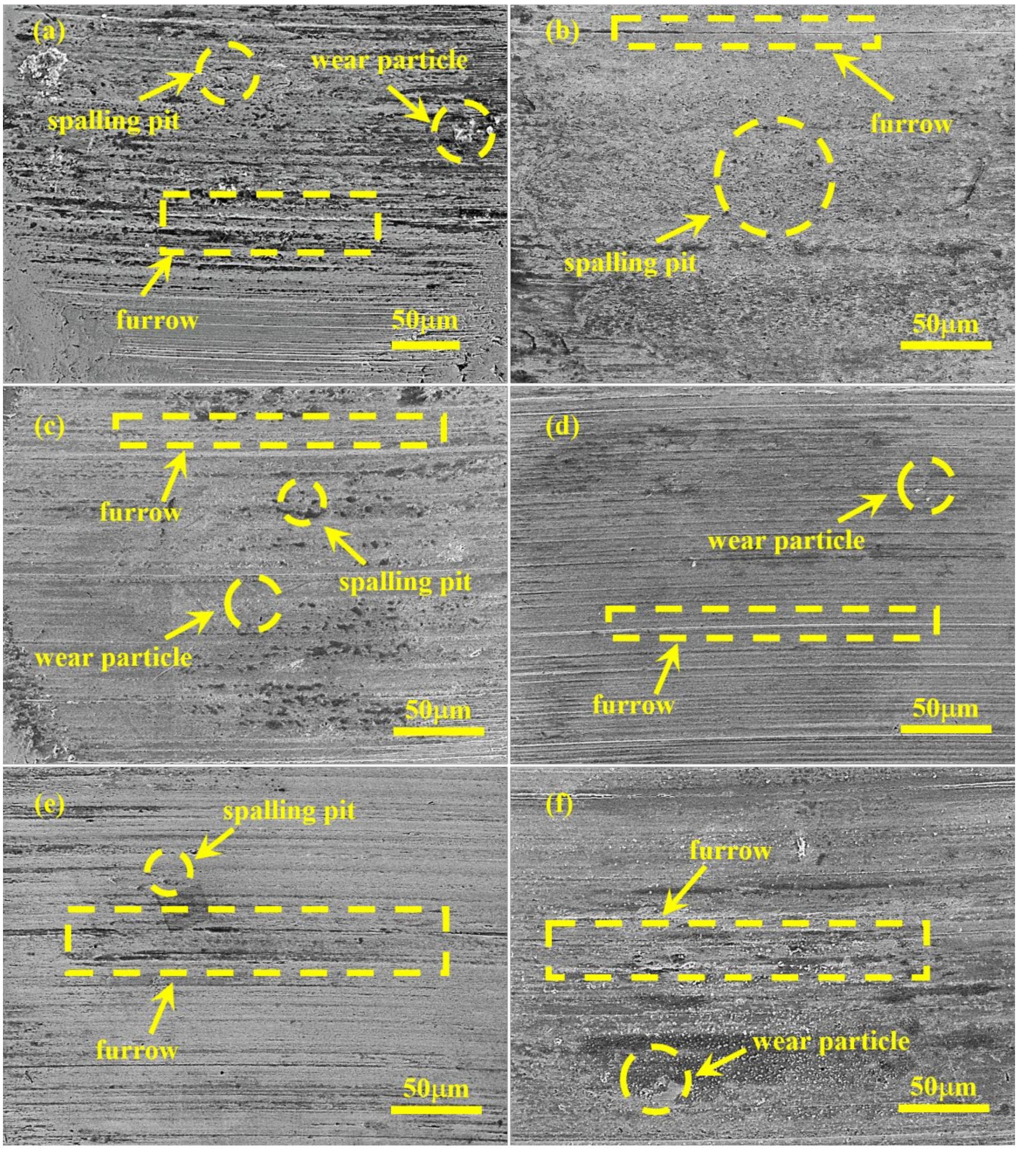

Fig. 7. SEM images of wear surfaces: (a) base grease; (b) 0.1% h-BN grease; (c) 0.2% h-BN grease; (d) 0.3% h-BN grease; (e) 0.4% h-BN grease; (f) 0.5% h-BN grease

In addition, Figure 8 illustrates the surface topography and contour curves of the worn surface under varying h-BN mass fractions. Fig. 8(a) shows the surface topography and surface contour diagram of worn surfaces of base grease. The surface topography reveals severe wear and pronounced scratches on the worn surfaces. The surface contour diagram indicates significant surface fluctuations, with the maximum wear depth reaching 3.11 μm. Compared to the base grease, the worn surface is smoother with no obvious scratches when the h-BN mass fraction ranges from 0.1% to 0.5% (Fig. 8(b)-(f)). With the increase of h-BN mass fraction, the maximum abrasion depth initially decreases and then increases, with the minimum value of 0.67 μm observed at a mass fraction of 0.2%. Fig. 8(c) shows the surface topography and contour curves of the worn surface of the grease with 0.2% h-BN. It can be seen that the surface is the smoothest and its undulations are the least. These results indicate that the presence of h-BN nanoparticles effectively improves surface quality and reduces the wear depth of the worn surface.

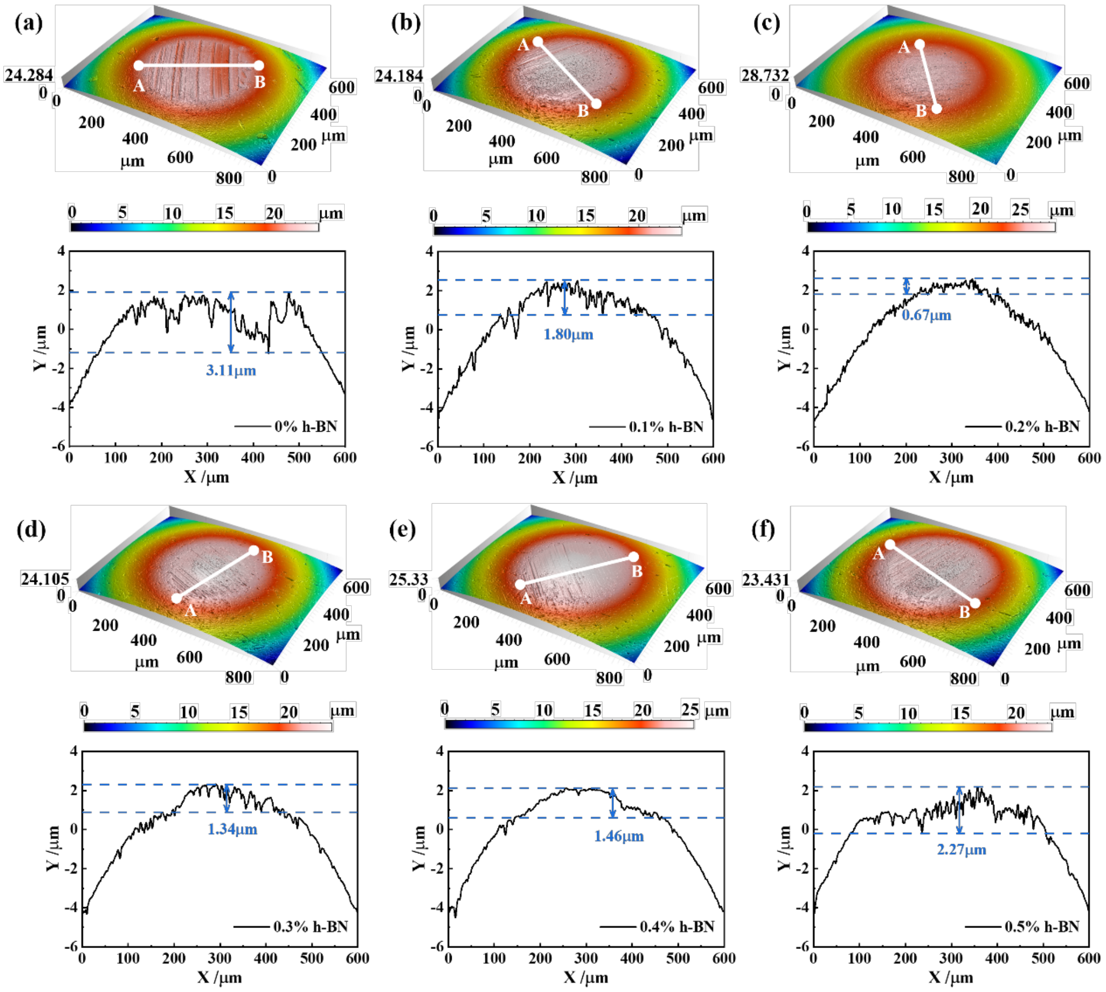

Fig. 8. Surface morphology and contour curves of worn surfaces: (a) base grease; (b) 0.1% h-BN grease; (c) 0.2% h-BN grease; (d) 0.3% h-BN grease; (e) 0.4% h-BN grease; (f) 0.5% h-BN grease

Elemental analysis of wear surfaces

To further investigate the anti-friction mechanism of h-BN nanoparticles, XPS analysis was conducted on the surface of 0.2% h-BN grease. Table 1 lists the elemental composition of the wear surface of h-BN grease. Detected elements on the wear surface include C, O, Si, B, N, Fe, and S. The B element originates from h-BN, while the S element is from the sulfur-containing additive in the base grease.

Table 1. XPS element composition on the ground surface of the grease with 0.2% h-BN

Name	Peak (eV)	Half peak width (eV)	Area (CPS. eV)	Atomic ratio (%)
C1s	284.92	2.74	210789.29	83.03
O1s	531.97	3.22	69199.62	10.74
Si2p	101.99	2.77	6352.95	2.58
B1s	189.00	0.45	1946.76	1.95
N1s	400.30	2.28	3341.38	0.83
Fe2p	709.93	6.73	13617.34	0.48
S2p	163.90	0.90	1331.96	0.40

Figure 9(a) presents the photoelectron spectrum of C1s on the wear surface. The characteristic peak at 284.84 eV corresponds to the C-C functional group, indicating the presence of carbon in the air. The characteristic peak at 287.69 eV represents the C=O functional group, corresponding to the organic compounds in the grease, indicating their adsorption on the wear surface. Fig. 9(b) illustrates the Fe2p photoelectron spectrum of the abrasive spot surface. The characteristic peak at 707.91 eV represents FeS_2. The presence of sulfur-containing additives in the base grease results in the formation of FeS_2, generated by the reaction of sulfides in the grease with metal at high temperatures during friction. The characteristic peak at 710.61 eV represents Fe_2O_3, formed by oxidation on the metal surface at high temperatures. Fig. 9(c) illustrates the photoelectron spectrum of O1s on the wear surface. The characteristic peak at 530.36 eV corresponds to Fe_2O_3. The peak at 531.94 eV represents the C=O functional group, while the peak at 533.3 eV indicates B_2O_3, suggesting that h-BN forms a B_2O_3 oxide film on the wear surface during testing. Fig. 9(d) presents the N1s photoelectron spectrum of the wear surface. The characteristic peak indicates that the N element primarily originates from h-BN. The analysis indicates that a chemical reaction film comprising Fe_2O_3, B_2O_3, and FeS_2 forms on the wear surface of h-BN grease, while organic compounds in the grease deposit on the wear surface, creating a lubricating film. The grease with h-BN reduces friction and wear through the combined effects of the chemical reaction film and the organic lubricating film.

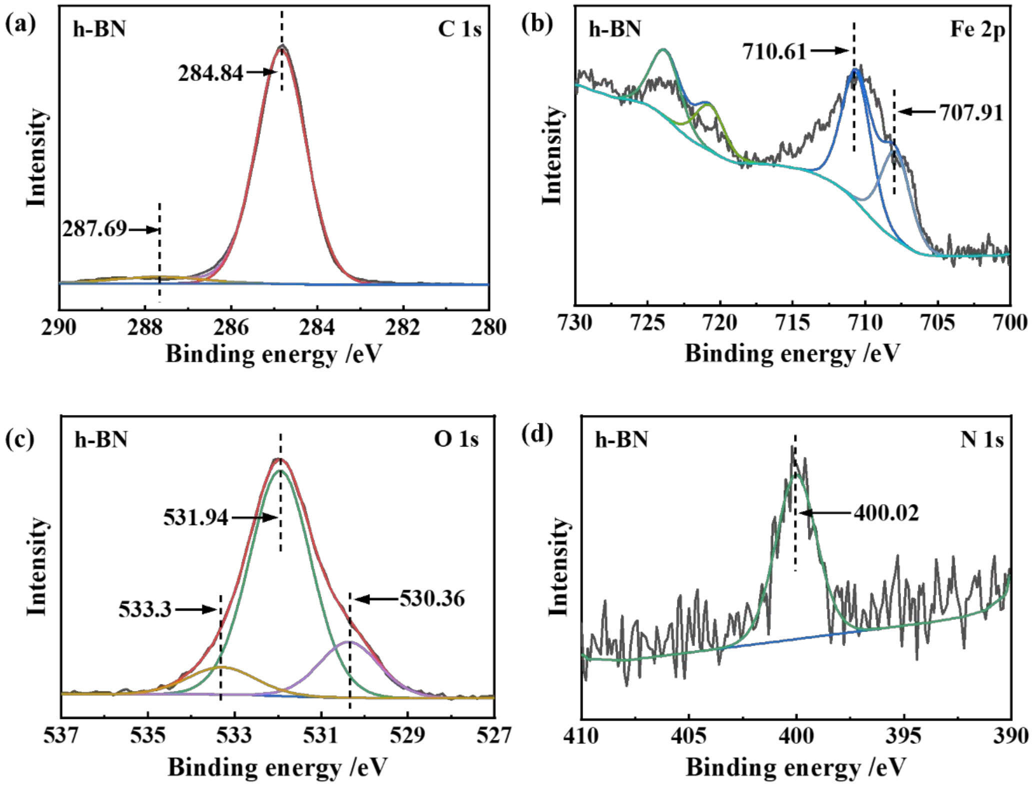

Fig. 9. Photoelectron spectra of the wear surface of the grease with 0.2% h-BN: (a) C1s; (b) Fe2p; (c) O1s; (d) N1s

Friction reduction mechanism of h-BN nanoparticles

The schematic in Figure 10 illustrates the friction reduction mechanism of h-BN nanoparticles. The SEM image of h-BN nanoparticles reveals small particle sizes with a lamellar and relatively loose structure. During friction, h-BN nanoparticles can enter between friction pairs, sustaining applied loadings and reducing friction through the slip between lamellar layers. Additionally, h-BN nanoparticles can deposit on the wear surface, helping to repair the friction interface. The h-BN nanoparticles can not only shield the asperities contact but also act as abrasive particles. When the mass fraction of h-BN reaches 0.2%, the higher the mass fraction, the more obvious the polishing effect and the more serious the wear. Under high speed and heavy load, the generated high temperature between friction pairs causes h-BN nanoparticles to react, forming a B_2O_3 oxide film that reduces friction and wear. Therefore, the h-BN nanoparticles and the B_2O_3 oxide film formed on the wear surface work together to improve the anti-friction capabilities. The tribochemical reaction plays a significant role in the enhancement of friction performance and load-carrying capacity (Ren et al., 2021). These results provide a new insight into the friction reduction mechanism of h-BN nanoparticles in lithium grease.

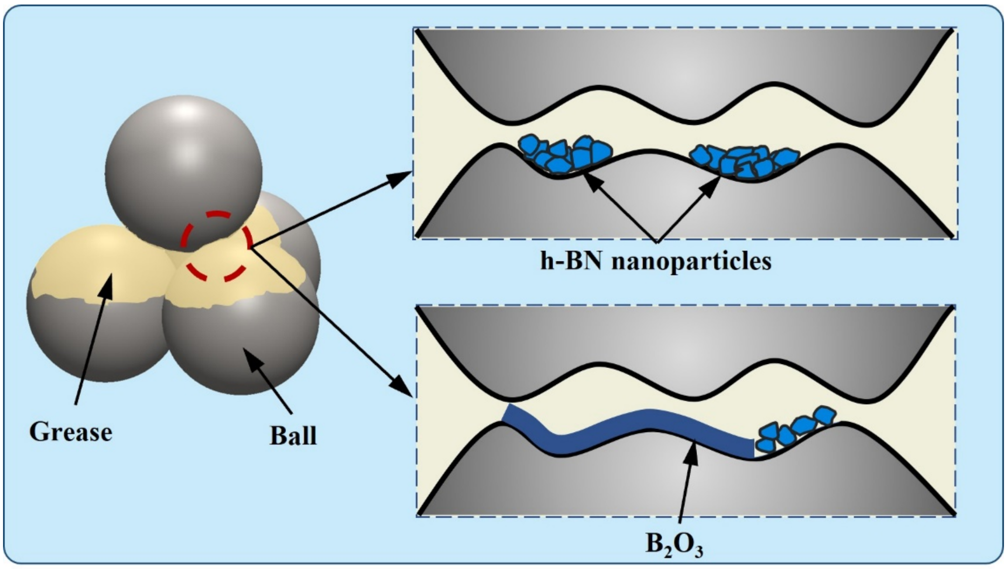

Fig. 10. Anti-friction mechanism of h-BN nanoparticles

CONCLUSION

In this paper, the effects of h-BN nanoparticles with different mass fractions on the tribological properties of lithium compound grease were investigated. The main conclusions can be drawn.

(1). h-BN nanoparticles effectively improve the tribological performance of compound lithium base grease, achieving optimal performance at a mass fraction of 0.2%, with the average friction coefficient and the average wear scar diameter reduced by 24.2% and 14.2%, respectively.

(2). h-BN nanoparticles fill the wear surface, significantly shortening the unstable wear period, allowing the friction pair to quickly enter the stable wear stage.

(3). On one hand, h-BN nanoparticles, due to their small size, can enter between friction pairs and fill worn surfaces, reducing friction and wear. On the other hand, at high temperatures, h-BN undergoes oxidation to form a B_2O_3 oxide film, endowing the compound grease with excellent tribological properties.

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